



NEWS

March 2017

Foreword by the Chair:

Traceability: a Need or a Necessity?



Welcome to our latest Newsletter. Firstly, I would like to apologise for not being able to publish the CITAC Newsletter for the last few years. As I begin my fourth year as Chair of CITAC, I am looking forward to working with my fellow members to bring more enthusiasm and involvement to CITAC activities. I am really proud of what CITAC has accomplished so far and congratulate my predecessors on their good work and leadership, and am sincerely thankful and honoured to have been elected as the chair of CITAC.

Since the inception of CITAC in 1993, members have worked persistently around the globe to promote traceability in analytical chemistry. From the initial 22-members, the group has now grown to a truly global network combining invaluable skills, knowledge, and dedication. It comprises of members from 28 countries and 32 organisations from six continents. CITAC celebrated its 30th meeting in April 2015 in Paris by inviting past chairs and former mem-

bers. It was a special meeting and celebrated in a truly international way to commemorate the achievements. We were lucky to have 5 of our previous chairs in the meeting. The one day meeting was truly inspirational and attended by about 30 people. We had a chance to review and discuss the CITAC strategy for the upcoming five years. The first chair of CITAC and the former director of LGC UK, Dr. Bernard King, gave a talk about the origin of CITAC and its initial activities including the spinoff of CCQM from CITAC. Various collaborative options with the liaison organisations were discussed to promote traceability in analytical measurements.

Each year CITAC invites nominations of published articles from members for its best paper award. The articles can be from any field of chemistry and from any organization that follows metrologically valid methods and principles. It is a recognition of the individual or team's high calibre in chemical science and research, and acceptability of the work from their peers. Last year we had more than 10 nominations to consider and a large number of members participated in reviewing the articles to decide the top three places.

During the 2014 Annual Meeting, two new members were proposed to the

meeting: **Dr. Tong Kooi Lee**, Director of the Chemical Metrology Division, HSA Singapore; and **Dr. Osman Bin Zacharia**, Director of SIRIM Malaysia. Both were elected and they will bring a wealth of experience to the CITAC community. The nomination of **Dr. Michela Segna**, Istituto Nazionale di Ricerca Metrologica INRIM, Italy was adopted at the 2015 meeting. Michela is a well-known expert in chemical metrology and she will be a great asset to the group. Last year **Dr. Bernd Güttler**, Director of Physikalisch-Technische Bundesanstalt PTB Germany, and **Dr. Rola Bou Khozam** from Lebanese Atomic Energy Commission became members of CITAC. Dr. Güttler is the past chairman of the Technical Committee for Metrology in Chemistry (TCMC) of EURAMET and the current chair of CCQM *ad hoc Working Group on the Mole*. Dr. Khozam is the first CITAC member from the Arabic countries. CITAC will hugely benefit from their membership. **Dr. Teemu Naykki**, principal metrologist at Laboratory Centre, Finnish Environment Institute and **Dr. Della W. M. Sin**, the Government Chemist of Hong Kong Government Laboratory were elected as new members replacing our long serving members Prof Timo Hirvi, Center for Metrology and Accreditation, Finland and Dr Tai Lun Ting, Government Laboratory

Foreword by the Chair:

of Hong Kong respectively. During the past year, we revisited the existing membership list and followed up inactive members to see if they still have an active role in analytical chemistry. Some of the members are retired and are no longer active in the field, so the meeting decided to remove them from the list.

CITAC has resurrected from the need of international traceability in analytical measurements and many activities have been developed to help testing laboratories demonstrate traceability in routine analytical measurements. A number of useful guides have been developed individually and in cooperation with Eurachem/IUPAC and are available on the CITAC website <http://www.citac.cc/>. We have accomplished a lot since 1992, but there's always more to do. Next year, as we did this

year, we will continue to add new members to the committee to meet new challenges and add fresh ideas.

The CITAC encompasses both chemical and biological disciplines and its goal is to enable laboratories to demonstrate traceability from their working level to the SI (International System of units) wherever possible. National metrology institutes around the world are already in the process of delivering traceability and comparability of measurements. Some countries have taken it seriously and some are still in the 'wait and see' approach.

There is no coordinated approach among economies about how to disseminate chemical measurement traceability to testing laboratories and the connectivity between field labora-

tories and national metrology institutes are little or nowhere near the desired level. CITAC as an organization can enhance this level and work closely and collaboratively with national metrology institutes, industries, accreditation agencies and field laboratories. As the demands being placed on chemical measurements are increasing, governments across the globe have regulations to track commodities that are directly concerned with consumer health. New guidelines, measurement procedures and methods are vital and disseminating them to the desired level is a job that CITAC can sponsor. I hope we as a group could enlighten the awareness and elevate the measurement quality that affects human life.

Laly Samuel
CITAC Chair, New Zealand



CITAC 30th meeting Celebrations. Past Chairs: from left to right Prof. Dr. Wolhard Wegscheider (1999-2002), Dr. Laly Samuel (2013-17), Dr. Wolfgang Richter (member since 1999), Dr. Ilya Kuselman (2007-10), Dr. Vera Poncano (2004-07), Dr. Bernard King (1993-96) and Mr. Alan Squirrell (1996-99).

Picture Credit: W. Wegscheider, Leoben

Message from the Vice Chair



Since 1993, when CITAC was born, metrology in chemistry and in biology has undergone an enormous spread worldwide. A lot of work has been carried out to implement the metrological approach to chemistry-related topics.

This turns out in the flourishing of amount of substance activities both at national and international level, with an increasing commitment of National Metrology Institutes (NMIs) and Designated Institutes (DIs) and the development of metrology in chemistry sectors in emerging economies. At the same time, the number of accredited calibration and testing laboratories in the chemical field has rapidly increased.

Despite such a rapid evolution, there is still a very long way to go. A fundamen-

tal aspect that needs to be addressed is the capillary diffusion of the metrological concepts and the sharing of documents to tear down the barriers that are still in place, among which the terminology and languages specific for each measurement sector. In this context, the contribution that CITAC is being made in co-editing guidance documents is of outmost importance and the work with international organizations, among which IUPAC and Eurachem, is a great opportunity to share information and concepts within different communities.

Among its initiatives that aim at spreading the information on metrology in chemistry and traceability related issues, CITAC runs every year an award for the most interesting papers on Metrology in Chemistry. The following three papers were selected for special mention in 2015:

◇ ***Use of assigned reference values: revisiting a small-scale inter-laboratory comparison for residual pesticides in tea***

By: *Della Wai-mei Sin, Yiu-chung Wong*
Accred Qual Assur (2015) 20, 495-500.

◇ ***Achieving comparability with IFCC reference method for the measurement of hemoglobin A_{1c} by use of an improved isotope-dilution mass spectrometry method***

By: *Hong Liu, Lingkai Wong, Sharon Yong, Qinde Liu, Tong Kooi Lee*
Anal Bioanal Chem (2015) 407, 7579-7587.

◇ ***Validation of ISO 6974 for the measurement of the composition of hydrogen-enriched natural gas***

By: *Adriaan M.H. van der Veen, Paul R.Ziel, Jianrong Li*
International Journal of Hydrogen Energy (2015) 40(46), 15877-15884.

The award ceremony was held during the annual CITAC meeting, held in Sèvres on 21st April 2016.

I would like to thank everyone for the support given in the nomination process. Special thanks to Aleš Fajgelj, the past CITAC vice-chair, who coordinated the entire process.

I would like to thank also the CITAC members and the Executive Committee, who gave me the honour and the opportunity to act as vice-chair. On behalf of INRiM, I wish to all the CITAC Newsletter readers a fruitful 2017.

Michela Segal,
INRiM, Italy

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

CITAC Most Interesting/Important Papers on Metrology in Chemistry: Abstracts

- ◇ "Achieving comparability with IFCC reference method for the measurement of hemoglobin A_{1c} by use of an improved isotope-dilution mass spectrometry method", *Anal Bioanal Chem* (2015) 407, 7579-7587. **H. Liu, L.Wong, S. Yong, Q.Liu, T. K.Lee** 37
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CITAC Member's List

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Picture Credit: W. Wegschieder, Leoben.

Paul De Bièvre was a founding member of CITAC and an active promoter of traceability in chemical measurements. He received his Ph.D. degree from Gent University, Belgium, in 1959 and he was an Assistant/ Lecturer at Gent University from 1959 to 1961. In 1961, he joined the EC-Central Bureau for Nuclear Measurements, GEEL in Belgium where he worked on Isotope Mass Spectrometry. He had been a Group Leader for Mass

R I P : Paul De Bièvre (1933-2016)

THE LEGEND OF CHEMICAL METROLOGY

Spectrometry from 1976 onwards, and later the Division Head for "Isotope Measurements". Since 1972, he had also been part-time Professor in Isotope Mass Spectrometry at the University of Antwerp (Belgium). For many years, he worked on the production and certification of Isotopic Reference Materials at IRMM, has led a project to improve the Avogadro constant since 1982 and worked on the development of Metrology in Chemistry (MiC) since 1985.

Prof. De Bièvre has been a member of the IUPAC International Committee on Atomic Weights and Isotope Abundances since 1971 and of its Inorganic Division Committee from 1985 until 1995. He was a founding member of CITAC and of EURACHEM, and Chairman of EURACHEM (1993-1995). He was the elected

President of the National Committee on Chemistry of the Belgian Academies in 1990 and was member of CCQM from its inception (1993). Since 1998 he was the Adviser of the Director of IRMM on 'Metrology in Chemistry' and advises/ lectures on MiC around the world. His columns on metrology in chemistry appeared frequently in Accreditation and Quality Assurance of which he was the Founding Editor-in-Chief. Paul was a constant presence in all of the CITAC meetings and we enjoyed his jokes and strong opinions about traceability in analytical measurements. We miss him dearly and RIP, dear friend.

Laly Samuel

New Zealand

AN AMIABLE GENTLEMAN



My first encounters with Paul De Bièvre go back to the second half of the 1980's, when we started the first discussions on the need for a global organization, charged with the process of establishing

and maintaining global comparability of chemical measurement results, preferably through traceability to the SI. Although, for example, the European Commission's Bureau de Référence (BCR) supported already within the European Union technical/scientific developments improving international comparability in metrology in physics and produced a number of BCR CRMs underpinning some chemical analysis, it did not undertake new and more fundamental initiatives in metrology in chemistry.

However, that a system of global comparability through traceability to the SI

was needed, was strongly felt by an increasing number of laboratories being active in the field of chemical analysis in support of judicial, forensic, safety and security measures, as well as in the field of environmental measurements and trade measurements. Also Paul was convinced about the need and the possibilities to establish global metrological comparability in chemical measurements. His laboratory had obtained unique experience and measurement capabilities by developing advanced isotope dilution mass spectrometry, among others to be used for "finger

R I P : Paul De Bièvre

print" measurements of radioactive materials. In the 1980s these were a major part of the aims and activities of the EC JRC Central Bureau for Nuclear Measurements (CBNM) in Geel, Belgium. But Paul's views were not limited to these. For example, he also was promoting and establishing links with the clinical measurement community through participation in regular clinical symposia held in Antwerp, Belgium.

Paul's enthusiasm and restless energy in support of metrology in chemistry was great. As a result, together with equally enthusiastic scientists of the LGC (UK), BAM and BASF (Germany), VSL (The Netherlands) and the University of Strasbourg (France), EURACHEM was created in July 1989, with the aim of promoting and establishing global comparability of chemical measurement results through traceability to the SI, whenever possible, or to other international references, if traceability to the SI was not (yet) feasible.

In order to bridge the considerable gap in metrological thinking and the simple, pragmatic views and opinions existing between the majority of chemical analysts and the few with a more physical and metrological background, Paul, NIST staff and myself published a number of papers making clear the essence of metrological traceability. This was for Paul the beginning of many, many papers he would write about metrology in chemistry ever since.

In the early 1990's when EUROMET (now EURAMET) and the CIPM became active in the field of metrology in chemistry the development of chemical metrology real-

ly started. So, not amazingly, Paul on behalf of the IRMM (the renamed JRC CBNM Geel institute) became from the beginning in 1993 an active member of the CCQM.

In the same year, during a blizzard in Atlanta, when we were not able to fly back home after having attended the PittCon conference, Paul was also involved in the creation of CITAC. Already in the 1990's it became clear that Paul likes scientific and related debates, for example on what is a "primary measurement method".

After his retirement from the IRMM Paul continued as a CCQM member, now also representing the IUPAC. And, after his retirement, having more time, Paul also became a member of the BIPM Working Group on international metrological vocabulary VIM. Paul's right views were that metrological terminology should not only be restricted to the physics area, but should also cover the chemical area. And that then based on a consistent coherent, almost mathematical, model of defined and derived terminology, like the SI is a coherent system of units. This unfortunately in many cases lead to unprofitable, unnecessary and unpractical conflicts with the widely used and understood different terminology by the chemical community.

Finally, Paul also contributed actively to the discussions on the redefinition of the fundamental constants. In particular, Paul started long and intense debates among those scientists, having sharp and quite different opinions with respect to the status of the "mol" as a base unit of the SI, and its relationship to the Avoga-

dro constant, as a fundamental constant. Paul was a lively debater and having developed his own philosophy on measurements, comparisons and metrological traceability, it was not always easy to speak the same language and agree on issues at stake, although I think we fully agreed on the final aims to be achieved. During his long period of retirement Paul travelled all around the world. He enjoyed it very much, as a great promotor, to give lectures on what is a measurement, and on the international metrological vocabulary (VIM).

But above all, Paul was an extremely amiable man, a gentleman! He and his wife Lieve enjoyed always inviting scientists, colleagues and friends at their home in Kasterlee for a very pleasant chat, a nice drink and/or dinner. Sometimes even brightened with music played by one or more of their children and friends.

Having worked together with Paul on global comparability of chemical measurement results and metrological traceability over a period of some 30 years, we can conclude that, although work is certainly not yet ready and finished, during this period a very good basis for reliable and much more accurate chemical measurement results has been established.

We will remember Paul as a gifted scientist with undimmed energy and enthusiasm, always open for debate, and a good glass of wine, and having very well contributed to the further development of the chemical metrology community.

Robert Kaarls

Past Secretary CIPM
Past President CCQM
Former Director VSL

RIP : Paul De Bièvre

TO THE MEMORY OF PAUL DE BIÈVRE



It was on 14 April, 2005, Paul De Bièvre approached me with his friendly smile in the Pavillon du Mail of BIPM where the 11th meeting of CCQM in Sèvres, Paris, was about to begin, and handed me a special volume of "Traceability in Chemical Measurement" edited by him with Helmut Günzler of Springer. I was honored to be listed as one of the 79 authors who contributed to this special volume on metrology in chemistry which is the compendium of the articles related to the traceability in chemistry, published in the Journal of Accreditation and Quality Assurance from 1996 to 2004.

His dedication says: To Yoshito Mitani, with admiration for the way he has planted his MiC programme and his people on the world scene over the last ten years.

When he mentioned about the way we planted our MiC program and our people, he referred to the process that we had experienced in Mexico in establishing chemical metrology program, which began in 1992, very surprising at that time and in Latin America region.

He was one of the leaders on chemical metrology, who had promoted to create CCQM in the framework of the meter convention, and whom I knew for his active role in the international arena, since I was involved in the processes of estab-

lishing chemical metrology program in Mexico in 1992, and he was one of the experts whom we planned to invite as a consultant, because we considered necessary to transmit his strong message and passion to the chemical metrology to our officers in the Secretary of Commerce (SECOFI at that time) which was responsible to coordinate CENAM's construction and start-up in Queretaro, a city located in northwest from the capital of Mexico. For any political decision, it was critical to make clear to the officers why we need to invest to metrology and particularly to chemical metrology, for which financial need for investing and maintaining the measurement instruments is considerable, particularly when we want to establish higher-order measurement capabilities in the metrological areas defined by so diversified analytes in diversified matrices.

In the original project of CENAM at Queretaro site drafted in 1990, no consideration was given to the investment for the establishment of chemical metrology measurement capabilities at CENAM, but only considered to provide some physical spaces to maintain and distribute reference materials to the users. The drastic change of the project to include chemical metrology program was introduced under the responsibility of the newly appointed Director General Dr. Jaime Gonzalez-Basurto in 1991, and thanks to the support of the World Bank team led by Kathleen De Tullio and NIST adviser, Dr. Stephen B. Carpenter. When I was invited to join to the team in 1992, the first task was to draft a modified version of the CENAM project, which was approved officially by the governmental officers from Ministry of Commerce (SECOFI) and Ministry of Finance (SHCP).

Immediately, we began to draft a new building for chemical metrology, parallel to the modification of one of the 8 existing buildings to allocate initial 15 laboratory spaces for chemical analysis, whose project was approved in 1993. It was the year, when the first CITAC meeting was held in Atlanta, chaired by Dr. Bernard King of LGC.

In the spring of 1994 Dr. Robert Kaarls, who was chairing the newly established Working Group on Quantity of Substance of CIPM, which was later renamed as CCQM in 1995, paid a visit to CENAM. We could show him proudly the blueprint of the laboratory, which was drafted thanks to the strong support of NIST advisers like Dr. John Moody.

