CITAC
Co-Operation on International Traceability in Analytical Chemistry

news

FEBRUARY 2003

Foreword by the Chairman

It is my great pleasure to take the opportunity to send this Chairman's message to all who have been involved in CITAC activities. At the last CITAC members meeting held on October 3, 2002 at Curitiba, Brazil, I was very pleased to have fruitful discussions with 22 participating members and observers all over the world. First of all, I thank Dr. Ed de Leer (NMi, the Netherlands), Dr. Laly Samuel (MSL, New Zealand), Dr. Wynand Louw (NML, South Africa) for joining us as new CITAC members. We also invited Mr. Yu Yadong, Director of NRCCRM China, in replacement of Dr. Zhao Min of the same institute. I think the participation of the new members will greatly assist us in promoting CITAC activities, particularly reflecting on their broad experience in chemical metrology and geographical distribution of the membership. We are going to cooperate through CITAC liaison more closely with other relevant international organizations working on metrology, health, environment and accreditation. Presently, CITAC has liaison officers with EURACHEM, BIPM/CCQM, ISO/REMCO, ILAC, EA, APLAC, EUROMET, SIM, APMP, SADCMET, IFCC, IUPAC, AOACI, IAEA and so on.

Achievement of traceability in chemical measurements is still difficult in many cases, particularly when dealing with real-world samples. A draft CITAC/EURACHEM Guide on "Traceability in Chemical Measurement", was prepared by a CITAC/EURACHEM joint working group. It was discussed at the CITAC/EURACHEM Workshop on "Measurement Traceability and Uncertainty in Analytical Chemistry" in June 2002 in Lucerne, Switzerland. In laboratories engaged in the analysis of clinical and biological materials, the importance of measurement traceability is currently well-recognized in order to guarantee comparability and commutability of their analytical results. In June 2002 the Joint Committee on Traceability in Laboratory Medicine (JCTLM) was established at BIPM jointly with IFCC to focus on traceability issues in this difficult but important area. Since analytical chemistry plays a most important role in measurements in clinical and biological laboratories, I think CITAC should actively participate in discussions such as what categories of traceability is being required in each area, i.e., traceability to the SI, to the standard or to the document; how to establish traceability, if required, in the area, and so on. Traceabilty in laboratory medicine is becoming an increasingly important issue for National Metrology Institutes (NMIs), clinical and biological laboratories, accreditation bodies, industries and academia, and therefore I think it is a most important task for CITAC to prepare a Guide on Traceability in Laboratory Medicine in cooperation with other relevant national and international organizations.

CITAC was established in 1993 on the occasion of PittCon in Atlanta, USA. We will celebrate the 10th anniversary of the establishment of CITAC at a CITAC/BERM-9 joint workshop during the 9th International Symposium on Biological and Environmental Reference Materials (BERM-9) in June 2003 in Berlin, Germany. All BERM-9 participants are invited to the workshop, where we will take the opportunity to review the 10 years activities with the past CITAC chairpersons and discuss with the participants about our future activities in order to lead CITAC in the proper direction. CITAC will also host a workshop on "Traceability, Comparability, Reliability and Quality in Chemical Measurements" during the 2nd International Conference on Metrology from 4 - 6 November 2003 in Eilat, Israel, as well as other CITAC events (see page 2). Your participation in future CITAC events, your suggestions and comments on CITAC activities are very welcome.

Dr. Kensaku Okamoto
CITAC Chairman,
NMIJ, Japan

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Contribution of Past — Chairman

In ancient days the most important unit of time was the Olympiad, the span from one celebration of the Olympic games to the next. For CITAC it is the span of three years, from one chairmanship to the next. CITAC chairmen have so far been Bernard King (as founding chair), Alan Squirrel and myself. After three years I have turned over this position in spring of 2002 to Vice-Chair (in the tradition of CITAC, the Chair Elect) Kensaku Okamoto of Japan. At the same time the term of office for the Secretary of CITAC also expired. I take this opportunity to thank Ioannis Papadakis for the splendid and collegial cooperation from 1999 — 2002 and wish him the very best for his personal and professional future. It is also very appropriate to mention the role of Manfred Grasserbauer the former director of IRMM in Geel who made this cooperation possible while Ioannis was member of the staff of IRMM. The position of Secretary of CITAC is now being held by Ilya Kuselman of Israel who is doing a remarkable job serving our membership.

When I took over from Alan Squirrel the time was about right for CITAC to issue the first concise document on traceability in analytical chemistry and I remember the struggle for understanding and subsequently for words to give CITAC a voice. We are now at the point where the common understanding is accompanied by a good dissemination of this very understanding on a worldwide basis. Indeed, the progress of the joint CITAC-Eurachem Working Group on Measurement Uncertainty and Traceability (Chairman: A. Williams) will likely lead to the completion of a more comprehensive guidance document on traceability in analytical chemistry in 2003.

In the meanwhile Maíre Walsh, David Holcombe, Alan Squirrel and Bernard King have completed the update of the first guidance document of CITAC ever, the CITAC-Eurachem Guide to Quality in Analytical Chemistry that has the potential of becoming a de-facto standard for chemistry laboratories all over the world.

All of these accomplishments also demonstrate the place and the position CITAC has occupied over the years: it is at the interface of individual laboratories and national metrology institutes. Not surprisingly, the strongest stakeholders at this interface these days are accreditation bodies who vastly benefit from the scientific background and from the strong technical leadership CITAC provides in the field of chemical measurements. It is against these facts that the growing number and wide distribution of our

membership all over the world is so beneficial.

