CITAC NEWS
Co-Operation on International Traceability in Analytical Chemistry
FEBRUARY 2008

Foreword by the Chairman: CITAC Strategy for 2007-2010

I am honored to have been elected the CITAC Chairman and grateful to my colleagues for their confidence. These were the first CITAC elections when voting was organized electronically by e-mail. In spite of the virtual system of elections, I am entirely approachable, and anybody who was interested in testing this could shake hands with me after the adoption of the voting results at the 22nd CITAC Members Meeting, July 17, 2007, São Paulo, Brazil, at the IV International Congress on Chemical Measurement Traceability and Quality Assurance (IV Metrochem).

I am pleased that the following officers were elected at the same time:
- Dr. Wynand Louw, the National Metrology Institute of South Africa, as Vice-Chairman and CITAC Award Coordinator;
- Dr. Philippe Charlet, the Laboratoire National de Metrologie et d'Essais, France, as Secretary and CITAC News Editor; and
- Prof. Dr. Wolfhard Wegscheider, the Leoben University, Austria, as Treasurer and CITAC Website Administrator.

What more could any chairman dream of than working with so qualified and capable a team?

Dr. Koichi Chiba, National Metrology Institute of Japan, Prof. Venkatesh Iyengar, Tufts University, USA, and Dr. Chainarong Cherdchu from the National Metrology Institute of Thailand were elected as new CITAC members. This favorable addition comes after the resignations of Mr. John Hammond, Optiglass Ltd., UK, because of a change in his field of activity, and by Dr. Kensaku Okamoto, National Metrology Institute of Japan, due to his retirement from the Institute. Many thanks for their contributions over the years and their involvement in CITAC activity. I wish them the best for their future endeavors!

I would also like to thank my predecessor, Dr. Vera Ponçano (2004-2007), Instituto de Pesquisas Tecnologicas, Brazil, for her warm, heartfelt attitude towards every CITAC member during her term as chairperson, especially at the IV Metrochem, and Dr. Laurie Besley, the National Metrology Institute of Australia, past CITAC Honorary Secretary, for his humor, optimism and help in discussing and overcoming problems. It is a subject of pride for CITAC that Vera was able to prepare at this time her Ph.D. thesis "Network Organization in Chemical Metrology" and to defend it successfully on 1 November 2007 at the University of São Paulo, Brazil. Congratulations, Vera!

It is my pleasure as well to congratulate the first CITAC award winners, authors of the most interesting/important papers on metrology in chemistry by CITAC version 2007: Dr. David L. Deuwer, National Institute of Standards and Technology, USA, Dr. Bertil Magnusson, Swedish National Testing and Research Institute, and his co-authors from NORDTEST, and Prof. Venkatesh Iyengar, Tufts University, USA. More details can be found in the message from Dr. Wynand Louw, the Award Coordinator, and in the papers of the award winners.

As a government employee, I cannot attend any meeting abroad without permission from a special committee for business trips at the Ministry guiding the National Physical Laboratory of Israel (INPL). Discussing my request to take part in the 22nd CITAC Members Meeting in conjunction with the IV Metrochem, one of the members of the committee for trips said that in his opinion CITAC is not such an important organization: 1) he can also establish an "international forum" with his friend from USA by internet whenever he wants; 2) his "international forum" website decorated with graphic art and a video (with pretty girls) will be richer than the CITAC website; and 3) decisions of his "international forum" will not be obligatory for laboratories and institutions as the CITAC decisions are. Why should the Ministry pay for activity of its employee at CITAC and for the trip, and not CITAC itself? Why is CITAC necessary at all? Even if such questions are natural, they were unexpected for me before the meeting where I planned to start my CITAC Chairmanship (I rather expected to hear compliments - but life is life).

My answers were:
- Really, CITAC is a voluntary organization active in the field of scientific and applied, not legal metrology. However, participation in the Metre Convention and in the International Organization for Standardization, for example, is also voluntary. Decisions and documents of such organizations are not obligatory, but they are helpful, as a rule. In particular, guides for analytical (testing) laboratories developed with CITAC participation are useful for the laboratories and their accreditation bodies, as well as for users of the laboratory measurement (analytical/test) results.
- Of course, website graphics depend on the money invested, but the value of the CITAC website is to be assessed by its scientific, professional chemical/ metrological content.
- The CITAC budget is based on membership payments only.
- I am proud that the CITAC members are my friends. However, it should be taken into account that CITAC members were elected as leading specialists in metrology in chemistry, and their contributions are known internationally.

I also explained the CITAC's aims. In the end, the trip was approved.

I returned to these questions after the trip to formulate with CITAC members our strategy for 2007-2010. The draft strategy prepared using email communication is available at the website www.citac.cc (page "About CITAC") and in the present CITAC News issue, p. 37. This draft will be further discussed at the 23rd CITAC Members Meeting (in conjunction with the CCQM meetings in Paris, 30 March 2008). Therefore, at this time I would like to invite CITAC News readers to contribute to improving the strategy and to the realization of CITAC's goals.

Dr. Ilya Kuselman
CITAC Chairman
INPL, Israel

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Editorial

The 22nd CITAC Members' Meeting in São Paulo, July 17, 2007

In July, only eight members assembled in Hotel Feller, near the most famous avenue of São Paulo,Brazil to conduct the 22nd CITAC Members' Meeting and enjoyed the pleasant Brazilian wintertime. Members came from very different parts of the world, from Finland, USA, Russia, Israel, South Africa, France, Czech Republic and, of course Brazil. We were very honored to also have Dr. Alejandro Herrero, Director of IRMM (EU, Belgium), as observer. The meeting preceded the IV International Congress on Chemical Measurement, Traceability and Quality Assurance (IV Metrochem) on July 18th- 20th in São Paulo.

After the welcome words from the Chair, Mrs. Vera Ponçano, the agenda was adopted and the minutes of the previous meeting were accepted. The results of the election of CITAC officers and new members, as well as resignations are described in the Foreword by Dr. Ilya Kuselman, the new Chairman.

The financial report indicated that a limited number of members are currently paying their annual fees. Therefore, there is a clear need to explain, particularly to less motivated members, why CITAC is unique as a facilitator of contacts by disseminating traceability information. In order to get more flexibility, a proposal from the Chair was adopted to establish a new membership payment, a collective one for organization, institution or company. The "collective" member could appoint his representative having a specific expertise. On the whole CITAC is financially sound.

The liaison reports by Dr. Laly Samuel (APMP), Dr. Robert Kaarls (CCQM), Dr. Wynand Louw (SADCMET), Dr. Yoshito Mitani (SIM), Mr. Alan Squirrell (ILAC) and Dr. Willie May (JCTLM) have been received electronically by the members. The reports and the draft guidelines for CITAC liaison officers which were circulated before the meeting and discussed by e-mail and included the updated list of the CITAC liaison officers, were adopted. It was decided to include in the Agenda of the 23rd CITAC Members' Meeting oral reports of the liaison officers, taking into account the importance of the information conveyed by the officers.

The Terms of Reference of CITAC have been revised and the updated version was accepted during the meeting. These Terms of Reference will be the base for the development of the CITAC Strategy Plan 2007-2010 presented in this CITAC News issue by Dr. Ilya Kuselman.

Reporting on CITAC activities, Dr. Kuselman informed members about the recently published EURACHEM/CITAC guides "Use of uncertainty in the assessment of compliance" and "Estimation of measurement uncertainty arising from sampling". He asked members to be more active in development of the guides "Assessing performance in qualitative analysis"; "The fitness for purpose of analytical methods: a laboratory guide to method validation and related topics" (revision); and "Selection and use of proficiency testing schemes for a limited number of participants". These guides, currently being developed, demonstrate the strong emphasis of CITAC on the dissemination of the concepts of metrology in chemistry throughout industry and society.

Furthermore, in order to arouse attention and input of the CITAC members regarding the development of the guides, it was decided to include in the agenda of the next CITAC Members' Meeting oral reports of the CITAC representatives who are involved in the Working Groups developing the guides.

The CITAC procedure for nomination and awarding the most interesting/important papers on metrology in chemistry was adopted. Dr. Wynand Louw, the new CITAC Vice-Chairman and Award Coordinator, was asked to initiate the first round of the procedure already in the current year.

Dr. Kuselman stressed that it is important for CITAC, as an international organization active in the field of metrology in chemistry, to support the journal "Accreditation and Quality Assurance. Journal for Quality, Comparability and Reliability in Chemical Measurement" (ACQUAL), in the same way that a number of chemical societies support the "Analytical and Bioanalytical Chemistry" journal. This will be of particular major importance when ACQUAL changes its sub-title to "Journal for Metrology and Quality in Chemistry" or similar.

CITAC is also continuing to present its activities worldwide with involvement planned for April 2008 in Berlin (Workshop on Uncertainty in Sampling and in Compliance Assessment) and in Paris (the CITAC 23rd Member's Meeting in conjunction with the CCQM meetings), in May in Athens (EURACHEM GA and Workshop on Education and Training), in October in Rome (Workshop on Proficiency Testing), and in November in South Africa (Test and Measurement Conference). CITAC also plans to take part at the Joint Committee on Traceability in Laboratory Medicine (JCTLM) in Fortaleza, Brazil. The policy paper related to the procedure for CITAC to highlight important papers in metrology in chemistry was adopted. Finally, Dr. Ilya Kuselman, the new Chairman, expressed gratitude to Mrs. Vera Ponçano for her contribution by enlightening the vision of CITAC's Mission during the past three years and the hope that Vera will continue her activity as the CITAC Past-Chair.

Functioning also as the CITAC News Editor I would like to draw your attention to the contents of this issue. As usual, the reports of international organizations and regional metrology organizations are presented but you will also find the full text of the awarded "most interesting papers on metrology in chemistry in 2007". European NMIs have been fully involved in 2007 in the elaboration of joint research projects in the framework of iMERA (Implementation of Metrology in the European Research Area). A paper related to a selected project, in the Targeted Program "Health", on the new approach of Bayesian statistics to improve reference values in metrology in chemistry is also presented in this issue.

Meeting reports and announcements of upcoming meetings on metrology in chemistry are also included. May I draw your attention on the MEFNM 2008, The International Conference on Metrology of Environment, Food and Nutrition Measurements, to be held in Budapest next year. This conference will join the activities of three IMEKO technical committees and two of them, Food and Nutrition, and Chemical Measurements are chaired by CITAC members (Venkatesh Iyengar and myself).
The updated list of CITAC members is available at the end of this issue.

Dr. Philippe Charlet
CITAC Secretary
LNE, France

Message from the Vice Chair

Since its official inception a little more than a decade ago, metrology in chemistry spread its wings over the entire world; huge strides were made in bringing the concept of metrology to each country especially in the developing countries. More importantly, documents and traceability solutions must reach each and every accredited (and other) analytical and testing laboratory. I believe that it is in this important final frontier of analytical chemistry that CITAC can play a vitally important role to disseminate, interpret and assist to implement the advances in traceability in metrology in chemistry being made at the NMI level.

Being the Vice-Chair of CITAC not only creates the opportunity to make a contribution to the international goals of CITAC, but also paves the way to further the activities of CITAC in Africa. This is especially important in the next year or two with the recent establishment of the Intra-Africa Metrology System (AFRIMETS) where CITAC can play a role to distribute metrology related documents in an easily accessible location and form. May I also, in my capacity as the AFRIMETS Chairperson (on behalf of my NMI and African colleagues), wish CITAC a fruitful and productive 2008.

Dr. Wynand Louw
Vice-Chair, CITAC
NMI, South Africa

Message from the Past Chair

Usually a compass identifies directions and ensures that one knows his/her location and/or stays on a pre-defined course. It is important in life to have a compass in order to know where you are going, to set your priorities and goals and to be totally focused on a cause while trying to function in the middle of a myriad of situations that appear daily. These perturbations demand our attention, while many times taking us off the "North" course that was originally set. So, to exist in this life routine one needs a compass that will hold the preset direction or priorities intact. To set these priorities we need to know what matters most in life, our personal values. One needs to reconfirm the direction and be sure about the goals that were set. In this way I tried to define my day by spending time on issues that would provide intense personal and professional pleasure. But this was possible only now, at this time in my life, since my four children have grown up and are going their own individual ways.

My initial course of direction was my work at IPT as Director for Metrology in Chemistry and Coordinator for International Affairs. Simultaneously I was coordinating a Brazilian Network for Chemical Measurement Laboratories (BNCM) which today is comprised of colleagues from 47 technology institutions and universities from 12 Brazilian states; this was in addition to CITAC activities that brought a stimulating input for the development of activities like the Metrochem IV, a high level congress supported by CITAC and BNCM members. During my professional career it became more evident how important it was to work in a partnership mode where one could get together results of common interest and act in a complimentary way which increases knowledge through interacting with each colleague and institution involved in the work being developed. It has been a great way to advance in providing relevant technological tools for the community and country. Particularly in chemical metrology, it provides relevant metrological tools for laboratories to get quality results and generate confidence and safety for society.

Having clearly developed these concepts and using the opportunity of this environment, it became a challenge to study in depth subjects related to management and relationships between institutions with focus on common interest, in an academic way. I remember a dear friend who insisted that I go ahead with my PhD; he said it would be like flying in another dimension where I could see the whole thing, having the relationship of each part better defined... and that was what happened.

So, three and half years ago it became another priority, developed in conjunction with all the other activities in my life. Even with a harmonic compass, melody and faith it was not easy to carry on all issues under my responsibility and become a student again after such a long time. There were no weekends, a mixture of day and night, notes and observations were constant companions. But the primary direction to travel, the North, was always there and I was fixed on reaching this point without deviating or stopping. The participation of friends and colleagues was fundamental in stimulating and providing the best information for this work.

The dissemination of chemical measurement traceability at the national level is a concern and an important task for managers with this kind of responsibility. In this context cooperation among national institutions has a relevant role. With this in mind, I decided to develop a study for my PhD at the University of Sao Paulo, concluded in 2007, with a focus on the Organization and Management of Laboratory Networks.

This research focused on identifying the benefits and increasing the amount of understanding associated with operation and performance of a network system focusing on chemical metrology. Its development comprised the identification of factors of influence and dimensions of improvement. The experimental research was conducted through a multiple case study that included national and international experiences developed through interviews and questionnaires with 33 senior researchers from the Brazilian Network for Chemical Measurements (formerly the Brazilian Program on Metrology in Chemistry) and 58 managers of chemical metrology area from 54 institutions in 46 countries that were mainly signatories to the Metre Convention. The conclusion was that cooperative actuation like networks are especially beneficial because of the diversity of current demands for chemical metrology. In a network, these demands can be resolved with less investment, in a timely manner, using national specialists and equipment, and following global standards. However, management of such a network system requires careful monitoring of all factors that effect its performance and maintenance, including those factors needed for its improvement. Future researchers are needed to validate these results, applying them to other network systems. In addition, complimentary studies should be done to identify the "weight" of the influencing factors to networks and their relation using mathematical models such as modeling techniques. Also practical experiences like the ones presented in this study can be applied in the development of theories like the Actor Network Theory.

Dr. Vera Ponçano
CITAC Past Chair
IPT, Brazil

Reports of International Organizations

An Update on the CCQM Activity in 2007

The interest of NMIs and other (potential) Designated Institutes to participate in the activities of the CCQM and its working groups has continued to increase. Inasmuch as metrology in chemistry is increasingly recognized to be of high importance for the quality of life and the economy (export) of countries we still expect a further increase of the activities in the field of metrology in chemistry.

The CCQM has met on 19-20 April 2007 at the BIPM. As usual the meeting was attended by more than 70 persons of about 45 organizations, not only representing NMIs and other Designated Institutes, but also representing intergovernmental organizations and international bodies, like the IAEA, WMO, Codex Alimentarius Commission, IFCC, ILAC, JCTLM, WADA, AOAC International, CITAC, IUPAC, ISO, ENGL (European Network of GMO testing Laboratories) and CropLife International (association of multi-national GMO producers). Due to illness a contribution by the representative of the ENFSI (European Network of Forensic Science Institutes) had to be cancelled.

All seven CCQM Working Groups met during the days before the meeting of the CCQM. The meetings of the CCQM and its working groups were also attended by representatives from the NIBSC (WHO) and the US pharmacopoeia (USP). Both organizations contributed actively to the work in the BAWG and the OAWG.
The CCQM Working Groups on Organic Analysis, Inorganic Analysis, Gas Analysis Electrochemical Analysis and Bio Analysis also met during the second half year of 2007.

The following are the points discussed and activities carried out.

Redefinition of the Mole

The CCQM is of the opinion that there are no fundamental objections with regard to a re-definition of the mole based on a fixed value for the Avogadro constant. Such a change would not yet have any effect at the practical level of chemical measurements and measurement uncertainty. However, it should be ensured that any possible modifications to widely-used equations was communicated clearly, so that the large number of scientists who depend on measurement results expressed in amount of substance units are clear as to how any change should be handled. The CCQM concluded that a careful approach will be needed that would lead to a considered opinion. The CCQM agreed on Recommendation CCQM 1 (2007) *"On the possible redefinition of the mole and the kilogram"*.

Further the CCQM established a working group on the redefinition of the mole chaired by Dr. M. Milton. Other members are Dr. L. Besley, Prof. de Bièvre, Dr. Marc Salit and Dr. R. Wielgosz. The CCQM will cooperate with the IUPAC Commission on Isotope Abundance and Atomic Weights (CIAAW) and the International Avogadro Coordination (IAC). The issue will be discussed again at the next meeting of the CCQM in 2008.

CCQM Working Groups

All CCQM working groups reported about the work carried out over the period 2006-2007. At this very moment about 108 Pilot Study comparisons and 67 Key Comparisons have been carried out, are in progress or are planned for the near future. This is an increase of about 15% compared to a year ago. This again demonstrates the rapidly growing interest in reliable, comparable and traceable measurements in chemistry by a growing community representing all areas of trade, industry and society. In addition, several Key Comparisons are followed up by a subsequent Key Comparison under the same code number, or are repeated by a RMO as a RMO Key Comparison. Also, the RMOs are now organizing more supplementary comparisons.

In order to limit the number of Key Comparisons as much as possible and to work in the most efficient and effective way, the CCQM and its working groups have started a study on how use can be made of the results of pilot study comparisons and how we can best test the capabilities and competences of the NMIs and other designated institutes by testing more generally the competences and available capabilities (techniques and procedures) in the institutes.

CCQM Workshop

A CCQM workshop on calculating the KCRV and its uncertainty and new approaches to a more efficient and effective way of testing the capabilities and competences of NMIs and other DIs has been held on 18 April 2007 at the BIPM. It has resulted in the creation of two ad hoc CCQM Working Groups:

- the KCRV and Its Uncertainty, chaired by Prof. M. Cox, NPL;
- Efficient Testing of Claimed CMCs, chaired by Dr. G. Turk, NIST.