When Paul visited us in Queretaro in 1998, we had already inaugurated the new building on the commemorative date for Mexican, December 12, in 1997, after the 2-year suspension of the construction, due to the economic crisis began in December 1994. This task was completed, however, under the leadership of newly appointed Director General Dr. Hector Nava-Jaimes, appointed in June 1996, and we could establish several chemical metrology programs, such as electrochemistry, gas, inorganic organic in this new building and began to participate in CCQM comparisons from 1999. Our idea to invite Paul was not only for us to receive lectures on chemical metrology, but also for high ranked officials of the Ministry to convince the needs to strengthen chemical metrology in Mexico for competitiveness.

Paul mentioned always that people who is promoting something new like chemical metrology at that time is like a missionary in the new continent, facing at

RIP : Paul De Bièvre

every kind of difficulties in the process of conversion. Obviously, it seemed to me that it was an exaggeration, but finally it turned out to be true, because until now after more than 20 years of our existence as NMI we have still similar difficulties, namely we cannot convince yet easily our stake-holders. We cannot show sufficient evidences nor clear argument to convince them that they have tools with NMI, instead of burden for the nation's economic development. Or even we cannot identify and quantify clearly benefits to the stake-holders.

I must add that the most of the people Paul referred to in his message are not with us anymore. But some critical number of them he met are still with us with common dream to develop chemical metrology program strong enough to support the economic development of the country, and also luckily those people who left us are also working as pioneers of chemical metrology in different organizations.

The way we planted our chemical metrology program was to copy NIST's chemical metrology program in smaller scale comparable to our economy, and was supported by the governmental officers at that time, since Mexico had almost signed NAFTA with US and Canada, from our understanding. Of course we had an option to choose less expensive and selective in the scope of CENAM as an NMI,

which could be sustainable in a long-run, but we are convinced that we made a right decision.

With these lessons in mind, we also intended to expand our capabilities to the new fields of metrology, such as molecular level measurements and nanoscale materials with those people who experienced the difficult period of its birth, but the effort as missionary should still continue to house those new fields of metrology, that CCQM is now covering. Every six years we do our planning according to the cycle of government, and we proposed a new project in 2000, to continue expanding our capabilities in the period of 2000-2006. The project was partially supported but its progress was very limited, and finally suspended, without completing the chemical and biological components in 2015, due to the recent financial difficulties in the country.

Paul and I met mostly at every CCQM meetings, including CITAC meeting since 1999, but since 2004 when CENAM started to participate formally at the CCQM meeting as a member, we could always exchange friendly conversations, including personal issues on his beloved wife, who accompanied Paul to visit Queretaro, and on his son, who is working in Ecuador. I appreciated very much for his supportive comments to our particular development in Latin America on the world scene, as he stressed in his message. I do not think I

am a faithful follower of his idea on the metrology in chemistry and on his idea on the redefinition of the mole, but I valued his passion to the metrology in chemistry, and his motto "back to the basic". I appreciated really the nice opportunity that we could share and participate in the drafting process of traceability document of IUPAC in Vienna in 2007, which was published as an IUPAC Technical Report [IUPAC Recommendations 2008], coauthored by Rene Dybkaer, Ales Fajgelj and D. Bryan Hibbert, who are also active members of CITAC. This opportunity was given thanks to the support of PTB, which paved the basics of metrological traceability in chemistry thanks to the efforts of Dr. Wolfgang Richter and his crew. This publication of IUPAC is now almost the bible of our lectures on metrological traceability of measurement results in chemistry here and in Latin America.

However our mission is still on the way, until we could deploy appropriate metrological activities that require the society. The effort of new planning is under the leadership of our new Director General Dr. Victor Lizardi-Nieto since September 2015, who does not know Paul personally, but at least his metrological mind through our actions.

December 2016

Yoshito Mitani

**Director General of Materials Metrology
Centro Nacional de Metrología (CENAM)**

RIP : Paul De Bièvre

A GENTLEMAN & A GREAT LEADER IN MiC



I first met Paul at a Eurachem PT seminar somewhere in chilly Holland in December 1991.

He gave some thought-provoking talks about PT & Metrology in Chemistry (MiC) --What is this "new" topic all about I thought?-- & I was stimulated to have further (& lengthy!) discussions with him in the bar that night.

So began our 25 years of friendship.

We met often at meetings (CITAC, Eurachem, CCQM etc) & Conferences. Also at his home in Belgium where his lovely wife Lieve played the super host. His office there (downstairs by lift) was jammed full of papers, mainly relating to MiC...his filing system was not for the faint-hearted!

Discussions continued during walks across the fields of Flanders...he was passionate about improving chemical measurements worldwide & stimulated my sparse brain cells..".its all about ratios of numbers" he would constantly tell me.

Some of our debates were quite robust ---Paul was not a great supporter of laboratory accreditation...."look at those poor PT results from accredited labs he said..."accreditation provides no guarantees" (he was right there!)...but I thought his argument

(comparing NMI performance in Key comparisons with accredited labs in regular commercial PT programs) was rather "unbalanced". This, however, did not damage our friendship & I continued to enjoy his company, including time during his regular visits to Australia.

We continued to work & play together until I retired from ILAC in early 2014.

Paul was a gentleman & a great leader in MiC. The results of his efforts have manifested themselves around the world & filtered in to various organisations (including ILAC!)..& the work continues through Cooperations like CITAC .

He is missed by many.

Alan Squirrel

Australia

OBITUARY PROF. PAUL DE BIÈVRE



It is with great sadness that Eurachem and all of the scientific community have learned about the death of our colleague and friend Prof. Paul De Bièvre. As Eurachem members we would like to offer our condolences to his wife Lieve and all of his large family.

Paul has been and will always remain a great source of inspiration to our com-

munity of chemical metrologists. He had a bright mind, a witty and inquisitive nature and a big heart for metrology in any culture of the world. This can best be seen from his widespread cooperations with diverse international groups and through his contributions to the work of these groups. His good command of numerous languages made him a perfect initiator and moderator of many discussions about technical and the communicational difficulties in worldwide cooperation. And it was through his spirited queries that he encouraged so many of us to join the debate and thereby constantly widening the constituency interested in quality matters in chemical measurement. The conduction of workshops that he liked to call "thinkshops" became legendary.

It can be stated without exaggeration

that more than a handful of today's leading figures in the field of metrology -in-chemistry were first exposed and "caught fire" for this subject while working in his laboratory at IRMM in Geel. As the first Editor-in-Chief of a new Journal, ACQUAL, he was able to reach an even wider audience. His numerous Editorials, "food for thought" as he himself chose to call them, remain legendary and inspirational to all of us.

It is with the pledge to keep working in "his" field that we all are fondly cherishing his memory for the times to come. Might this be a little consolation to Lieve and to all his family.

R.I.P.

Wolfhard Wegscheider

Austria

Discussion Paper: Redefinition of the S.I. Unit

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In 2018, the SI will be completely revised by defining a set of seven fundamental constants with fixed values and no associated uncertainties at all, among them N_A . This in turn triggers the redefinition of the unit mole. The XRCD (x-ray crystal density) experiment for the determination of the Avogadro constant with the aid of a silicon single crystal contributes substantially to the redefinition of the kilogram, but also of the mole (so-called Avogadro Project).

The physical quantity *amount of substance* is measured in the unit *mole* (symbol: mol). It is one out of a total of seven units of the International System of Units (SI). Although the kilogram and the mole are defined as independent base units, they are closely related to each other. 1 mol of the carbon isotope ^{12}C has, by definition, a mass of exactly 12 g. Usually, the instrument used to determine the mass of an object is the balance; it is, however, also used to determine the amount of substance. The mass divided by the molar mass yields the amount of substance expressed in mol.

The molar mass has the unit g/mol. The amount of substance differs from the

mass in particular by the fact that identical amounts of different substances contain the same number of atoms, molecules or other particles, but do usually not have the same mass.

The *Avogadro constant* N_A expresses the number of atoms, molecules or other particles in one mole ($N_A = 6.02214076(12) \times 10^{23} \text{ mol}^{-1}$) [1]. When $m_a(\text{X})$ is the mass of an atom, a molecule or of another particle X and $M(\text{X})$ is the molar mass of this particle, the Avogadro constant N_A is obtained as follows:

$$N_A = \frac{M(\text{X})}{m_a(\text{X})} \quad (1)$$

HISTORY

The concepts "mole" and "Avogadro constant" are based on the interpretation of chemical processes as connections between atoms and molecules. In 1808, John Dalton (1766-1844) declared that atoms of an element do not differ from one another and that they have a defined atomic mass. In 1811, Avogadro (1776-1856) said that the same volumes of all gases contain - at the same temperature and the same pressure - the same number of molecules. In the first instance, his observation was forgotten until Stanislao Cannizzaro revived it again and published, in 1858, a consistent system of chemical formulas and "atomic weights" (relative atomic masses, see below) of all elements. After that, the concepts of atomic and molecular weight as well as other concepts borrowed from atomic theory developed in chemistry [2].

The origin of the concept "mole" is assigned to Wilhelm Ostwald [3]. In his

"Manual and Auxiliary Book for the Performance of Physicochemical Measurements" of 1893, he wrote: "Let us generally refer to the weight in grams - which is numerically identical to the molecular weight of a specified substance - as one mole, ..." [4]. Similar concepts such as, for example, "g-molecule" or "g-mole" with a comparable meaning were, however, at the same time also used by others [5]. According to this definition, the unit "mole" was, therefore, closely related to the mass and for a long time it was interpreted as "chemical mass unit". Although the atomic perception - which links the mole up with a particle number and, therefore, requires the introduction of an additional base quantity, the "amount of substance" - had existed since Dalton and Avogadro, experimental results, which could confirm these models, were first of all missing [5]. The experimental confirmation of the atomic theory and the determination of the Avogadro constant (particle number per mole) finally led to two different perceptions of the mole, which Stille [6] differentiated by the concepts "mole" (as chemical mass unit) and "mole number" (as a unit related to a particle number which is defined by the Avogadro constant). The integration of the unit "mole" into the SI system of units solved this contradiction and made a differentiation of the concepts superfluous. It took place much later, in October 1971, after the 14th General Conference of the Metre Convention had decided to introduce the "amount of substance" as the 7th base quantity. The English concept "amount of substance" was derived from the German concept "Stoffmenge", according to Stille [7,8]. First, there had been a corresponding recommendation of

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the International Union of Pure and Applied Physics (IUPAP), of the International Union of Pure and Applied Chemistry (IUPAC) and of the International Organization for Standardization (ISO), together with the note that the carbon isotope ^{12}C had to be selected as the reference point [9]. To handle questions related to the base quantity "amount of substance" a committee of the Metre Convention (Comité Consultatif pour la Quantité de Matière - CCQM) was founded in 1993 [10].

THE MOLE AS AN SI UNIT IN CHEMISTRY

With the aid of the mole, results of measurements in chemistry can be traced back to SI units and therefore internationally compared

The definition of the SI unit "mole" is [9]:

1. The mole is the amount of substance of a system which contains as many elementary entities as there are atoms in 0,012 kilogram of carbon 12; its symbol is "mol".
2. When the mole is used, the elementary entities must be specified and may be atoms, molecules, ions, electrons, other particles, or specified groups of such particles.

Unlike Ostwald, whose definition related only to the mass, reference is now made to a particle number. The number of particles corresponds to the numerical value of N_A which is today determined with smallest uncertainty within the scope of the so-called *Avogadro Project*.

In practice, traceability of the amount of substance $n(X)$ of a substance X is real-

ized via its mass m and molar mass $M(X)$:

$$n(X) = \frac{m}{M(X)} \quad (2)$$

The molar mass $M(E)$ of an element E is calculated from its mean, relative atomic mass A_r and the molar mass constant M_u (from the definition of the mole it follows that M_u is exactly 1 g/mol). All relative atomic masses use ^{12}C as the reference point: by definition: $A_r(^{12}\text{C}) = 12$ and $M(^{12}\text{C}) = 12$ g/mol. The mean A_r of an element is calculated from the $A_{r,i}$ of its isotopes ${}^i\text{E}$ and their respective amount fractions $x({}^i\text{E})$ [9, 11]:

$$M(E) = A_r(E) \times M_u \quad (3)$$

$$A_r(E) = \sum_i [x({}^i\text{E}) \times A_r({}^i\text{E})] \quad (4)$$

Traceability to the mole without reference to the mass via a determination of the particle number is, however, also imaginable, once the Avogadro constant is fixed as it is planned in the revision of the SI system. Then, counting elementary particles may also be based on other properties of a particle than the mass such as its charge or its optical or magnetic properties. They may be related to observable transitions between nuclear or electronic energy levels that can be specific for a particular molecular entity and also correlate with the particle number. Concepts for measurements of the number of elementary entities have been available for a rather long time, although this had been limited to special cases. The measurement can, for example, be performed by determining the amount of substance of a crystalline solid with the

aid of its microscopic, crystallographic lattice parameter and its macroscopic volume. In principle, this is also done within the scope of the *Avogadro Project*: a high-purity ^{28}Si single crystal serves to perform these measurements. Similar - although very much more simple experiments - have been carried out since the discovery of X-ray diffraction. There are also experiments for the (direct or indirect) counting of elementary entities, e.g. of ions [12] and electrons (single-electron-tunnelling - SET circuits) which could become relevant, for example, within the scope of a quantum-metrological redefinition of the SI base unit ampere [13,14,15].

REDEFINITIONS

The kilogram is the only one of the seven base units of the SI that is defined by a material embodiment as the mass of the international kilogram prototype. The other base units are defined by reference to a fundamental constant of physics and/or by an experimental procedure [9]. Some units also depend on other base units. The metre, which is defined as the path travelled by light in vacuum in a specific fraction of a second is, for example, defined by a specified value of the speed of light. In that definition, the second is referred to as a unit of the time. The definition of the ampere describes an idealized arrangement of two electrical conductors and states for it the values of measurands in the units *kilogram*, *metre* and *second*. These values in addition define the magnetic constant. According to the present state of knowledge, it is assumed for such definitions that the unit is invariable although its practical realization is always affected by a certain uncertainty. Also, definitions of this kind enable the unit to be realized at any location and

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at any time.

It has also been found that the masses of the national prototypes of the kilogram have significantly drifted during the last hundred years with respect to that of the international prototype [16,17]. On average, they have put on about 0.05 mg. As the international prototype does not basically differ from the other prototypes as regards its physical properties, it is rather probable that it has lost weight compared, for example, to an atomic mass. Also, all prototypes might be subject to an additional drift which is still unknown.

Therefore, for more than 30 years the attempt has been made to define also the kilogram via an atomic constant or a fundamental constant of physics. The redefinition of the kilogram is becoming more and more probable and scheduled for 2018. It will be based on the Planck constant h .

In addition to a redefinition of the kilogram, it is planned to redefine also the ampere, the kelvin and the mole. According to the current definition of the mole, the mole is linked directly to the kilogram definition via the mass of ^{12}C [9]. To break this link is the most important reason to re-define the mole. Another reason is the general demand to base all unit definitions solely on fundamental constants [18]. For the mole, the definition valid so far shall be reworded in such a way that it is based on a fixed value of the Avogadro constant N_A without relating to the unit "kilogram" as it is the case in the current definition. The most recent drafts reads [19,20]

The mole, symbol mol, is the SI unit of amount of substance of a specified elementary entity, which may be an atom, molecule, ion, electron, any other particle or a specified group of such particles. It is defined by taking the fixed numerical value of the Avogadro constant N_A to be

$6.022\,140\,857 \times 10^{23}$ when expressed in the unit mol^{-1} .

SILICON SINGLE CRYSTAL AND THE AVOGADRO CONSTANT

The *Avogadro Project* has its origins in the 1970s, when German researchers started for the first time in determining the lattice distances in a silicon crystal by X-ray interferometry. This allowed the kilogram to be linked up with the atomic mass unit. As a connecting link between a macroscopic mass and the mass of an atom acts the Avogadro constant which indicates the number of atoms in a mole.

The N_A is named after the Italian earl and advocate Amedeo Avogadro who - at the beginning of the 19th century - dealt with atomism which had been explained by Dalton and who brought in line all observations connected with it. Dalton had demonstrated that all gases expand in the same way when they are heated. This, concluded Avogadro, could be explained only by the fact that the number of gas particles involved is also always the same: Identical volumes of all gases had to contain - in the case of identical external conditions - the same number of smallest particles. But it seems that Avogadro did not really know how to determine this number.

Only in 1865, Josef Loschmidt calculated the particle number per volume with the aid of the mean free path of gas molecules which was determined by Oskar Meyer and, later, by James Maxwell. At that time, this value deviated by only 20 % from the value recognized today. Forty years later, Albert Einstein tried to indicate the Avogadro constant more precisely: In his doctoral thesis of 1905, in which he determined the number of molecules per mole on the basis of a novel diffusion equation from measurements on a sugar

solution, his values deviated - due to a calculation error - by a factor of three.

In 1917, Robert Millikan reduced the error to less than one percent by clearly improving his famous oil droplet test for determination of the elementary charge. Today, this accuracy is no longer sufficient. Managed by PTB, scientists from several metrology institutes have joined forces to determine - with the aid of a nearly perfect silicon single crystal - the Avogadro constant as exactly as never before [1].

The achievement of this objective required, however, many years of development. At the beginning, silicon crystals of natural isotopic composition were used for the determination of N_A . At several metrology institutes such as, for example, PTB, the National Institute of Standards and Technology (NIST USA), the National Physical Laboratory (NPL, Great Britain), the National Measurement Institute of Japan (NMIJ) and the Istituto Nazionale di Ricerca (INRIM, Italy), measurements of N_A were started at the end of the 1970s with the establishment of an X-ray interferometer for measuring the lattice distance in the silicon single crystal. Despite the increased efforts made at all institutes involved, it has - in the coming years - been impossible to achieve a measurement uncertainty of less than 3×10^{-7} . Success was reached only when some years ago, the newly established International Avogadro Coordination (IAC) and other institutions and companies repeated the measurements with highly enriched ^{28}Si applying the XRCD method [21, 22].