On handing over the position of Chairman of CITAC I urge all readers to support the new team Kensaku Okamoto — Ilya Kuselman wholeheartedly. It will be for the 2003 Members Meeting in Berlin that CITAC is celebrating its 10th anniversary. For me personally this is the time to pledge, that my own future contributions toward the goals and to the work of CITAC will be nothing less than my intense striving for professional credibility as analytical chemist.

Prof. Wolfhard Wegscheider
Institute for General and Analytical Chemistry, University of Leoben, Austria

Future CITAC Events

In the following table one can find information on the CITAC events in 2003 — 2004 according to the decisions of the 17th CITAC Members Meeting in Curitiba — Parana, Brazil, October 3, 2002 (more details on the meeting are available in pp.5-7 and in the CITAC website: www.citac.cc).

Subject	Location	Date	Contact
Workshop and members meeting in conjunction with: Ninth International Symposium in Biological and Environmental Reference Materials (BERM 9)	Berlin, Germany	15-19 June 2003	NMIJ, Kensaku Okamoto Tel: +81-298 61 4420 Email: kensaku-okamoto@aist.go.jp
Workshop and members meeting in conjunction with: 2nd International Conference on Metrology - Trends and Applications in Calibration and Testing Laboratories	Eilat, Israel	4-6 November 2003	INPL, Ilya Kuselman Tel: +972-2-5661856 Email: ilya_kus@netvision.net.il
Workshop in conjunction with: PittCon 2004	Chicago, USA	March 2004	NETL, Gerst Gibbon Tel: +1-412-386 6092 Email: gerst.gibbon@netl.doe.gov
Workshop and members meeting in conjunction with: Annual Meeting of the Japan Analytical Chemical Society	Tokyo, Japan	September-October 2004	NMIJ, Kensaku Okamoto Tel: +81-298 61 4420 Email: kensaku-okamoto@aist.go.jp

Dr. Ilya Kuselman, CITAC Secretary

Joint CITAC — NCSLI Workshop at Pittcon 2002

As a long standing tradition CITAC is helping to organize symposia and workshops on technical issues in analytical quality assurance the world around. An important continent — by all standards — is the North American one where the good cooperation of CITAC with NIST and A2LA has led to a much larger number of CITAC events than on any other continent on an almost regular basis. For Pittcon 2002 in New Orleans, LA, the forces were joined with the recently founded Chemical Metrology Committee of NCSLI to stage a Joint Workshop on "Traceability and Uncertainty: Key Technical Issues and Laboratory Accreditation" .

NCSLI — National Conference of Standards Laboratories International — was founded in 1961 to support US calibration laboratories, but is now an international volunteer and not-for-profit organization with over 1500 member organizations. The Chemical Metrology Committee is headed by Thomas Ouimet of Kodak who also served as co-chair of the workshop.

The aim of the workshop was to highlight key technical aspects of advanced laboratory operation. There are numerous implications around the new ISO 17025 Standard that cause serious problems in the minds of the laboratory community, the most serious of these concern the requirements regarding traceability and uncertainty of results. The Workshop addressed practical interpretations and solutions for these issues on the basis of the technical guidance that was developed through the efforts of Eurachem and CITAC — Cooperation in International Traceability in Analytical Chemistry (www.measurementuncertainty.org). It focused on the needs of laboratories worldwide by bringing together experts in the respective areas with practitioners from the laboratory community. This format was found very successful in supporting the proliferation of good practices in laboratory operation in earlier CITAC symposia and therefore chosen again for this Workshop.

The workshop started with an introduction of NCSLI and its strategic vision (*"Promote competitiveness of member organizations by improving the quality of products and services through excellence in calibration and testing"*) by R.B. Pettit of Sandia. A. Squirrell of NATA contributed a first glimpse on the revised (now) CITAC-Eurachem Guide on the interpretation of the ISO 17025 requirements for chemical laboratories. T. Ouimet highlighted the important role of in-house standards particularly in a research and production environment, while Reenie Parris of NIST introduced the proficiency testing scheme set up by her organization for the providers of environmental standards on a US-wide basis.

For the institutions providing accreditation services the actual everyday requirements imposed on the laboratories dictate the (breadth and depth of) implementation of ISO 17025. Views on traceability and measurement uncertainty practices from the two sides of the Pacific were ably presented by W. Merkel of A2LA and B. King of NARL. The workshop was concluded by two contributions of an even more technical nature: S.L.R. Ellison of LGC discussed qualitative analysis ("*Identification, Measurement and Decision in Analytical Chemistry*") and the related issues in quality assurance, while W. Wegscheider, Univ. of Leoben, provided a fresh look at an old subject in "*Method Validation Revisited: Its Role in Traceability of Results*" where specific conditions were laid out for the validation protocol in order to provide input to the estimation of measurement uncertainty.

It was the general view that Pittcon again provided an excellent forum for bringing together interested parties from around the world. This is the most important mechanism to avoid a drifting apart of interpretations of a common world-wide standard document. A selection of these presentations is offered on www.citac.cc and it is anticipated that CITAC and the Chemical Metrology Committee of NCSLI will join forces again in the future for the benefit of chemical metrology.

Prof. Wolfhard Wegscheider
Institute for General and Analytical Chemistry, University of Leoben, Austria

EURACHEM/CITAC Workshop on Measurement Traceability and Uncertainty in Analytical Chemistry

Meeting the requirements of ISO/IEC 17025

Traceability and uncertainty are important elements of measurement results and for this reason they are given greater emphasis in ISO/IEC 17025 than in Guide 25. The main objective of this workshop was to share experiences and to develop guidance on how these requirements can be met in a cost-effective manner. Over 120 delegates attended the three-day workshop, which took place in the Culture and Congress Centre in Luzern, Switzerland from June 16-18, 2002.