The ad hoc working groups will report back in April 2008 to the CCQM. In the meantime interesting and creative discussions within the CCQM Working Groups concerned are taking place.

The **CCQM Working Group on Organic Analysis** reported results of key comparisons and pilot study comparisons on:

- volatile organic compounds (VOCs) in a solution of methanol
- ethanol in aqueous matrix
- cortisol and progesterone in human serum
- nutrients in infant formula

The excellent results obtained in a pilot study on 19 - norandrosterone, carried out in cooperation with the WADA was published. This drug belongs to the group of second most abused anabolic steroids. A next comparison in cooperation with WADA is being prepared.

Work in progress includes VOCs in solution and PCBs in mussel tissue. Future work will include study comparisons on theophylline and digoxin, malachite green in fish, residues of veterinary drugs, apple juice spiked with pyrethroids, acrylamide in potato chips and organic components in alcoholic drinks. The work by the BIPM in taking a leading role on purity analysis (theophylline) has been well received.

The **CCQM Working Group on Inorganic Analysis** reported results of key comparisons and pilot studies comparisons on:

- Pb, Fe, Cu and Cd in wine
- Toxic elements in bovine liver
- stable isotope delta values
- As, Hg, Se and methyl-mercury content in marine fish
- Assay of chloride content of KCl
- Total Se and Se-speciation analysis of Se-rich wheat flour.

Other comparisons are in progress:

- analysis of copper alloy (key comparison)
- trace elements in phosphogypsum
- Sr isotopic ratio measurements

- Cd, Cr, Hg, Pb in poly propylene (plastics) for the EU RoHS Directive
- Purity of zinc

A workshop on Neutron Activation Analysis as a primary method has taken place. NAA has been added to the short list of potential primary methods in metrology in chemistry.

The **CCQM Working Group on Gas Analysis** reported results of key comparisons and pilot study comparisons on, carbon dioxide in air, oxygen in nitrogen and hexane in methane. Proposed comparisons include:

- Purity of methane
- Mercaptans in methane (odourisation of natural gas)
- Stack emission gases (NO, SO_2, CO, CO_2, propane in nitrogen)
- Species at trace levels typical of environmental measurements
- Reactive species.

Further, the collaboration with the WMO Global Atmospheric Watch programme (WMO GAW) has been reported. The WMO GAW has now also requested that the **CCQM Working Group on Gas Analysis** takes responsibility for maintaining a "scale" for atmospheric VOCs on their behalf in support of the WMO VOC global network.

The CCQM GAWG has participated together with the WMO GAW's VOC group in a workshop in Garmisch-Partenkirchen, Germany.

The organization of a BIPM – WMO symposium *"On Metrology and Climate Change"* in the spring of 2009, among others addressing green house gases, ocean salinity and irradiation issues, will be very welcome.

The **CCQM Working Group on Electrochemical Analysis** reported results on carbonate buffer, phosphate buffer and nitrate and nitrite in calibration solutions and natural water.
Proposed comparisons include:

- electrolytic conductivity
- pH of carbonate and oxalate buffers
- assay of potassium chloride

It has been proposed to also consider pH in organic solutions such as bio-fuels.

Work on measuring the salinity of ocean water is being prepared in order to improve the metrological traceability of these measurements. This is also a EURAMET project lead by the PTB
Presentations have been made on the potential for stripping voltammetry to perform as a primary method and activity measurements in complex mixtures.

The **CCQM Working Group on Surface Analysis** reported results on:

- silicon dioxide on silicon (key comparison)
- C and N concentrations in coatings on steel
- C in bulk steel
- Nitrogen concentration in a "diamond-like" carbon film
- Fe-Ni concentration in alloy films on silicon

In carrying out the key comparison different technologies have been applied, delivering different results. Investigations have identified the existence of an effect in some results due to the existence of hydrocarbon and water layers on the samples (ultra-thin surface contamination). The work of the SAWG group will be extended to encompass work on organic layers on surfaces.

The **CCQM Working Group on Bio Analysis** reported results of comparisons on quantitative polymerase chain react ion (PCR) (key comparison), protein structural measurements by circular dichroism, protein quantification, quantification of DNA methylation and assay by ELISA.
Proposed work includes:

- glycan species measurement in digested glycoprotein mixture
- quantification of cells with specific phenotypic characteristics
- multiplexed RNA gene-expression bio-markers
- genomic DNA quantification

The NIBSC (WHO) and the Pharmacopoeia (USP) are actively cooperating and participating in the activities of the BAWG.

The **CCQM Working Group on Key Comparisons and CMC Quality** reported that now 3736 CMCs published in the KCDB database of the BIPM are related to the work under the CCQM (www.bimp.org/kcdb).

BIPM Programme of Work

The BIPM programme of work has been presented by Dr. Wielgosz. The comparison on purity analysis of theophylline was successful. Work is now underway on digoxin and steroid hormones. In close cooperation with the OAWG the BIPM will coordinate a series of key comparisons covering a wider field based on a model on "molecular weight versus polarit space".

The BIPM QM-K1 is an on-going key comparison of ozone. Standard reference photometers have been upgraded with kits from NIST.
Comparisons of NO in nitrogen carried out by the BIPM have delivered very good results with noticeably improved uncertainties for analytical values.

JCTLM

Work in the scope of the JCTLM was reported by Dr. W. May, chair of the JCTLM Working Group 1 on Reference Materials and Methods of Higher Order, and Prof. L. Siekmann, chair of the JCTLM Working Group 2 on Reference Measurement Laboratory Services and inter-laboratory comparisons. WG 2 is organizing series of regular inter-laboratory comparisons. The results of laboratories listed with their services in the JCTLM database are of clearly good quality. Results of the inter-comparisons are available at the web-site of the DGKL.

Up-dated lists of CRMs and measurement procedures "of higher order" as well as available reference measurement laboratory services are now regularly published in the JCTLM database. (www.bimp.org)

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

Codex Alimentarius Commission

The cooperation with the Codex Alimentarius Commission and the Inter Agency Meeting is developing satisfactorily. Dr. Doyran from the Codex Alimentarius Commission outlined the background of the Commission and described the remit of the Commission on Methods of Analysis and Sampling. The Codex Alimentarius Commission is considering the application of measurement uncertainty including allowance for sampling uncertainty. The Codex Commission is also assessing the safety and labelling of biotechnology products.

Reports of International Organizations

Working Group 7

Mr. Finidori, secretary of Working Group 7 (GMOs and derived products) of ISO TC 34 (food products) gave an overview of work on DNA-based methods, protein-based methods, definitions and sampling strategies.

ENGL

Dr. Moens from the Institute of Health and Consumer Protection (IHCP) at the EU JRC in Ispra, in his function as deputy chair of the European Network of GMO Testing Laboratories (ENGL), gave an overview of the Activities under the ENGL. The ENGL laboratories operate within the EU Framework Directive on GMOs. This includes harmonization and standardization of measurement methods and quality systems. Sampling strategies are a hot topic. IHCP is also an EU Reference laboratory for GMO measurements that is also involved in method validation.

CropLife International

Mr. Dana from CropLife International described the interest of the multi-national GMO industry in reliable, comparable and traceable measurements. He gave an example of the disruption of trade due to independent adoption of measurement methods and materials. He mentioned the importance of applying standardised PCR methods and the need for adequate CRMs. Further discussions should take place in order to identify the role for the NMIs in this area.

Dr. Robert Kaarls
President CCQM
The Netherlands

ILAC Update for CITAC News 2008

CITAC

ILAC has maintained its close cooperation with CITAC and thus has enabled us to continue our efforts in strengthening the links between accreditation and metrology (particularly in chemistry) – an essential link to assist accreditation bodies and accredited laboratories in delivering traceable and reliable measurement results which are fit for their intended use. Issues of common interest include metrological traceability, proficiency testing, reference materials and further applications of measurement uncertainty (including qualitative analysis) and CITAC's input on these issues, through their membership of ILAC's Laboratory Committee (which is currently chaired by Dr Máire Walsh, a CITAC member) is much appreciated. A general update on recent activities within ILAC follows:

ILAC Meetings

The 2006 Annual Meetings for ILAC and IAF, were hosted by the Entidad Mexicana de Acreditación A.C. (EMA) in Cancun, Mexico, from 6-14 November 2006. Representatives of several international liaison organizations attended and the meetings were both productive and informative. Delegates participated in a very full meeting schedule but were also fortunate to experience some very enjoyable cultural events arranged by our Mexican colleagues.

The first ILAC Executive Committee and Arrangement Committee (ARC) meetings for 2007 were hosted by COFRAC in Paris, France during the first week of March. As is usual for the first Executive meeting of the year, planning of the work program for 2007 was high on the agenda, with the updated work programs of the Committees also being reviewed. Another standard activity for the first meeting of the year is the review of the General Assembly Resolutions and the status of those requiring action.

The June meetings in 2007 were hosted by CNAS in Beijing, China. Meetings of the ILAC and IAF Executive Committees, the ILAC/IAF Joint Committee for Closer Cooperation (JCCC), the ILAC Arrangement Management Committee (AMC), the IAF MLA Management Committee (MC) and a joint session of the ILAC AMC and IAF MLA MC were held during this period. Terms of Reference for the operation of the joint sessions of the ILAC AMC and IAF MLA MC were approved by each organization at the ILAC-IAF Joint General Assembly in Cancun in November 2006.

The 2007 Annual Meetings (ILAC and IAF) were co-hosted by NATA and JAS-ANZ in Sydney, Australia, on 19–31 October. Please visit the ILAC website for a list of the Adopted Resolutions at the ILAC General Assembly. The 2008 meeting will be held in Stockholm, Sweden, during the period 10-22 October.

This year, the developing country seminar was held jointly in Sydney on 31st October 07, between ILAC, IAF and the Asia-Pacific Metrology Programme (APMP). The three organisations took advantage of the proximity and overlap between both sets of annual meetings to conduct this joint seminar on the topic of "Interactions between National Metrology Institutes and Accreditation Bodies for Laboratories, Inspection Bodies and Certification Bodies - with particular focus on issues for Developing Economies". UNIDO was a co-sponsor of the seminar, as one of the partner organisations of the Joint Committee on Coordination of Technical Assistance to Developing Countries in Metrology, Accreditation and Standardisation (JCDCMAS).

The ILAC Arrangement

As of 19 June 2007, there were fifty-eight Signatories (Full Members) to the Arrangement, representing forty-six economies.

The Inter American Accreditation Cooperation (IAAC) received formal recognition of their Multilateral Arrangement (MLA) during the meetings in Cancun. This recognition was granted following an extensive peer evaluation process that culminated in a ballot amongst ILAC Arrangement Council members. The IAAC recognition is for Testing & Calibration and signatories to the IAAC MLA who are also members of ILAC, are now

eligible to also become ILAC Full Members, i.e., Signatories to the ILAC Arrangement. ILAC now has three Recognised Regional Cooperation Bodies - Asia Pacific Laboratory Accreditation Cooperation (APLAC), European Co-operation for Accreditation (EA) and Inter American Accreditation Cooperation (IAAC).

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

Joint ILAC/IAF Activities

The joint activities between ILAC and IAF continue to be managed through the Joint Committee for Closer Cooperation (JCCC). Currently operating under the stewardship of this committee are the following:

- Joint Working Group for Inspection (JWGI);
- Joint Development Support Committee JDSC);
- Joint Working Group on Maintenance of A-Series Documents;
- Joint Working Group on Training of Peer Evaluators;
- Joint Working Group on Guidance for ISO/IEC 17011:2004;
- Joint Meetings of the ILAC Arrangement Management Committee (AMC) and the IAF Multilateral Arrangement Management Committee (MLA MC);
- Joint Working Group on Communications.

Each of these Joint Working Groups reports on their work programs and progress at the Joint Committee for Closer Cooperation (JCCC) meetings and also at the annual Joint General Assembly.

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. To assist with the management of the ILAC liaisons, the Liaison Database was established in the last quarter of 2005. The volume of information in the database has increased substantially since that time and it is available for the information of all ILAC members. It can be accessed via the members' area of the ILAC website. (ILAC members who have not as yet sought access to the Members Area of the website, can do so on-line, via the 'Home' page of the website).

The BIPM/ILAC Accreditation Committee (AIC) working group's work on the dissemination of traceability (eg Best Measurement Capability (BMC)/Calibration and Measurement Capability (CMC)) was further progressed during the recent meetings in Paris. A joint statement on the future use of CMC was recently approved by both BIPM and ILAC. A metrology workshop was held on 9 March 07 and representatives of the Regional Accreditation Cooperations and Regional Metrology Bodies once again shared ideas on the topics of common interest to the accreditation and metrology communities. Further meetings are scheduled for March 2008.

A meeting of the CCQM (Consultative Committee on Amount of Substance – Metrology in Chemistry) was held in April 07 – and this included developments in Proficiency Testing and Reference Materials. ILAC was represented at meetings of the Joint Committee on Traceability in Laboratory Medicine (JCTLM) in Paris in December 2006 and is very active in the accreditation of medical laboratories.

ILAC's continuing close cooperation and liaison activity with EURACHEM and CITAC supports these important metrological initiatives in chemical and biological measurement including work on method validation, measurement uncertainty and compliance with limits, the use of "good quality" reference materials and proficiency testing.

The Memorandum of Understanding (MoU) signed between ILAC and OIML in November 2006 has been expanded to include IAF in a Tripartite MoU. Final preparations are underway for the signing of this MoU during the Sydney meetings in October. Progress is also continuing on the ILAC and OIML Joint Work Programme, with related items scheduled for consideration and progression during the ILAC/IAF annual meetings in October 2007.

ILAC continues its very active role in many ISO Technical Committees and CASCO Working Groups. This year ILAC liaison officers (sometimes more than one) have participated, and are scheduled to participate, in meetings of the CASCO CPC (Chairman's Policy Committee), WG 27 (Drafting requirements for use in conformity assessment applications), WG 29 (Revision of ISO Guide 65 – Product Certification), CASCO WG28 (Revision of ISO Guide 43 – Proficiency Testing), CASCO Plenary, ISO TC212 (Technical Committee - Clinical laboratory testing and in vitro diagnostic test systems), ISO TC 69 (Technical Committee - Applications of statistical methods), ISO TC 176 (Technical Committee - Quality Management and Quality Assurance) and the IAF, ILAC and ISO Joint Working Group.

ILAC was also actively represented at ISO REMCO (reference materials) which met in Tsukuba, Japan on 5-8 June 2007 and the APLAC Workshop on reference materials held in conjunction with this ISO REMCO meeting. The meetings focussed on the review of ISO Guide 34 *General Requirements for the competence of reference material producers.*. ILAC and its Regional Cooperation Body members are continuing to contribute to the revision of this important guide together with other related work on reference materials.

ILAC has also continued its cooperation with various ISO technical committees - eg TC 176 (ISO 9000 series), TC 69 (Statistical Methods), TC 34 (Food Safety) and ISO TC212 (Medical Testing).

ILAC and the World Anti Doping Agency (WADA) have continued the cooperation begun in 2003. ILAC now holds a seat on the WADA Laboratory Committee (LC). WADA held its fourth training course for (Accreditation Body) Assessors in April 2007. It is pleasing to note the good progress this collaboration continues to make, as reflected by the fact that a Memorandum of Understanding between the two organisations was signed in November 2007 in Madrid during the World Conference on Doping in Sport.

Secretariat Staff

The staff of the ILAC Secretariat has recently undergone some changes for 2007. We are pleased to welcome Sharon Kelly as the Senior ILAC Coordinator. Sharon, who previously held the role of Assistant Technical Manager at

NATA, brings extensive accreditation experience to the role and will be a valuable asset for the Secretariat.

We are also pleased to welcome Amanda Stubbs who joined the ILAC Secretariat on 15 May 2007, as the Administrator – ILAC Secretariat, a role she shares with Alison Hay. Amanda replaced Nilla Merrigan who left the ILAC Secretariat, to take on a role with another organisation, on 2 May 2007.

From 1 January 2007, Annette Dever has taken on the role of ILAC Secretary from Alan Squirrell. Annette and Alan have worked together in the Secretariat for the past 6 years. Alan continues to support the work of ILAC and the Secretariat as ILAC Executive Liaison on a part-time basis.

The Work of the ILAC Secretariat

The new ILAC website was launched on 15 October 2006 and it incorporates changes requested by the ILAC members and other users. In addition, the new website is easier to navigate and there have also been a number of back end changes to make the website more readily able to adapt to the changing needs of the ILAC membership. As always we welcome all feedback from members and other users.

The ILAC-MRA Mark registration process continues and, as at 31 January 2007, 39 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 ILAC Secretary met with the Secretaries of the International Accreditation Forum (IAF) and the Regional Cooperation Bodies, both in Cancun and Sydney, to consider opportunities for, and benefits of, closer cooperation and communication between the Secretariats. The meeting was very productive and several initiatives were put forward for consideration and some of these are now in the process of being implemented.

ILAC Membership

ILAC membership as of 27 November 2007 is as follows:

- 58 Full Members (Signatories to the ILAC Arrangement) representing 46 economies;
- 19 Associates representing 18 economies;
- 20 Affiliates representing 19 economies;
- 5 Regional Cooperation Bodies
- 1 National Coordination Body
- 24 Stakeholders (including CITAC)

The ILAC membership (total 127 bodies) now covers a total of 83 different economies world wide and approximately 30,000 laboratories and 5,000 inspection bodies are accredited by the 77 ILAC Full Members and Associates.

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

Mr. Alan Squirrell
ILAC Secretariat/CITAC Liaison
Australia

IMEKO Technical Committee (TC-23) on Food and Nutritional Metrology

International Measurement Confederation known as IMEKO is a non-governmental federation of 36 member organizations individually concerned with the advancement of measurement technology. Its fundamental objectives are the promotion of international interchange of scientific and technical information in the field of measurement and instrumentation and the enhancement of international co-operation among scientists and engineers from research and industry. Founded in 1958, the Confederation has consultative status with UNESCO and UNIDO. IMEKO secretariat is based in Budapest, Hungary (for details please refer to www.IMEKO.org

IMEKO has recently approved a Technical Committee (TC-23) on Food and Nutritional (F&N) Metrology. This is yet another important milestone for strengthening the reliability of F&N measurements by an international organization supported by a host of outstanding metrology experts. The TC-23 facilitates addressing food safety issues, expanding the activities in the area of inorganic chemical metrology relevant to F&N measurements.

Food Safety Concerns and Measurement Issues

In the world of food as a trading commodity, we are moving towards a global but borderless-trade situation. Food safety being a prime concern under this scenario, reliable F&N measurements take the centre stage in decision-making. A measurement process characterized by metrological concepts enhances the reliability of analytical results and ensures sustainability to the quality assurance (QA) needs. These steps infuse authority to the F&N analytical results and enhance the user confidence in the ensuing public health decisions. Importantly, QA is also firmly linked to economic benefits at the national levels.