To determine N_A , the volume V and the mass m of the highly enriched silicon single crystal sphere are measured:

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$$N_A = \frac{N \times M(\text{Si}) \times V_{\text{sphere}}}{m \times v_{\text{cell}}}$$

(5)

Additional samples of the same crystal serve to determine the molar mass M and the volume v_{cell} of the unit cell. N is the number of atoms in the unit cell (in case of Si: $v_{\text{cell}} = 8$). All quantities must, of course, be measured traceable to the SI units. The volume of the silicon sphere is determined by interferometric measurements of the sphere diameter. This provides a complete surface topography of the sphere. The spheres exhibit a deviation from a perfect spherical shape of only a few 10 nm which in turn is required to achieve the necessary uncertainty associated with V_{sphere} . The silicon sphere is coated with an oxide layer, some nm in thickness, which geometry and mass must be determined. On top of this layer additional adsorbed layers of water and hydrocarbons are present and have to be characterized. The mass of the silicon sphere is linked up with the kilogram prototype by weighing. Due to the large density differences between silicon and platinum-iridium, the accuracy requirements for the mass determination can, at present, be achieved only under vacuum conditions, i.e. without air buoyancy correction. Volume and mass determinations must – in addition – be corrected with respect to the coating layers. The volume of the unit cell is determined from the lattice distance of the crystal with the aid of an X-ray interferometer. A sufficient knowledge of the impurities and of the crystal defects is also indispensable. The molar mass is determined by mass spectrometry from the atomic masses of the three isotopes ^{28}Si , ^{29}Si and ^{30}Si and from their amount-of-substance fractions. The atomic masses are linked up with the



Figure 1: Single crystal consisting of silicon highly enriched in ^{28}Si of approximately 4.7 kg. Source: Leibniz-Institut für Crystal Growth (IKZ), Berlin, Germany.

mass of the carbon isotope ^{12}C by means of penning traps. Using natural silicon, the measurement uncertainty associated with the molar mass could not have been improved any further. This is why the investigations for the determination of N_A were stopped some years ago when a measurement uncertainty of approx. 3×10^{-7} was reached [23].

The possibility of using enriched ^{28}Si on a large scale for sample preparation gave new momentum to the *Avogadro Project*. Estimates showed that the uncertainties associated with the isotopic composition would be considerably reduced. In 2003, in a cooperation with research institutes in Russia, several national and international metrology institutes therefore launched the ambitious plan to prepare approx. 5 kg of silicon highly enriched with respect to ^{28}Si ($x(^{28}\text{Si}) > 0.9999$ mol/mol). With this single crystal material a measurement uncertainty of approx. 2×10^{-8} by the year 2010 was targeted.

In 2008, the material was successfully

prepared with the growing of a perfect single crystal at the Institute for Crystal Growth (IKZ) in Berlin (Figure 1) and two 1 kg ^{28}Si spheres could be polished at the Australian ACPO.

Despite the theoretical prediction the targeted uncertainty associated with N_A could not be achieved, because again the molar mass determination was the limiting quantity due to problems with the chemical blank caused by ubiquitous natural silicon and the much larger dynamic range necessary to do the mass spectrometric measurements. Switching from gas mass spectrometry to inductively coupled plasma mass spectrometry and the development of the so-called “virtual element isotope dilution mass spectrometry” (VE-IDMS) (Figure 2) solved these new problems inherent to the enriched silicon [24,25,26,27].

First results using the enriched material have shown that a more exact value of N_A with an uncertainty of 3×10^{-8} could be obtained and the cause of the observed deviation with respect to other fundamental constants be detected [22]. Meanwhile (2015), the uncertainties associated to almost all quantities contributing to the determination of N_A (equation 5) were substantially improved [28] resulting in a relative uncertainty associated with N_A of less than 2×10^{-8} [1,28]. Most notably the relative uncertainty associated with the molar mass was reduced during the last decade by nearly three orders of magnitude down to less than 1×10^{-9} [27], shifting its relative contribution to the uncertainty of N_A from 60 % (ten years ago) to a mere 6 % today.

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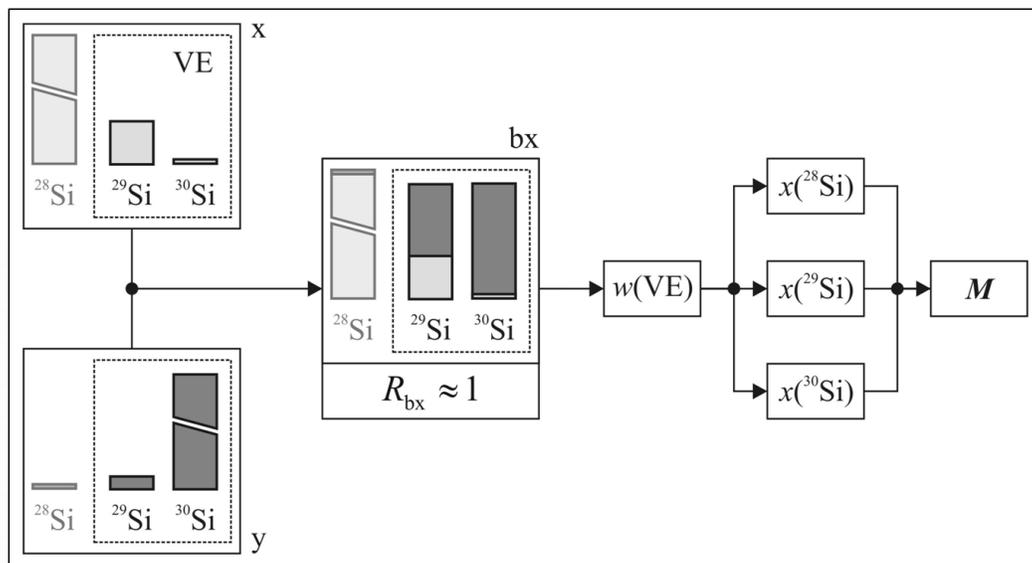


Figure 2: The principle of the so-called “Virtual Element Isotope Dilution Mass Spectrometry” (VE-IDMS) is based on ignoring the ^{28}Si signal completely and mixing the original ^{28}Si -enriched silicon x with a ^{30}Si -enriched material y in a way to adjust a $^{30}\text{Si}/^{29}\text{Si}$ ratio in the blend bx close to one. From the measurement of this ratio, the mass fraction $w(\text{VE})$ of the VE can be calculated. This mass fraction yields the amount-of-substance fractions $x(^i\text{Si})$ of all three isotopes and as the final result the molar mass of silicon in the original sample. Source: *Metrologia* 47 (2010) 460-463.

REALIZATION AND DISSEMINATION IN ACCORDANCE WITH THE REDEFINITIONS

Also after the planned redefinition of the mole by fixing the numerical value of N_A , the amount of substance will only in rare cases be determined by the counting of atoms or molecules. This means that ratios of amounts of substances will also in future be measured with mass spectrometers. Weighing of the reference material will remain the method for the determination of the quantitative reference point. Here, the question arises whether – or how – the molar masses will change after a redefinition.

If the mole will, in the future, be defined by a fixed value of the Avogadro constant N_A , and the kilogram by a value of the Planck constant h – as currently planned – this could lead to a redundant dimensioning as N_A and h depend on each other via other constants forming the so-called “molar Planck constant” $N_A h$ (c speed of

light in vacuum, $A_r(e)$ relative mass of the electron in u , $M_u = 10^{-3} \text{ kg mol}^{-1}$ molar mass constant, α fine-structure constant, R_∞ Rydberg constant) [29]:

$$N_A \times h = \frac{c \times A_r(e) \times M_u \times \alpha^2}{2R_\infty} \quad (6)$$

Then, in equation (6), c , h and N_A are defined in the definitions of the metre, the kilogram and the mole, whereas α and R_∞ remain independent measurands. This means, only $A_r(e)$ or M_u have to be considered as variables which depend on these measurands. If the value of $A_r(e)$ changed as a result of new measurement results of α and R_∞ and a corresponding adjustment, all relative atomic masses would change as well. In particular $A_r(^{12}\text{C}) = 12$ would no longer be exactly valid as a reference. As the relative atomic masses are measured with uncertainties which are comparable

to that of α , future changes would be significant and require the compilation of new tables of the relative atomic masses. This is why the proposal to introduce M_u as a variable [30] seems to be more useful and would, in addition, have negligible impacts. As the molar mass of an element X is calculated according to $M(X) = A_r(X) M_u$, the molar masses would change with M_u .

From the moment of the redefinition until the availability of new values of α or R_∞ , $M_u = 10^{-3} \text{ kg mol}^{-1}$ will be valid. After the redefinition, the relative uncertainty of M_u and, correspondingly, that of the molar mass of ^{12}C , will depend predominantly on the measurement uncertainty of α^2 which is at present in the order of 10^{-9} , future relative changes will probably not exceed this value. In view of the smallest relative uncertainties which are achieved in the realization of the mole and the molar masses (approx. 10^{-5}), no changes will result in practical applications.

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References

1. Azuma Y, Barat P, Bartl G, Bettin H, Borys M, Busch I, Cibik L, D'Agostino G, Fujii K, Fujimoto H, Hioki A, Krumrey M, Kuetsgens U, Kuramoto N, Mana G, Massa E, Meeß R, Mizushima S, Narukawa T, Nicolaus A, Pramann A, Rabb S A, Rienitz O, Sasso C, Stock M, Vocke Jr R D, Waseda A, Wundrack S, Zakel S, Metrologia, 52 (2015) 360.
2. Becker, P., Rep. Prog. Phys., 64 (2001) 1945.
3. Sommerfeld, A., 1929, Atombau und Spektrallinien, Harri Deutsch Verlag (reproduction 1978)
4. Ostwald, W., 1893, Hand- und Hilfsbuch zur Ausführung Physiko-Chemischer Messungen, Leipzig, W. Engelmann Verlag
5. Nernst, W., 1893, Theoretische Chemie vom Standpunkte der Avogadro'schen Regel und der Thermodynamik, Stuttgart, Enke Verlag
6. Stille, U., 1955, Messen und Rechnen in der Physik, Braunschweig, Friedrich Vieweg Verlag
7. Guggenheim, E. A., The J. Chem. Ed., 38 (1961) 86.
8. Milton, M.J.T., Phil. Trans. R. Soc. A, 369 (2011) 3993.
9. Bureau international des poids et mesures (BIPM), 2006, Le Système international d'unités (SI) – The International System of Units (SI). 8th edition, Paris/Sèvres
10. Bureau international des poids et mesures (BIPM), 1993, Procès-verbaux des séances du Comité International des Poids et Mesures, 82nd meeting (1993), Sèvres, France: BIPM
11. De Laeter, J.R., et al., Pure Appl. Chem., 76 (2003) 683.
12. Schlegel, C., Gläser, M., Scholz, F., Mecke, M., Kiekenap, G., Thomsen-Schmidt, P., Hoffmann, K.P. and Ahbe, T., Metrologia, 47 (2010) 146.
13. Bylander, J., Duty, P. and Delsing, P., Nature, 434 (2005) 361.
14. Zimmermann, N.L. and Keller, M.W., Meas. Sci. Technol., 13 (2003) 1237.
15. Ebbecke, J., Bastian, G., Blöcker, M., Pierz, K., Ahlers F.J., Appl. Phys. Lett., 77 (2000) 2601.
16. Quinn, T., IEEE Trans. Instrum. Meas., 40 (1991) 81.
17. Girard, G., Metrologia, 31 (1994) 317.
18. Fischer J, Ullrich J, Nature Physics, 12 (2016) 4.
19. CCQM/16-04, Draft mise en pratique for the definition of the mole, BIPM, <http://www.bipm.org/en/measurement-units/new-si/>
20. BIPM, DRAFT 9th edition of the SI Brochure, <http://www.bipm.org/en/measurement-units/new-si/>
21. Deslattes, R.D., Henins, A., Bowman, H.A., Schoonover, R.M., Carroll, C.L., Barnes, I.L., Machlan, L.A., Moore, L.J., and Shields, W.R., Phys. Rev. Lett., 33 (1974) 463.
22. Andreas, B., Azuma, Y., Bartl, G., Becker, P., Bettin, H., Borys, M., Busch, I., Gray, M., Fuchs, P., Fujii, K., Fujimoto, H., Kessler, E., Krumrey, M., Kuetsgens, U., Kuramoto, N., Mana, G., Manson, P., Massa, E., Mizushima, S., Nicolaus, A., Picard, A., Pramann, A., Rienitz, O., Schiel, D., Valkiers, S., and Waseda, A., Phys. Rev. Lett., 106 (2011) 030801.
23. Fujii, K., Waseda, A., Kuramoto, N., Mizushima, S., Becker, P., Bettin, H., Nicolaus, A., Kuetsgens, U., Valkiers, S., Taylor, P., De Bièvre, P., Mana, G., Massa, E., Matyi, R., Kessler, E.G., Hanke, M., IEEE Trans. Instrum. Meas., 50 (2005) 854.
24. Mana G, Rienitz O., Int. J. Mass Spectrometry, 291 (2010) 55.
25. Rienitz O., Pramann A., Schiel D., Int. J. Mass Spectrometry, 289 (2010) 47.
26. Pramann A., Rienitz O., Schiel D., Güttler B., Valkiers S., Int. J. Mass Spectrometry, 305 (2011) 58.
27. Pramann A., Rienitz O., Anal. Chem., 88 (2016) 5963.
28. Fujii K., Bettin H., Becker P., Massa E., Rienitz O., Pramann A., Nicolaus A., Kuramoto N., Busch I., Borys M., Metrologia, 53 (2016) A19.
29. Mohr, P. J., Taylor, B. N. and Newell, D. B., J. Phys. Chem. Ref. Data, 37 (2008) 1187.
30. Taylor, B. N., Metrologia, 46 (2009) L16.

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AFRIMETS REPORT TO CITAC

Paris // April 2016

1. Summary of general issues

AFRIMETS held its 9th General Assembly (GA) hosted by NMISA in partnership with the PTB of Germany at Emperors Palace Convention Centre, Johannesburg, from the 28th to the 30th of July 2015. The GA and related meetings were attended by more than 80 delegates from all over the continent and international partners.

Three Technical Committee (TC) Working Groups (TC-Mass & Related Quantities, TC-Legal Metrology and TC-Quality Systems) met to report on comparisons and to draw up work plans for the next year. More than 50 delegates from all over Africa attended the three meetings.

The TC-Mass meeting were attended by representatives from 18 countries. It started with country reports where members gave feedback on metrology developments in their respective countries. It was stressed by a number of the members that metrology is necessary not only to comply with standards and requirements for the international system, but also to facilitate trade and to eliminate technical barriers to trade in the sub-regions. The committee discussed current and future ILCs required for the region and the recent reissued certificates to NMIs by BIPM for calibration of mass Prototypes and the consequences thereof. It was indicated that the impact was minimal in the region since majority of the NMI's standards are calibrated at lower levels.

Presentations were also made about the redefinition of the SI envisaged

for 2018. Discussions followed and proposals were made about possible collaborations between interested NMIs to invest in resources to have one or two experiments/systems to realise the unit for mass in Africa. NMISA will facilitate discussions with interested NMIs and coordinate the collaborations which might come from the discussions.

The TC-QS discussed the submission from Tunisia for the approval of the Quality system of a designated institute, DEFNAT. The future submission of QS for approval were also discussed. In total 7 Associates of the CGPM (5 from SADC) reported that they planned to submit their QSs for approval. To date 5 of the 7 QSs were submitted and are in the final TC-QS review phase.

The Executive Committee of AFRIMETS meeting (EXCOM) was attended by representatives from five of the six (CEMACMET could not be present) sub-regional metrology organisations, the BIPM, the OIML, the African Union Commission (AUC), the PTB and the Pan African Quality Infrastructure (PAQI). The meeting discussed the progress in metrology in the sub-regions, the activities of BIPM/CIPM and BIML/OIML and received reports from the PTB, UNIDO and the AUC on projects in metrology in Africa. The meeting expressed support for the projects in anticipation of the re-definition of the SI by 2018, but some members cautioned that the bulk of activities in metrology in Africa should remain at the level that most countries need.

The EXCOM was followed by a short

GA where for the period 2015 to 2017, Mr Dennis Moturi of KEBS (Kenya) was elected as the Chair of AFRIMETS, Mr Wondwosen Fisseha of NMIE (Ethiopia) as the Vice-Chair Scientific Metrology and Mr Jaco Marneweck from NRCS (South Africa) as the Vice-Chair Legal Metrology. The GA also welcomed the efforts to establish the BIPM Capacity Building and Knowledge Transfer Programme to include scientific work on traceability for mycotoxins, encouraged participation by AFRIMETS members and encouraged donors to support these activities.

The next General Assembly is to be hosted by the NMI of Mauritania in Nouakchott during the latter part of July 2016. All RMOs are invited to send a representative. Details will be available soon on the AFRIMETS website.

2. RMO Contact Details

See the following page.