The first day, an introductory afternoon on measurement uncertainty in chemical measurement, was well attended (about 80 delegates), and used a mixture of presentations and subsequent working groups to illustrate the basic principles.

The first plenary sessions concentrated on the experience gained using the second edition of the EURACHEM/CITAC Guide "Quantifying Uncertainty in Analytical Measurement" with particular emphasis on the use of validation data in uncertainty estimation. After a sound review of the requirements of ISO 17025 from Alan Squirrell (ILAC secretariat), the first session covered the theory and practice of uncertainty estimation in chemical measurement. A well-structured summary of the current Eurachem approach from Dr. Matthias Roesslein (EMPA) was followed by a consideration of the applicability of validation data (Dr. Steve Ellison). An important practical issue is that screening tests (such as ruggedness tests) intended to test the significance of a possible effect are typically insufficient to quantify uncertainty if the effect is significant. It follows that such tests are better used as confirmation of insignificance. The problem of treating uncertainty for purity assessment was considered by Dr Van der Veen (NMi, The Netherlands); this is a common problem in pure reference material certification and is not yet dealt with in internationally agreed guidance. Turning to practical implementation in the laboratory, Dr King described the structured approaches used by the Australian national laboratory to handle uncertainty estimation for their standards work, and Lionel Spack demonstrated that at Nestlé, uncertainty estimation is a valued tool in developing new methods. Individual Working Groups considered such topics as practical examples of uncertainty evaluation, design of validation studies to provide uncertainty data and experience on implementation of ISO/IEC 17025. In addition topics not covered in the current edition of the EURACHEM/CITAC Guide, such as evaluation of uncertainty near detection limits and in qualitative analysis have been discussed, together with current work being carried out to update GUM. One practical action arising from the workshop sessions was an agreement to form a new Eurachem working group especially to deal with uncertainties associated with qualitative analysis (interested parties should contact Dr. Steve Ellison, steve.ellison@lgc.co.uk).

Dr. Matthiaas Roesslein and the Williamses in Luzern

The second session covered the EURACHEM/CITAC draft guide on measurement traceability, giving practical guidance on how to establish the traceability of results obtained using existing methods. Dr Wegscheider and Alex Williams covered the principles of traceability and the philosophy of the new guide. The guide is based on the overall philosophy that, given a calculation, from measurable inputs, valid understated and measurable conditions, traceability of all the inputs (whether in the calculation or conditions of measurement) is sufficient for traceability of the calculated result. This philosophy is simple for testing laboratories to implement, yet reinforces the necessity for traceable measurement standards in all aspects of the measurement, the crucial role of validation in checking the validity of the 'model', and the necessity for uncertainty in reference values in order to quote reliable uncertainties in the final result of the measurement. Dr Salit (NIST) reinforced these three basic points in his description of the selection and use of reference materials for establishing traceability with particular attention to the work necessary for high-level reference materials work. Finally, Dr Kaarls (Chairman, CCQM) provided an excellent description of the work in progress to develop an international traceability infrastructure through BIPM, including the establishment of a database of National Measurement Institute capabilities. Working Groups on traceability for existing methods, traceability and method development, selection and use of reference materials, accreditation and the development of international traceability system were held, and the comments on the new Guide will be incorporated in the revised document, intended for circulation early in 2003.

Dr. Matthias Roesslein, EMPA, Switzerland
Dr. Steve Ellison, LGC, UK

CITAC Workshop at the III International Congress of Metrology in Chemistry

October 2, 2002, Curitiba — Brazil

The relationship between metrology, economy and society is complex and not always easy to discern. It is known that measurements or metrology does impact trade, commerce, the environment, health protection, safety and quality of life, while allowing developed and developing countries to be on a level playing field when it comes to international trade. Additionally, metrology provides the society with the opportunity for consumer protection, fairness in trade and trust in merchandise in domestic markets of all countries.

For many years metrology activities, especially physical metrology, in international trade were almost invisible and chemical metrology was just about non-existent. However, as tariff barriers began to fall, technical barriers have become more prevalent and visible, especially those barriers associated with measurements or metrology. As a result of this, there is evidence for the need of more and better chemical measurements. Many countries, including Brazil, are seeing the rising use of chemical metrology to resolve trade issues and eliminate technical barriers and are beginning to evaluate the current situation of chemical metrology.

Brazil has found better chemical measurement to be extremely important for both social and economic reasons to review all areas of its measurement technology. Additionally, Brazil's studies have indicated that the impact of measurement technology on international and domestic trade can be felt in many directions. This illustrates the need to continue interactions between industries and research centers as well as to increase technical competence in several areas and to develop linkages internationally with other metrology institutions.

The accelerated growth and acceptance of metrology in chemistry within the global market means that a few years from now there will not be place in the international market for products without the quality of all the measurement being assured. Therefore, it is of extreme importance to all countries, including Brazil, to hasten the improvement measurements of all of the laboratories in the country. This can be achieved by extending the availability of basic metrological tools, in order to make possible a fast and homogeneous metrological improvement within the national laboratory system, assuring credibility and trust in their results and products that they are testing.

Brazil is attempting to improve measurements through the Brazilian National Program on Metrology in Chemistry, whose main objective is to improve the quality of the Brazilian Laboratory Measurement System as a whole, through the dissemination of a metrological culture, measurement traceability, and the deliverance of confidence in the Brazilian measurement system and network.