Objectives of TC-23

F&N metrology is an emerging discipline that is helpful in strengthening the reliability of analytical data, and integration of metrological concepts into the measurement process is the need of the hour. This step is necessary also to ensure sustainability of the analytical QA process. These tasks are looked upon as a twin assignment under TC-23: (i) enhancing the reliability of F&N measurements by introducing metrological concepts, and (ii) strengthening the metrological capability of the professional pool (capacity building for young investigators), since the academic system in the F&N areas generally do not provide for adequate training that includes teaching metrology.
For details how you can contribute to this effort, please contact:

Prof. Venkatesh Iyengar
Chair of TC-23, IMEKO
USA

Reports of International Organizations

EURAMET METCHEM Activity in Metrology in Chemistry

METCHEM is the technical committee of EURAMET (European Metrology Association) dealing with metrology in chemistry: a joint committee between EURAMET and EURACHEM (the network association aiming to promote good practices in analytical chemistry). METCHEM delegates meet annually in February at one of the European NMIs. The following points were covered at the last meeting.

Mandate of the Chairman

The mandate of Philippe Charlet (LNE, France) ended at the Euromet General Assembly in May 2007 (two mandates, 2003-2007). During 2006, the METCHEM chairman asked METCHEM contact persons to provide names of candidates for the position of chair. Two names were proposed to the Euromet executive and elections were carried out in December 2006. The new METCHEM chairman for the period 2007-2009, at least, is Bernd Guettler from PTB, Germany.

The current convenors of the METCHEM sub-committees are:

- Electrochemistry: Petra Spitzer, PTB, Germany
- Inorganic Analysis: Christophe Quétel, IRMM, EU
- Gas Analysis: Martin Milton, NPL, UK
- Organic analysis: Franz Ulberth, IRMM, EU

Meetings

In 2007, the METCHEM meeting plenary was held at IPQ, Caparica, Portugal, February 8-9. Two sub-committee meetings were held on February 6th (electrochemistry and organic) and two others (inorganic and gas analysis) on February 7th. Therefore, the meetings of the four sub-committees were spread over two days in order to allow specialists to attend 2 or 3 SC meetings. This created stimulating exchanges, discussions and proposals. Visits of IPQ laboratories were planned during these two days. 40 persons from 24 European countries (plus IRMM representatives) attended the plenary meeting.

During the Plenary Session a follow up to last year's forum discussions between NMIs and EURACHEM delegates on the "Strategies of NMIs to ensure adequate metrology capabilities to users" was given by organising a special session with invited speakers. Specific subjects were chosen and Portuguese field partners, particularly from EURACHEM, were invited to present their view and experience. As usual, part of the session was dedicated to the most recent developments regarding training and education in Metrology in Chemistry.

The following eight topics were presented:

- Water analysis laboratories - European directive
- Proficiency assays suppliers - traceability
- Producers of reference materials
- Emergent sector - traceability in medical sector
- Emergent sector - traceability in bio metrology
- Measurement uncertainty revisited
- Alternative approaches to uncertainty evaluation by Bertil Magnusson (SP, Sweden)
- The status of the European training and education initiatives by Philip Taylor (IRMM)
- International master study programme by Ivo Leito (University of Tartu, Estonia)

Research / Co-operation Trends

(status of projects and new cooperation developments)

Report on inorganic analysis

Christophe Quétel reported on the activities of the Inorganic Analysis WG and Bernd Guettler reported on the on-going and proposed projects. 22 participants attended the WG. The two mains subjects were the CMCs (Calibration and Measurement Capabilities) review and the projects. A mini workshop was organised on the traceability and characterisation of isotopic CRMs by means of synthetic isotopic mixtures.

In addition two projects have been registered: Project n° 894 deals with "Surface Enhanced Raman Spectroscopy - SERS - based metrology at a primary level". A work-plan was proposed by the coordinators NPL and PTB. It will be interesting to add the project to the European Metrology Research Program.

Project n° 924 topic is on "the development of a sustainable traceability and dissemination system providing Europe-wide comparable measurement results in water analysis under the WFD". The project aims to support the Water Framework Directive, started in December 2006 and should be completed in February 2009.

Report on electrochemistry

Petra Spitzer reported on the activities of the Electrochemistry WG. 16 participants attended the meeting. At the meeting three presentations were given: one on some developments for the measurement of dissolved oxygen by INRiM (Italy), another on electrochemical metrology activity in Estonia by the University of Tartu, and the last on electrochemical measurements of simple ions and glucose in complex mixture by METAS (Switzerland).

Concerning the projects, three are on-going: Project n° 843 on the evaluation and the calibration of pH on site measurement instrument is scheduled to end in December 2008. The two other projects are Project n° 898 on electrolytic conductivity at pure water level and Project n° 918 on traceability of salinity measurements in sea-water. Another project has been proposed: a research project on traceability on ion activity measurements (electrolytes in e.g. serum by ion selective electrode).

Report on gas chemistry

Martin Milton reported on the activities of the Gas Analysis WG. The attendees were from 22 countries and 26 laboratories. The two mains topics were CMCs and projects.

Martin Milton informed the assembly that a workshop on "dynamic measurements of NO2, SO2, CO at low ambient level and their comparability" will be held in March 14-17, 2007. Concerning the comparisons, Milton gave the new results on Projects n° 708, 883, 867 and 900.

Four new projects were proposed on the following topics:

- optical spectroscopy as a potential primary method
- purity analysis comparisons of nitrogen at sub 50 nmol/mol
- bilateral comparison
- ozone pre-cursor, already a Euromet

Project n° 886 with 20 participants. A lot of components have to be studied from solvent, cleaning, chemical industry, bio-genics, active components, difficult to control and treat.

Report on organic analysis

Franz Ulberth reported on Organic Analysis WG. The report concerned the CMC Cycle VIII review

and the on-going projects. The question was raised on the necessity of keeping this group as there are no new proposed projects at the moment.

Project n° 878 was updated for "Crude protein in cereals". Some new results were presented on Project n° 833 concerning PCB congeners in organic solution to build up measurements capacities. The results improved when compared to the results given in 2004-2005. A questionnaire was sent in 2004-2005 on the activity and needs in the organic field. the questionnaire showed that the community expressed a great interest in training exercises and comparisons. However, there is no new proposal for future comparisons and no real reason given. In Europe, some NMIs have well-established capabilities, and it seems that they have other priorities.

A discussion started on the difficulties encountered in this WG related to active participation. There are many designated institutes (DI) in this field. DIs need encouragement from NMIs to participate in the activity of this group. There is certainly also a question of funds. It seems important to the Metchem contact persons to attract laboratories and potential DIs to share ideas and experiences. The lack of communication and transfer of information to those laboratories was also a point raised. The laboratories are not intrinsically aware and interested by metrology but just in the delivering of CRMs. For some countries it can be difficult to motivate the laboratories to participate in such a group. The question of the future of the organic group is still open. No consensus was expressed by the Metchem contact person group.

Mutual Recognition Arrangement

European NMIs often participate in CCQM activities and coordinate CCQM comparisons. CCQM Working Groups are mainly chaired by European Convenors (Inorganic by LGC, Electrochemistry by SMU; Gas by NMi, Bioanalysis by LGC, Surface Analysis by BAM) and these convenors are also very involved and active in METCHEM activities.

In 2006 and the beginning of 2007, as usual, a large part of the activities of the convenors and delegates of the Technical Committee were devoted to the review and the submission of the next CMCs (Calibration and Measurement Capabilities) Cycle (Cycle VIII). 102 claims (new and revised) proposed by 9 institutes were accepted during the discussion at METCHEM in mid-February and proposals sent to JCRB on March 1. They mainly concern the inorganic analysis field. These proposals will be discussed at the next inter RMOs (Regional Metrological Organisations) meeting in April.

Valuable contacts with the other RMOs and the CCQM were maintained at a high level (meetings, exchange of information, invitation to seminars and events, etc.). Furthermore, METCHEM was fully involved (TC chair) in the ad hoc working group of BIPM that is currently elaborating a strategy regarding activities to be carried out at the CIPM level on Materials Metrology.

All during the year European NMIs have been fully involved in the preparation of JRPs (Joint Research Projects) within the framework of a ERANET Plus program. Metrology in Chemistry was particularly concerned by the Targeted Program "Health". With these programs, European NMIs are entering into new cooperative activities aiming to reach sustainable financial support from the European Commission, through Article 169. The new structure of the European Metrology Association with the creation of EURAMET was specifically designed to support the development of research collaboration between NMIs.

Dr. Philippe Charlet
Past METCHEM Chair
LNE, France

Plenary meeting of METCHEM

Reports of International Organizations

Bayesian Statistics to Improve Reference Values in Metrology in Chemistry: an ERANET-Plus Project

The European Framework

In May 2007, EURAMET e.V., the European Association of Institutes of Metrology, submitted the proposal for an ERANET Plus (European Research Area Network) funded European Metrology Research Program (EMRP) to the European Commission. The three pillars of the EMRP are fundamental metrology, innovation and quality of life, and technologies of the future with the aim of demonstrating that coordination of European Metrology is sustainable. As a part of this program, a horizontal targeted program of the EMRP has been devised on metrology underpinning "health". Indeed, not only are health advances a policy priority in Europe but also an area where metrology can make a significant impact swiftly.

Starting from the observation that data and uncertainty analysis in the field of laboratory medicine and clinical chemistry are not yet well established, European chemical experts have developed Joint Research Projects (JRPs). Among them the JRP entitled "Tracebioactivity" has been selected so that the European Commission will provide its financial support, and it is not incidentally that this JRP includes a work package "Modelling and data analysis: - New methods and algorithms employing a Bayesian approach to compute reference values from interlaboratory comparisons in laboratory medicine" which will be conducted by PTB (Germany), LNE (France) and SP (Sweden) by 2008.

An important aspect underlying this project is the application to Certified Reference Materials (CRM), so that this paper aims to shed light on how Bayesian statistics can improve the reliability of the certified values of reference materials. Before going any further let us first have a brief look at the crucial role of CRMs.

Certified Reference Materials: the tool of the chemical analyst

Accreditation of laboratories for chemical analyses is an essential quality management tool and a means to obtain confidence and comparability of results. Due to the introduction of the international standard ISO/CEI 17025, field laboratories have expressed their need of specific traceability schemes to ensure the reliability of chemical measurements. Actually these traceability schemes are mainly based on the use of CRMs. These CRMs are usually produced by National Metrology Institutes (NMIs) and provided to laboratories for different specific purposes of Quality Control and Quality Assurance. In addition, NMIs have implemented so called primary methods of measurements which ensure the traceability of measurements to the SI and that are also used by NMIs to assign the certified value to materials.

Value-assignment can also be based on data from an interlaboratory study. Consensus values are based on the results of all participating laboratories or only on selected laboratories. According to the development or improvement of analytical techniques by manufacturers and laboratories, NMIs are used to certify their reference materials from time to time in order to obtain more reliable and accurate certified values through a re-certification process.

Re-certification: situation

Re-certification of a certified reference material can be performed either through interlaboratory comparisons or by a single laboratory, typically a NMI carrying out a primary method of measurement. In both cases old and new data are at hand. Old data are past measurements used to compute the past certified values. When they were computed, the past certified values represented our best knowledge of the true value of the CRM. Nevertheless, by now this valuable information have been left out when computing each new certified value.

Challenge: To take into account the most data at hand to get more accurate certified values and associated uncertainties and thus to improve traceability.
Answer:: Bayesian statistics have a widespread approach to science and technology. Recently researchers have been trying to apply it to metrology and, more specifically to metrology in chemistry.

Bayesian Approach

Bayesian approach is a learning method based on the Bayes' formula, designed to update our knowledge of a quantity through the combination of prior information about this quantity and new observations. As a result, our knowledge, called posterior, about the targeted quantity and its associated uncertainty is improved. More specifically Bayesian estimates give a complete description of the uncertainty given observations and prior knowledge. Here prior means before observations are made, so that prior information is elaborated from expert judgements and past results. This prior information is modelled by a probability density function (pdf) which is meant to best represent our degree of belief about the possible values of the quantity before new observations.

So what's new with Bayesian approach? Unlike classical frequentist statistics which works with the sole new observations modelled by the likelihood, Bayesian approach allows, via the introduction of a prior, to take into account the most information at hand within a specific statistical modelling framework to obtain more accurate estimates of parameters.

Bayes' Formula : from prior to posterior

Let θ be the random variable modelling the targeted quantity and $x = (x_1,\ldots,x_n)$ be n new measurements. Let us define the following notations for the different notions introduced above:

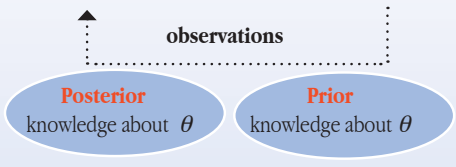
- The prior knowledge about θ is modelled by the pdf $\pi(\theta)$.
- The posterior updated knowledge is modelled by the pdf $\pi(\theta \mid x)$.
- The data are still modelled by the likelihood $p(x \mid \theta)$.

The Bayes' formula is then given by:

$$\pi(\theta \mid x) = \frac{p(x \mid \theta)\pi(\theta)}{p(x)}$$

where $p(x) = \int p(x \mid \theta)\pi(\theta)d\theta$ is the normalizing constant so that the left member integrate to unity and be a density.
Using the symbol *of* proportionality $\propto$, it appears then more clearly that the posterior density is proportional to the prior times the likelihood:

$$\pi(\theta \mid x) \propto p(x \mid \theta) \times \pi(\theta)$$



Estimates of the mean and the variance of the quantity are then given by the mean and the variance of the posterior distribution $\pi(\theta|x)$ and are usually computed numerically.

Let's give an easy example. Just consider that the prior pdf of the quantity θ and the pdfs of the observations $x_1,\ldots,x_n$ are both Gaussian as described below:

$$x_i|\theta \sim N(\theta,\sigma^2)$$
$$\theta \sim N(\mu,\tau^2)$$

In that case the posterior distribution of θ is:

$\theta|x_1,\ldots,x_n \sim N(\theta_1,\phi_1)$

Where: (P)

$$\theta_1 = \frac{\sigma^2/n}{\sigma^2/n+\tau^2}\mu + \frac{\tau^2}{\sigma^2/n+\tau^2}\bar{x} \quad \text{and} \quad \phi_1^{-1} = \frac{1}{\tau^2}+\frac{1}{\sigma^2/n}$$

Notice that:

- The posterior mean θ_1 is the weighted mean of the prior mean μ and of the sample mean of the observations $\bar{x}$ where $\bar{x}=\frac{1}{n}\sum_{i=1}^{n}x_i$ and $\bar{x}|\theta \sim N\left(\theta,\frac{\sigma^2}{n}\right)$
- The posterior inverse variance ϕ_1^{-1} is the sum of the inverse variance of the prior $\frac{1}{\tau^2}$ and of the inverse variance of the sample variance of the observations $\frac{1}{\sigma^2/n}$.

which shows that Bayesian approach is a compromise between the prior and the observations.

Bayesian Approach in Metrology in Chemistry

Undoubtedly Bayesian approach can be applied to the recertification process of CRMs. In this case the recertified value and its associated uncertainty are the updated values of the past certified values and their past associated uncertainty through Bayesian analysis. In particular the recertification process through intercomparisons is illustrated in the Figure 1.

In the case where all the distributions are Gaussian, the example above may be considered as a special case. Indeed if one supposes that:

- θ is the certified property
- the x_i $i=1\ldots n$ are the results of a sole laboratory (case N=1)
- the x_i are issued from the same Gaussian distribution (mean θ, variance σ^2)

then the posterior distribution of θ is given by expression (P).

This particular model can thus be applied when recertification is performed by one laboratory only.

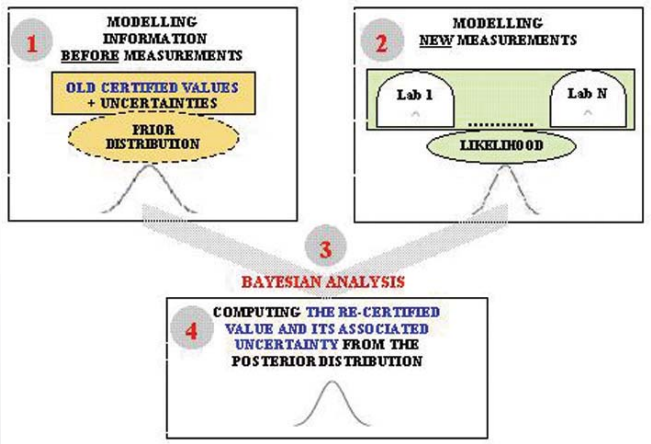


***Figure 1:** Recertification of a certified property of a CRM in a Bayesian statistical framework*

When N laboratories are involved the expression of the posterior distribution is more complicated because the expression of the likelihood is then heavier. Indeed, now let x_{ij} be the j^{th} measurement of the i^{th} laboratory where each laboratory makes n_i measurements that is j is between 1 and n_i for i between 1 and N. In addition, suppose that the variances of the measurements in each laboratory are different, say σ_i^2 for laboratory i. The model then becomes:

$$x_{ij}|\theta \sim N(\theta,\sigma_i^2)$$
$$\theta \sim N(\mu,\tau^2)$$

The posterior distribution of θ is then given by:

$$\theta|x_{ij} \sim N(\theta_N,\phi_N) \quad i=1\ldots N \quad j=1\ldots n_i$$

Where:

$$\theta_N = \frac{\sum_{i=1}^{N}\frac{1}{\sigma_i^2/n_i}\bar{x}_i+\frac{1}{\tau^2}\mu}{\sum_{i=1}^{N}\frac{1}{\sigma_i^2/n_i}+\frac{1}{\tau^2}} \quad \text{and} \quad \phi_N^{-1} = \sum_{i=1}^{N}\frac{1}{\sigma_i^2/n_i}+\frac{1}{\tau^2}$$

We observe that the posterior estimates of the mean and the inverse variance are still a weighted mean and the sum of the inverse of the variances respectively. In this respect it is worth noticing that this time, not only do we obtain a weighted mean between the prior and the observations but also the term standing for the observations is a weighted mean of the means of the laboratories. Thus all the information available has been taken into account and weighted according to the new measurements and prior knowledge. And that's precisely this idea of compromise that makes Bayesian approach so attractive.

Improving Reliability in Recertification: providing confidence on measurements

In conclusion, working on new measurements together with previous reliable measurements gives more accurate and reliable updated values. That way the new certified value and its associated uncertainty is consistent with the previous certified values and their uncertainties. In addition, when updating the certified value, metrologists increase the traceability of CRMs by improving the knowledge of the best estimate of the true value of the CRM.