3. Current TC and Working Group Chairs and Contact Details

The AFRIMETS structure includes five technical committees of which TC-1A is responsible for Metre Convention affairs, TC-1B for OIML and BIML affairs, TC-2 for Metrology Infrastructure, TC-3 for Metrology Training, TC-4 for Metrology Legislation and TC-5 for Metrology Awareness. The scientific metrology technical working groups in AFRIMETS fall under Technical TC-1A. The working groups are identified as TC-(parameter), to mirror the CC-WGs.

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The contact details of the TC-Chairs important to Chemistry are listed below:

Function	Name	Details
TC – QM	Dr Angelique Botha (NMISA)	National Metrology Institute of South Africa, Private Bag X34, Lynnwood Ridge, 0040, RSA Tel: +27-12 8413800 E-mail: abotha@nmisa.org
Vice-Chair (Bio analysis)	Mrs Desiree Prevoo (NMISA)	Tel: +27-12 8414576 E-mail: dprevoo@nmisa.org
TC-Mass and Related Quantities (Convener)	Dr Alaa Eltaweel (Egypt)	National Institute for Standards (NIS) Tersa Street, El Haram, Giza, 12211 , Egypt Tel: +202 33867451 Fax: +202 33867451 E-mail: eltaweel@nis.sci.eg
Vice-Chair (Convener)	Mr Thomas Mautjana (South Africa)	National Metrology Institute of South Africa, Private Bag X34, Lynnwood Ridge, 0040, RSA Tel: +27 12 8413457 Fax: +27 12 8412131 E-mail: TMautjana@nmisa.org
TC – QS	Dr Noha Emad Khaled	National Institute for Standards Tersa Street, El Haram , Giza, 12211 Egypt
Vice-Chair (CMCs)	Dr Wynand Louw	Tel: ++(202)33862322 Fax: ++(202) 33862322 Email: nemadnis@yahoo.co.uk or nemadnis@netscape.net
Vice-Chair (QS review)	Mr Peter Kahihia	National Metrology Institute of South Africa, Private Bag X34, Lynnwood Ridge, 0040, RSA Tel: +27 12 841-4227 Fax: +27 12 86 530 5916 E-mail: wlowu@nmisa.org Kenya Bureau of Standards Popo Road, PO Box 54974-00200, Nairobi, Kenya Tel : +254 20 6948431 Fax : +254 20 6005673 E-mail : pkahihia@kebs.org

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4. RMO Membership Update

AFRIMETS has 6 sub-regional RMOs as Principal members representing 45 countries in Africa. In addition, Sudan became a member in 2013 through the Sudanese Standard and Metrology Organisation (SSMO) and Sierra Leone and the Gambia became Ordinary country members during 2015, bringing the total number of member countries of AFRIMETS to 44 (if Libya is included, 45, but currently no contact can be made with the original signatory of the NEWMET MOU).

The Associate members are the PTB, LNE, the NIRPR (National Institute of Radiation Protection and Research – Nigerian Nuclear Regulation Authority), GRPI -Ghana Radiation Protection Institute, TAEC (the Tanzania Atomic Energy Commission), INSTN of Madagascar and the IAEA. Observers include EURAMET, the Arab Federation of Metrology (AFM), the African Committee of Metrology (CAFMET) and the African Electrotechnical Standardisation Commission (AFSEC).

The Members of the BIPM and Associates of the CGPM are shown below, as well as

the participants in the CIPM MRA. Sudan signed the CIPM MRA on 26 June 2014.

5. AFRIMETS CMCs

There is a total of 451 CMCs accepted in Appendix C of the BIPM Key Comparison Database (KCDB) (415 from South Africa, 35 from Egypt and 1 from Kenya). This includes a total of 42 CMCs in Chemistry (39 South Africa, 2 Egypt and 1 Kenya). A further 24 are in the inter-RMO review process (to be concluded by mid-year).

6. Development work in Chemistry

During 2015, NMISA launched a Food and Feed Reference Material Programme dedicated to providing measurement support to Food and Feed testing laboratories through the provision of Certified Reference Materials, Reference analysis and Proficiency testing schemes. The capability to produce and export safe food and feed relies heavily on the testing of their nutritional and contaminant content. For testing laboratories to competently and accurately test these, they need assurance that their measurement results are accurate, reliable and internationally comparable. This is achieved through the use of certified reference materials to

calibrate measurement instruments and proficiency testing, all requirements for accreditation of a testing/measurement capability.

The programme started with a unique opportunity to all laboratory managers, QC managers, researchers and industry regulators in the food and feed industry to participate in a Food and Feed Reference Material Needs Analysis Workshop on the 9th June 2015, in Pretoria and on the 13th October 2015 in the Western Cape. Over 110 representatives from Retail, Commercial, Government and Research Testing Laboratories in South Africa participated in these workshops. The aim was specifically to assist NMISA to identify and prioritise the types of reference materials required, measurement applications and analytical challenges laboratories encounter and then to propose the best strategy to produce fit-for-purpose reference materials and PT schemes that are relevant to South Africa and the African continent.

NMISA currently has expertise in food contaminant and nutrient analysis that includes heavy metals, pesticides, mycotoxins, dioxins and other industrial contaminants; Fatty Acid Methyl Ester (FAME) profiling for determining meat

Member Country	Members of the BIPM	Associates of the CGPM	Signatories to CIPM MRA
Egypt	X		X
South Africa	X		X
Kenya	X		X
Tunisia	X		X
Botswana		X	X
Ghana		X	X
Mauritius		X	X
Namibia		X	X
Seychelles		X	X
Zambia		X	X
Zimbabwe		X	X
Sudan		X	X

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authenticity, veterinary drug residues, and quantifying amino acids for confirming protein quality. Gaps in capabilities were identified according to the feedback from the industry representatives and the required capabilities will be established over the next three years.

The process focused the programme on food matrices particular or very important to Africa –white maize, cassava, ground and tree nuts, coffee, cacao and the main food products of Southern Africa. It will also assist the testing of imported food and feed (NMISA tested a few hundred grain samples for heavy metal and other contaminants for the Southern African Grain laboratories in 2015/16).

Going international, a NMISA-initiated AFRIMETS proposal was submitted to the metrology organisations of the Regional Economic Blocks in the Americas, Asia and Europe and finally to the International Committee for Weights and Measures (CIPM) during 2015/16, for building the

core capability of developing NMIs to provide reference materials in support of food safety. This has culminated in the signing of a Memorandum of Understanding (MoU) between NMISA, the International Bureau of Weights and Measures (BIPM) and NIM, the metrology institute of China. The MoU's are based on the activities of the Mycotoxin Metrology Capacity Building and Knowledge Transfer "Joint Technical Project". The MoU with NIM China allows for cooperation on various scientific activities, which would also support knowledge sharing and the production of matrix reference materials in the African Food and Feed Reference Material Programme.

To further enhance Africa's public health and regional/international trade in foodstuffs through laboratory networking. NMISA is working together with the Agricultural Research Council-Onderstepoort Veterinary Institute (ARC-OVI) on the International Atomic Energy Agency IAEA -AFRA RAF5/0/67 project, started January

2016, that will result in a wider and functional network of food safety laboratories using nuclear and complementary analytical techniques that meet international standards. During 2015/16 three NMISA MSc studentships were funded for food safety projects including 1) the determination of mycotoxins in maize, 2) the determination of amino acids in infant formula, and 3) arsenic speciation in foods.

7. Conclusion

All AFRIMETS structures including the technical and quality system working groups are functioning well. Key and Supplementary comparisons are being conducted and it is expected that a number of new CMCs will be submitted by Associates during the next 1-2 years.

Wynand Louw

NMI, South Africa

APMP LIAISON REPORT TO CITAC

31st CITAC Meeting, Paris

20 April 2016
Highlights

National Institute of Metrology China hosted the 31th APMP GA and related meetings from 1 to 5 November 2015, in Beijing. The activities included:

1. The APMP General Assembly;
2. Meetings of the governance bodies: the APMP Executive Committee and Technical Committee Chairs' (TCC);
3. Meetings (and, in some cases, Workshops) of APMP's 12 Technical Committees (TCs);
4. Meetings of the APMP Developing Economies' Committee (DEC);
5. The 6th APMP NMI Directors' Workshop;
6. A national Symposium organized by the host NMI;

7. MEDEA Project Consultation and Planning Meetings; and Laboratory tours.

A total of 57 participants attended TCQM meeting. Along with the above activities a TCQM workshop was also held on 30-31 October 2015 in Beijing discussing :

- Traceability to the definition of SI units for inorganic analysis
- MEDEA symposium on metrology in chemistry
- Establishing metrological traceability in advanced lipoprotein testing: the BioSITrace project

Gas analysis workshop

- 13th meeting: 3 August 2015, Bangkok
- Next workshop in Mongolia hosted by MASM (in July 2016)

Membership

New Full Membership applications:

- Central Geological Laboratory of Mongolia (CGL Mongolia)
- Department of Chemistry, Malaysia (KIMIA)
- Centre for Technology of Radiation Safety and Metrology – National Nuclear Energy Agency of Indonesia (PTKMR-BATAN)

New Associate Membership applications:

- Emirates Metrology Institute (EMI)
- Central Organization for Standardization and Quality Control (COSQC), Iraq

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- Membership query received from Department of Research and Innovation (DRI), Myanmar

Important developments

- Technical workshops in electrical and mass metrology
- MEDEA (Metrology-Enabling Developing Economies in Asia) Meeting
- Training workshops in:
 - * quality systems & CMC preparation (July, Philippines),
 - * strategy and performance management (July, Malaysia),
 - * mass (Sep, China);
 - * electrical (Nov, China)
 - * Training attachments in chemical metrology

New strategic initiatives:

- Proposal to GA: TCI budgets to be increased by \$100k for 2015 to fund Focus Group activities
- EC will seek legal advice regarding whether APMP needs to become a legal entity

The 2014 APMP General Assembly (GA) endorsed a work program under a revised APMP Strategy that recognised that in order to increase its impact, APMP's resources and activities must be better focused on issues that are priorities at both national and regional levels while continuing to deliver on its current functions. The following projects were identified as the regional priority with lead NMI in brackets:

- Renewable Energy/Energy Efficiency (NIMT, Thailand);
- Food Safety (NIM, China);
- Medical Metrology (CMS/ITRI, Chinese Taipei);
- Metrology for Climate Change (KRISS, Korea).

The first workshop was held in China during the APMP week and discussed the following:

- Prioritising funding through APMP's Technical Committee Initiatives (TCI) and Developing Economies' Committee (DEC) budgets;
- Establishing and funding one-off projects;
- Coordinating projects and collaborations;
- Identifying issues to which TCs and/or the DEC should consider and respond; and
- Initiating and strengthening ties with external stakeholders by prioritising APMP participation in meetings and other engagements.

Decision was taken in such a way that each WG Chair will draft a work program to be submitted to the APMP Executive Committee (EC) for review at APMP's mid-year meetings in June. TCQM submitted a proposal for 2016 TCI project titled: Research on metrology for low-cost sensors used for urban air quality measurements in Asia Pacific region.

Metrology: Enabling Developing Economies within Asia (MEDEA Project):

Under the APMP-PTB MoU, APMP is working with PTB and the Asia Pacific Legal Metrology Forum (APLMF) on a joint regional capacity building project, the MEDEA Project. A number of PTB-APMP-APLMF meetings have taken place since the project started in late May 2014. Ten Work Packages have been identified and are at various stages of progress. The 4 joint APMP-APLMF Work Packages cover:

- Development of national metrology infrastructure in developing economies;
- Raising stakeholder awareness;
- Exchanging information on different approaches to technical assistance; and
- Communication skills.

There are 5 APMP-specific topics covering, e.g. training courses and comparisons in various physical metrology areas, metrology in chemistry and preparation of

CMCs.

APMP-APLAC Cooperation:

Joint Proficiency Testing Working Group proposed Joint PTs:

The APMP-APLAC Joint Proficiency Testing (PT) Working Group established under the APMP-APLAC MoU continues to make steady progress under the care of the co-convenors Dr Della Sin (APMP) and Dr Koichi Nara (APLAC). NMIs/DIs coordinate PTs and provide homogeneous and stable samples with certified reference values as assigned PT reference values, with registered relevant CMCs or KCRVs.

Three joint PT Schemes were completed in 2014 and draft reports are being finalised: Two projects were initiated in 2015 and five new projects proposed for future studies.

Projects completed in 2014 are:

- APLAC PT T093: Toxic elements (Pb and Cd) in Cabbage (KRISS/KOLAS)
 - ◇ 83 participants
 - ◇ Completed
- APLAC PT T094: Pesticide residues (*p,p'*-DDE and alpha-endosulfan) in Cabbage (KRISS/KOLAS)
 - ◇ 70 participants
 - ◇ Completed
- APLAC PT T095: Elements (Ca and Cd) in drinking water (NMIJ/GLHK)
 - ◇ 99 participants
 - ◇ Measurement finished
 - ◇ Parallel to CCQM-K124/P158

Two new joint PT programs relating to food safety have been initiated in 2015/2016:

- APLAC PT T100: Toxic elements (Pb and Cd) in wheat flour <KRISS/KOLAS>
 - ◇ 91 participants
 - ◇ Measurement finished
- APLAC PT T102: Pesticide in fruit juice <GLHK> - Proposal approved.

Joint PT's proposed:

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- Elements in food supplement (GLHK)
- Organochlorine pesticides in ginseng root (GLHK)
- Nutritional elements (Fe and Zn) in wheat flour (KRISS)
- Cadmium in milk powder (NIM)
- Phthalate esters in edible oil (NIM)

Stakeholder engagements

APMP's has linkages with APEC and Asia Pacific Legal Metrology Forum (APLMF) Meeting

Upcoming stakeholder meetings for 2016:

- GULFMET 1st Gulf Metrology Forum Doha, Qatar,
- 14-15 Dec 2015 – APMP Chair
- APEC SCSC and SRB Forum Jan/Feb 2016
- Advances in Metrology Symposium (AdMET) NPLI, Feb 2016
- CIPM MRA Working Group 14-15 March 2016
- 35th Meeting of the JCRB 16-17 March 2016
- APMP Mid year Meetings, Hosted by NIM Thailand and held in Bangkok,

June 2016

Key and Supplementary Comparisons

Data on key and supplementary comparisons in APMP is available on the KCDB website.

From 1999 APMP TCQM organized:

- 12 key comparisons (9 completed)
 - Categories: gases (9) and pH (3)
- 11 supplementary comparisons (7 completed)
 - Categories: gases (7) and food (4)
- 30 pilot studies (26 completed, 1 cancelled)
 - Categories: food and biological materials (21), pH (5), cosmetics (2), inorganic solutions (1) and surface (1)

New proposals

- APMP.QM-K22 Volatile organic compounds in air (KRISS)
- APMP.QM-Sxx 1000 µmol/mol N₂O in N₂ (NIM)

- APMP.QM-Sxx Elements in food supplement (GLHK)
- APMP.QM-Sxx Organochlorine pesticides in ginseng root (GLHK)
- APMP.QM-Pxx Cadmium in milk powder (NIM)
- APMP.QM-Pxx Phthalate ester in edible oil (NIM)
- APMP.QM-Pxx E. coli plate count in milk (NIM)

Chairperson and Secretariat:

New Chair Elect for APMP from GA 2016-19 is Dr Toshi Takatsuji from NMIJ, Japan

Laly Samuel

APMP Liaison

New Zealand

ANNUAL REPORT OF COOMET 1.8 "PHYSICO-CHEMISTRY" TO CITAC

2015 - 2016

1 GENERAL INFORMATION

Activities of the COOMET Technical Committee 1.8 "Physico-Chemistry" covers those measurement services, which could be referred to "Metrology in chemistry" category.

COOMET TC 1.8 members

25 NMIs from 20 COOMET Member-Countries are represented in TC 1.8 now. They are: AZSTANDART (Azerbaijan), NIM (Armenia), BelGIM (Belarus), BIM (Bulgaria), IMBIH (Bosnia and Herzegovina), PTB and BAM (Germany),

KazInMetr (Kazakhstan), NISM (Kyrgyzstan), CIM (North Korea), INIMET (Cuba), VMT (Lithuania), NIM (Moldova), INM (Romania), VNIIM, VNIIFTRI, UNIIM, VNIIOFI, VNIIMS (Russia), SMU (Slovakia), Tajikistan (Tajikstandart), TUBITAK UME (Turkey), Ukrmetrteststandart (Ukraine) as well as CNS Uz (Uzbekistan) and GEOSTM (Georgia).

In 2015/16 the work of COOMET TC 1.8 "Physico-Chemistry" was carried out in the following directions:

- Organization and realization of the work on the preparation of the NMI CMC data of the COOMET Member States that have signed the MRA.

- Organization and realization of the internal review of CMC for COOMET NMIs and the interregional review of NMI CMCs for other regional metrological organizations.

- Planning and organizing of international comparisons and interlaboratory researches.

- Getting the TC 1.8 Members acquainted with the CCQM and COOMET documents aimed at the realization of the MRA.

- Improvement of the TC 1.8 structure.

- Promotion in rendering metrological services in the field of physico-chemical measurements.

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2 TC 1.8 PROJECTS

The NMIs of the COOMET Member States as well as interested metrological centers and designated laboratories of other countries that have an appropriate basis of standard equipment can take part in the TC projects connected with carrying out of international comparisons and pilot researches. The TC projects are coordinated at present by VNIIM, UNIIM and VNIIFTRI.