The Ministry of Science and Technology has supported this national Program on chemical metrology — PADCT/TIB, through a Program for the support of scientific and technological development, and as one of its activities, the III International Congress on Metrology in Chemistry was held. This activity is one of several within the project entitled "Implementation on Metrology in Chemistry in Brazil: Establishment of Networking Laboratories and Organization of Activities for the Production of Reference Materials".

About the Congress

The Congress, held in Curitiba, Brazil, from October 1st to October 3rd, was organized to establish a linkage between the national and international communities, to identify areas where these linkages could exchange information and researchers, and to identify areas that would lead to collaborations of mutual benefit. The main focus area of the Congress was technical issues related to various aspects of metrology in chemistry. However, other areas included discussions on the political aspects of the framework, its operation, its hierarchy, and the role of the national metrology institutes and use of the national competences that may exist in other laboratories in the country for specific measurement disciplines .

The various focus areas were interwoven, such that the contributions and experience from the international specialists linked the entire Congress together for the participating attendees. Through this forum, technical questions, measurement needs, national priorities, and the understanding of how to utilize existing capabilities were shared.

The global focus of the Congress on the issues proved beneficial and essential, especially to countries with developing economies, because the Congress identified the importance of chemical metrology and emphasized the need to have available an established competence recognized worldwide, in order to guarantee these economies a level playing field and respect in the global marketplace when chemical measurements are required.

A valuable outcome of this Congress was the organization of a one-day CITAC Workshop on October 2, where the participation of CITAC members played a fundamental role towards the success of METROCHEM III. The focus was related to the identification and to the scheme of necessary activities that assure the traceability, comparability and confidence for the measurements to be available to the end user or the workshop floor. So, the aim of the Workshop was to provide a description of the chemical measurement

infrastructures currently in place in different countries and identify their specific experiences. The traceability chain, the national metrology system and the role of NMIs (National Metrology Institutes) were discussed. The main objective of the Workshop was to show how the field laboratories could achieve reliable, comparable and traceable measurement results.

In a parallel session, Prof. Wolfhard Wegscheider, from the University of Leoben, Austria, taught a course entitled "UNCERTAINTIES IN ANALYTICAL PROCESSES (Cases)", which brought examples of how traceable measurements are achieved at the level of the field laboratory in various chemical areas.

Also, on October 3rd, the 17th CITAC members meeting was held, attending were 12 official members and 10 observers.

The comments about the panels, courses, technical sessions, round tables and debates that occurred during the Congress will be accessible at the web site www.metrologiaquimica.org.br

The Workshop

Dr. Kensaku Okamoto, NMIJ, Japan, and Dr. Laurie Besley, NARL, Australia, were chairmen of the first session "How to disseminate traceability to the workshop floor, the role of CRMs, an international recognizable structure".

The session was begun from the lecture by Dr Wolfgang Richter, PTB, Germany, "Traceability structure for chemical measurements in Germany". Dr Richter explained that in Germany traceability in chemistry has been established through a combination of several institutes. He mentioned that in Germany, chemical calibration laboratories accredited within the framework of the German Calibration Service (DKD) act as "multipliers" between the national standards level and the user level. They provide the user with calibration means, which are traceable to the SI via primary standards provided by the national metrology network. He mentioned that because of the breadth of chemistry, the national standards for Germany will also be provided through a decentralized system. The major players in this network are: PTB (German National Metrology Institute), BAM (German Federal Institute for Materials Research & Testing), UBA (German Federal Environmental Agency) and DGKC (German Society for Clinical Chemistry).

PTB will provide oversight for the network and maintain competencies for organic analysis, elemental analysis, gas analysis, electrochemistry and laboratory medicine. BAM will be responsible for the development and dissemination of primary pure element standards, primary standard gas mixtures and metal alloy CRMs, among others. UBA will be responsible for the development and dissemination of environmental gas mixture standards and DGKC for the development and distribution of standards for laboratory medicine, such as enzyme activity, hormones etc. Each institute within the National Metrology Network will provide CMCs (Calibration and Measurement Capabilities) for the BIPM database that are representative for their unique contributions to the German national metrology system and participate in appropriate CCQM comparisons. In this way, the four mentioned institutions together provide reference materials and dissemination mechanisms for the German community. Different dissemination structures were employed for different application areas. A key element was the provision of a range of certified reference materials. Another was the use of the German Calibration Service for metrology in chemistry. Dr Richter gave examples related to the arrangements for traceability in clinical measurements. He showed how a partnership with the German Physicians Council could be used effectively to ensure the accuracy of measurements in that arena.

Dr. Mike Sargent, LGC, UK, talked on "UK delivery of traceable chemical measurements in the 21st century — building on the foundation of the VAM program". He first gave a brief history of LGC, emphasizing that LGC is perhaps the world's oldest national standards laboratory, having been founded as The Excise Laboratory for England. He mentioned that the National Physical Laboratory is the National Metrology Institute in UK, while LGC had designated responsibilities for much of chemistry.

Dr. Sargent described the important development of the Valid Analytical Measurements (VAM) program and the principles upon which it was based. He talked about the multi-facetted nature of the program, describing in detail some of its aspects including the array of educational initiatives it undertook. He stated that promotion of the concepts of metrology in chemistry was a very important aspect of VAM.

Reinforcing some aspects, he said that the demanding for chemical metrology, specifically for traceability is growing very rapidly in the UK as elsewhere in the world, what means that much had been done and much must be done. He informed us that LGC is addressing this need by establishing a new UK Chemical Calibration Facility and an industrial outreach activity called "Achieving traceability in ISO 17025 accredited laboratories". During the question time, Dr. Sargent answered questions about the area of pharmaceuticals, where LGC does not have an active role.