Finally many decisions in the clinical sector are based on results of content measurements. That's the reason why the JRP project will first investigate the requirements of the European clinical sector for improved mathematical tools and then intends to make the demonstration that Bayesian probability theory is really the most promising approach to provide appropriate solutions for the improvement of reliability and comparability of measurements.

Séverine Demeyer
LNE-CNAM, France

Dr. Philippe Charlet
LNE, France

Reports of International Organizations

APMP Activity Report 2007

General Report

The 22nd APMP General Assembly and 6th TCQM meeting were held at the "Habitat Centre" New Delhi, India, December 13–16, 2006 in cooperation with the National Physics Laboratory of India (NPLI) and APMP. Approximately 150 metrologists from member NMIs and guests from CIPM, BIPM, RMOs (EUROMET and SADCMET), metrology societies (IMEKO, NCSLI) and SRB (APLAC, APLMF, PAC) were present. Brief reports were presented from NSCLI meeting and the CMC issue, COOMET GA 2006, APLAC GA 2006, APLMF GA 2006, SIM GA 2006 and APEC/SCSC meeting 2006. Jordan National Metrology Institute (JNMI) joined as a new associate member of APMP.

Update on APMP data in Appendix C

Based on the agreement at 16th JCRB in April 2006, APMP returned the list of QM CMCs to the JCRB Executive Secretary declaring the status of the QS supporting those of AU+CN+JP+KR. In response to JCRB's inquiry, APMP sent additional explanation about JP+KR on 24th July 2006 and about AU on 28th July 2006.

Asia-Pacific Metrology Programme (APMP) the 6th Technical Committee on Amount of Substance (TCQM) Meeting

- About 40 delegates from 18 member states/economies attended the meeting.
- Guests from other RMOs and BIPM were invited to TC meetings to participate in comprehensive discussions on issues relevant to the APMP members and organizations.
- Dr. Robert Kaarls, Secretary of CIPM and President of CCQM was invited to give a presentation entitled ""CCQM progress report 2006". DR. Kaarls outlined the history and development of CIPM and its relationship with other inter-governmental and international organizations such as JCTLM, ISO-REMCO, IAEA, ILAC, IUPAC, WADA etc.
- Delegates from member organizations who regularly attended various CCQM Working Group meetings gave updates on the latest activities and programmes organized by the CCQM Working Groups.
- Representatives from member states gave reports on the activities and future plans of their respective countries/economies.
- Results of calibration and measurement capabilities (CMC) reviews, pilot studies, key comparisons and cooperation between APMP members and international cooperation with other RMOs and BIPM were reported.

APMP TCQM Activities in 2006: Highlights

- An International Symposium and Workshop on Metrology in Chemistry was held in Malaysia on 20-21 February 2006 to promote the awareness of MiC and to draft a guideline document for setting up national measurement system in MiC.
- 4th Workshop on Gas CRM for Calibration and Testing was held in Thailand from 22-24 February 2006. The objective of the workshop was to exchange knowledge between APMP TCQM and CCQM GAWG members on the production of gas CRMs, gas analysis, traceability system and uncertainty evaluation.
- 5th APMP TC Chairs meeting was held on 30-31 March 2006 in Singapore to facilitate and prepare the APMP's presentation to the 16th JCRB meeting describing the QS review process in the region.
- The DEC meeting, QS workshop and APMP strategic planning session were held on 10-14 July 2006 in the Philippines. The main theme of the programme was to take a wider view and develop an overall strategic plan for APMP through collaborative efforts.
- A working group was formed to establish a guideline document for creating or improving a national infrastructure for chemical measurement. Dr. Laurie Besley presented a draft document at the meeting and invited members to give opinions and feedbacks.
- A Chemical metrology symposium was held along with Admet India 2007 in cooperation with CITAC, APMP and NPL India.

Progress Reports on Outstanding Pilot Studies/Key Comparisons

Progress reports were given by representatives of coordinating institutes on the following studies:

- APMP.QM-P05 (Cd in Oyster tissue)
  The coordinating lab KRISS reported that six NMIs (GLHK, KRISS, LIPI, NFI, NMIA and TISTR) participated in the programme and those NMIs using IDMS methods showed a preferential agreement on reference values and MU than those of the non-IDMS.
- APMP.QM-P07 (trace elements in soybean powder)
  This programme was running parallel to CCQM-P64. Eleven NMIs participated in the APMP study and 19 NMIs participated in the CCQM study. According to the coordinating lab, agreement of the data from CCQM-P64 was better than the APMP QM-P07.
- APMP.QM-P08 (SiO_2 thickness on Silicon)
  SiO_2 thickness on Silicon
  Coordinating laboratory reported that 11 organizations (CSIR-NML, ITRI, KRISS, KOBELLO, NIMT, NMIJ, NRCCRM, NTT-AT, RENESUS, SAIT, and TRC) participated and employed a range of measurement techniques such as ellipsometry, HR-RBS, SIM, TEM, XPS and XRR.
- APMP.QM-P09 (pH determination of phthalate buffer)
  Eleven organizations from seven economies(CERI, DSS, IDTI, Kato Co, KRISS,LIPI, NIMT, NMIJ, NML, NPLI and Wako Co) participated. Two NMIs used pH primary method with hydrogen electrode and others used glass electrode method.

Progress report on the following supplementary, bilateral and trilateral comparison were also presented.

- APMP.QM-SC1(Gravimetry)
- APMP.QM-SC1(Impurity analysis in methane)
- APMP.QM-K4.1(Ethanol in N_2)
- APMP.QM- K1.d (SO_2 in N_2)
- APMP.QM- k1.c (NO in N_2)

The 5th APMP TC Chairs' Meeting

The 5th APMP Technical Committee Chairs' Meeting was held on 30 - 31 March 2006 in Singapore. hosted by SPRING, Singapore.

- Each TC Chair and the delegate reported Quality System review status and APMP Key/Supplementary comparisons status.
- APMP Secretary explained the specific procedure for Quality System review.
  It has been agreed that CMCs of APMP member NMIs already published in Appendix C will be reviewed once a year in accordance with the decision of APMP General Assembly.
- APMP TC Chairs have arranged BMC/CMC definition to the satisfaction of APMP members in preparation for the 16th JCRB meeting held in April 2006.
- APMP TC Chairs have also agreed to a

tentative procedure of APMP Quality System review criteria, which is effective until December 2006.

Future Activities and Planning during the Meeting:

- 5th APMP TCQM Gas Analysis Workshop on 23-25 May 2007 in Xian, China.
- Workshop on MiC in mid June in China.
- The next (23rd) APMP GA and related meetings will be held in Sydney in October 2007 hosted by NMIA Australia.
- The General Assembly elected Indonesia as the venue of the 24th GA in 2008.

New Appointments

The General Assembly elected Dr Kwang-Haw Chung (from KRISS) as the chairperson-elect, Dr Vikram Kumar (NPLI) as the new DEC chair and Dr Vikram Kumar (NPLI) and Dr Hussein Akin (KIM_LIPI) as two new members of the Executive Committee. The General Assembly also approved the following new TC chairs: Dr R P Singhal (NPL, India) – TCL, Dr Saito Terubumi (NMIJ, Japan) – TCPR, and Dr Ho Seong Lee (KRISS, Korea) – TCTF.

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SADCMET Amount of Substance Annual Report to CITAC: 2007

In addition to strengthening metrology in the areas of trade measurement, the current focus in the SADC region is on traceability for measurement results in analytical chemistry and medical diagnostics. Special programmes are being conducted to provide reference materials for food analysis and various proficiency testing schemes have been piloted. Third party accreditation of food health testing laboratories is progressing and traceability in medical diagnostics is receiving special attention.

Metrology in chemistry activities in the region (outside South Africa) focus primarily on the establishment of accredited analysis or testing facilities in support of local export products. Traceability is sourced from outside the region and South Africa. Training is provided particularly by the National Metrology Institute of South Africa (NMISA) and the Physikalisch-Technische Bundesanstalt (PTB). Training in metrology (measurement toolkit) was provided to more than 100 small and medium enterprises in 5 countries, including Brazil (Table 1).

Further projects included assistance to fourteen food testing laboratories in Tanzania to the level where they could be assessed to ISO 17025 (sponsored by the Danish International Development Agency - DANIDA). The PTB also sponsored assistance to mass laboratories in Botswana and Zimbabwe.

Table 1: ***Measurement Toolkit rollout***

COUNTRY	DATE
Tanzania	18-19 May 2006
Ethiopia	13-14 June 2006
Lesotho	2-3 July 2006
Mauritius	4-5 July 2006
Brazil	31 July to 4 August 2006

A SADC water proficiency testing (PT) workshop was held in Gaborone from 20-24 November 2006 to discuss the completed third phase of a regional water PT scheme (sponsored by the PTB with assistance from the SADCMET secretariat at NMISA). The fourth phase including biochemistry, has since commenced.

Participation in the activities of the Consultative Committee on Amount of Substance (CCQM) of the International Committee of Weights and Measures (CIPM) in SADC is primarily performed by the NMISA. Kenya and Egypt (associate members of SADCMET) are in the process of submitting their first calibration and measurement capability (CMC) claims. A summary of metrology in chemistry and especially CCQM activities is provided below.

Gas Metrology

The national measurement standards for ambient air monitoring were maintained by stability testing and updating of the primary standard mixtures. The measurement standard for carbon monoxide (CO) in nitrogen consists of primary standard mixtures over the concentration range of 1 μmol.mol^{-1} to 10% mol.mol^{-1} and carbon dioxide (CO_2) in nitrogen ranges from 100 μmol.mol^{-1} to 20% mol.mol^{-1}. The primary standard mixtures for nitric oxide (NO) and sulphur dioxide (SO_2) in nitrogen range from 10 μmol.mol^{-1} to 1 % mol.mol^{-1}. Established stability data are now available for carbon monoxide and carbon dioxide for over 2 years and for nitric oxide and sulphur dioxide for a period of 1 year and 6 months, respectively. All these reference materials are now also commercially available.

The laboratory participated in a key comparison for carbon dioxide (CO_2) in synthetic air at ambient levels (360 μmol.mol^{-1}). This was the biggest key comparison organised by the CCQM gas analysis working group to date and the laboratory performed well compared to the other participants. The result of the laboratory was within 1% of the KCRV. However, the uncertainty quoted on the result was conservative compared to the other participants.

The laboratory also participated in a key comparison for the gravimetric preparation of 100 μmol.mol^{-1} oxygen (O_2) in nitrogen. Only preliminary results for this comparison have been discussed at the CCQM gas analysis working group meeting in April 2007.

The laboratory also participated in a bilateral comparison with NMi VSL in the Netherlands. The comparison was registered as a EUROMET project. The purpose of the comparison was to serve as a feasibility study to evaluate the capabilities of the NMISA to act as coordinating laboratory for a key comparison of carbon monoxide (CO) in nitrogen at 5 μmol.mol^{-1}. After several comparison rounds the two laboratories compare well, but the CCQM gas analysis working group indicated that they require more work to improve the uncertainty on the analytical capability of the NMISA, as well as the purity analysis of the CO parent gases.

For other regional comparisons, the laboratory participated in an APMP comparison for the gravimetric preparation of 12,5 % nitrogen (N_2) in helium with very good results. Finally the laboratory also participated in a pilot study for the gravimetric preparation of nitric oxide (NO) in nitrogen (30 to 70 μmol.mol^{-1}). The laboratory's results were excluded from the regression analysis of the results from the participants for the determination of the KCRV due to problems with purity analysis of the NO parent gases.

The laboratory is in the process of developing a methodology for the purity analysis of reactive parent gases, such as nitric oxide (NO) and sulphur dioxide (SO_2) by Fourier transform infrared spectroscopy (FTIR). The purity analysis of carbon monoxide by gas chromatography with flame ionisation detection in high purity nitrogen has been improved to the level where the carbon monoxide can now be detected at concentrations of as low as 20 nmol.mol^{-1}. Primary standard mixtures for carbon dioxide (CO_2) have been developed to include the balance gas of synthetic air over the concentration range of 100 μmol.mol^{-1} to 1000 μmol.mol^{-1} for the key comparison.

The development of the national measurement standard for ambient air monitoring will be completed in the coming year with the development and preparation of the primary standard mixtures for sulphur dioxide (SO_2) and nitrogen dioxide (NO_2) in synthetic air. The laboratory will then continue to develop a suite of multi component mixtures of permanent and reactive gases, primarily for the automotive industry as well as in preparation of a CCQM key comparison. The laboratory will also develop the capability to prepare condensable gas mixtures in the coming year for the development of a primary standard for ethanol-in-nitrogen for evidential breath testing. This technology will also be especially applicable to provide reference materials to SASOL and other polymer based industries.

During 2006 the accreditation of the laboratory was maintained with an extension of the scope to include the gravimetric preparation of nitric oxide (NO) and sulphur dioxide (SO_2) mixtures. The laboratory also underwent its initial assessment to ISO Guide 34 for the production of the certified gaseous reference materials.

Organic Metrology

The organic group kicked off 2006/7 by filling a large order for aqueous ethanol and sodium fluoride certified reference materials (preparation, assay and certification). This coincided with the preparation of three concentration levels for the second proficiency testing (PT) scheme for ethanol. The matrices for the previous scheme were blood and urine, while the second scheme was aqueous ethanol. The PT scheme continued for the rest of the year and comprised 12 different ethanol concentrations over 4 rounds. The scheme has served to assist laboratories with accreditation and demonstrating in-house laboratory capability for the analysis of ethanol in blood. Shortly after this, two more large orders for CRMs were received.

The Organic laboratory received accreditation to ISO 17025 during October 2006 and ISO guide 34 during February 2007. This was the first laboratory in SADC to receive SANAS accreditation to ISO guide 34 for the preparation of certified reference materials.

During April 2007, the NMISA attended the Organic Analysis Working Group meeting (CCQM OAWG) at the BIPM in Paris. The results for participation in CCQM-P31a.1 and P31c.1 were within 2-5% of the gravimetric values for the pesticides (lindane, 4,4'-DDT and 4,4'-DDE) and the PAHs (phenanthrene, fluoranthene, benz(a)anthracene, benzo(a)pyrene, benzo(ghi)perylene).

The laboratory also participated in the purity assessment pilot inter-comparison. The samples were distributed during September 2006 and the results were submitted during January 2007. PTB presented a possible study for the determination of chloramphenicol (CAP) in milk (a veterinary drug, CCQM-P90). Since the analysis of CAP is of concern for SA, the laboratory decided to develop and validate a method for participation in this study. The call for participation and study protocol was presented at the fall meeting in Korea and the sample distribution was planned for January 2007. Unfortunately due to homogeneity issues, the study has been delayed and the samples were only received in April 2007. The results are due in July 2007.

The laboratory was invited to present a paper on the production of certified reference materials at the Biological and Environmental Reference Materials (BERM 10) conference held in Charleston, South Carolina, USA. This was a great opportunity to showcase SADC and some of the reference materials that are provided. Two posters were also presented to highlight the ethanol and sodium fluoride certified reference solutions. Mention was also made of the gas primary standards that are in production at our laboratory. The paper was well received and published in the Accreditation and Quality Assurance Journal "M.Archer, B-J de Vos, MS Visser, *The Preparation, Assay and Certification of Aqueous Ethanol Reference Solutions*, September 2006.

Dissemination of measurement criteria is vital and the NMISA was invited to present a lecture at the Laboratory Management seminar on "A practical approach to method development and validation in metrology". Dissemination of analytical skills were also effected through the presentation of a one day "Basics of Gas Chromatography" course. The course was given during May 2007 and was attended by 29 delegates from industry and government laboratories.

The laboratory was also invited to visit PTB as part of a student program and a NMISA metrologist received training in the development of an isotope dilution liquid chromatography-tandem mass spectrometry (LC-MS-MS) method for the analysis of chloramphenicol extracted

from milk. This was an excellent opportunity since SA had agreed to participate in the CCQM-P90 inter-comparison.

Other activities included research into method development and validation;

- automation of the sodium fluoride primary method, thus implementing an additional reference method for the established CRM,
- re-modelling of a headspace sampler to improve the peak area repeatability from 4% to under 1,5%. This will assist with the development of a headspace gas chromatographic method using flame ionization detection for ethanol stability studies,
- a theophylline purity assessment involved research into exactly how to assign a value to a high purity material,
- pesticide research is ongoing, as are extraction and recovery methods for target analytes from complex matrices. A new project was also started for the preparation of industrial solvent CRMs in the Forensic field.

Inorganic Plasma Spectroscopy (IPS)

Primary and certified, single and multi-element calibration standards for inorganic elemental analysis are provided to the SADC analysis and testing community. The standards are obtained from National Metrology Institutes (NMIs), accredited institutions for the production of reference materials or prepared in the IPS laboratory at NMISA. The calibration standards are only used within their validity period and replaced as required. They are kept in a clean and temperature controlled environment. All measurement standards are used according to the ISO 17025 requirements and the IPS quality control procedures.

The IPS laboratory participated with particularly good success in a number of CCQM pilot and key intercomparison studies. For most of the measurands, the NMISA results and their respective associated uncertainty values compare quite well and were one of the better comparison results reported.
The relative differences for Ca, Fe, Cu and Zn contents in non-fat soybean powder from the recommended IDMS comparison values were in the range of 0.03 to 1.1%. Selenium in pharmaceutical supplement was determined with a relative difference of 0.3% from the comparison value. The estimated deviation of the result for Pb in wine (a key comparison) from the projected KCRV is 0.35%. The relative differences for Cu and Fe contents in wine (a pilot study) are expected to be well within 1% from the projected comparison values. Very good results were achieved for Pb, Cd and particularly for Fe and Zn determinations in bovine liver. The relative differences for Fe and Zn in bovine liver are expected to be less than 0.5 % from the comparison values.

Plans for participation in future comparisons are in progress. It is envisaged that the scope of intercomparisons will include a number of new measurands in the field of food nutrients, food contaminants, plastic materials and metal purity. Preparation of primary element standards by the IPS laboratory and their use in the comparison studies will be of high importance.

Surface and Microanalysis

The surface and microanalysis (SAM) laboratory continued with the project in assisting the automotive sector with the measurement of substances of concern (SOCs). Awareness, through roadshows and popular articles, was created among the various OEMs (original equipment manufacturers) in the automotive industry. This will now be extended to include the electrical and electronic equipment (EEE) industry and the SOCs will include the two brominated flame retardants (BFRs) targeted by the restriction of hazardous substances RoHS Directive 2002/95/EC.

Most of the original equipment manufacturers (OEMs) in the automotive industry were contacted and their analytical capabilities accessed. A definite need for laboratories providing reliable results was identified. In this process a laboratory database was compiled by NMISA and is hosted by the National Laboratory Association (NLA). The database will be extended as more and more laboratories come to the fore and express interest to do analysis of the substances of concern. These laboratories will have to be evaluated in some kind of round robin in the near future.