VNIIM is the coordinator of:

1 COOMET Project № 576/RU/12 (COOMET.QM-S5) «Supplementary comparison of national standards in the field of analysis of gas mixture of CO₂, CO, C₃H₈ in nitrogen (automotive emission gases)». Participants: VNIIM, KazInMetr, BelGIM, Ukrmetrteststandart and BAM. Status of the comparison is changed from 'Key' to "Supplementary" in accordance with recommendations of GAWG CCQM. Final report.

2 COOMET Project № 615/RU/13 (COOMET.QM-K93) «Key comparison in the field of measurements of mass fraction of ethanol in nitrogen». Participants: VNIIM and Ukrmetrteststandart. Final report.

3 COOMET Project № 596/RU/13 Pilot comparison "Pesticides in tea". Participants: VNIIM, BelGIM, Ukrmetrteststandart, KazInMetr, CSM ME (Kyrgyzstan). The Final report.

4 COOMET Project № 622/RU/13 Supplementary comparison "C₃-C₅ components in mixtures of liquified hydrocarbons". Participants: VNIIM, BelGIM, Ukrmetrteststandart, KazInMetr. Deferred to 2016.

5 COOMET Project № 611/RU/13 Pilot comparison "Melamine in milk powder". Participants: VNIIM, BelGIM, Ukrmetrteststandart, KazInMetr. Status of the Project is "proposed". The samples are prepared for distribution.

6 COOMET Project № 519/RU/11 "Pilot comparison in the field of measurements of ethanol mass fraction in aqueous solutions". Participants: VNIIM, Ukrmetrteststandart, CSM ME (Kyrgyzstan), NIM (Moldova). Draft report is prepared.

7 COOMET Project 649/RU/14 «Pilot comparison "Study in the field of measuring of formed elements of blood: erythrocytes (RBC), leukocytes (WBC)» Participants: VNIIM, CSM ME (Kyrgyzstan). The Draft report is sent to CSM ME for agreement.

8 COOMET Project 654/RU/14 «Pilot comparison in the field of measurements in human serum. Cholesterol in human serum». Participants: VNIIM, CSM ME (Kyrgyzstan), VNIIOFI. The pilot laboratory is preparing comparison samples.

9 COOMET project № 664/RU/14 (COOMET.QM-S3.2015) Supplementary comparison of standard gas mixtures "Atmospheric air pollutants: CO in Nitrogen, 5 µmol/mol" Participants: VNIIM, KazInMetr, BelGIM. The Draft A report is prepared.

10 COOMET project № 678/RU/15 (COOMET.QM-K111) «Key comparison of national measurement standards in the field of analysis of gas mixture of C₃H₈ in nitrogen» Participants: VNIIM, KazInMetr, BelGIM, Ukrmetrteststandart. Comparison samples are sent to the participants.

11 COOMET project № XXX/RU/16 Pilot comparison «Determination of polychlorinated dibenzo-p-dioxins and dibenzofurans in fat matrices (pork fat)» Participants: VNIIM, PTB, BelGIM, Ukrmetrteststandart, CSM ME. The samples are ready for delivery to participants. The protocol is sent for registration in COOMET.

UNIIM is the coordinator of:

1 COOMET Project 645/RU/14 «Pilot comparisons in the field of measuring of mass fraction of copper and impurities in the oxygen-free copper wire rod KMB M001b brand for determination of cop-

per purity» Participants: UNIIM, Ukrmetrteststandart, KazInMetr. Draft A report is sent to the participants for agreement.

2 COOMET Project 672/RU/15 «Pilot comparisons in the field of measuring the mass fraction of iron in iron» Participants: UNIIM, NIM, Ukrmetrteststandart, BelGIM, VNIIFTRI, VNIIOFI. Measurements in progress.

VNIIFTRI is the coordinator of:

1 COOMET Project № 574/RU/12 "Pilot comparison in the field of measurements of count concentration of aerosol particles in the range of sizes from 0,5 µm and higher". Participants: VNIIM, VNIIFTRI and KazInMetr. Report in progress.

2 COOMET Project № 575/RU/12 "Pilot comparison in the field of measurements of count concentration of aerosol nanoparticles in the range of sizes from 20 to 500 nm». Participants: VNIIM, VNIIFTRI and KazInMetr. Preparation to the comparison.

3 COOMET Project 655/RU/15 «Pilot study on pH measurement of phosphate buffer (pH=7.0)».

Participants: VNIIFTRI and KazInMetr, CSM ME. Preparation to the comparison.

3 COOPERATION WITH THE INTERNATIONAL ORGANIZATIONS

CCQM

Representatives of TC 1.8 have participated in CCQM activity practically since the time of its establishment and became the members of the working groups KCWG (Key Comparison working group), QAWG (Organic Analysis working group), GAWG (Gas Analysis working group), IAWG (Inorganic Analysis working group), NAWG (Nucleic Acids working group), EAWG (Electrochemical Analysis working group), SAWG (Surface analysis working group) with the purpose of formation of International System of Uniformity of Measurements.

Reports of International Organizations

APMP

Since 2008 VNIIM (Russia) has become the full member of APMP, KazInMetr (Kazakhstan) is an associated member of this Regional Metrological Organization.

At present VNIIM participates in bilateral comparison with KRISST in the field of measurement of mass concentration of aerosol particles.

EURAMET

VNIIM is an associated member of EURAMET. Cooperation with TC Metchem EURAMET consists mainly of participation in the projects of international comparisons and realization of bilateral agreements with NMIs of EURAMET Member-Countries.

VNIIM participates in one EURAMET project in the field of gas analysis.

SIM

Bilateral comparison of VNIIM with INMETRO in the field of gas analysis was completed in 2015 within the frames of the program of joint researches of ROSSTANDARD and INMETRO.

Ukrmetrteststandart participates in one SIM project in the field of electrochemical analysis.

ISO TC

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

OIML

Representatives of VNIIM and VNIIFTRI are the heads of OIML TC 17 "Physicochemical measurements" and its subcommittees (SC) (SC2 "Saccharimetry", SC3 "pH-metering", SC4 "Conductometry", SC5 "Viscosimetry", SC6 "Gas Analysis", and participate in a development and expertise of the International Recommendations and Documents.

4 ACTIVITIES ON IMPLEMENTATION OF MRA

Organization and realization of works on preparation of the CMC data:

The review of cycle XVI COOMET CMCs was completed.

The results published in the BIPM KCDB are as follows:

7 new and 11 revised positions in the field of gas analysis (1 position is deleted), 3 new positions in the field of inorganic analysis (on the part of VNIIM);

2 new positions in the field of electrochemical analysis (on the part of BelGIM);

1 new position in the field of electrochemical analysis (on the part of KazInMetr).

At present the International CMC Database (BIPM KCDB) contains 534 positions of VNIIM, 17 positions of UNIIM, 5 positions of VNIIFTRI, 24 positions of Ukrmetrteststandart, 17 positions of BelGIM, 1 position of KazInMetr.

Distribution of the COOMET CMCs according to measurement categories is the following:

gas analysis – 452; organic solutions – 24; inorganic solutions – 15; metals – 14; sediments, soils, ores and particles – 9; high purity chemicals – 30; biological fluids and materials – 1; foods – 8; water – 9; electrochemical analysis – 21, advanced materials – 12, other materials – 3.

Arrangements made for the COOMET CMCs review of cycle XVII are as follows:

The intraregional review was completed.

The CMCs were claimed by

VNIIM in the field of gas analysis (33 new), organic analysis (3 new in the category «biological fluids and materials»), electrochemical analysis (1 new) and inorganic analysis (1 revised in the category «biological fluids and materials»);

UNIIM in the field of inorganic analysis (12 new in the category «high purity chemicals»);

VNIIFTRI in the field of electrochemical analysis (1 new and 1 revised);

BelGIM in the field of gas analysis (3 revised);

Ukrmetrteststandart in the field of gas

analysis (2 new and 3 revised) and in the field of electrochemical analysis (1 new);

KazInMetr in the field of in gas analysis (3 new).

Reviewers: BelGIM, VNIIM, UNIIM, Ukrmetrteststandart.

All CMCs were presented for interregional review.

Note: CMC positions are shown as CMC lines.

Participation of COOMET NMIs in international comparisons : COOMET has participated in approximately 35 past and current key and pilot comparisons worldwide, the details of which are available.

5 EVENTS AND MEETINGS

The last meeting of COOMET TC 1.8 "Physical chemistry" was hold in May 21-22, 2015 (St. Petersburg, VNIIM);

The next meeting of TC 1.8 "Physical chemistry" will be hold in May 24-25, 2016 (St. Petersburg, VNIIM).

Meeting of the Coordination Counsel on Traceability in Chemistry was hold in March 23-24, 2016 (St. Petersburg, VNIIM). The meeting was devoted to implementation of Primary Reference measurement procedures and problems of traceability in Clinical Diagnostic Laboratories.

Prof. L.A.Konopelko

The Chairman of COOMET TC 1.8 "Physical chemistry"

Yu.A.Kustikov

The Deputy chairman of COOMET TC 1.8

Coordinator - O.V.Efremova

March 2016

Reports of International Organizations

EURAMET: TC-CHAIR ANNUAL REPORT 2014/2015

*TC for Metrology in Chemistry TC-MC Chair
Michela Segà 18 May 2015*

1. General Aspects

The work of the Technical Committee for Metrology in Chemistry, TC-MC, continues in its well-established form. The TC-MC members are actively participating in the European metrology programmes, i.e. the European Metrology Research Programme (EMRP) and the European Metrology Programme for Innovation and Research (EMPIR).

The eighth meeting of EURAMET TC-MC was held in Malta hosted by MCCA, from 3rd to 6th February 2015. All the

four Sub-Committees (SCs) had separate meetings on the 4th of February and the TC-MC plenary meeting was held on 5th–6th February. About 80 representatives from various European NMIs/DIs participated in these meetings. An additional day on the 3rd of February 2015 was reserved for the periodic meetings of the EMRP project ENV52 and for the Convenors' meeting.

Running EURAMET and EMRP projects were discussed in detail. A workshop in preparation of the upcoming EMPIR calls was held during the TC-MC plenary meeting.

It was the last TC-MC meeting chaired by Michela Segà (INRiM), whose second

mandate of TC-MC chairperson will end in June 2015; she will be replaced by Hanspeter Andres (METAS), who had already been nominated by EURAMET as new TC chairperson.

Pia Tønnes Jakobsen (DFM) was nominated as the new convenor of the Sub-Committee for Electrochemical Analysis (SCEA) in the plenary meeting. She replaces Francesca Durbiano (INRiM), who ended her second mandate of Convenor and had acted as the Convenor of SCEA for four years. Béatrice Lalere (LNE) was reappointed for a second two-year mandate as Convenor of the Organic Analysis SC (SCOA).

2. Projects

Many EURAMET projects are currently active. Most of them are comparisons and mainly carried out in the field of gas analysis. Detailed information on the projects is given in the TCMC project webpage. During the first months of 2015 one comparison was agreed and one further comparison was proposed.

Year	Projects started/proposed	Projects completed
2003	5	4
2004	4	3
2005	5	4
2006	8	3
2007	4	4
2008	4	3
2009	3	3
2010	3	4
2011	9	8
2012	-	1
2013	2	3
2014	7	2
2015	2	-

3. Comparisons

Nine EUROMET.QM Supplementary Comparisons and nine Key Comparisons are registered in the BIPM KCDB:

About the 80% of these comparisons are carried out in the field of gas analysis.

KC/SC	Name	Status
KC	EUROMET.QM-K1.c	completed
KC	EUROMET.QM-K3	completed
KC	EUROMET.QM-K4	completed
KC	EURAMET.QM-K4.1	completed
KC	EURAMET.QM-K12	completed
KC	EUROMET.QM-K17	completed
KC	EURAMET.QM-K26.a	completed
KC	EURAMET.QM-K111	in progress
SC	EUROMET.QM-S1	completed
SC	EUROMET.QM-S2	completed
SC	EUROMET.QM-S3	completed
SC	EURAMET.QM-S4	completed
SC	EURAMET.QM-S5	completed
SC	EURAMET.QM-S6	completed
SC	EURAMET.QM-S7	completed
SC	EURAMET.QM-S8	completed
SC	EURAMET.QM-S9	completed

Reports of International Organizations

4. CMCs

In 2015, a review of new claims as well as a re-review of a range of existing claims was carried out in the regional CMC review process under cycle XVI of the CMC claim period. The files and accompanying documents were received by the relevant TC-MC SC convenors after discussion and evaluation in the SCs. Prior to the SC meetings appointed experts from different European NMIs/DIs pre-reviewed the submitted claims and the evaluation was completed during SC meetings on 4th February 2015. 130 new and 133 revised and re-reviewed claims (24 of which were withdrawn) have been proposed by 24 NMIs and DIs from 19 countries, including the European Union, covering 12 categories. The collated claims from all the sub-committees were sent to the chairperson of the CCQM KCWG on the 20th of February 2015, according to CCQM rules, for the inter-regional review. During the inter-regional review, the claims were divided into Fast Track and Non Fast Track ones and only 1 EURAMET claim was put among the Non Fast Track ones. The Cycle XVI claims are presently undergoing inter-regional review. EURAMET experts also took part in the inter-regional review process of 323 claims submitted by AFRI-METS, APMP, COOMET and SIM.

5. Activities of the Sub-Committees

Meetings of all Sub-Committees were conducted together with the TC-MC plenary meeting in Malta in the week from 3rd to 6th February 2014. A review of new claims as well as the compulsory re-review of a range of existing claims were carried out under Cycle XVI of the CMC claim period and a great part of the SC meetings was devoted to a face-to-face review of the claims of Cycle XVI. Running and new projects in the framework of EURAMET and EMRP and also proposals for the upcoming EMPiR calls on "Metrology for Health", "SI Broader Scope" and "Metrology research for pre- and co-normative projects" were discussed in the sub-committees. Unfortunately, no inputs were given for the "Research Potential" call. An overview of the technical work carried out in the four

SCs is reported.

SC for Electrochemical Analysis (SCEA)

A major issue dealt with the presentation and discussion of possible future cooperation activities to be carried out within the SC. Some of the participants gave presentation concerning the recent developments in their NMIs/DIs. Various members of the SCEA are successfully taking part in EMRP projects and the outline of various JRPs related to electrochemistry was given during a joint meeting of the SCIA and SCEA, held on the 4th February in the afternoon.

SC for Inorganic Analysis (SCIA)

A large part of the technical work in SCIA is at present concentrated on ongoing EMRP projects. During the meeting, two project proposals were presented:

- Determination of transferrin in human serum, as outcome of the EMRP project HLT05 "Metalloomics – Metrology for metalloproteins", coordinated by PTB
- Comparison of analytical capabilities for gold determination in gold jewellery alloy coordinated by IMBiH.

Two members gave technical presentations on the activity in inorganic analysis carried out in their institutes. The outline of various JRPs related to inorganic analysis was given during a joint meeting of the SCIA and SCEA, held on the 4th February in the afternoon.

SC for Organic Analysis (SCOA)

For the SCOA is considered to be of vital importance to be proactive and to arrange EURAMET activities that complement OAWG activities. During the meeting, a project proposal related to a comparison on determination of Aflatoxins in dried figs coordinated by UME was discussed. The outline of the EMRP project ENV 08 "WFD traceability - Traceable measurements for monitoring critical pollutants under the 'European Water Framework Directive' (WFD- 2000/60/EC)" was also given. Some ideas for the upcoming EMPiR calls were discussed.

SC for Gas Analysis (SCGA)

The SCGA is very active in various EURAMET projects, some of them also registered as EURAMET Supplementary or Key Comparisons in the KCDB. Its activities involve also many participants from outside Europe. Eleven active projects were discussed during the meeting.

- EURAMET 708 is an ongoing bilateral comparison between VSL and NIST dealing with different challenging analytes.
- EURAMET 1112 (PTB) is a comparison on ethanol in water saturated air with the purpose of comparing the realisation process of mixtures to be used in the breath ethanol analysis
- EURAMET 1220 (NPL) is a project for the analysis of trace level impurities in hydrogen used in fuel cells.
- EURAMET 1255 deals with coal mine methane and is coordinated by BAM
- EURAMET 1274 is a bilateral comparison between SMU and MKEH on automotive mixtures.
- EURAMET 1280 (PTB) deals with establishing the equivalence of TILSAM and gravimetry-based analytical capabilities.
- EURAMET 1258 "Guidelines for CMC", which concentrates on a document that clarifies existing guidelines for setting up CMCs and to specify for a couple of scenarios what supporting evidence is to be delivered, e.g., extension of ranges, extension of range of components, claims for purity analysis, smaller uncertainties and guidance on CRMs with reference to

In addition, the activities of The Hungarian Accreditation Board (NAT), Hungary, were terminated on 31 December 2015 by decree of the Hungarian Government. As such the signatory status of NAT to the ILAC MRA and NAT's membership of ILAC have therefore been withdrawn. Additional information on the recognition of accreditation in Hungary is available from the [EA website](#).

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existing CCQM GAWG documents.