The lecture by Dr. Willie May, NIST, USA, was titled "NIST programs and practices for providing chemical measurement traceability: our mandate and the impact of our work". He talked about the general mandate that NIST has from its government to provide technical leadership for the USA measurement and standards infrastructure, and assure the availability of essential reference data and measurement capabilities. He described how NIST's research and service programs in Metrology in Chemistry are structured to provide the reference base and underpinning for chemical measurements made throughout the U.S. and on a *de facto* basis — much of the world. Examples were given regarding the impact of several current NIST measurement and standards activities that impact quality of life - such as healthcare, food and nutrition, and the environment. The talk concluded with a brief discussion of NIST's measurement and standards role in areas of contemporary interest such as nutraceuticals, genetically modified foods and global climate change. Dr. May mentioned that NIST provides a number of traceability routes to its clients, including the provision of a very large range of reference materials, and a designated supplier scheme whereby responsibility for an area is partially devolved to a third party or parties, often in the private sector. He described how this latter scheme has been very successful in the gas standards area but less so for the delivery of optical filter standards. Like Dr Sargent, he stressed that in the USA the demand for traceability was increasing very rapidly and that NIST was having

difficulty meeting the demand. He emphasized that nonetheless NIST was zealous in maintaining a high level of science in its work, quoting as evidence a range of academic success performance measures. He described how NIST sets its priorities and gave the results of several formal studies that had been conducted to assess the impact of the measurement services that NIST provides.

The topic of the lecture by Dr. Kensaku Okamoto, NMIJ, Japan, was "Traceability of reference materials in Japan: the role of the new NMIJ". He explained that the new National Measurement Institute of Japan was rearranged as part of the general reform of government and aimed at designing to provide greater integration of activities and greater transparency of operation. In providing traceability, the aim was to shorten the traceability chain, providing higher accuracy at lower cost. A 5-year plan had been put into place for the generation of suites of reference gas mixtures, with emphasis on standards for environmental air monitoring. A group of matrix reference materials was being developed for marine sediment analysis, metallic solution standards and high purity materials. Food analysis was to be another major area of focus.

Dr. Okamoto presented NMIJ's long-term plan for development of new gas mixture standards that included VOCs, global warming, and "sick building" gases (styrene, n-butyl acetate, etc.). He emphasized NMIJ's commitment to participating in relevant CCQM comparisons and talked about their plans for linking NMIJ's production of new CRMs with relevant CCQM International Comparisons.

The second session "The role of quality assurance and proficiency testing schemes" was chaired by Prof. Wolfhard Wegscheider, University of Leoben, Austria, and Dr. Marco Antônio Grecco D'Elia, IPT, Brazil.

The session started with a lecture by Alan Squirrell, NATA, Australia, "Proficiency testing in Australia". He explained that in Australia, the National Association of Testing Authorities — NATA is providing the laboratories with a range of proficiency testing programs, according to the guidelines of ISO/IEC Guide 43 (1997). He mentioned that 27 programs were offered by NATA related to several areas: biology, chemistry, mechanics, electricity and others. Alan Squirrell also mentioned 39 programs offered by APLAC (Asia Pacific Laboratory Accreditation Cooperation), some of them coordinated by NATA and by IMEP (The International Measurement Evaluation Programme), which is operated by IRMM (Institute for Reference Materials and Measurements, Belgium). The available proficiency testing programs were not sufficient to attend all needs although approximately 35 were running each year. An increasing demand for these programs is foreseen, due to the participation of accredited laboratories through the National Metrology Institutes and larger number of implemented quality systems. NATA would be reviewing its accreditation programs in a near future due to interest proficiency testing providers in other areas such as medicine, veterinary and forensic area.

Dr. Ed de Leer, NMi, The Netherlands, reported on "A quality control program for Dutch environmental laboratories". He explained that 5 years ago in The Netherlands organizations were compulsory to sign any contract with government agencies. However, analysis of interlaboratory studies with participation of laboratories of these agencies showed coefficient of variation values from 50 up to 200%. It required an effort from the government to initiate an extra program than those based on the accreditation process. Therefore a program was initiated improving the environmental quality measurements, emphasizing the improvement and validation of existing standards and development of new validated standards. Currently there are 8 laboratories involved in the testing according to the required specifications. Their work contributed to minimize the dispersion of the results along the time.

Dr. Yoshito Mitani, CENAM, Mexico, talked on "Programs in Mexico". He presented 8 programs of proficiency testing coordinated by CENAM in Mexico since 1995, with the main objectives to evaluate the chemical measurement competence of analytical laboratories from different sectors, such as environmental analysis, food, agriculture, clinical analysis and chemical industry, to establish a networking laboratories for method validation, to assign property values to materials, to estimate uncertainty budgets, as well as to disseminate the concept of accuracy and traceability of measurements in respective sectors. He emphasized the unique methodology used in these PT schemes. He reported also on CENAM's participation in SIM (Sistema Interamericano de Metrologia) and in international comparison studies.

Vera Maria Lopes Poncano
IPT, Brazil

The CITAC members and observers participated in the Workshop and the 17th CITAC members meeting

Meeting ISO/IEC 17025 Traceability Requirements: A New Guide with Worked Examples

Many of the issues associated with traceability are well-established components of good measurement practice in chemical laboratories. However, some of the formalities are new and, until recently, the views of experts concerning the details of how to achieve the traceability of chemical measurements differed considerably. This has made it difficult to implement the traceability requirements of ISO/IEC 17025, in chemical testing laboratories.