After the successful commissioning and training on the Time of Flight Secondary Ion Mass Spectrometer (TOF SIMS), the SAM laboratory participated in an inter-laboratory comparison study with NPL on the repeatability and consistency of TOF SIMS measurements on a polymer sample. The relative quantification of an organic matrix was also successfully completed as part of this study. A further study in progress on TOF SIMS is the quantification of trace metal analysis. This study already resulted in very positive results which will be published after the results are verified with other primary techniques.

The SAM laboratory provided measurement support to the CSIR nanotechnology group on various projects. A joint project on nano-phosphors between the University of the Free State, University of Kwazulu Natal, CSIR Build Environment and the SAM laboratory delivered positive results and the project will be continued into 2007/8.

On behalf of the South African Bureau of Standards (SABS), the SAM laboratory hosted the 13th plenary meeting of the ISO TC 202 Microbeam Analysis Working Group. Several members of the SAM group is actively involved in the development of ISO standards for electron microscopy, and host a local mirror working group to ensure that members of the South African microscopy community are up to date with developments in the field.

The SAM laboratory also participated in a pilot study on the determination of the nitrogen concentration in "Diamond-Like Carbon" (DLC) layers. In all the specimens provided, the SAM laboratory delivered high-quality results.

The scope of measurements for polymer analysis has been expanded with the successful commissioning of a thermogravimetric analyzer (TGA) and a pyrolysis-GC-MS.

An international project on developing vapour phase corrosion inhibitors for the packing industry have been secured, and a collaborative research project with the Royal

Table 2. ***NMISA Substance comparisons***

	International Number	Laboratory	Description
1	CCQM-K27	Organic	Ethanol in aqueous solution
2	CCQM-P20e	Organic	Theophylline purity
3	CCQM-P73	Gas	30 to 70 µmol/mol CO in nitrogen
4	EUROMET 900a	Gas	5 µmol/mol CO in nitrogen
5	CCQM-K52	Gas	360 µmol/mol CO_2 in air
6	CCQM-K53	Gas	100 µmol/mol O_2 in nitrogen
7	CCQM-P85	Inorganic	Determination of Essential Toxic elements in Bovine liver
8	CCQM-P86	Inorganic	Analysis of total Se in Pharmaceutical supplements
9	CCQM-K30	Inorganic	Analysis of Pb in wine
10	CCQM-P12.1	Inorganic	Analysis of Fe, Cu and Cd in wine
11	CCQM-P75	Inorganic	Stable isotope data of H,C, N, O & S in methionine
12	VAMAS 2002	SAM	Static TOF-SIMS Inter-laboratory study
13	CCQM-P80	SAM	C amount in precipitates in Fe
14	CCQM-P81	SAM	N amount in surface layers of Fe
15	CCQM-P-95	SAM	Determination of light elements-N doped C layers

Institute of Sweden on measurement techniques to characterise biodegradable polymers/plastics have been established through the National Research Foundations South Africa/Sweden Memorandum of Understanding. The measurement requirements of the South African plastics, polymer and rubber communities in South Africa will be further explored in 2007/8 through the First Polymer Technical Advisory Forum in August 2007.

Participation in International Comparisons

In total, the NMISA participated in 15 amount of substance comparisons, listed in Table 2.

Finally, a significant event in the region was the establishment of the National Metrology Institute of South Africa on 1 May 2007 through the Measurement Units and Measurement Standards Act (Act No. 18 of 2006) of South Africa. The new institute was established from the National Metrology Laboratory (NML), which was part of the CSIR. All personnel and assets were transferred to the new entity.

The NMISA is an independent public entity under the auspices of the Department of Trade and Industry of South Africa, the DTI.

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Report from SIM

Since 1995 when SIM was reactivated, Chemical metrology has been one of the most strategic fields to be developed among member countries in SIM. Especially since 1997 when the first SIM Chemical Metrology kick-off meeting was organized in Rio de Janeiro, many efforts have been made to promote chemical metrology in SIM.

In the last 10 years the SIM Program in Chemical Metrology had particular characteristics: NIST and NRC have led this initiative and the newly born CENAM played a complementary role to demonstrate what a small economy can do.

In order to most effectively address the unique needs of all 34 countries within SIM, whose capabilities in chemical metrology span a very broad range, SIM has focused its Chemical Metrology Working Group activities on training and capability assessment rather than participation in MRA-driven key and supplemental comparisons This has been accomplished through:

- Outreach and awareness activities within the ANDIMET CAMET, CARIMET and SURAMET subregions
- Chemical measurement proficiency assessment comparison studies and follow-up activities
- Training in CMC preparation and review

The long-term goal is to promote improved capabilities in chemical metrology in the region and increase participation of SIM members in CCQM and CIPM MRA-related activities, and now, Brazil, Chile Uruguay and Argentina are participating in CCQM activities.

In the last SIM Chemical Metrology WG meeting in Rio de Janeiro, 07-08 program plans were discussed. Some of the topics discussed were:

- Chemical metrology in the Americas Symposium to be held in conjunction with 2007 SIM GA during the week of Sept 24-28, 2007 in Ottawa
- Subregional activities focused on Impact of Chemical Metrology on Trade and Quality of Life

– CARIMET (St. Lucia in 07?)
– ANDIMET (Ecuador in 07)

- awareness seminar focused on various approaches for delivery of chemical measurement services
- guidelines for establishing a chemical metrology program

– CAMET (Honduras in 08 in conjunction with GA)
– SURAMET (Paraguay in 08)

- Interlaboratory Proficiency Assessment Studies
- Database on CCQM and RMO key comparisons and CRM development plans Potential CMWG Studies for 07 and 08
- SIM.8.12P1 Automotive Exhaust Emissions
- SIM.8.21P Natural Gas
- SIM.8.16P1 Toxic Metals in Seafood II (could include pesticides)
- SIM.8.22P Cholesterol and Toxic Metals in Meat Homogenate (include pesticides?)
- SIM.8.19P Carbon Dioxide in Stack Emissions (carbon dioxide?)
- Trace Elements in Water (connect to CCQM Key Comparison)

In May 2007, SIM CMWG Awareness Seminar "Various Approaches for Delivery of Chemical Measurement Services of Higher Metrological Order in the Food Sector" was organized in Inen Ecuador.

The SIM-CMWG conducted discussion rounds in the context of its subregional awareness raising activities in Lima, Perú (June 2005) and Montevideo, Uruguay (May 2006) on how to design Metrology in Chemistry Programs on the national level. The need for orientation of responsible management level staff on the existing possibilities to deliver measurement services on reference level became evident. The objective of this seminar was to enhance knowledge at SIM NMIs and/or designated institutes on the range and nature of existing possibilities to deliver reference measurement services of a higher metrological order.

As an invited speaker, Dr. Laurie Besley of NMIA gave a talk on value-assigning PT sample, and also Dr. Robert Kaarls, Dr. Willie May (NIST) and Dr. Mariana Arce (CENAM) presented their experiences on metrological services in chemical metrology.

It is expected that SIM NMI metrology in chemistry managers are aware of the possibilities and characteristics of different approaches to deliver reference metrological services of a higher metrological order. In this event PTB technical cooperation provided conceptual input and detailed report.

The follwing are recommendations derived from this seminar for a second phase project in MiC: Being that political support is critical to implement an infrastructure in MiC, it is recommended to organize specific activities, such as seminars or individual meetings, to address policy decision makers. During these activities, the impact of MiC on trade, environment and health should be clearly explained.

Also recommended to ensure the continuity of the effort:

- How could the project fulfill the expectations stated during this first workshop?
- What kind of interaction will be among groups?
- How could the project motivate the transfer of knowledge and experience among countries in each group and among groups?
- How could the project provide assistance to a whole group and at the same time attend to specific needs of a country?

Under the theme of Chemical Metrology in the Americas: Current Practices and Future Needs, a symposium was held in conjunction with 2007 SIM GA during the week of Sept 24-28, 2007 in Ottawa.

- Plenary Session on Current State of the Practice
  - Impact on trade and quality of life
  - Worldwide infrastructure
  - 10-years progress in SIM
- Poster session on current activities for each country
- Needs for developing economies (focused on trade and quality of life)
- New emerging technologies
  - Energy
  - Bio and Health
  - Nano

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Sampling - Validation, Quality Control and Uncertainty Estimation

Method validation and quality control have been implemented in analytical laboratories since the late 1970s, e.g.: [1]. With the publication of guidance documents for uncertainty estimation, e.g.:[2], implementation of uncertainty estimation in analytical laboratories has been well under way since the mid 1990s. Still, the use of concepts such as validation, quality control and uncertainty estimation has been confined to the analytical processes. A recent inquiry to the 20 major Danish operators of groundwater investigations demonstrated the all used laboratories accredited according to ISO 17025 [3] for analysis, an accreditation that requires validation and quality control of applied analytical methods. Most operators specified quality requirements to the analysis, although only half of them actually verify the analytical quality obtained. On the other hand, only 1/3 of the operators applied quality control procedures of sampling and only 10% required accredited sampling.

As sampling uncertainty may be considerably larger than analytical uncertainty (examples in [4]), estimation and reduction of sampling uncertainty are easily identified as the next important targets in controlling measurement uncertainty.

With the guidance of the recently published Eurachem/EUROLAB/CITAC/Nordtest/AMC Guide: *Measurement uncertainty arising from sampling: a guide to methods and approaches* (available at, http://www.eurachem.org) the first steps towards making this practicable have been taken. Accordingly, quality control of sampling is recommended in the guidance document

on groundwater monitoring published by the European Commission [5].

The Nordtest handbook on uncertainty from sampling

A handbook has now been prepared supported by Nordtest under the Nordic Innovation Centre on uncertainty from sampling [4]. The Nordtest handbook is an extract of and based upon the principles, methods and text of the Eurachem guide on uncertainty from sampling [6]. The handbook provides practical guidance on sampling uncertainty estimation in the Nordtest handbook format and will be freely available at the Nordtest web site at http://www.nordicinnovation.net/nordtest.cfm, under NT technical reports, report number NT tec 604. Until the final report is available on the Nordtest web site, a final draft of the Nordtest handbook is available at http://www.samplersguide.com

Sampling purpose and quality requirements

The first part of the handbook emphasizes the fact that all measurements are done for a purpose and that sampling in most cases is an integrated part of the measurement process. Therefore the target of the sampling and the required sampling quality depends upon the purpose. The role of sampling in the uncertainty and decision chain can be seen from Figure 1. In the handbook, examples are given on how to quantify the required sampling uncertainty, e.g.: on setting sampling quality objectives.

Sources of uncertainty

To support efforts to control sampling uncertainty, the contributions to sampling uncertainty are discussed and divided into the well known concepts of random and systematic errors. A range of tools are presented for the estimation of different types of uncertainty, see e.g.: Table 1, including equations for calculating sampling uncertainty.

Furthermore, the requirements for competence of both samplers and samplings planners are discussed and the required documentation of sampling is described in details. The minimum documentation stipulated is:

Table 1 ***Uncertainty contributions from random and systematic effects***

	Random (precision)	*Systematic (bias)*
Analysis	Analytical variability – including sample splitting/preparation and handling (combined contribution of random effects)	Analytical bias (combined effect of bias sources)
Sampling	Sampling variability (dominated by heterogeneity and operator variations)	Sampling bias (combined effect of selection bias, operator bias etc.)

Validation and quality control of sampling

The concepts of method validation and quality control familiar from analysis are then explained as tools that can be used for sampling as well. A major barrier is here, that sampling may appear either as one or a few samples taken at each occasion, or as large sampling campaigns at one site. The use of validation and quality control is described in order to accommodate to different types of sampling without requiring an excessive number of quality control samples (and derived costs) compared to the samples required anyway for the measurements.

The methods presented are the replicate method for both validation and quality control, in addition to a time series based control chart type of quality control.

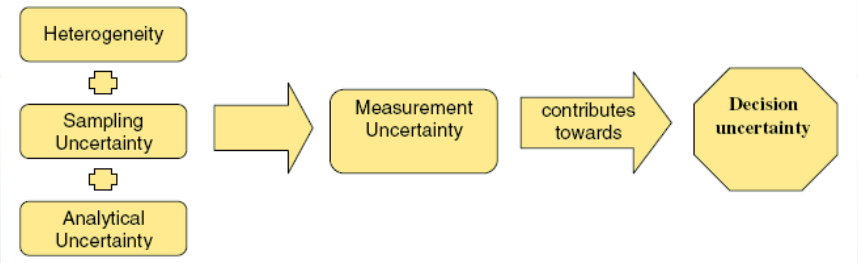

Figure 1. ***Sampling in the uncertainty and decision chain***

- Written sampling procedures based upon defined sampling methods
- sampling field report
- sampling report stating uncertainty of sampling

Whereas this may seem trivial as seen from the world of analytical chemistry, these requirements are not commonly met for sampling. The Danish inquiry mentioned above demonstrated that ¼ of the major Danish operators of groundwater investigations did not apply a written procedure and even when a written procedure existed, only 15% of the procedures were based upon a standard method. Less than 1/3 of the operators would be able to provide estimates of sampling uncertainty.

Uncertainty estimation

The major part of the Nordtest handbook describes the methods that can be used for estimating the sampling uncertainty. The methods are based upon the replicate design, and the statistical methods are range statistics and two types of ANOVA. The methods are described in details showing all calculations in order to facilitate implementation. Guidance is given with respect to selecting the most appropriate statistical methods for the purpose, although the differences caused by using different statistical approaches in some case may seem very minor, see Table 2.

Finally, the use of variography as an alternative tool is described.

Table 2. ***Example of calculation of uncertainty contributions using different statistical methods Data from sampling vitamin A in baby food***

	$s_{analysis}$	$RSD_{analysis}$	$s_{sampling}$	$RSD_{sampling}$	$s_{measurement}$
	µg/100 g	%	µg/100 g	%	
Range – single split	-	-	-	-	42
Range – double split	30	8.6	19	5.5	35
ANOVA	29	8.3	17	5.0	34
Robust ANOVA	31	8.8	21	6.1	37

Practical examples

Perhaps the most important part of the Nordtest handbook is a series of worked examples demonstrating in details the use of the methods presented for different sectors: groundwater (environmental monitoring), iron ore (production control), baby porridge (product quality control) and wastewater (environmental/ process control). The examples are given showing all data used and all steps of the calculations in order to facilitate understanding of the principles and methods.

Perspectives

The main message of the handbook is that uncertainty from sampling can be estimated with reasonable effort and following steps that are comparable to those used in quality assurance of laboratory analysis: method validation and quality control. Still, the main emphasis has until now been upon the uncertainty contributions from random effects.

We urgently need practical tools for controlling the uncertainty contributions to sampling from systematic errors comparable to those available in analytical chemistry: proficiency tests and certified reference materials. Examples of such methods that are still only sparsely available are:

- Sampling method studies
- Sampler proficiency tests
- Sampling reference sites

Among the efforts currently in progress in order to further enhance implementation of uncertainty control in sampling are:

- Preparation of horizontal sampling standards for sampling of dynamic and stationary systems irrespective of sector supported by Nordtest/Nordic Innovation Centre
- Implementation of personnel certification of environmental samplers, also in a Nordtest scheme
- Preparation of textbooks and exams for education of sampling staff supported by the European Leonardo program for the implementation of a European Community vocational training policy.

Although the process of understanding and controlling sampling uncertainty to the same extent as analytical uncertainty has thus been started at the technical level, implementation and enforcement will not be realized until required by regulations and by the customers.

Acknowledgements

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Measurement Uncertainty Revisited: Alternative approaches to uncertainty evaluation

This EUROLAB Technical Reports, Measurement Uncertainty Revisited: Alternative approaches to uncertainty evaluation is focussed on reviewing, comparing and giving some examples of the currently available approaches for evaluating measurement uncertainty of quantitative test results. After more than ten years since publication of the 1st edition, the *Guide to the Expression of Uncertainty in Measurement*, known as the GUM, is acknowledged as the master document on measurement uncertainty throughout the testing community. The term "measurement uncertainty" is recognised to apply to all types of quantitative test results, and the GUM principles are fully accepted. Among others, these principles require that

- uncertainty evaluation is comprehensive, accounting for all relevant sources of measurement error;
- uncertainties arising from random and systematic effects are treated alike, i.e. are expressed and combined as variances of associated probability distributions;
- statistical evaluation of measurements (Type A) and alternative techniques, based on other data / information (Type B), are recognised and utilised as equally valid tools;
- uncertainties of final results are expressed as standard deviations (standard uncertainty) or by multiples of standard deviations (expanded uncertainty) with a specified numerical factor (coverage factor).

However, when it comes to evaluating the uncertainty of the results for a (quantitative) test procedure, the GUM almost exclusively treats a single approach for uncertainty evaluation: the modelling approach based on a comprehensive mathematical model of the measurement procedure. Following the GUM principles experimental approaches have only recently received greater attention. They are based on whole-method performance investigations designed and conducted so as to comprise the effects from as many relevant uncertainty sources as possible. The data utilised in these approaches are typically precision and bias data obtained from within-laboratory validation studies, quality control, interlaboratory method validation studies, or proficiency tests. Such approaches are fully compliant with the GUM, provided that the GUM principles are observed.

Focusing on the first principle from the bullet points above, the basic requirements for any valid uncertainty evaluation are:

- a clear definition of the measurand, i.e. the quantity to be measured
- a comprehensive specification of the measurement procedure and the test items
- a comprehensive analysis of the effects impacting the measurement results.

Given a comprehensive list of relevant effects/ uncertainty sources, uncertainty evaluation may be carried out using various different approaches. They range from individual quantification and combination of input uncertainties to collective quantification, e.g. using a reproducibility standard deviation for a standard test procedure. Handling of uncertainty information requires due attention to the scope and the form of the data concerned. For example, the results obtained using "empirical" approaches normally refer to the typical performance of a specified test procedure on specified test objects, while uncertainty estimates obtained using the modelling approach most often refer to individual measurement results.

Concerning empirical approaches for uncertainty evaluation, the use of the reproducibility standard deviation from an interlaboratory method validation study has recently been firmly established (see ISO/TS 21748). The use of within-laboratory data, that is, data from method validation studies and quality control carried out in the laboratory is also widely recognised as a valid approach. Concerning the use of laboratory performance data from proficiency tests, some proposals have been published but the approach is still under debate.

Outline of the Eurolab Report:

Chapter 1 provides a summary of the current main approaches for uncertainty evaluation: (i) the modelling approach, (ii) the single-laboratory validation approach, (iii) the inter-laboratory validation data approach and (iv) the proficiency testing approach. The use of PT data is still under debate and authoritative references are few. Therefore in chapter 2 a full description of the approach using PT data is given. Chapter 3 is devoted to technical issues pertinent to comparison, validation and revision of uncertainty estimates. As the core part of this report, chapter 4 presents a range of examples. These are case studies from various testing fields, where different approaches were used to evaluate the relevant uncertainty and the results so obtained were compared. Some conclusions and recommendations complete the main body of the document. Finally the Annex presents a compilation of selected standards, guidelines, books and websites on measurement uncertainty.