- EURAMET 1290 (VSL), which is also registered as Key Comparison EURAMET.QMK111, deals with C₃H₈ at 1000 µmol/mol in N₂. This is a core comparison in the gas field and the first organised CCQM KC linked with regional KCs. From each RMO there are only 2 participants (as a maximum) in the CCQM KC and one of those has to organize the regional comparison. For EURAMET VSL and NPL are participating in the CCQM KC and VSL is coordinating both the CCQM and the EURAMET KCs.
- EURAMET 1293 (NPL) on C₆ - C₁₀ hydrocarbons in methane. Previous natural gas comparisons have only covered hydrocarbons up to n-C₆. VSL and NPL are the only participants.
- EURAMET 1305 (NPL), bilateral comparison between NPL and VSL on 5 µmol/mol of oxy-VOCs to underpin global monitoring of key components which alter the oxidising capacity of the atmosphere and therefore is important for the WMO-GAWG.
- EURAMET 1329 (LNE) on SO₂ at 100 nmol/mol in synthetic air.

No new projects were proposed. The SCGA members are actively involved in ongoing EMRP projects.

6. EMRP and EMPIR

The ongoing European Programmes EMRP and EMPIR represent a very important factor in the work and prospect of the TC-MC.

TC-MC has been strongly involved in the EMRP process and participates in about 15 running projects in all the EMRP fields, i.e. "Health", "Energy", "Metrology for environment", "Metrology for industry", "SI broader scope", "New technologies", indicating the cross-disciplinary nature of the TC itself.

The periodic meeting of the JRP ENV52 "HIGHGAS – Metrology for high-impact greenhouse gases" was carried out on 3rd February in Malta prior to the TC-MC meetings. Chemical aspects related to the upcoming EMPIR calls on "Metrology for Health", "SI Broader Scope" and "Metrology research for pre- and co-normative projects" were addressed in a specific workshop organised during the TC-MC plenary meeting. Eleven presentations were given by representatives of various European NMIs/DIs, dealing with the various TPs. The large amount of discussed topics underlines the interdisciplinary features of metrology in chemistry and the deep involvement of metrology in chemistry community in these subjects.

7. Meetings

TC-MC annual meeting 2015: 3rd to 6th February 2015 (Sub-Committees and TC-MC plenary meeting) in Malta.

Highlights:

Reports on:

- News from EURAMET, BoD and EMPIR, Wolfgang Schmid, Head of EURAMET Secretariat
- CCQM/CIPM February 2015 up date, Robert Kaarls, CIPM Secretary and Immediate Past President of CCQM
- Activities of the EURAMET Task Groups "Health", "Environment", Energy, Hans Koch, Convenor of the TG Health, Richard Brown on behalf of Andrea Merlone, Convenor of the TG Environment, Hugo Ent on behalf of Gert Rietvelt Convenor of the TG Energy
- BIPM metrology programmes in support of Health, Food Safety and the Environment, Robert Wielgosz, Head of BIPM Chemistry Department
- Eurachem 25 years – looking back and forward; Eurachem Guide "The fitness of purpose of analytical meth-

ods", Bertil Magnusson, SP (Sweden), Eurachem Past-Chair

- Training in Metrology in Chemistry, Ivo Leito, University of Tartu (Estonia)
- CCQM WG activities, Representatives of the 6 technical CCQM WGs

An ad-hoc working group on TC-MC perspectives on European Metrology Centers was established, chaired by the TC-MC incoming chair Hanspeter Andres. Its members are the TCMC present, past and incoming chairs (Michela Segal, Bernd Guettler, Hanspeter Andres), the SC Convenors (Hugo Ent, Pia Tønnes Jakobsen, Béatrice Lalere, Nilgün Tokman) and the Contact person from UK (Richard Brown).

An issue on how to give representativeness within TC-MC to the bioanalysis activities was raised. The possibility of having a new TC-MC subcommittee on Bioanalysis had already been considered in the past and within the TC-MC it had been decided not to create it as the activities of the European NMIs and DIs were well represented in the CCQM corresponding Working Group (BAWG). Considering the increasing number of European DIs, the spread up of activities in biology and also the change of CCQM name from "Consultative Committee on Amount of Substance", the issue was taken into account again in the 2015 meeting. The decision was not to constitute any new SC waiting for the reorganisation of CCQM structure and to use the SCOA as a forum to discuss also the bio-related issues, thus inviting the chair of the CCQM BAWG, or other representatives, not only at the TC-MC plenary meeting but also to the SCOA meeting.

Workshop: "EMPIR workshop in preparation of the 2015 calls":

- "Health metrology requirements in support of stratified medicine; improved cancer & neurodegenerative disease diagnosis & treatment; and anti-microbial resistance detection",

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Helen Parkes, LGC

- "LNE's proposals for next Health call", Vincent Delatour, LNE
- "A metrological platform for drug/metabolite imaging to support improved targeted therapies & diagnostics", Heidi Goenaga-Infante, LGC
- "Metrology for biomarkers important for the diagnosis of widespread diseases", Claudia Swart, PTB
- "Cells and tissues metrology needs for advanced therapies and diagnostics", Maria Paola Sassi, INRiM
- "Contemporary metrology requirements in diagnosis and therapy", Alex Knight, NPL
- "Establishment of SI traceability of electrolytic conductivity at pure water level from 5°C to 50°C", BSteffen Seitz, PTB – Francesca Durbiano, INRiM

- "Pre-normative research requirements for future European air quality and emissions legislation", Richard Brown, NPL Further information; none 22 May 2015

- "Pre-normative research ideas on nanoparticles and critical chemical substances", Sophie Vaslin-Reimann, LNE

- "qNMR for advanced purity measurement, & other LGC SI/prenorm PRT ideas", John Warren, LGC

- "Measurement of size distribution and associated concentration of nanoparticles in matrix", Rita Cornelis, SMD

8. Issues

- CMCs submission: need to further develop the concept of difference between calibration and testing activities for emerging NMIs and DIs
- Re-review process of existing CMCs exceeding new CMCs

- Involvement of emerging NMIs/DIs in comparisons and in the activities of the technical SCs
- TC-MC contribution in the development of EURAMET Strategic Research Agenda
- Representation of the bioanalysis community in the TC-MC Subcommittees.

9. Strategic planning/ 10. Outlook for 2015/2016

- Discussion about participation in future EMPIR projects
- Activities related to CMC submission and revision
- Perspectives on European Metrology Centers
- Relationship with Eurachem: need to establish common topics of interest

Michaela Segal

TC Chair

ILAC UPDATE TO THE CITAC MEMBERS MEETING

ILAC Meetings

The 2015 ILAC-IAF mid-term meetings were held at the Le Meridien Park Hotel in Frankfurt, Germany during 9-16 April 2015.

The 2015 ILAC-IAF Joint Annual Meetings were hosted by L'Ente Italiano di Accredimento (ACCREDIA) at the Marriott Hotel in Milan, Italy from 28 October - 6 November 2015. The Adopted Resolutions of the 19th ILAC General Assembly in Milan, Italy can be downloaded from the ILAC website at Resolutions of the Nineteenth ILAC General Assembly, 6 November 2015

The 2016 ILAC and IAF mid-term meetings will again be held at the Le Meridien

Park Hotel in Frankfurt, Germany during 30 March – 6 April 2016. The ILAC Accreditation Committee (AIC), Proficiency Testing Working Group (PTWG), Inspection Committee (IC), Laboratory Committee (LC), Joint Working Group A Series, Arrangement Committee (ARC), Arrangement Management Committee (AMC), Joint meeting of the ILAC and IAF Management Committees (JMC), ILAC Executive Committee and Joint meeting of the ILAC and IAF Executives (JEC) will be held as usual during this period. The ILAC Marketing and Communications Committee (MCC) will hold their meeting in South Africa in mid-April 2016. Information about the ILAC committee meetings can be found in the ILAC Events Calendar.

The ILAC MRA

As at 3 March 2016, there are **89 ILAC MRA signatories**, representing **86 economies**. The ILAC MRA covers recognition for accreditation in the areas of calibration (ISO/IEC 17025), testing (ISO/IEC 17025), medical testing (ISO 15189), and inspection (ISO/IEC 17020). The list of signatories to the ILAC MRA is available from the ILAC MRA Signatory Search.

It should be noted that the signatory status of The Kyrgyz Center of Accreditation (KCA), Kyrgyz Republic, to the ILAC MRA for the accreditation of testing laboratories using ISO/IEC 17025 was suspended on 4 November 2015. This suspension is a result of the decision taken at the 15th Meeting of the ILAC Arrangement Council held on 4 November 2015.

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The ILAC MRA acts as an internationally recognised 'stamp of approval' to demonstrate compliance against agreed standards and requirements. Many specifiers, such as government agencies, have recognised the importance of credible accreditation programs that are developed against internationally recognised standards. Accreditation and the ILAC MRA help regulators meet their own legislated responsibilities by providing a globally recognised system to accept accredited test reports. The ILAC MRA is recognised and referenced by a number of regulators. Further details and examples are available from the [Public Sector Assurance web-site](#).

A resolution was adopted at the 2014 ILAC General Assembly (GA) in Vancouver that will allow the implementation of the extension of the ILAC MRA to include accreditation of proficiency testing providers (ISO/IEC 17043), when the appropriate peer evaluation documents within ILAC are updated to cover this new scope.

The *ILAC MRA Annual Report 2014* is available from the ILAC website.

ILAC Membership

ILAC membership as at 3 March 2016 is as follows:

- 89 Full Members (signatories to the ILAC MRA) representing 86 economies;
- 17 Associates representing 22 economies;
- 12 Affiliates representing 12 economies;
- 26 Stakeholders
- 6 Regional Cooperation Bodies

The ILAC membership consists of 150 organisations from 112 different economies worldwide. Over 49,000 laboratories and almost 8,000 inspection bodies are accredited by the ILAC Full Members (signatories to the ILAC MRA).

ILAC Executive

The revised version of *ILAC-R7 Rules for the Use of the ILAC MRA Mark* was published in May 2015 following a successful

30 day ballot and shortly after this the *ILAC-R7-F1 Agreement for the Use of the ILAC MRA Mark* was distributed to eligible ILAC MRA signatories for signing.

The revised version of *ILAC-R4 Use of the ILAC Logo and Tagline* was also published in May 2015 following a successful 30 day ballot and shortly after this *Annex 1 - Declaration Form to Use the ILAC Logo and Tagline* was also distributed to eligible ILAC members for signing.

The ILAC Executive Committee continues to work on the implementation of *ILAC-R3 ILAC Strategic Plan 2015-2020* and the actions detailed in *Supplement 1 to ILAC R3*. The ILAC Strategic Plan is also a standing item for the agenda of each of the ILAC committee meetings.

ILAC-R7, ILAC-R4 and ILAC-R3 can be downloaded from the [Publications and Resources section](#) of the ILAC website.

A revised MoU between ILAC, IAF and IEC was re-signed in October 2015. In addition, the review of the MoU between ILAC and the BIPM has also now been completed and the MoU will be re-signed in March 2016.

Copies of signed MoUs are available from the [Partnerships page](#) of the ILAC website.

ILAC Liaisons

ILAC has been represented at a number of meetings and other liaison activities throughout 2015 and this will continue during 2016.

The ILAC Chair and Vice-Chair both attended the regular round of ISO-CASCO meetings in May and December 2015, including the Chairman's Policy Committee (CPC), the Strategic Alliance and Regulatory Group (STAR) and the Technical Interface Group (TIG). ILAC liaison officers have also participated in the ISO-REMCO, ISO TC69 and ISO TC212 meetings in 2015. ILAC representatives are also actively participating in each of the following ISO-CASCO working groups:

- **ISO CASCO WG42: ISO/IEC 17011 revision**

ILAC Representatives: Ms Jennifer Evans

(NATA) and Dr Andreas Steinhorst (EA).

ILAC Support Committee: Arrangement Committee (ARC).

ILAC has been represented at all of the WG42 meetings to date, including the subcommittee meeting and drafting group meeting.

- **ISO CASCO WG44: ISO/IEC 17025 revision**

CASCO Convenor: Mr Warren Merkel (nominated by ILAC).

ILAC Representatives: Ms Mingxia Zhang (CNAS) and Ms Johanna Acuna Loria (ECA).

ILAC Support Committees: Accreditation Committee (AIC) and Laboratory Committee (LC).

ILAC has been represented at all of the WG44 meetings to date, including the drafting group meeting.

- **ISO CASCO/REMCO JWG 43: transformation of ISO Guide 34 into ISO 17034**

ILAC Representatives: Ms Lorraine Turner (UKAS) and Mr He Ping (CNAS).

ILAC Support Committee: Accreditation Committee (AIC).

ILAC has been represented at all of the JWG43 meetings to date, including the drafting group meeting. The ILAC Representatives were also on the ISO-REMCO ad-hoc advisory group (AHG3) that prepared the ground work for the transformation to an international standard.

In addition to the ISO meetings noted above, ILAC representatives have participated in numerous meetings and events of other international organisations where ILAC maintains a liaison.

The first such activity in 2015 was the annual bipartite working group meeting between ILAC and the International Bureau of Weights and Measures (BIPM), together with the quadripartite meeting between ILAC, BIPM, OIML and ISO. These meetings took place on 4 and 5 March. The ILAC delegation to the meeting with BIPM consisted of 4 people and

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ILAC was represented by two delegates at the quadripartite meeting. In 2016, the ILAC delegation to each of these meetings will consist of three representatives.

ILAC was also represented at two extra events at the BIPM in 2015, presenting at the Workshop on Measurement Uncertainty and participating in the Workshop on Global to Urban Scale Carbon Measurements in June. ILAC liaison officers also participated in the CIPM MRA Review Preparatory Workshop in October.

In March 2015, the ILAC Chair presented on behalf of ILAC and IAF at the United Nations Industrial Development Organisation (UNIDO) Workshop on Trade Capacity Building (TCB) in Maputo, Mozambique and participated in the annual meeting of the Network on Metrology, Accreditation and Standardization for Developing Countries (DCMAS Network). The ILAC Chair also presented at the CROLAB International Conference of Laboratory Competence in October.

The ILAC Vice-Chair presented on behalf of ILAC and IAF at the World Trade Organisation (WTO) Advanced Technical Barriers to Trade (TBT) Course in Geneva during March 2015 and the Vice-Chair also represented ILAC at the Organisation for Economic Co-operation and Development (OECD) meeting on "Fostering the contribution of international organisations to better rules of globalisation" on 17 April 2015.

ILAC and IAF again hosted a working session on "Accreditation: Making Trade Work" at the World Trade Organisation (WTO) Public Forum on 1 October 2015 in Geneva, Switzerland. ILAC also participated in the WTO TBT @20 meeting on 4 November 2015. In addition, ILAC presented at the IEC-ISO-ITU World Standards Cooperation (WSC) Conformity Assessment Workshop and participated in the United Nations Economic Commission for Europe (UNECE) Working Party on Regulatory Cooperation and Standardisation Policies (WP. 6) Annual Session in December 2015.

ILAC was represented at the 50th Meeting of the International Committee of

Legal Metrology (CIML) and the 60th Anniversary of the International Organisation of Legal Metrology (OIML) in October 2015, the Joint Committee for Guides in Metrology (JCGM) WG₁ (GUM), WG₂ (VIM) and Plenary meetings in June, October and December, the IEC-ILAC-IAF Steering Committee meeting in November and the Joint Committee for Traceability in Laboratory Medicine (JCTLM) Executive and Stakeholder meetings in December 2015.

The Liaison information page, located in the member's area of the ILAC website, continues to serve as the main repository for the reports and documents produced as part of ILAC's liaison activities. Members can also find copies of all ILAC comments submitted during ISO/CASCO ballots on international standards pertinent to the work of ILAC. The *ILAC Liaison Procedure*, which provides all the pertinent information relating to ILAC's liaison activities, can also be downloaded from this page.

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

ILAC Secretariat

The ILAC Secretariat is currently staffed as follows: Annette Dever, Sharon Kelly, Rana Baleh, Rose De Rota and Joëlle Nicolas.

World Accreditation Day will be celebrated on 9 June 2016 and the theme this year is "Accreditation: A Global Tool to Support Public Policy".

ILAC publications can be downloaded from the ILAC Website. Documents and brochures published since February 2015 are as follows:

ILAC P4:02/2016 *ILAC Mutual Recognition Arrangement: Policy and Management*

ILAC P5:02/2016 *ILAC Mutual Recognition Arrangement: Scope and Obligations*

ILAC G7:02/2016 *Accreditation Requirements and Operating Criteria for Horseracing Laboratories*

ILAC B1:10/2015 *Why use an Accredited Laboratory?*

ILAC B3:10/2015 *How does using an Accredited Laboratory benefit Government and Regulators?*

ILAC B7:10/2015 *The ILAC Mutual Recognition Arrangement*

ILAC B13:09/2015 *Why become an Accredited Reference Materials Producer*

ILAC R4:05/2015 *Use of the ILAC Logo and Tagline*

ILAC R7:05/2015 *Rules for the Use of the ILAC MRA Mark and ILAC R7-F1 Agreement for the use of the ILAC MRA Mark*

IAF/ILAC B7:2/2015 *Accreditation: Supporting the Delivery of Health and Social Care*

It should be noted that many of the ILAC documents and brochures have been translated into a range of different languages.

ILAC and the International Federation of Clinical Chemistry and Laboratory Medicine (IFCC) have also recently produced a presentation, with commentary, on "Medical laboratory accreditation: what is it and why is it important?" The presentation is freely available from the [IFCC e-Academy](#).

Follow [@ILAC Official](#) on Twitter to receive the latest ILAC news, including information on meetings, events, liaison activities and new publications.

[Subscribe to the ILAC Newsletter](#) to receive updates from ILAC members and liaisons. The latest edition of the newsletter can be accessed via the following link: [ILAC News 48, October 2015](#).