In recognition of this problem, CITAC and Eurachem have been working for over ten years to develop a widely agreed strategy applicable to different types of chemical measurements. The result of this effort is a new guide that describes a simple, but metrologically robust strategy and provides worked examples to illustrate what laboratories need to do to establish the traceability of their measurements. The guide is currently at the 'advanced draft' stage. It has been the subject of an international workshop in Lucerne in June 2002 (1) and is available on the Eurachem web site (www.eurachem.org). The final document is expected to be published during 2003. The following is a summary of the key issues covered in the guide, coloured by the personal views of the author. The paper was presented at the ILAC Berlin Conference in September 2002 and is scheduled to be published in full in ACQUAL in June 2003.

An important precursor of measurement traceability, is an adequate description of what is to be measured (the measurand), which needs to include the units of measurement and consideration of the acceptable level of measurement uncertainty (MU). Most chemical measurements can be made in SI units and by appropriate calibration measurements can usually also be made traceable to the SI. The strategy described in the guide is summarised in Box 1.

Box 1

Strategy for Establishing the Traceability of Chemical Measurements

- Define the measurand including measurement units and the acceptable level of MU
- Establish the measurement equation during method validation
- Establish the traceability of each quantity in the measurement equation
- Establish the traceability of any other quantities that significantly affect the measurement result

For measurements to be traceable to the SI, they need to be made using equipment that has been calibrated using measurement standards, that have themselves been calibrated using higher level standards that are traceable to the SI. Often such measurement standards are obtained from reference or calibration laboratories, simplifying the task of the testing laboratory.

Chemical measurements are invariably made indirectly, by measuring other quantities (such as sample weight, volume of sample solution, signal response from the instrument relative to the response from a series of chemical standards) and calculating the chemical measurement result using an appropriate measurement equation. If the measurement of these 'influence quantities' is carried out with equipment calibrated using SI traceable standards, then the chemical measurement calculated from these results can also be expected to be traceable to the SI. If there are additional quantities, such as time, temperature, pH etc influencing the measurement process, then their effect can often be eliminated by keeping them constant. When 'control quantities' have a significant effect, then the measurements used to control them also need to be made with equipment calibrated using SI traceable standards. This strategy is illustrated by the example in Box 2.

An important related issue is the uncertainty associated with both the measurement standards used to make a measurement, and the uncertainty of the final test result. The value assignment of measurement standards, including reference materials (RMs), need to be at levels of uncertainty such that they do not contribute more than about one third of the uncertainty of the final test result. However, often the uncertainties of measurement standards are small compared with other sources of uncertainty in chemical measurement, and hence, whilst important, the standards are not usually a major source of difficulty.

Nonetheless, questions may arise concerning the traceability of the values carried by the chemical reference materials. The essential requirement is for the traceability of the assigned value to be established at a level of uncertainty appropriate to the final test result. Where the RMs have been obtained from National Measurement Institutes, or from accredited calibration / reference material producers, then the traceability of the standards is assured. However, as is often the case, where the RMs are obtained from a non-assured source, it is the responsibility of the user to establish their traceability, at an

Box 2

Example — The Measurement of Cadmium in Soil

- Measurand including units: total Cd in soil, on a dry weight basis, measured in mg/kg
- Equation established during method validation —

$$C = C_c \cdot V / m \cdot R, \quad mg/kg$$

where
C = concentration of Cd in the soil,
C_c = concentration of Cd from calibration graph,
V = final volume of test solution,
m = weight of test portion,
R = recovery (or bias correction factor).

- Traceability:
  - C_c is traceable to SI through the pure substance Cd RM used to prepare the standard Cd solutions
  - m and V are traceable to SI through the balance and volumetric flask calibrations
  - R is traceable to SI through the matrix RM used to evaluate the overall measurement bias
  - The sample drying time and temperature measurements are traceable to SI through the timer and thermometer calibrations
  - Since the equation was established during method validation, the model is valid and therefore C is also traceable to SI

appropriate level of measurement uncertainty. The traceability of such 'in-house RMs' can be established as shown in Box 3.

The remaining question concerns the QA of chemical reference materials and the competence of RM producers. Requirements for the accreditation of RM producers are available, but there is a need for a more active programme and a more balanced approach to accreditation practice related to measurement standards.

Box 3

In-House RMs

- Should be related to higher level RMs of the same type, where:
  - The in-house RM uncertainty contributes significantly to the MU;
  - It is feasible.
- Where commercial chemicals, or other materials are the only RMs available, the laboratory needs to:
  - Assess available data;
  - Where necessary, characterize the materials.
- In addition to property value assignment, based on traceable methods, in -house RMs should be stable and homogeneous, at an appropriate level of uncertainty, and stored appropriately

In conclusion, the advent of this guide will facilitate the implementation of the traceability requirements of ISO/IEC 17025. This will no doubt take time to implement, but as with measurement uncertainty, the scale of the task is less daunting than might appear at first sight. Success will depend on the willingness of chemical laboratories to embrace this element of 'good measurement practice' and on enforcement of the ISO/IEC 17025 requirements by accreditation bodies.

Acknowledgements
The author would like to acknowledge the work of the joint Eurachem/CITAC working group who are producing the guide, particularly the contributions from the chair, Dr. Alex Williams and the secretary, Dr. Steve Ellison.