The report is available under Documents at www.eurolab.org

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on behalf of the Working Group that consisted of ***Werner Hässelbarth, BAM (convenor) Stephen Ellison, LGC, Manfred Golze, Anita Schmidt BAM, Ulf Hammerschmidt, PTB, Wilfried Hinrichs, Materialprüfanstalt für das Bauwesen, Ulrich Kurfürst, Fulda University of Applied Sciences, Teemu Näykki, Finnish Environment Institute, Marc Priel, LNE, Burkhard Peil, DACH, and Pedro Rosario Ruiz, Real Casa de Moneda.***

Reports of International Organizations

Information from the EuCheMS Division of Analytical Chemistry

After nine year in office, Heiner Korte retired as the secretary of the DAC and the secretary elect is Jens E.T. Andersen of the Danish Chemical Society. The new secretary, the former secretary and the chairman Bo Karlberg met in Copenhagen in October to prepare for the takeover. The decision was made at the annual meeting of DAC held in conjunction with the Euroanalysis XIV conference held in Antwerp, September 9-12. At this meeting it was also decided that DAC supports continuation of the Study Group of Quality Assurance. Jens E.T. Andersen took over from Wolfhard Wegscheider after his long time position as head of the Study Group. Jens E.T. Andersen was also appointed as the liaison person to EuraChem and to CITAC.

The Euroanalysis conference is the main event of the DAC, and was excellently organised by Koen Janssens and Luc Van't Dack under the auspices of Flemish Chemical Society KVCV. The conference was held under the general theme "The role of analytical chemistry in the preservation of mankind's natural and cultural environment", and it was attended by 650 participants from 50 countries world wide providing more than 800 contributions. The stimulating scientific program, including fully booked poster sessions, was flavoured with interesting booths of instrument manufacturers and publishers. Prof. Alfredo Sanz-Medel of Oviedo University, Spain held the Robert Kellner Lecture that is sponsored by Springer Verlag. The Merck Award was awarded to Alexander Makarov of Thermo Finnigan for his development of the orbitrap analyzer of mass spectrometry and to Prof. Shuming Nie of Georgia Institute of Technology for introducing quantum dots to clinical analysis and diagnosis.

Euroanalysis XV with the motto "The Impact of Analytical Chemistry on Quality of Life" is planned for Innsbruck, September 6-11, 2009, can be viewed at www. EUROANALYSIS2009.at.

In 2008 DAC will contribute to the Second General Conference on Chemistry in Turin September 16-20. DAC focuses on metrology in chemistry and thus promotes the continuing effort within the field of quality assurance Prof. Manfred Grasserbauer will present a lecture entitled "The Environmental Challenge for Analytical Sciences". This work continues the process of involving in DAC the analytical chemists of the public sector, industry and academia.

Europe has a central role over the centuries in developing analytical chemistry and Prof. Duncan Burns continues his effort within the framework of DAC with "Contributions to the History of Analytical Chemistry in Europe made via DAC-EuCheMS". This work resulted in several publications including the latest contribution to Analytical Chemistry of Italy published this year preceding the conference in Turin 2008.

It is important to DAC to maintain networking with the other divisions of EuCheMS which are concerned with parallel scientific issues. This cooperation is ensured by appointing liaison persons who relate to education, history, life sciences, food, environment, electrochemistry, computations, microsystems, IUPAC and Eurachem. The developments of quality assurance and quality control are followed with great interest because these subjects have a profound impact on a wide range of applications that are also considered and supervised by the European commission. Although highly specialised conferences are increasing in number and popularity, it is important to communicate actively across the borders defined by technologies to ensure a high level of science, education and innovation. The contact with other divisions, with supranational boards and with non-European societies is maintained by observers. Updated information on DAC activities may be found at www.dac-euchems.org

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JETA, Denemark

Heiner Korte on the left, Jens E.T. Andersen on the centre and Bo Karlberg on the right

Award Winners 2007

CITAC Most Interesting/Important Papers on Metrology in Chemistry 2007

The aim of the CITAC award for the most interesting/important papers on Metrology in Chemistry winners for 2007 is to highlight remarkable papers in the field of Metrology in Chemistry which may not have been "discovered" by the wider community after their publication in a national journal (for example in a local language) and are not readily accessible to the larger metrological/analytical community. The award is normally awarded to three papers. CITAC also strives to "explain" publications in known international journals written in "sophisticated language" to communities. Finally, it aims to draw attention to papers with scientific content important to the wider community.

The nomination is coordinated by a CITAC member elected for three years at a CITAC members meeting. For 2007-2009, Dr Wynand Louw, the current Vice-chair, was selected as the "CITAC Coordinator for Nomination". Any member can nominate a paper by sending a supporting letter and an abstract (by e-mail) of the paper in English. The supporting letter should include the name of the paper's corresponding author and his/her e-mail address. The deadline for the nomination is 1 September, to enable the process to be concluded by 1 October of the same year. The Coordinator sends the nominations to CITAC members by e-mail to vote for the three most appropriate papers. All votes received by 1 October are counted. The winners are notified by 1 November.

For 2007, three nominations were received. After verification that the nominated papers fulfill the selection criteria, all three were selected as winning papers, and the CITAC committee on behalf of the members wants to congratulate Venkatesh Iyengar, Bertil Magnusson et al and David L. Deuwer with the selection of their papers and the contribution made to metrology. The authors will each receive a "CITAC Award Winner" certificate at the next CITAC Members' meeting in 2008. Results of the CITAC award procedure (including abstracts of the papers) will be published in ACQUAL and other journals. The links can be followed for the complete articles and brief abstracts are provided below.

David L. Deuwer, How to combine results having stated uncertainties: to MU or not to MU? published in: Ales Fajgelj, Maria Belli, Umberto Sansone (Eds.) Combining and reporting analytical results. Royal Society of Chemistry, London (2007), pp 127-142.

This comprehensive report paper deals with the widely accepted issue that knowledge of the expected uncertainty of a measured value is necessary for judging the measurement's fitness-for-purpose, but some experienced data analysts argue against using measurement uncertainty (MU) in even relatively simple univariate applications such as using interlaboratory data to assign the expected value of a measurand in particular materials. The question is then if MU is not expected to be useful for univariate tasks, can it be useful in multivariate applications? More generally, can estimates of MU provide quantitatively useful chemical information when there may be significant bias among the data analyzed, such as when measurements are from more than one source or are taken over a long period of time?

This report describes the use of publicly available CCQM (Consultative Committee for Amount of Substance) data to explore whether appropriately evaluated MU estimates can be useful in combining results from interlaboratory studies. Three different approaches to using MU are evaluated (weighting, bootstrap resampling, and mixture-models) relative to the performance of a number of commonly used or recently proposed evaluation metrics.

Bertil Magnusson, Teemu Näykki, Håvard Hovind and Mikael Kryssel, Handbook for Calculation of Measurement Uncertainty in Environmental Laboratories published by Nordtest Tekniikantie 12, FIN-02150 Espoo, Finland; www.nordtest.org

The handbook is written for environmental testing laboratories in the Nordic countries, in order to give support to the implementation of the concept of measurement uncertainty for their routine measurements. The aim is to provide a practical, understandable and common way of measurement uncertainty calculations, mainly based on already existing quality control and validation data. Practical examples, taken directly from the everyday world of environmental laboratories are presented and explained.

The handbook covers all steps in the analytical chain from the arrival of the sample in the laboratory until the data has been reported. The approach is very general and should be applicable to most testing laboratories in the chemical field. It is therefore envisaged that many more laboratories over the world will benefit from this wider exposure

Venkatesh Iyengar, **Metrological concepts for enhancing the reliability of food and nutritional measurements** published in the Journal of Food Composition and Analysis 20 (2007) 449-450,

The paper deals with the concept that to incorporate metrological approaches into the measurement process enhances the reliability of analytical findings. These developments include: high-quality reference standards, validated methods, robust sampling practices, proven calibration approaches, natural matrix reference materials, speciation chemistry, assessment of measurement uncertainty and establishment of traceability links, certified reference materials to facilitate one aspect of traceability, and proficiency testing, among others. Abbreviated versions of the papers are published in this newsletter. The readers of CITAC News are invited to access the full documents.

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Award Winners 2007

How to Combine Results Having Stated Uncertainties: to MU or not to MU?

It is widely accepted that knowledge of the expected uncertainty of a measured value is necessary for judging the measurement's fitness-for-purpose. Considerable effort continues to be expended on how to best estimate and report measurement uncertainty (MU) for chemical and biological measurands. It is therefore perhaps surprising that some experienced data analysts argue against using MU to help estimate consensus values for interlaboratory studies [1,2].

The primary concern of those who advise against using MU is that estimating MU is not yet sufficiently routine and standardized to ensure that different participants interpret and report the uncertainties in their values in a consistent manner. Any enquiry into the potential utility of MU for assigning consensus values to interlaboratory study materials therefore requires, at a minimum, that there be philosophically consistent MU estimates for all of the reported measurements and trustworthy reference values (RVs) for the studied measurands. Few, if any, "routine" interlaboratory studies currently meet this standard.

Beginning in 1993, a number of very non-routine interlaboratory studies involving various chemical measurands have been performed under the auspices of the Consultative Committee for Amount of Substance – Metrology in Chemistry (CCQM). The member organizations of the CCQM have considerable expertise in chemical metrology and experience with MU evaluation. All of the samples used in the CCQM studies are thoroughly characterized for homogeneity and stability. All of the individual values are reported with a symmetrical uncertainty estimate of defined confidence. The source of every datum in these studies is fully attributed. Reliable RVs, assigned either by gravimetric sample preparation or extensively debated expert review, are available for all measurands.

Data

As of October 2007, data are publicly available for 171 Key Comparison (KC) measurands evaluated by the member organizations of the CCQM [3]. KCs are formal studies designed to establish the extent of measurement agreement among the participating CCQM member organizations and their official delegates for particular measurands. All of the measurement values submitted for a particular KC are made public with the formal acceptance of the study's Final Report. Depending on the type of sample and measurands investigated, some form of RV is established for all measurands. Many of these RVs are assigned through gravimetric preparation verified by measurement, others are assigned through a combination of consensus technical judgment and statistical analysis. No participant's KC result can be withdrawn from publication once submitted; however, results that are technically suspect are generally not used in assigning RVs.

Measurement results for all KCs must be reported as values with an associated MU of defined confidence. For all of the data in the published CCQM studies, the confidence intervals are symmetric about the expected value and are expanded with the intent to include the true value with about 95 % confidence. The data for the i^{th} participant in the j^{th} data set can thus be denoted: $x_{ij} \pm U_{95}(x_{ij})$ and displayed as a dot-and-bar in a measurement equivalence graph as in Figure 1. However, since all of the MUs in these CCQM studies are expressed as symmetric 95 % confidence intervals, the CCQM measurements can also be interpreted as normal kernel densities with expected value x_{ij} and standard deviation proportional to $U_{95}(x_{ij})$ but covering the true value with about 68 % confidence, $u(x_{ij})$ [4]. For normal distributions $u(x_{ij}) \cong U_{95}(x_{ij})/2$. Just as each measurement kernel, $N(x_{ij},u_{68}(x_{ij}))$, represents the expected probability density function (PDF) of the true location given the measurement, the sum of the n_j kernels defines a mixture-model PDF (MM-PDF) for the combined set of measurements $\mathrm{PDF}_j = \sum_{i=1}^{n_j} N(x_{ij},u(x_{ij}))/n_j$. This alternate kernel density and PDF representation of the CCQM measurements is also displayed in Figure 1. The PDF_j at the right of the graph is a marginal distribution for the combined results.

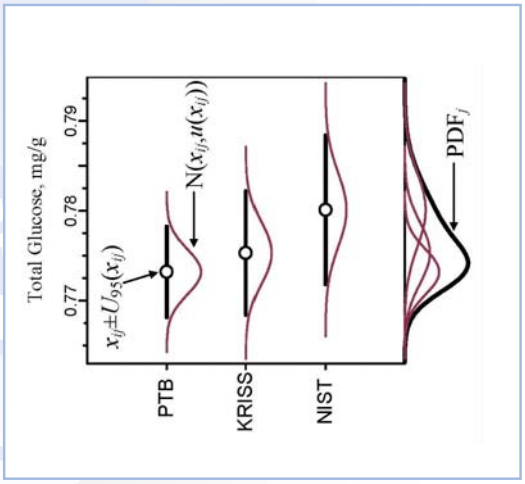

Figure 1. Measurement Equivalence Graph for CCQM-K11: Total Glucose in Human Serum, Material I

Location Estimators

There are a very large number of summary statistics that are routinely used or have been proposed for estimating a measurand's "true value" from interlaboratory study results. Each of these location estimators makes somewhat different assumptions about the nature of the data being combined and summarized. The extent of agreement between the estimated location and "truth" depends upon how well the observed data match these underlying assumptions. Only a few, hopefully representative, estimators are examined in this report: the MU-ignoring mean, median, H15, L1 ½ and shorth; and the MU-using Graybill-Deal (WtU) and Mandel-Paule (WtMP) weighted means and the relatively novel MM-

PDF-based MMmode, MMmedian and MMshorth. Except for the mean, the MU-ignoring estimators are relatively robust to "outlier" values. The WtU has been recommended as the "best" estimator when all of the x_{ij} are believed to represent a single normally distributed population and all $U_{95}(x_{ij})$ are credible [5]. The WtMP has been recommended for use when some of the $U_{95}(x_{ij})$ may be underestimated. The MMmode is the location where the MM-PDF is largest, the MMmedian is the location which divides the PDF into two sections of equal area, and the MMshorth is the mid-point of the shortest interval that spans half of the area of the PDF. These and other estimators are fully defined in the complete report and elsewhere [6,7].

Results and Discussion

As discussed in the complete report, the majority of the CCQM KC measurement values agree well with the assigned RVs and most MU estimates appear realistic. However, about 10 % of the x_{ij} are location "outliers" (more than two dispersion units from the RV) and about 30 % of the $U_{95}(x_{ij})$ are under-estimated (the $x_{ij} \pm U_{95}(x_{ij})$ interval does not include the RV). Given these imperfections and the number and diversity of the measurands, the KC data should present the various estimators with a variety of non-ideal data structure to challenge their performance. Comparison of the estimator values to the assigned RVs over all of the KC data sets may thus provide a realistic assessment of estimator properties.

The expected location of each of the 171 publicly available CCQM KC data sets was estimated with the various location estimators described above using purpose-built spreadsheet software [8]. The redundancy in the resulting 11 row (10 location estimates plus the assigned RV) by 171 column (measurands) data matrix was removed using principal components analysis [9]. The resulting 11 row by 10 abstract-factor score matrix captures 100 % of the covariance information contained in the original data matrix. Location estimators that respond similarly to the various challenges presented in the evaluation of the 171 measurands will have similar score coefficients, c_{mn}, where m is the estimator (row) index and n is the factor (column) index. It is convenient to quantitatively summarize the similarities of the different estimators with the Euclidean score distance: $D_{p,q} = \sqrt{\sum_{n=1}^{10} (c_{pn} - c_{qn})^2}$, where p and q are the row designations for two estimators. Should the two estimators have exactly the same properties, the value of $D_{p,q}$ will be 0.0; the value of $D_{p,q}$ will increase as the similarity between the estimators' behavior decreases.

Figure 2 displays the $D_{p,RV}$ for the ten estimators examined relative to the RV. The estimators are sorted in order of decreasing similarity to the RV. These distances suggest three broad conclusions. First, none of the estimators provides locations that are closely related to the RV: unguided statistics do not replace expert evaluation. Second, of the estimators evaluated, the MMmedian is the most similar to the RV: chemical MU estimates can provide quantitatively useful location information. Third, the (MU-using) WtU and WtMP weighted mean estimators are less similar to the RV than are most of the robust MU-ignoring estimators: while chemical MU estimates can provide useful information, as always the devil is in the details of how that information is used.

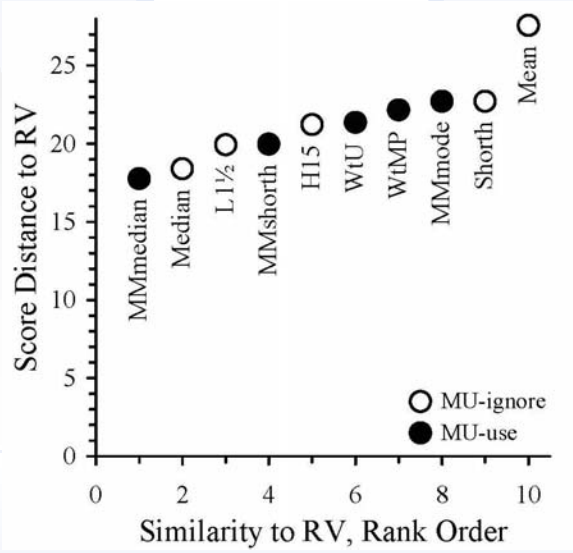

Figure 2. Factor-Score Distance of Location Estimators to Reference Values

The article is extracted (and lightly updated) from: Ales Fajgelj, Maria Belli, Umberto Sansone (Eds). Combining and reporting analytical results. Royal Society of Chemistry, London (2007), pp 127-142.

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Award Winners 2007

Nordtest Handbook for Calculation of Measurement Uncertainty Based on Quality Control and Method Validation

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The aim with this handbook is to provide a practical, understandable and common way of measurement uncertainty calculations based on already existing quality control and method validation data for test procedures in routine use. To illustrate the approach the estimation of measurement uncertainty for some routine methods in environmental laboratories are presented.

The approach follows the general ISO and EA guidelines for measurement uncertainty and uses the possibility of grouping the individual uncertainty components into different categories. The first category is the intermediate precision within a laboratory, here called reproducibility within laboratory, u(Rw) and the second category is the method and laboratory bias, u(bias). The third category is the reproducibility from interlaboratory studies, u(R). The error model used is a simplification of the model presented in the ISO guide 21748.

However the first and most important step is always definition of the measurand and specification of the required measurement uncertainty. Starting with this information the handbook then tries to cover all steps in the analytical chain from the arrival of the sample in the laboratory until the data has been reported. It is important to notice that vital parts of the total measurement uncertainty are not included, e.g. sampling, sample transportation.

It is recognised that while the recommendations presented do form a valid approach to the evaluation of measurement uncertainty for many purposes, other suitable approaches may also be adopted especially the EURACHEM/CITAC Guide (www.eurachem.com) which, in the second edition, also stresses the use of method validation data. Another good source is the Eurolab guide 2007 (Measurement Uncertainty revisited, www.eurolab.org). The English version [1] is available at www.nordicinnovation.net, Nordtest, Tecn. Report TR537.