Further information can be obtained from the [ILAC website](#) or by emailing the [ILAC Secretariat](#).

Annette Dever

ILAC Secretary

3 March 2016

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REPORT FROM ISO/REMCO TO CITAC

April 2016

The 38th Meeting of the Reference Material Committee of ISO, ISO/REMCO was held in Pretoria, South Africa from 9– 2 June 2015. It was hosted by the South African Bureau of Standards (SABS). The progress achieved during the meeting and in the following months can be briefly summarized as:

Conversion of ISO Guide 34 into ISO 17034:

Within ISO a joint Working Group between ISO/REMCO and ISO/CASCO has been established for the conversion of ISO Guide 34 into an ISO standard 17034. It was expected that this conversion would follow a similar route to that of the ISO Guide 43 for Proficiency Testing to ISO standard 17043. However, this transformation has been complicated by a required updating of the general structure for all the '17000 series' standards under the control of ISO/CASCO, to be implemented here, and in the updating of ISO/IEC 17025, currently also under revision.

The second meeting of JWG 43 in July 2015 discussed the comments on the Committee Draft version of ISO/CD 17034 and decided on the way forward to reach the DIS (draft international standard) stage. The corresponding ISO ballot took place from October to December 2015. The DIS has been approved and received many comments. A drafting group has reviewed them and the proposed changes and responses to the comments will be presented and discussed with the JWG 43 in April 2016. Provided that JWG 43 agrees, the document can move forward to FDIS balloting in the summer of 2016.

ISO Guide 31

The third edition of ISO Guide 31 has been published in November 2015. It advises on the contents of certificates, labels and accompanying documentation for reference materials. The guide contains requirements not only for reference material certificates, but also for product information sheets (i.e., information sent with non-certified reference materials). Natu-

rally, requirements for certificates are more comprehensive. Mandatory information of RM certificates are besides the certified values and their uncertainty, intended use, minimum sample intake, validity period of the certificate, storage information, instruction for handling and some administrative information. RM certificates require a clear specification of the measurand and the reference to which the values are traceable to, and where the value assignment is method specific, the methodology used.

ISO Guide 35

Given the extensive use of ISO Guide 34:2009 by accreditation bodies worldwide, the guidance on homogeneity and stability assessments of RMs and their characterisation (ISO Guide 35), as a document supporting also the accreditation of Reference Material Producers, is currently extensively being revised. As part of this revision process the guide has been renamed "Reference Materials—Guidance for the characterization and the assessment of the homogeneity and stability of the material". The DGuide version is in the ISO ballot process until 01 April 2016, and the outcome will be discussed at the next ISO/REMCO meeting in June 2016. **ISO TR 79**

ISO TR 79 was published on 15 April 2015, and summarizes the state of the art of the production and certification or characterisation of qualitative properties of reference materials. The need for guidance documents for the production of RMs certified for qualitative properties was recognized by many experts. At the same time, the available information was found to be too immature to develop an internationally accepted guidance document. Additionally, the lack of an international vocabulary for terms and definitions for qualitative properties made it more difficult for the experts from various testing areas to communicate with each other. For instance, the investigation of such properties is referred to differently in various specialized areas (examination, classification, identification, testing, observation, etc.).

Therefore, ISO/TR 79 summarizes the available expertise. It aims to contribute to the on-going discussion on qualitative (nominal) properties and the production of such RMs. It tries to foster the future development of an internationally harmonized guidance document. As an on-going project, the corresponding ISO/REMCO Working Group 13 will collect further information on qualitative RMs and will report on advancements in harmonized approaches and contacts made with experts in the field at the next meeting. WG13 will also to continue to interact with JCGM/WG2.

New study items

In order to study how requests for guidance in specific RM areas could be addressed, ISO/REMCO has created two new Ad-Hoc Groups, tasked with investigating and reporting on:

- "Chemical purity CRMs" (AHG 4);
- "Development of new approaches to the establishment and monitoring of stability and homogeneity" (AHG 5).

AHG 4 will review current best practice for the production of high purity chemical CRMs (without duplicating the work of the IUPAC project on methods for the SI value assignment of the purity of organic compounds). AHG5 will also analyse current practices as the provisions in ISO Guide 35 can only offer examples of the many protocols available. Both AHGs have been requested to prepare reports on their reviews of corresponding existing documents, consultations with relevant committees and bodies, inside and outside ISO, as well as on proposals for the way forward, for discussion during the ISO/REMCO meeting.

Next Meeting

The 39th Meeting of ISO/REMCO will be held in Ekaterinburg, Russia, on 14-17 June 2016.

Hendrik Emons

on behalf of ISO/REMCO

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ILAC LABORATORY COMMITTEE REPORT

Frankfurt, April 1st and 2nd 2016

The greater part of the meeting was devoted to the revision of 17025. Jeff Gust NCLSI is co-ordinating the Laboratory Committee response and the Accreditation Committee is the committee charged with coordinating the ILAC input.

The Chair of Wr Gr 44 is Warren Merkel from NIST's AB, the vice-Chairs are Steve Sidney (LC Chair) and a representative from IEC. The Wr. Gr. Has 73 members, 18 liaisons and 8 observers.

Committee Draft (CD) 1 was voted on last year and received an 86% positive vote but as 2606 comments were received it was deemed necessary to proceed to CD 2. The comment/voting period for CD 2 is March 24 to May 23rd. If LC members wish to comment through ILAC the deadline for submission to Jeff (jeff.gust@fluke.com) is April 24th as these have to go to the joint ILAC working group and then to the ILAC secretariat for collation, etc. Alternatively, individuals are free to send their comments to their National Standardisation Organisation

(check their deadline for receipt of comments). Note most Standards organisations have Mirror Groups for examining such documents.

There has been a lot of discussion as to whether the Standard should apply to sampling as a standalone entity. There was no consensus within ILAC or within the ISO/CASCO working group.

The visual appearance of the Standard will be quite different from the 2005 version as it must conform with the common format for all ISO/CASCO Standards. Its contents page will look like:

- 1 Scope
- 2 Normative references
- 3 Terms and definitions
- 4 General requirements
- 5 Structural requirements
- 6 Resource requirements
- 7 Process requirements
- 8 Management requirements
- Annex A (informative)

- Metrological traceability
- Annex B (informative)
- Management system
- Bibliography

There are several additions/modifications to the Draft including "Terms and Definitions", one of which is the inclusion of "Decision Rules".

The next meeting of the ISO working is scheduled for Sept when it is hoped to have a DIS prepared for circulation.

The remainder of the meeting was devoted to actions from previous meetings, finances and reports from member organisations.

2016 is an election year in ILAC (every 2 years) and nominations will be requested for the LC and other Committee Chairs about 2 months before the next meeting scheduled for the end of October in India.

Máire C. Walsh

11th April 2016

Meeting Reports

THE 14TH INTERNATIONAL SYMPOSIUM ON BIOLOGICAL AND ENVIRONMENTAL REFERENCE MATERIALS (BERM 14)

October 11-15, 2015

The 14th International Symposium on Biological and Environmental Reference Materials (BERM 14) was held at the Gaylord Conference Center at National Harbor, Maryland (just outside Washington DC) on October 11-15, 2015. For more than 30 years, the BERM symposia have been the premier meeting for discussions on needs for and production and use of reference materials. The symposium series originated in 1983 as a one-day session with 25 participants at the 10th annual meeting of the Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) in Philadelphia, PA, USA. Since then the BERM symposia typically have alternated between the United States and Europe every 2-3 years (with the exception of a trip to Japan in 2007); however, BERM 14 was back in the U.S. after an absence of nearly a decade.

BERM 14 was unique in the BERM symposia series in that the host was Sigma-Aldrich, a commercial producer and distributor of reference materials. Stephen A. Wise of the National Institute of Standards and Technology (NIST) served as the chair of the symposium; the local Organizing Committee consisted of Stephen Wise (Chair), Hendrik Emons (Institute for Reference Materials and Measurements, IRMM), and a group of Sigma-Aldrich staff led by Alan Nichols and Peter Jenks. The technical program was organized with significant input from the international Scientific Committee composed of representatives from national metrology institutes (NMI), federal government agencies, and the commercial sector. The members of the Scientific Committee, representing 10 countries, were: Stephen Wise (Chair), Hendrik Emons (IRMM, Belgium), Joseph Betz (National Institutes of Health, Office of Dietary Supplements, NIH-ODS, USA), Angelique

Botha (National Metrology Institute of South Africa, NMISA) also representing ISO-REMCO, Derek Craston (LGC, UK), Peter Jenks (Sigma-Aldrich, UK), Stephen Long (NIST, USA), Lindsey Mackay (National Measurement Institute of Australia, NMIA), Zoltan Mester (National Research Council Canada), Alan Nichols (Sigma-Aldrich, USA), Ulrich Panne (Federal Institute for Materials Research and Testing, BAM, Germany), Eric Reiner (Ontario Ministry of Environment, Canada), Kate Rimmer (NIST, USA) also representing AOAC Technical Division on Reference Materials (TDRM), Sang-Ryoul Park (Korea Research Institute of Standards and Science, KRISS), and Takeshi Saito (National Metrology Institute of Japan, NMIJ).

The technical program consisted of 8 plenary and 13 keynote lectures, which opened each morning and afternoon session and were intended to present critical overviews of reference material needs, development, and use in various sectors including environmental, clinical, food, dietary supplements, biosimilars/pharmaceuticals, elemental speciation/isotopics, and accreditation/proficiency testing. There were 14 topical sessions, each consisting of 5 to 7 invited and contributed presentations, and two sessions ran concurrently. A total of 100 oral presentations were made during the 4-day symposium. In addition, there were 110 poster presentations that were discussed during two poster sessions on Tuesday and Wednesday afternoons.

The symposium opened with a plenary lecture by Hendrik Emons (IRMM) titled "Demands on Reference Materials from European Legislation". Dr. Emons discussed the increasing emphasis on "measurement results for decisions" in three areas (safety of citizens, marketing

of products, and quality of life) and the need for reference materials to support current and upcoming regulatory demands and policies in these areas.

Plenary and keynote lectures related to environmental measurements were provided by Eric Reiner, Ontario Ministry of the Environment and Climate Change, and by Don Patterson, President of EnviroSolutions Consulting, Inc. (retired from Centers for Disease Control and Prevention, CDC). Dr. Reiner discussed the analytical challenges associated with providing accurate measurements for persistent organic pollutants and the important role that CRMs play in validating analytical methods and assessing and monitoring the quality and performance of laboratories. Dr. Patterson addressed CRMs in the environmental analysis arena from a different perspective in "Applications of Environmental and Biological Reference Materials: Forensics; Litigation and Mediation; National Human Level Surveys; and Human Exposure Studies." He discussed the important role of CRMs in measurements associated with human exposure studies and environmental forensics, particularly with regards to their use in litigation and dispute resolution between commercial laboratories.

An area of considerable interest at BERM 14 with four plenary/keynote lectures was reference material needs and use in the food and dietary supplement communities. Darryl Sullivan, Director of Scientific and Regulatory Affairs at Covance Laboratories, discussed "Critical Needs for CRMs in the Contract Analytical Laboratory" emphasizing the important role of CRMs in providing defensible measurement results that are required in the contract laboratory environment. Melissa Phillips described the broad scope of food

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-matrix CRMs developed at NIST to address nutrition labeling regulations in the U.S. Representing the U.S. Food and Drug Administration (FDA), Center for Food Safety and Applied Nutrition, which has regulatory authority over most U.S. foods, dietary supplements, and cosmetics, Steven Musser addressed the use of reference materials to ensure that FDA has accurate and reliable data, which is essential for making regulatory decisions, developing policy, and taking enforcement actions. Dan Fabricant, CEO of Natural Products Association, in a presentation titled "Regulatory Requirements and Reference Materials: A Tale that Can No Longer Be Ignored" described the current situation in the regulation of dietary supplements and the challenges in implementation of Good Manufacturing Procedures (GMPs) within this community.

An entertaining "after lunch" plenary lecture titled "Humorous Health: Medicine, Diet, Dietary Supplements, Lifestyle as Seen Through Cartoons" was given by Mark Blumenthal, Founder and Executive Director of the American Botanical Council. This light-hearted and stimulating presentation consisted completely of cartoons from major American newspapers and magazines, which the author feels can be interpreted as reflections or mirrors of society and support his hypothesis that 'seriousness is one of the most prevalent diseases on planet Earth' and that scientific symposia should include more humor in their programs. The cartoons depicted medical practice, the safety (or toxicity) and ubiquity of pharmaceutical drugs, various aspects of diet, dietary supplements, exercise, alternative medical modalities, drug (i.e., coffee) addiction, and environmental problems.

The 14 technical sessions each addressed a topic within the scope of

BERM including: reference material production; CRMs for clinical, environmental, food, and/or dietary supplement analyses; commutability of clinical CRMs; accreditation and proficiency testing; biosimilars and pharmaceuticals; CRMs for isotopics and speciation analyses; and reference materials and microbiology. The Technical Division on Reference Materials (TDRM) of AOAC organized a session with invited speakers titled "Confidence in Identification for Preparation of Biological Reference Materials", with presentations discussing approaches for authentication of botanical materials using chemical and DNA fingerprints.

The scope of BERM 14, and the diversity of participants, was expanded with several notable sessions on purity assessment and quantitative NMR, accreditation and proficiency testing, and microbiology. The "Purity Assessment and qNMR" session was kicked off by a Keynote lecture by Takeshi Saito describing the role of qNMR at NMIJ in disseminating high purity organic CRMs. The session presentations addressed the significant impact qNMR is having within the NMIs (presentations from LGC, NIST, National Institute of Metrology China) and the commercial sector (presentation by Sigma-Aldrich and Spectral Service). The session on Reference Materials and Microbiology was preceded by a Plenary lecture by Ray Cypess, ATCC titled "Closing the Reproducibility Gap with Standards and Best Practices". Presentations in the session highlighted efforts that are currently underway to develop reference materials for microbiology at NMIs (NIM China, NIST, and IRMM) and the commercial sector (BTF bioMerieux Industry). A session on "Accreditation, Proficiency Testing and the Use of Reference Materials" attracted a previously underrepresented community of participants. Representatives of several accreditation groups (ANAB, A2LA, Neptune and

Company) made presentations with titles such as: Challenging Priorities and Issues with Reference Material Producers (RMP) Accreditations, The Benefits of RMP Accreditation, Should RMP be Accredited?, The Need for Statistical Expertise in Accreditation, and What to Look for on Reference Material Certificates.

The closing plenary/keynote session included three stimulating lectures addressing aerosols, nanoparticles, and future RM needs. "Aerosols?! - Reference Material Needs Challenges" was the topic of the lecture by Ulrich Panne (President of BAM, Germany). Dr. Panne addressed the basic measurement challenges and needs in characterization of aerosols of inorganic, organic, and biological origin with respect to applications in environmental chemistry, occupational hygiene, and safety and security issues. A stated conclusion was that aerosol reference materials are needed to define size, chemical composition, and optical and physical values. The keynote lecture by Ruud Peters of RIKILT, the Dutch Food Safety Institute, provided the only presentation addressing reference materials in the emerging area of nanotechnology. His lecture titled "Nanotechnology Applications and Analytical Challenges" focused on "Nanofood" and the analytical methods and challenges in detection of nanoparticles in food matrices (e.g., TiO₂ in food and silver nanoparticles in chicken) and the measurements needed to support the EU nanomaterial regulations. Finally, Derek Craston (The Government Chemist in UK) provided an exciting look into the future with "(C)RMs – A Future Perspective". In this presentation, he highlighted the important role of measurement quality assurance in underpinning regulation and international trade with emphasis on emerging markets citing examples in cell therapy, stratified medicine, nanomaterials, imaging and disease diagnosis.

Meeting Reports

BERM 14 was attended by 290 participants from 28 countries, which represents the largest number of participants and geographical diversity of any previous BERM symposia. Another successful aspect of BERM 14 was the large number of partners (10) and exhibitors (23) that contributed not only in financial support but in the content of the technical program. The exhibitors' tables and booths were set up directly outside the main ballroom, overlooking the National Harbor atrium with a beautiful view out toward the Potomac River. The coffee breaks each day were convened in the Exhibitor Area to facilitate interactions between the participants and the exhibitors. The participation of the commercial reference material producers and accreditation organizations, as well as the general commercial analytical chemistry industry, as partners and exhibitors, broadened the scope and diversity of the technical program

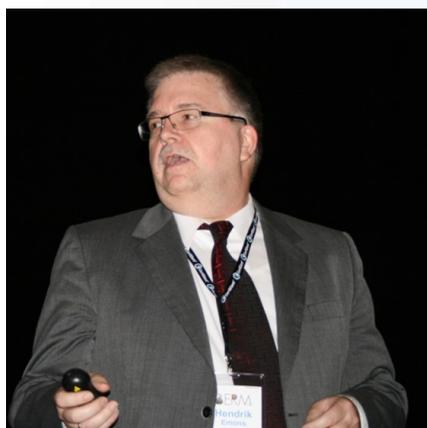
and contributed significantly to the overall success of the symposium. The complete technical program for BERM 14 is still available on the BERM 14 website, sigma-aldrich.com/berm.