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Ten years on: A Review of CITAC's Achievements

Those who stayed for the end of the 1993 Pittsburgh Conference in Atlanta, will remember the hurricane that caused havoc on the final day. But for a small band of analytical quality enthusiasts, 12 March will also be remembered as the birth date of CITAC. Whilst holed-up in the Peachtree Hotel in downtown Atlanta, with gale force winds sweeping vehicles down the street and lumps of ice falling from the fortieth floor onto the plastic roof of the foyer, the first meeting of CITAC was taking place. During the height of the storm, no one could enter or leave the hotel, so there was ample time to discuss aims and objectives and to plan the work programme (1).

CITAC began under snow — a view from the hotel

All of this followed a survey of the views of analytical measurement scientists in 1992, which concluded that there was a need for a new initiative, to develop a metrologically based, international, chemical measurement system.

The first workshop attracted forty five invited delegates from Africa, Australia, Canada, Europe, Japan, Russia, South America and the USA, and included input from international organizations such as AOACI, BIPM, ISO, ILAC and IUPAC. Despite the existence of numerous national and international organizations dedicated to various aspects of measurement quality, there was seen to be a need for a new umbrella organization, to help co-ordinate and harmonize the work of the different sectors and functions, such as metrology, accreditation, standards making and knowledge transfer. The requirements for CITAC membership were established as a willingness to contribute to its work and payment of a modest, US$ 300 pa, membership fee. Over the years, membership has grown and currently stands at input from delegates from twenty countries and ten international organizations.

One of the outputs from the first meeting was a description of the mission and objectives of CITAC, as summarised in Box 1. Although they have been subsequently refined, they have remained substantially the same and stood the test of time, as a description

Box 1

CITAC Mission

To improve the comparability of measurements made in different parts of the world

CITAC Objectives

- To facilitate the practical realization of metrology in chemistry
- To promote & harmonize quality practices in the analytical chemistry laboratory
- To foster collaboration as a means of effecting technology transfer and cost sharing
- To examine the practicality of traceability structures for various chemical measurements

of the task to be performed.

The other output from the first meeting was a 'wish list' of topics that needed to be addressed. These are listed in Box 2, and as can be seen from the following summary of achievements, progress has been made on nearly all of the issues, often with or through other organizations. The one exception is the provision of adequate funding. As with all such organizations, funding is a problem and progress on work items depends on the input from individual enthusiasts and their sponsoring organizations.

Box 2

Work Items Identified at First CITAC Meeting

- Prepare a directory of CRMs under development
- Provide advice on quality requirements for RM producers
- Prepare a guide on good QA practice
- Prepare a directory of chemical metrology activities
- Describe the criteria for establishing the traceability of chemical measurements
- Develop strategies for evaluating the uncertainties of chemical measurements
- Prepare a guide on the QA requirements for non-routine analysis and analytical R&D

From the beginning, as highlighted in the organisation's name, it was recognised that traceability was the key to improving the comparability of analytical measurements on a global basis. Although progress has at times seemed to be painfully slow, looking back over the ten years, it is clear that we have indeed come a long way, in part at least, due to the work of CITAC. As expected, it took considerable time to understand some of the more subtle aspects of subjects such as measurement traceability and uncertainty of measurement. However, also as expected, when we did come to understand the issues, they proved to be much more simple than sometimes imagined.

As a result of CITAC's work, a number of 'good measurement practice' guides have been developed, as an aid to the establishment of an internationally harmonized measurement system. Also, traceability is now emphasised as a requirement for ISO/IEC 17025 accreditation. CCQM is helping to make this a reality by establishing a system of primary measurements, whose equivalence has been demonstrated on an international basis. This will help reference laboratories all around the world produce the measurement standards (including reference materials) traceable to the SI. A fuller list of the activities and achievements of CITAC, over the past ten years, is given in Box 3.

Box 3

Summary of CITAC Achievements

- Organised or co-organised fifteen international symposia/workshops on topics such as:
  - Analytical QA
  - RMs and their quality
  - Measurement traceability
  - Education and training
  - Food analysis
  - ISO/IEC 17025
- Produced or contributed to guides on:
  - Analytical QA
  - R&D
  - Method validation
  - Measurement uncertainty
  - Measurement traceability
- Contributed to ISO/ILAC documents on:
  - Laboratory accreditation (ISO/IEC 17025)
  - RM accreditation (ISO Guide 34 / ILAC Guide 12)
  - PT accreditation (ISO Guide 43 / ILAC Guide 13)
- Organized interlaboratory studies on trace elements and ethanol standards
- Liaised with and lobbied for other international organizations to adopt a metrological approach to chemical measurement at a 'fit for purpose' level

Some of the current topics of interest are summarised in Box 4. Although many of these topics were in the original work programme, over the years the debate has progressed and the current work is at a more focused and advanced level. For those that have been involved, it is clear that the networks established through CITAC have facilitated both formal and informal collaboration, leading to cost savings, harmonization of measurement systems and improved comparability of measurements.

A good part of the success of CITAC stems from the friendly spirit developed amongst the members. This has allowed us to tackle some of the hard issues - and following robust debate - to achieve compromise where appropriate. As one of the members of CITAC, and its first chairman, I am delighted to record my thanks to CITAC colleagues for their friendship and forbearance. It will be a pleasure to celebrate CITAC's tenth birthday and to wish it an interesting and challenging future.

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1. B King and R Walker, Anal Chem, 1994, 66, 1168A-1171A.