Introduction

There are several ways that a laboratory can choose to estimate measurement uncertainty for results from a method in routine use. We propose a model where either the reproducibility within-laboratory (Rw) is combined with estimates of the method and laboratory bias, or the reproducibility sR is used more or less directly, ISO Guide 21748.The alternative way is to construct a detailed fish-bone diagram and calculate/estimate the individual uncertainty contributions. This approach may prove very useful when studying or quantifying individual uncertainty components. It has been shown, though, that in some cases this methodology underestimates the measurement uncertainty, partly because it is hard to include all possible uncertainty contributions in such an approach.

By using existing and experimentally determined quality control (QC) and method validation data, the probability of including all uncertainty contributions will be maximised.

Flow Scheme for Uncertainty Calculations

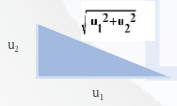
The flow scheme presented forms the basis for the method outlined in this handbook. The flow scheme, involving 6 defined steps, should be followed in all cases. The example with NH_4-N in water shows the way forward for calculating the measurement uncertainty using the flow scheme. For each step, there may be one or several options for finding the desired information.

Background for the NH_4-N example – automatic photometric method: The laboratory has participated in 6 interlaboratory comparisons recently. All results have been somewhat higher than the nominal value. The laboratory therefore concludes that there may be a small positive bias. On average, the bias has been +2.2 %. This bias is considered small by the laboratory and is not corrected for in their analytical results, but exists, and is thus another uncertainty component. The data from the interlaboratory comparisons are shown in Table 1. For this method, the main sources of uncertainty are contamination and variation in sample handling, both causing uncertainty components. These uncertainty sources are included in the calculations below.

Table 1. Results for a laboratory from interlaboratory comparisons of NH_4-N in water

Exercise	Nominal value xref	Laboratory result xi	Bias	sR	Number of labs
	µg/L	µg/L	%	%	
1999 1	81	83	2.5	10	31
2	73	75	2.7	7	36
2000 1	264	269	1.9	8	32
2	210	213	1.4	10	35
2001 1	110	112	1.8	7	36
2	140	144	2.9	11	34
$\bar{x}$			**+ 2.20**	8.8	34
RMS (Root Mean Square)			**2.26**		-

Table 2. ***Flow scheme for uncertainty calculations***

Step	Action	Example – Ammonium NH_4-N
1	Specify Measurand	Ammonium is measured in water according to EN/ISO 11732/11/. The customer demand on expanded uncertainty is ± 10 %
2	Quantify R_w comp. A control sample B possible steps not covered by the control sample	A: Control limits are set to ± 3.34 % (95 % confidence limit) B: The control sample includes all analytical steps.
3	Quantify bias components	From interlaboratory comparisons over the last 3 years the % difference from nominal value were 2.4; 2.7; 1.9: 1.4; 1.8: and 2.9. The root mean square (RMS) of the bias is 2.26 %. The uncertainty of the nominal values is *u(Cref)* = 1.5 %. (see Table 1)
4	Convert components to standard uncertainty u(x)	Confidence intervals and similar distributions can be converted to standard uncertainty [2,3]. *u(Rw)* = 3.34/2 = 1.67% $u(bias) = \sqrt{RMS_{bias}^{\ 2} + u(Cref)^2}$* $=\sqrt{2.26^2+1.5^2} = 2.71\%$
5	Calculate combined standard uncertainty, u_c  	Standard uncertainties can be summed by taking the square root of the sum of the squares $u_c = \sqrt{u(R_w)^2 + (u(bias))^2} = \sqrt{1.67^2 + 2.71^2} = 3.18$
6	Calculate expanded uncertainty, $U = 2 \cdot u_c$	The reason for calculating the expanded uncertainty is to reach a high enough confidence (app. 95 %) in that the "true value" lies within the interval given by the measurement result ± the uncertainty. $U = 2 \cdot 3.18 = 6.36 \approx 6$ % or 7 %.

* (u(bias) denotes the uncertainty contribution from the bias estimate to the measurement uncertainty. It is not the uncertainty of the bias value. The bias is here estimated by single results (differences) from PT rounds. If CRM is used several determinations over a longer timeperiod of each CRM is used to estimate the bias.

The measurement uncertainty for NH_4-N will thus be reported as ± 6 % or 7 % at this concentration level.

Summary table for uncertainty calculations

The results of the calculations done in the flow scheme will then be summarised in a summary table.

Ammonium in water by EN/ISO 11732 Measurement uncertainty U (95 % confidence interval) is estimated to ± 7 %. The customer demand is ± 10 %. The calculations are based on control chart limits and interlaboratory comparisons.

Combined uncertainty, u_c is calculated from the control sample limits and bias estimation from interlaboratory comparisons. The s_R from interlaboratory comparisons [4] using the same method can also be used if a higher uncertainty estimation is acceptable.

Table 3. ***Summary table for uncertainty calculations***

		Value	Relative u(x)	Comments
Reproducibility within-laboratory, R_w				
Control sample $\bar{x}$ = 200 µg/L	Rw	Control limits is set to ± 3.34 %	1.67 %	
Other components		--		
Method and laboratory bias				
Reference material	bias	--		
Interlaboratory comparisons	bias	RMS_{bias} = 2.25 % u(Cref) = 1.5 %	2.71 %	$u(bias) = \sqrt{RMS_{bias}^2 + u(Cref)^2}$
Recovery test	bias	--		
Reproducibility between laboratories				
Interlaboratory comparisons	R	--	8.8 %	Data - see Table 1
Standard method	R	--	8 %	EN/ISO 11732 at level 200 µg/l

Table 4. ***Measurement uncertainty for Ammonium in water***

Measurand	Combined Uncertainty u_c	Expanded Uncertainty U
Ammonium	$\sqrt{1.67^2 + 2.71^2} = 3.18$	$3.18 \cdot 2 = 6.4 \approx 7$ %

Theoretical Part of the Handbook

In the following sections of the handbook the components within-laboratory reproducibility and bias are treated in detail as well as obtain measurement uncertainty from interlaboratory data.

Examples Part of the Handbook

In this section are given some examples of uncertainty calculations and also an example with the concentration dependence of measurement uncertainty

Conclusions

This handbook has now been used for several years and has been found to give reliable estimates of measurement uncertainty for methods in routine use where a quality control is in place and validation data for the bias estimate can be obtained.

It is full compatible with the GUM principles, however not giving and details of the magnitude of different uncertainty sources that would be needed for further optimization of the method but this approach combining within-laboratory reproducibility with bias uncertainty covers most of the individual uncertainty components.

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Award Winners 2007

Metrological Concepts for Enhancing the Reliability of Food and Nutritional Measurements

Metrology is a specialized discipline that deals with the science of measurements irrespective of the field of application and regardless of the size of the measurement uncertainty. Incorporating metrological approaches into the measurement process enhances the reliability of analytical findings. These developments include: high quality reference standards, validated methods, robust sampling practices, proven calibration approaches, natural matrix reference materials, speciation chemistry, assessment of measurement uncertainty and establishment of traceability links, certified reference materials to facilitate one aspect of traceability, and proficiency testing, among others.

A comprehensive assessment of dietary intake of nutrients and toxicants involves measurements that encompass physiological and metabolic processes. In this context, it is essential to understand (i) the impact of the potential sources of errors arising from the "bio"dimension of a living system, and (ii) how to minimize such errors to enhance the reliability (i.e. reduce uncertainty) of the analytical findings. The sources include: (i) conceptual errors arising from our limited understanding of the presampling factors such as biological variations, short- and long-term variations reflecting influence of food intake, environmental impact and seasonal variations (e.g. variation of iodine content of milk), among others (Iyengar 1982); (ii) analytical errors stemming from sampling, sample preparation and matrix effects. Variations arising from biological properties and pathological shifts are identifiable but are not always quantifiable (Iyengar 1981). Yet, the extent of variability of results due to this must be understood for a meaningful data interpretation (Iyengar 1989). The sampling part of the metrology reflects its own problems: valid sampling through sample quality, quantity, validity and stability (Heydorn 1984). Identifying sources of uncertainty followed by establishment of traceability (i.e. internationally accepted credibility links) of measurements in life sciences can be accomplished by adopting metrological concepts used in physical and chemical measurements.

Metrology in physics and chemistry is well advanced, although not in all cases with respect to chemical measurements. The high accuracy measurements in physics for length, mass, time, temperature, electric current, etc., are carried out by definitive methods (measurements based on a specific natural phenomenon). Metrology in chemistry is also advanced as demonstrated by physical chemistry (e.g. molar relationships, volume, pressure, etc), which follows the principles of metrology in physics. Further, examples are available through classical analytical chemistry (e.g. gravimetry for preparing high purity standards, related definitive analytical techniques, among others). However, a transition is seen in dealing with chemical measurements in complex chemical matrices (mixed entities, complex compositions), as it adds an additional parameter, namely indirect measurements (e.g. AAS determination of Zn in foods, mixed diets). This practice is common in field assays and calls for extra steps to account for traceability to satisfy reliability concerns.

Metrology in biology (i.e. metabolic, physiologic, clinical, food, nutritional and toxicological measurements, etc.) requires that biological parameters should also be considered as part of an analytical evaluation. This is particularly true when measurements such as the bioavailability of nutrients are carried out in human subjects. Typically, the bioavailability measurement involves knowledge of nutrient composition of the food, chemical species of the nutrients, methodologies used (definitive or indirect methods, and if indirect, what measures are adopted to account for traceability and how measurement uncertainties are assessed), and procedures adopted for assessment of sampling metrology issues. In addition, inter-disciplinary (physiological and nutritional) expertise is also required. Other measurements assessed by indirect methods include body composition, dietary energy intake and energy expenditure by traditional methods that lack evidence of a proven traceability link. However, 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. In this context, it should be stressed that systematic studies designed for estimating measurement uncertainties are lacking (Iyengar and Parr 2005). Bone mineral density as a tool for nutritional assessment (e.g. Ca nutrition and osteoporosis) using dual-energy X-ray absorption method is another example, since multiple parameters have to be met for a valid measurement to be accomplished.

Broader application of metrological concepts for food and nutrition (F&N) measurements is still in its infancy. The silver lining is that many of these measurement processes are amenable for harmonization and facilitate comparability (a vital requirement when results from different laboratories are evaluated). Comparability is broadly understood here as internationally recognized measurement procedures offering some degree of flexibility to address the SI issue because of practical reality faced in F&N laboratories (de Bievre 2004). Examples are: successful integration of metrological principles into food safety (i.e. food labeling) measurements by internationally accredited laboratories; certification of natural matrix food based materials for fat, protein, and other dietary measurements (Sharpless et al 1997); and the IAEA database for reference materials (Arunachalam et al 2006).

F&N metrology is an emerging discipline and the public health and nutrition investigators should develop awareness for current concepts in measurement practices. It is essential for projecting the reliability of food compositional data, among others. Food safety has become

a global issue and it should be recognized that in some parts of the world, movement of foods has assumed the status of borderless-trade. In this context, integration of metrology into F&N measurements strengthens the very base of the institutional measurement infrastructure efforts supported by the Food and Agricultural Organization (FAO 2004).

In conclusion, the society, as a client and as an end-user, looks at quality assurance (QA) in terms of net economic benefits gained by solving real-life problems that contribute to national development. Thus, economic benefit can serve as a measurable parameter, demonstrating the role of QA in a larger context. Steps undertaken to strengthen laboratory outcomes infuse authority to the F&N measurements as a whole and therefore, enhance the value of ensuing public health decisions. In addition, integration of metrology into F&N measurements strengthens the very base of nutrition education, a nearly forgotten agenda in our academic practices (de Bievre (2004).

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Prof. Venkatesh Iyengar
Tufts University, USA

IV International Brazilian Congress on Chemical Measurements, Traceability and Quality Assurance

The Congress was held in São Paulo, a city that does not deny its work mania and business vocation. It was the efforts of its citizens that made São Paulo the most important State of Latin America, in the economic aspect.

About 210 attendees including twenty four specialists from twenty different countries took part in the Congress. There were researchers and practitioners from the international and Brazilian communities of testing, calibration and other industrial laboratories, research centers, technological institutes and universities, from government and several economic sectors and geographic regions. In particular, participation of members of the Cooperation on International Traceability in Analytical Chemistry (CITAC) enhanced the quality and scope of the Congress. The participation of several members from the Brazilian Network of Measurements in Chemistry (RBMQ) provided insights on the impact of measurements of known quality to national productivity and international competitiveness.

The Congress was focused on issues concerning both theoretical concepts and practical aspects for providing traceability and increased quality of chemical measurements. It was an important forum for exchanging information among leaders of national and international communities. The presentations, courses and discussions focused on concepts and tools for establishing primary references and disseminating traceability for chemical measurement results.

The 22nd CITAC Members' Meeting was held during the Congress where new officers were elected to lead the organization for the next three years. This was an important moment for us, the user community and for the Congress.
At the beginning of next year we will publish a book for the proceedings, including papers, presentations, talks, summary of meetings and courses. The Congress photos are available at the website www.metrologiaquimica.org.br

In addition I can say, São Paulo wasn't all about the work. Thousands of cultural events, excellent restaurants, malls, shows, exhibitions, bars and one of the best nightlife in the world also attracted the attention of Congress participants. It was really pleasure.

Dr. Vera Maria Lopes Ponçano
President of the Organizing Committee
IV Metrochem
IPT, Brazil

Workshop on Sampling Validation, Quality Control and Uncertainty Estimation

The First International Workshop on Validation, Quality Control and Uncertainty Estimation of Sampling was held in Hillerød, Denmark April 14-15, 2007. The purpose of the workshop was to present and discuss a new Nordtest handbook on uncertainty from sampling. The workshop was attended by 60 experts from 15 European countries.

The Nordtest handbook

The Nordtest Handbook is an extract of and based upon the principles, methods and text of the recently published Eurachem/EUROLAB/CITAC/Nordtest/AMC Guide: Measurement uncertainty arising from sampling: a guide to methods and approaches (available at http://www.eurachem.org. The handbook provides practical guidance on sampling uncertainty estimation in the Nordtest handbook format and will be available at the Nordtest web site http://www.nordicinnovation.net/nordtest.cfm under NT technical reports, report number NT tec 604. Until the final report is available on the Nordtest web site, a final draft of the Nordtest handbook is available at http://www.samplersguide.com This handbook is the latest in a series of handbooks on validation, quality control and uncertainty estimation of laboratory analysis and field measurements, all available from the Nordtest web site.

The main message of the handbook is that uncertainty from sampling can be estimated with reasonable efforts and following steps that are comparable to those used in quality assurance of laboratory analysis: method validation and quality control. The most useful tools in sampling uncertainty estimation are replicate measurements (random errors) and sampler proficiency tests or method studies (systematic errors).

Workshop contents

The workshop presentations started with a general introduction to measurement, sampling, and analytical uncertainty. Methods for estimating the uncertainty were listed and discussed. Examples of an empirical method, the duplicate design, were presented more thoroughly and interpretation of the uncertainty estimations and fitness for purpose of the sampling methods were discussed.

After an introduction to the Nordtest handbook, the main general points in the handbook were highlighted:

- Design of the sampling program, including sampling purpose, sampling target, and quality objectives
- Uncertainty in measurements-sources and types of uncertainty
- Principles of quality assurance in sampling-validation and quality control.

Worked examples from the handbook were presented on ground water monitoring, industrial product control of iron ore, food sector control and waste water monitoring.

On the second day of the workshop, two presentations were demonstrating sampling uncertainty as the dominant source of measurement uncertainty: one on sampling of waste water using variography and one on sampling of contaminated soil. For the soil example, it was shown that the evaluation of the contamination level of a site was significantly different when sampling uncertainty was included. The calculation tool used was robust ANOVA, and a calculation program to perform this, ROBAN, was demonstrated. Finally, group work was done with the aim of giving the participants "hands on" experience of the tools presented.

Table 1: ***Illustration of the combined use of validation and quality control of sampling***

	One method used for many sampling targets	***One method used repeatedly for one sampling target***
Validation	Initial validation yielding generic performance data for the method	Target validation yielding the performance data for the specific target and the method used
Quality control	Quality control with target specific verification of generic method performance data	Spot quality control verifying the performance data consistency over time

Participant satisfaction

Feedback demonstrated that the participants were generally satisfied with 4 out of 5 (highest) being the most abundant mark. It is, however, difficult to satisfy all participants because of their very variable expectations, knowledge on the subject and theoretical background.

Perspectives

The workshop demonstrated, that methods and software are now available that allow for estimation of the random contribution to sampling uncertainty. For the systematic uncertainty component, tools such as method studies for method evaluation and sampler proficiency tests for sampler performance evaluation are not readily available, and sampling reference sites for both purposes have only been established for a few matrices.

An additional outcome of the workshop was a groundwater sampler proficiency test. It was organized by workshop participants in Denmark in the autumn of 2007 and further steps along this road are in preparation.

Furthermore, a Eurachem workshop is arranged for April 15-16, 2008 in Berlin entitled Sampling Uncertainty and Uncertainty for Compliance Assessment, see www.eurachem.org for more info.

Acknowledgements

The contributions of my co-authors of the Nordtest handbook, Bertil Magnusson, SP Technical Research Institute of Sweden, Mikael Krysell, Ulla O. Lund and Kirsten J. Andersen, Eurofins A/S, Denmark and Astrid Norbotten, Food Safety Authority, Norway, as well as the additional contributions as organizers and lecturers at the workshop by Anke Oberender and Jette B. Hansen, DHI, Mike H. Ramsey and Katy Boon University of Sussex and Pentti Minkkinen, Lappeenranta University of Technology, Finland are gratefully acknowledged.

The Nordtest handbook and the workshop were supported by Nordic Innovation Centre/Nordtest under contract 04130.

Dr. Christian Grøn
DHI, Denmark

The 1st International Proficiency Testing Conference, Romania 2007

Motto: *"There is no science without measurements, no quality without testing and no global market without standards."*

The Commission of the European Union

The First International Proficiency Testing Conference was organised by the Institute of Research, Engineering and Consulting for Cement, Lime and Plaster CEPROCIM SA, Bucharest, Romania under the auspices of the Research Excellence Programme CEEX initiated by the Romania's Ministry of Education and Research. The Conference was held between 11th and 13th October 2007, in Sinaia, Romania and was dedicated to the specialists from the analysis and testing laboratories in many fields (asphalt, biochemistry, bitumen, chemistry, construction materials, drugs, electrical engineering, environmental, food and feed, marine sediments, mechanics, metallography, metallurgy, microbiology, petrochemistry, physics of lasers, plasma and radiation, plastics, precious metals, soils, textiles, veterinary medicines, water, residual water, etc.).