Two Best Poster Awards, sponsored by the journal *Analytical and Bioanalytical Chemistry*, were awarded: First Place – Jonathan Grandy, University of Waterloo (Canada) for "Development and Validation of a Highly Reusable, In-vial Standard Gas Generating System", and Second Place – Nick Hauser, RTC/Sigma-Aldrich, for "An Alternative USP Method for the Analysis of Impurities in a Certified Reference Material (CRM) of Riboflavin (Vitamin B₂) Using LC-MS-MS". Benilda The poster "Development of a Reference Material for Histamine in Canned Tuna" by Ebarvia, Industrial Technology Institute (Philippines), was recognized as "Honorable Mention".

At the closing session, the announcement was made that the next BERM symposium, BERM 15, will be held in September 2018 at the Berlin Adlershof campus of the Federal Institute for Materials Research and Testing (BAM) with Ulrich Panne, President of BAM, as the chair of the symposium.

Stephen A. Wise

Symposium Chair, BERM 14



Hendrik Emons (IRMM) delivering opening plenary lecture.



Alan Nichols (Sigma-Aldrich), Stephen Wise (NIST), and Derek Craston (LGC).

Published Guides

IUPAC/CITAC GUIDE: CLASSIFICATION, MODELLING AND QUANTIFICATION OF HUMAN ERRORS IN A CHEMICAL ANALYTICAL LABORATORY

Pure Appl. Chem. Vol. 88 (2016), No. 5, pp. 477-515

Abstract

The classification, modeling, and quantification of human errors in routine chemical analysis are described. Classifications include commission errors (mistakes and violations) and omission errors (lapses and slips) in different scenarios at different steps of the chemical analysis. A Swiss cheese model is used to characterize error interaction with a laboratory quality system. The quantification of human errors in chemical analysis, based on expert

judgments, i.e. on the expert(s) knowledge and experience, is applied. A Monte Carlo simulation of the expert judgments was used to determine the distributions of the error quantification scores (scores of likelihood and severity, and scores of effectiveness of a laboratory quality system against the errors). Residual risk of human error after the error reduction by the laboratory quality system and consequences of this risk for quality and measurement uncertainty of chemi-

cal analytical results are discussed. Examples are provided using expert judgments on human errors in pH measurement of groundwater, multiresidue analysis of pesticides in fruits and vegetables, and elemental analysis of geological samples by inductively coupled plasma mass spectrometry.

Ilya Kuselman

Francesca Pennecchi

GUIDE TO QUALITY IN ANALYTICAL CHEMISTRY—AN AID TO ACCREDITATION, 3RD EDITION (2016)

Eurachem/CITAC Guide

This edition is a revision of the CITAC/Eurachem Guide published in 2002. The new version reflects changes that were introduced with the publication of the 2005 version of ISO/IEC 17025. The terminology has also been updated to take account of ISO/IEC 17000:2004, ISO 9000:2015 and the 3rd edition of the Inter-

national Vocabulary of Metrology – Basic and general concepts and associated terms (JCGM 200:2012 – VIM). The Guide focuses on the requirements of ISO/IEC 17025, but also be of use to organisations seeking accreditation or certification against the requirements of standards such as ISO 15189 or ISO 9001, or compli-

ance with the Principles of Good Laboratory Practice. The aim of this guide is to provide laboratories with guidance on best practice for the analytical operations they carry out. The guidance covers both qualitative and quantitative analysis carried out on a routine or non-routine basis.

Vicki Barwick

SETTING AND USING TARGET UNCERTAINTY IN CHEMICAL MEASUREMENT, 1ST EDITION (2015)

Eurachem/CITAC Guide

This Eurachem/CITAC guide discusses how to set a maximum admissible uncertainty, defined in the third edition of the International Vocabulary of Metrology as the “target uncertainty”, to check whether measurement quality quantified by the measurement uncertainty is fit for the

intended purpose. This guideline is applicable to analytical fields where the target uncertainty is not set by the regulator or the client, and discusses how to set the target uncertainty for process development and for applied or fundamental research. This guideline can also be useful

for authorities and stakeholders that feel the need to define or upgrade criteria for measurements quality.

Ricardo Bettencourt da Silva

Alex Williams

CITAC Most Important Papers in MiC 2016

ACHIEVING COMPARABILITY WITH IFCC REFERENCE METHOD FOR THE MEASUREMENT OF HAEMOGLOBIN A_{1c} BY USE OF AN IMPROVED ISOTOPE-DILUTION MASS SPECTROMETRY METHOD

Hong Liu, Lingkai Wong, Sharon Yong, Qinde Liu, Tong Kooi Lee, *Anal Bioanal Chem.*, 407 (2015), 7579-7587. DOI 10.1007/s00216-015-8961-2

Abstract

Hemoglobin A_{1c} or glycated hemoglobin (HbA_{1c}) is an important biomarker to assess glycemic control in diabetic patients, and is also often used as a diagnostic test for diabetes mellitus. The value is calculated from the ratio of HbA_{1c} and sum of HbA_{1c} and hemoglobin A₀ (HbA₀).

The development of reference measurement methods for HbA_{1c} is important for quality assurance in diabetes management. The IFCC reference method using purified proteins as calibration standards is the recommended accuracy-based reference method for the standardization of HbA_{1c} measurement. Our laboratory developed a liquid chromatography-isotope dilution tandem mass spectrometry (LC-IDMS/MS) procedure, which can serve as

an alternative accuracy-based method for HbA_{1c} measurement. The method involved two parts. Firstly, enzymatic proteolysis was carried out in sample preparation, followed by LC-IDMS/MS measurement of HbA₀ and HbA_{1c}, using two 'signature' hexapeptides for calibration. Secondly, the concentrations of the 'signature' hexapeptide calibration solutions were determined using a hydrolysis method with HCl, followed by LC-IDMS/MS measurement using amino acid solutions as calibration standards. These solutions were gravimetrically prepared from pure amino acid certified reference materials.

We participated in two IFCC ring trials for reference laboratories (RELA 2013 and 2014) for HbA_{1c}, where our LC-IDMS/MS results were compared with those using the IFCC reference method. The devia-

tions between our results and the means of IFCC results were found to be 0.4 to 1.7 mmol/mol, showing good comparability with the IFCC reference method. The relative expanded uncertainty of the LC-IDMS/MS method was in the range of 2.6% to 2.8%. With excellent method precision, good comparability with the IFCC reference method, and a small measurement uncertainty, the developed LC-IDMS/MS method may be used as an alternative accuracy-based reference method for HbA_{1c} measurement.

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Health Science Authority, Singapore

CITAC Most Important Papers in MiC 2016

USE OF ASSIGNED REFERENCE VALUES: REVISITING A SMALL-SCALE INTER-LABORATORY COMPARISON FOR RESIDUAL PESTICIDES IN TEA

Della W.M. Sim, Yiu Chung Wong, *Accred Qual Assur.*, 20 (2015), 495. DOI 10.1007/s00769-015-1157-9

Abstract

This paper compares reference values assigned by isotope dilution mass spectrometry with participants' consensus values in a small-scale proficiency test (PT) on the determination of residues of organochlorine pesticides (α -endosulfan, β -endosulfan and endosulfan sulfate) in a tea sample. The PT was specifically organized to benchmark the technical capability among the local participating laboratories in Hong Kong providing pesticide testing service to their customers and to identify possible areas for further improvement. Participant's performance, based on z-score, was evaluated using consensus values and standard deviation that was esti-

mated from the Horwitz function. Two out of the seven participating laboratories were identified to produce either questionable or unsatisfactory results (in terms of z-scores), showing that the performance of the majority was satisfactory. However, the number of questionable or unsatisfactory results increased to three if the consensus values were replaced by assigned reference values derived from gas spectrometry-isotope dilution mass spectrometry with higher metrological traceability than those used in obtaining the consensus values. Though the said PT program was fit for its intended purposes at that point of time, revisiting the program explicitly demonstrates the significant importance of assigned reference values in

achieving reliable assessment in reality. Unless the bias of consensus values is known with metrological traceability, PT scheme providers should be cautious in assessing the competence of participating laboratories using consensus values particularly in small-scale inter-comparison programs. The importance of pre-wetting dry tea samples to improve recovery of pesticide residues is also noted.

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VALIDATION OF ISO 6974 FOR THE MEASUREMENT OF THE COMPOSITION OF HYDROGEN-ENRICHED NATURAL GAS

Adriaan van der Veen, Paul R. Ziel, Jianrong Li, *Int J Hydrogen Energy.*, 40:46 (2015), 15877-15884.

DOI 10.1016/j.ijhydene.2015.10.005

Abstract

Hydrogen gas is considered as one of the means to store electrical energy generated from wind or solar sources. The produced hydrogen gas could be injected into natural gas grids and thus utilised. Currently, most fiscal metering systems for natural gas are not configured for measuring the hydrogen content, notwithstanding that hydrogen is found in some natural gases in low fractions. Neither do the current documentary standards ISO 6974 and ISO 6975 cover hydrogen levels above 0.5 cmol mol⁻¹. To support fiscal metering, measurement standards and validated

methods are needed to facilitate accurate composition and energy content measurement. The aim of this work was to develop measurement standards for hydrogen-enriched natural gas with uncertainties for the amount-of-substance fraction hydrogen similar to the analysis of methane and nitrogen in natural gas, as well as to validate ISO 6974 for use in this power-to-gas application. Measurement standards have been developed with state-of-the-art uncertainty for the composition for hydrogen fractions between 3 cmol mol⁻¹ and 16 cmol mol⁻¹. A natural gas analyser configured according to ISO 6974-3 was used to confirm the compo-

sition of the measurement standards. We conclude that the scope of the current ISO 6974 can be extended to cover natural gas compositions with hydrogen amount-of-substance fractions of up to 20%. The best measurement capability obtained is 0.12% (k_{1/2}), expressed as relative expanded uncertainty.

Adriaan M. H. van der Veen (avdveen@vsl.nl)

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VSL, The Netherlands

Messages from New Members



I am honoured to be elected as a member of CITAC in 2014.

I was first introduced to the subject of metrology in chemistry in 2008 when my institute, Health Sciences Authority (HSA), became a Designated Institute for Metrology in Chemistry in Singapore. I was tasked to establish a new laboratory to develop the capabilities and provide the relevant metrological services to support the local chemical testing industries. This was both a challenging and exciting change for me as I was involved in forensic drug analysis in the last 28 years of my career.

LEE TONG KOOI

During our initial years of developing the capabilities, we received a lot of help and support from members of the metrology in chemistry community; many of them were also long time members of CITAC.

We were able to send our young scientists to some of the established NMIs for training and attachments. I would like to take this opportunity to acknowledge the help and support from Dr Robert Kaarls, Dr Willie May, Prof Yu Yadong, Dr So Hun Young and the late Dr Laurie Besley. Both Prof Yu and Dr So had retired and were no longer with CITAC. It was Dr Kaarls who first invited us to the CCQM Plenary Meeting and the Working Group meetings as a guest of the President. He later led a team to conduct a peer review of our laboratory in 2012 and 2015, and made many suggestions to improve the quality and work processes of our laboratory. We were also grateful to Dr May for

helping us in many ways to develop and strengthen our measurement capabilities on clinical biomarkers. This has now become one of our focus areas.

Today, our team of young scientists have become competent chemical metrologists. We are actively involved in the activities of CCQM, APMP and JCTLM, and are now providing regular metrological services to our local testing industries. For me, it is the opportunity to get to know and interact with so many dedicated and passionate individuals from the metrology in chemistry community that make my career switch nine years ago so rewarding and fulfilling.

Lee Tong Kooi
Health Sciences Authority
Singapore



Internationally, the comparability of measurement results is remarkably important both to decision makers and the business community and the matter is of great economic importance. A vast number of important decisions – many of them industrial or environment-related – are based on measurement result. The reliability and comparability of the measurement results can be improved by realistic uncertainty estimates and by ensuring the traceability of analytical results.

The modern trend for governments or industrial operators is to streamline their activities mainly for economic reasons. New 'field measurement technologies' should more often be utilized in the collection of measurement data also in the environmental sector. Instead of carrying out laboratory analysis, you can measure for example pH, conductivity,

turbidity and nutrients in water using field sensors or elements in contaminated soil by portable XRF. You can also attach the sensors or hyperspectral camera to a drone (unmanned aerial or underwater vehicle) to carry out environmental measurements. Unfortunately, the field sensor measurements are often performed without knowledge of measurement uncertainty and without quality control procedures that are widely applied by accredited laboratories.

As working in the Designated Institute for Finnish NMI and as being responsible of CEN and ISO standardization work in environmental sector in Finland, I see that the anticipated pressure for utilization of field sensors in situ or continuously on-line in environmental measurements requires immediate actions in setting up tools for assessment of the quality of data produced by these instruments. The knowledge of uncertainty of

the measurement result is a key factor for comparability of the laboratories and field operators. Quality assurance procedures will help the operators of these instruments to improve the quality of the measurement results.

CITAC is one of the key players in the area of promoting comparability i.e. traceability leading to more reliable decisions. I am glad to be a member of CITAC and looking forward fruitful cooperation.

Teemu Näykki
Principal Metrologist
Finnish Environment Institute (SYKE)

TEEMU NÄYKKI



ROLA BOU KHOZAM

Being a Quality Manager at a research center and being in charge of leading analytical and calibration laboratories to obtain accreditation opened up in my mind that requirement of ISO 17025 would be much more easy for implementation if the technical people working at laboratories site understood the philosophy behind it. Proving the competence of laboratory is mainly related to "Method Validation" requirement, which covers among other characteristic parameters, the uncertainty measurement, considered as the "bottle-neck" of the standard. As Chemistry University Professor, those ideas brought me to introduce

"Quality Assurance course" in several diploma disciplines such as General Analytical Chemistry Science, Pharmacy Science, Medical laboratory Science and Nutrition Science. The most joyful lecture for students is when we discuss about "Metrology" where they start thinking that this is complete science taking part in every single activity in our daily life. In addition, that this science is the basis of whatever measurement results we produce in labs and that "Metrological Traceability" of results is a major component from the result values. I think we have been late to recognize the interest of such topics in underdeveloped countries due to the overall political situation in our region which overwhelmed the governments with the day to day problems, and shift their attention to other issues than dedicat-

ing effort to assuring the accuracy of information / data transmitted to consumers. I have been honoured to be selected as a member of CITAC, the first from the Arabic countries, raising the flag of dissemination of information related to metrology within the region as I am acting also as expert/consultant for the International Atomic Energy Agency in the field of quality management for laboratories. I look forward to meaningful interactions with CITAC members, and providing an active contribution in CITAC activities by acting as a link between the international metrology community and my region.

Rola Bou Khozam
Lebanese Atomic Energy Commission



BERND GÜTTLER

CITAC was founded in 1993. This was at the beginning of a process that changed metrology significantly on a global scale over the last 25 years and this process was, to a large extent, triggered by CITAC. The introduction of chemistry into metrology and, vice versa, metrological aspects into chemistry was probably the most important step in the science of measurements in this time.

The process was accompanied by the evolution of today's Consultative Committee for Chemistry and Biology within

the Meter Convention at the same time. Today, the CCQM is still the youngest but also already one of the largest CCs within the Meter Convention. This may serve as evidence for the relevance of the developments.

CITAC fostered collaborations between organisations involved in metrology in chemistry on a global scale and provided essential guidance for the work that's needed to achieve global comparability in analytical chemistry ever since.

Consequently, the work of CITAC also accompanied my own work since I joined the community of metrologists active in chemistry about 15 years ago when I started to represent PTB in CCQM and today's EURAMET TC-MC. During this time, the work of these organisations diversified into various working groups on all levels and extended, for example, into

biochemical subject fields. The European Union and EURAMET even set up their own metrology research programmes, EMRP and EMPIR, which were largely driven by questions related to chemical measurements. Today, the redefinition of the SI units including the kilogram and the mole are hot topics of the process. I had the honour to serve the communities during this time as chairman of EURAMET TC-MC and chair of the CCQM ad hoc working group on the mole. During all this time CITAC and its work gave essential guidance to our activities in the field.

It is a great honour for me to become a member of CITAC not only for these reasons and it also means joining a group of well-known friends.

Bernd Güttler
PTB, Germany



OSMAN BIN ZAKARIA

Dr. Osman Bin Zakaria is the Director of National Metrology Institute Malaysia (SIRIM). Dr. Zakaria represents Malaysia in various chemical metrology committees and meetings. He is a member of the Consultative Committee for Amount of Substances (CCQM), the Asia Pacific Metrology Programme (APMP), the Technical Committee for ISO/TC/WG

229 for Nanotechnology, ISO/TC 146 for Ambient Air Quality, Chairman of STC 10 (Reference Material Producers), the National Committee for Codex and Food Labelling (JKCK FL), the Technical Working Group (KKT) for Halal Analysis Products under the National Committee of Food Analysis Malaysia (JKAM) and many others.

Conference Announcements

- ◇ The 32nd **CITAC Members' Meeting and Workshop** will be held on **Saturday 29th April 2017** from 08:30 to 13:00 at:

Hotel Novotel, 11-13 Grande rue 92310 SEVRES, Paris, FRANCE



- ◇ 9th **International Workshop on Proficiency Testing** will be held from **9-12 October 2017**, and be organized by the Eurachem Proficiency Testing Working Group in association with EQALM and CITAC. It will be at:

St. Bernardin Resort, Portorož, Slovenia

- ◇ 3rd **INTERNATIONAL Congress RESAG** will be held on **13-15 September 2017** in :

Belo Horizonte, Brazil



- ◇ **Isranalytica 2018** will be held from the **23-24 January, 2018**, at:

David Intercontinental Hotel, Tel-Aviv, Israel

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