The themes of the conference included: Proficiency testing schemes (PTS); Reference materials (RM); Validation of testing methods

Local Organizing Committee

(VTM); Uncertainty of measurement (UM); Metrology and traceability (MT); Standardisation (St); Research, development and education in the laboratory activity (RDE); Accreditation, Quality management in testing laboratories (AQM). Approximately 150 specialists from 26 countries, namely, United Kingdom, Australia, Belgium, Bulgaria, China, Croatia, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Ireland, Israel, Lithuania, The Netherlands, Norway, Portugal, Romania, Russia, Spain, Sweden, United States, Thailand and Turkey shared and disseminated their experience, knowledge and innovative ideas.

The proceeding includes 64 papers, which were presented during the conference as oral, or poster presentations and they have greatly contributed to the scientific and research activities and also to the professional work. During the Conference proceedings, 8 exhibitors from Romania, United Kingdom and United States showed their products.

In the second day of the conference, the course "Training Metrology in Chemistry" which proceeds under the responsibility of TrainMiC® European Programme will provide new information on issues related to the quality of measurement results.

Dr. Graziela Guslicov
1st Proficiency Testing Conference Pres.
Romania

BERM 11
11th International Symposium on Biological and Environmental Reference Materials

October 29 - November 2, 2007, Tsukuba, Japan

The 11th International Symposium on Biological and Environmental Reference Materials (BERM 11) was held on October 29 - November 2, 2007, at the Tsukuba International Congress Center EPOCHAL in Tsukuba, Japan. BERM 11 was the first BERM held in the Asia-Pacific region and also the first big event on metrology in chemistry hosted by the National Metrology Institute of Japan (NMIJ) since its establishment in 2001. It was a very successful symposium and attended by about 180 participants from 19 countries and various fields/sectors.

As a continuation of the symposia series, held alternately in the EU and the USA since 1983, BERM 11 was intended as a forum to address issues related to the development of biological and environmental reference materials and their role in the quality assurance of analytical measurements. The scientific program of BERM 11 focused on the role of CRMs in the analytical measurement process, with emphasis on CRMs related to public health, such as clinical laboratory medicine, food safety and nutrition labeling, and environmental monitoring. A special emphasis was also placed on the Asian CRM (ACRM) network program between China, Korea and Japan.

The symposium comprised 11 oral sessions with 75 presentations including invited lectures and 2

poster sessions with 84 papers. The sessions and invited talks were as follows:

International activities on metrology in chemistry
- R. Kaarls (CIPM/CCQM) "CCQM activities on metrology in chemistry"
- R. Wielgosz (BIPM) "BIPM international comparisons in support of the equivalence of chemical measurements"
- H. Emons (IRMM) "Towards further internationally harmonized guidance on reference mateials - from REMCO to application notes"
- S. Wood (LGC) "Guidance for the production of non certified reference materials"
- S. Ellison (LGC) "International guides on measurement uncertainty"

Recent development of CRM production
- A. Herrero (IRMM) "New challenges for reference materials in the Omics era"
- S. Wise (NIST) "Recent trends in the development of environmental, food, dietary supplement, and clinical nutrition SRMs"
- K.Chiba (NMIJ) "Recent NMIJ Activities in the certified reference material program"
- S. Wood (LGC) "Production of certified reference materials at LGC"
- J. Vogl (BAM) "Reference material characterization by IDMS applying multi collector instruments"
- H. Emteborg (IRMM) "IRMM's reference materials for food and environmental analysis"
- L. Sander (NIST) "Analytical methods for analysis of botanical dietary supplements"

Biological RMs
- H. Emons (IRMM) "Reference materials for life sciences from IRMM"
- M. Salit (NIST) "Standards and measurement assurance for microarray gene expression measurements"
- S- R.Park (KRISS) "Development of higher order analytical methods for certification of DNA reference materials"

RMs and QA/QC for laboratory medicine
- J. Betz (NIH) "Development and use of certified reference materials for biomedical research applications"
- K. Kuwa (Tsukuba U) "Proficiency teting activities at JCCLS using human pooled serum and matrix reference materials in laboratory medicine"

RMs in food and safety
- A. Yasui (NFRI) "Food CRMs and PTs in Japan"

Environmental RMs
- H. Haraguchi (Nagoya U) "Certified reference materials required for metallomics research"

Greenhouse gas RMs
- H. Mukai (NIES) "Standards for GHGs and ozone monitoring in Japan"

Development of analytical techniques
- N. Furuta (Chuo U) "Bulk analysis and single particle analysis of size-classified airborn particulate matter"
- G. Turk (NIST) "ICP emission spectrometry for high-precision comparisons of elemental standard solutions"

RMs for proficiency testing
- Y. Mitani (CENAM) "roficiency testing scheme for harmonization and comparability of analytical measurement"
- I. Kuselman (INPL) "Traceability, comparability and compatibility of proficiency testing results based on the use of reference materials as test items"

Asian collaboration on CRM development
- H-Y. So (KRISS) "Asian CRM network program, activities at KRISS"
- C. Cherdchu (NIMT) "The first activity on chemical reference material production at National Institute of Metrology (Thailand)"

BERM 11 was closed after the presentation by Dr. K. Okamoto, Chair of BERM 11, on "30 years on reference materials; my first-, second- and third-generation reference materials"

Dr. Kensaku Okamoto
NMIJ, Japan

BERM 11 participants at the end of a conferece day

Announcements

THE REVISED VIM IS OUT!

The revised VIM: "International Vocabulary of Metrology – basic and general concepts and associated terms (VIM) ISO/IEC 99" can be purchased from ISO since 2007-12-14 on the address: http://www.iso.org
Look for "ISO/IEC 99".

In February/March 2008, it will be hyperlinked to the BIPM website where it can then be accessed and downloaded for free: www.bipm.org

In addition, a draft report of the IUPAC project "Metrological Traceability of Measurement Results in Chemistry" is out for public scrutiny on the IUPAC website: http://www.iupac.org/projects/2001/2001-010-3-500.html "Control + click" on the above html and you access the project description; at the end of the project description, go to "provisional recommendations"; at the end of these, you will find: "download full text of the Provisional Recommendations (pdf file - 1.28 MB)" from where you have access to the full document. I am available for further questions and information on both the revised VIM as well as on the IUPAC project, that includes specialized lectures and seminars on the above documents, if so desired.

Prof. Paul De Bièvre
Consultant, Belgium

Symposium: Advances in Understanding-Chemical Measurement Quality

Societal Impact, Turin, 18-19 September, 2008

The aim of this symposium, organised by the Division of Analytical Chemistry (DAC) of EuCheMS in cooperation with EURACHEM (website: http://www.euchems-torino2008.it/site/home.asp) is to demonstrate the impact of measurement quality on various societal activities based on analytical chemical measurements. Examples will cover measurement quality in areas such as environment, health, industry, trade, and forensics.

Short contributions and posters covering all the areas are welcome.
The conveners representing EURACHEM are Bertil Magnusson, SP Technical Research Institute of Sweden (bertil.magnusson(@)sp.se), Enzo Ferrara, Istituto Nazionale di Ricerca Metrologica, Italy (ferrara(@)inrim.it), and representing DAC is Bo Karlberg, Stockholm University, Sweden (bo.karlberg(@)anchem.su.se)

The 17th International Conference of the Israel Society for Quality

November 18-20, 2008 Jerusalem, Israel

It is a privilege to invite you to take part in the 17th International Conference of the Israel Society for Quality, to be an active partner in a challenging and fruitful endeavor and to contribute to its success.

The Israel Society for Quality biannual international conferences are very popular and generally attract 1700-2000 participants. Both specialists in metrology (measurement, calibration and testing, including chemical analysis) and quality professionals will again have an opportunity to network with each other, interact with senior management and learn how to bring the "message" to as many people as possible.

The 17th Conference is supported by CITAC, which will organize a metrology track at the event.

Joining us at the conference will also enable you to enjoy exploring the Land of Israel, birthplace of the three great monotheistic religions.

We look forward to welcoming you and your colleagues in Jerusalem.

Dr. Ilya Kuselman
CITAC Chairman
INPL, Israel

Metrology Track Topics

- Trends in metrology
- Metrology as a business
- Metrology in chemistry, petrochemistry, pharmaceuticals, environmental and clinical analysis
- Measurements of properties of nano-particles

- Measurement methods and their validation
- Measuring instruments and their qualification
- Measurement standards (etalons) and reference materials (RMs)
- Uncertainty estimation in measurement and testing/chemical analysis
- Traceability, comparability and compatibility
- Inter-laboratory comparisons and proficiency testing (PT)
- Conformity assessment
- Accreditation of calibration and testing/analytical laboratories, RM producers and PT providers
- Legal metrology
- Software for metrology
- Teaching metrology and quality
- Ethical problems in metrology

Abstracts of 200 words in MS WORD format are invited on all the suggested topics.
For further information please see www.isas.co.il/quality2008 or contact the Conference Secretariat at tel: +972-2-6520574, fax: +972-2-6520558.

6th Workshop on Proficiency Testing in Analytical Chemistry, Microbiology and Laboratory Medicine

Rome (Italy), (5)-6-7 October 2008

The EURACHEM Proficiency Testing Working Group www.eurachem.org, in co-operation with CITAC www.citac.cc and EQALM www.eqalm.org, welcomes you to attend the 6th event of a series of workshops addressing current practice, problems and future directions of interlaboratory comparisons in chemical and biological analyses. These events are structured to include both key-note lectures and discussions in working groups, to enable interactive participation and cross-fertilisation of ideas.

Scope of the Workshop

Focus is on developments in Proficiency Testing (PT) and External Quality Assurance/Assessment (EQA) in analytical chemistry, microbiology and laboratory medicine. Lectures, presented by invited speakers, and working group tasks will highlight the following topics:

- Frequency of PT/EQA and monitoring performance over time;
- Developments in PT/EQA within the EU;
- PT/EQA in developing countries;
- PT/EQA in microbiology;
- PT/EQA in forensic science;
- The new standard for PT/EQA: ISO 17043;
- End-user perspective of PT/EQA;
- Quality of test items used in PT/EQA.

Programme

The workshop starts on 6th October. Four lectures will be given each morning followed by the poster session and afternoon discussions within working groups. Feedback from working groups will be presented each day at the closure session.

The event shall be submitted for approval under the Italian Ministry of Health Programme for Continuous Education in Medicine.

A get together party and a workshop dinner will be organised for participants and accompanying persons.

Opportunities for Training

Two training courses, open to workshop participants only, and addressing the topics:

- Statistics for Proficiency Testing
- Selection, use and interpretation of PT/EQA Schemes

will be organised on the day before the workshop (5th October), providing opportunities for training for both PT/EQA organisers and end-users.

Who Should Attend?

PT/EQA scheme organisers, QA managers, internal and external assessors, participants in PT/EQA and the laboratories' customers.

Support to delegates from developing countries can be sought applying to:

Umberto Sansone, International Atomic Energy Agency (IAEA), Agency's Laboratories Seibersdorf, Chemistry Unit, A-2444 Seibersdorf (Austria) Email: u.sansone@iaea.org

Contributions and Exhibitions

Poster contributions are welcome.

Abstracts, according to the instructions given on the website, should be sent to the workshop secretariat by 15th June 2008.

Products and services related to PT/EQA and laboratory activities can be presented in the exhibition area. Requests should be sent to the workshop secretariat by 1st September 2008.

Proceedings

Abstracts of invited lectures and accepted contributions will be collected in an abstract book. All participants will receive a copy of the book of abstracts.

Full papers of invited lectures and accepted contributions will be considered for publication in a special issue of Accreditation and Quality Assurance (Springer Verlag). For example, contributions from the previous edition of this series of workshops were published in the special issue of Accred. Qual. Assur. 2006: 11(8-9), 371-484

Full papers, to be submitted for peer review, drafted according the journal recommendations, should be delivered to the Workshop Secretariat, before or at the workshop (electronic format or as 6 printed copies).

Venue

One of the most ancient cities in Europe, founded over 2700 years ago, Rome has become the biggest open-air museum in Europe. Heritage, complemented by pleasant weather, Italian cuisine and a lively atmosphere, continues attracting visitors over the centuries. Completely opposite styles of art and life manage to live side by side here making Rome a unique city. Rome is located in the middle of the Italian peninsula, at the centre of the Mediterranean basin. Connections with the rest of Italy and the world are assured by means of two airports and excellent highway and train systems.

The workshop will be held at
Centro Congressi Frentani
(http://www.congressifrentani.it/?lang=eng),
in the heart of the University district and on easy reach from both Termini and Tiburtina railway stations. Further information on how to get to the workshop venue is available on the workshop website and will be included in the second circular (March 2008).

Registration and Accommodation

The first circular, registration and hotel booking forms are available from the secretariat and on the workshop website.

Workshop Secretariat

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Marina Patriarca, Antonio Menditto
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Istituto Superiore di Sanità, Rome, Italy

Dr. Brian Brookman
Chair of EURACHEM PTWG
LGC Standards, UK

CITAC Strategy for 2007-2010 (Draft)

According to the CITAC Terms of References (TOR) adopted at the 22nd CITAC Members' Meeting in Sao Paulo, Brazil, 17.07.07, the CITAC strategy for the next three years includes the following:

1. To provide a broad international forum for the exchange of information with respect to worldwide metrological traceability of results of chemical measurements;
2. To assist analytical laboratories with tools for establishing traceability to "stated references";
3. To share views, clarify important concepts and raise awareness of the needs and possibilities leading to traceability in chemical laboratories;
4. To develop, distill and disseminate globally key traceability concepts and issues;
5. To prepare discussion and scientific papers for journals in relation to traceability, uncertainty and quality assurance issues, and guides for analytical laboratories, which could play a bridging role between industries, regulatory bodies, universities, metrology, standardisation and accreditation bodies;
6. To organise seminars, symposia and workshops and to participate in conferences to promote the message of traceability;
7. To work closely with other international organizations, e.g. CCQM/BIPM, ISO-REMCO, IUPAC, ILAC, AOACI, regional and national professional chemistry societies and institutions, like EURACHEM and DAC-EuCheMS, – without duplicating work already being conducted by other groups – using these societies and institutions to act as a conduit to the field laboratories;
8. To organize annual nomination of the most interesting/important papers on metrology in chemistry;
9. To prepare an annual newsletter "CITAC News" describing CITAC activities and international achievements in the field of metrology in chemistry;
10. To keep an updated website, informing the community about CITAC and its purposes, history, developed documents and planned activities.

In addition the following strategically important CITAC activities are planned for 2007-2010:

1. To prepare CITAC Bylaw;
2. To check expediency, cost and possibility of the registration of CITAC as a legal entity in one of the member countries;
3. To renew the CITAC logo;
4. To attract to CITAC membership active representatives of new NMIs, universities and industry (individual CITAC members), as well as organizations, institutions and companies (collective members);
5. To explore the possibility of consultation to industry as a new CITAC activity. In the framwork of the consultation to initiate and, where needed, coordinate work for the harmonisation and validation of analytical methods based on traceability and other metrological concepts.

CITAC
Cooperation on International Traceability in Analytical Chemistry

CITAC
Cooperation on International Traceability in Analytical Chemistry

Two new proposed logos

Messages from New Members

Message from Venkatesh Iyengar

CITAC's role in strengthening the application of metrology in various spheres is self evident. Its dynamic relationship with many national metrology institutes is a key feature in its successful and effective networking. CITAC's growing mutually supportive relationship with IMEKO through various technical committees is an excellent pathway to further strengthen its ability to be a crucial player in spreading the metrology message. We are likely to see further consolidation of CITAC-IMEKO relationship in specific future activities e.g. the proposed IMEKO TC-19, TC-23 and TC-24 international conference in Budapest in September 2008 and the proposed session on Food and Nutrition Metrology (F&N Metrology) at the International Congress of Nutrition of the International Union of Nutritional Sciences in Bangkok in late 2009.

While CITAC is striving to strengthen the metrological base in several areas across the world of measurement, it would be desirable to examine the ways and means for expanding the membership base. In my view, this should be done by casting the net to attract the student community (those doing advance degrees in several disciplines with links to metrology) as well as the young professionals. With the help of the existing members in various countries, an effort should be made to attract A Few Core Student Members From Each Country. This should be introduced in such a way that the chosen students appreciate that it is a privilege, and the membership should be free of cost. Further, a select group of young and upcoming professionals who are already in the measurement arena should also be contacted, and they should be offered membership with very modest fee (starting with a no-fee introductory year). With time, this concept would mature and hopefully a new and vibrant group of members would be developed.

I am glad to be a member and will strive to enlarge the young membership drive, if adopted by CITAC.

Prof. Venkatesh Iyengar
Tufts University
USA

Message from Chainarong Cherdchu

Having been elected as a new member of CITAC is not only the matter of honor but really attributed to several traceable consequences that I am experiencing now. After joining NIMT at the Department of Chemical Metrology and Biometry since October 2004 the value of best measurement and its traceability has become an important element of my daily practice. Metrology – the science of measurement – has shed some light and underpin my belief of science for human beings wealth and health. Certainly, it is a solution to one's quality of life providing that this quality system and the related ones have been implemented properly and fairly.

CITAC mission – to improve traceability of the results of chemical measurements everywhere – seems to catch our interest deeply since the growth of metrology in chemistry in Thai community has evolved enormously in all sectors during the past three years. I myself envision that participating closely and tightly with CITAC will further enhance the MiC activity for our Asian region chemical metrology community greatly. Therefore, I am looking forward to an active and a fruitful collaboration among our Thai chemical metrology, the CITAC and other organizations.

Dr. Chainarong Cherdchu
NMI
Thailand

Message from Koichi Chiba

It is my great honor to be a new member of CITAC. CITAC has been playing a great role in establishment of the traceability concept in chemistry and dissemination of the idea to the chemical measurement fields and it becomes a worldwide success in recent years. The traceability is now recognized as one of the most important elements for the measurement values and procedures. The coverage fields of CITAC have expanded from a conventional chemical metrology to new fields such as bioanalytical and clinical chemical ones, even after I joined chemical metrology in NMIJ at Chemistry Division in 2002. The chemical metrology is shining a light on an essential part of science for our quality of life, that is, human beings' wealth, health, safety and so on. CITAC mission - to disseminate the traceability to all the chemical measurements - is a main stream of analytical chemistry in all sectors, although the traceability in chemistry in the real world is still very complicated. In Japan, as you know, BERM11 was held in Tsukuba on October of last year, and I believe that BEAR11 was a great opportunity for Japanese scientists to reaffirm the significance of traceability and to understand an international trend in chemical metrology. I hope to contribute to a fruitful collaboration among CITAC, Asian regional chemical metrology community and Japanese communities including NMIJ.

Dr. Koichi Chiba
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