Box 4

Current Hot Topics

Measurement Traceability

- Continuous activity since 1993
  - Numerous workshops and papers
  - Simple four-page guide
  - Joint guide with Eurachem
- Issues
  - Describing and promoting a pragmatic strategy for establishing traceability
  - Linking working level measurements to the SI
  - Identifying the benefits and economic impact

Measurement Uncertainty

- Joint Eurachem/CITAC guide:
  - Second edition completed May 2000
- Future topics:
  - MU in compliance testing
  - MU near detection limits
  - MU near natural limits (0 and 100%)
  - MU in qualitative analysis
  - Advice for customers of measurements

Laboratory Accreditation

- ISO/IEC 17025
  - Lobbying for traceability and measurement uncertainty to be included in ISO/IEC 17025
  - Helping laboratories and accreditation bodies meet the traceability and measurement uncertainty requirements of ISO/IEC 17025
  - How? How rigorously? How soon?
- Accreditation of NMIs, reference laboratories, PT organizers and RM producers
  - Preparation of 'good practice' guides
  - Promoting the concept of accreditation

Dr. Bernard King,
Analytical Measurement Consultant, UK

UPDATE OF ILAC ACTIVITIES

2002 has been a year of both progress and consolidation for the International Laboratory Accreditation Cooperation (ILAC). Progress along the path to incorporation and in the activities conducted jointly with IAF and ISO/CASCO and consolidation of the ILAC Arrangement, now nearing the end of its second year of operation.

The ILAC Arrangement

As at December 2002, there were 42 signatories to the Arrangement, representing 34 economies. In the past 12 months, ILAC has been focussing on enhancing a more widespread understanding of the benefits of the Arrangement amongst the international community, particularly governments and regulators.

Key Strategic Issues for ILAC in 2002

Over the past two years, ILAC has been progressing towards incorporation — this is expected to be achieved in early 2003. In support of this, there has been a comprehensive revision of the ILAC Rules (Articles and Bylaws), following extensive consultation with the whole ILAC membership (including stakeholders).

From a strategic perspective, another crucial document which was finalised this year is the ILAC Strategic and Business Plan — this provides strategic directions and goals for ILAC over the next 3 years.

Another major focus for ILAC this year has been the provision of increased assistance to developing laboratory accreditation bodies in various countries (with the support of UNIDO, ISO and other bodies). Activities in this area have included training courses, workshops and pre-evaluation visits.

Stakeholder Members

There has also been increasing involvement of ILAC's stakeholders in the operations of ILAC. ILAC recognises the key role its stakeholders play in ensuring that ILAC retains a strong customer focus.

Stakeholders have played an active part in the development of the Strategic Plan and also overviewing the implementation of the ILAC Arrangement (Marie Walsh from Eurachem is a member of the Arrangement Management Committee).

A review of the issues relating to the participation of stakeholder members, including possible voting rights, will be conducted in early 2003 as a matter of priority.

CITAC was again represented by Dr Bernard King at the Laboratory Committee (LC) Meeting held in association with the ILAC General Assembly and Conference in Berlin (September 02). This Committee coordinates the laboratory (stakeholder) activity and inputs into the ILAC decision making process. The Committee has been very active and productive in 2002. This reflects both ILAC's desire to increase cooperation with all relevant stakeholders and a willingness on the part of stakeholders, and particularly the laboratory community, to actively contribute to the international laboratory accreditation process.

The LC work program is extensive and not surprisingly involves a lot of issues associated with ISO-IEC 17025:1999 — These include:

(a) The nature and timing of the next revision and possible "alignment" with the ISO 9000:2000 series.

(b) The development and promotion of "good practice" in traceability and measurement uncertainty, in ways that are both technically sound and affordable. (A survey instigated by CITAC will be conducted by ILAC in 2003 to ascertain the current "state of play" for accredited laboratories).

(c) Other "metrological" issues including the provision, selection and use of high quality reference materials in chemical laboratories. A 3rd party quality assessment program for both Reference Material Producers and Proficiency Testing Providers is being further investigated.

(d) The LC has also reviewed the recent revision (jointly by CITAC/EURACHEM) of CITAC Guide 1 (QA Practice). This is seen as a useful ISO-IEC 17025 guidance/application document for chemical laboratories and will be placed on the ILAC Reading List.

The next meeting of the LC is scheduled for 5/6 March 2003 in Orlando, USA and CITAC's new representative, Dr Laurie Besley for NARL/AGAL, Australia will attend. Thanks to Bernard for his contribution over the past 2 years.

All these activities will again provide the focus for work in 2003, and again CITAC will be making an active contribution (particularly for important technical matters). Progress will be reviewed at the annual ILAC General Assembly in Bratislava, Slovakia from 15 — 23 September 2003.

If you require more information about ILAC's activities please do not hesitate to visit our website on www.ilac.org or email the Secretary on ilac@nata.asn.au.

Alan Squirrell
NATA, Australia
CITAC Member/ ILAC Secretary

New CITAC Members

The following four new members were elected at the 17th CITAC Members meeting in Curitiba, Brazil, October 3, 2002: Dr. Ed W.B. de Leer, Scientific Director, the National Metrology Institute (NMi), The Netherlands; Dr. Laly Samuel, Project Leader Metrology in Chemistry, the Measurement Standards Laboratory of New Zealand; Dr. Wynand Louw, Manager, the National Metrology Laboratory of South Africa; and Mr. Yu Yadong, Director, the National Research Center for CRMs, China.

Dr. Ilya Kuselman,
CITAC Secretary

Dr. Ed W.B. de Leer

Dr. Laly Samuel

Dr. Wynand Louw

A message from Yu Yadong, China:

It's my honor to be accepted as a member of CITAC. As a representative coming from China's top laboratory of chemical metrology, I'm very pleased to participate in the activities of CITAC. I'm willing to play an active role in it and to cooperate with my colleagues from other countries to enhance the study of reference materials, the traceability of chemical measurement, the measurement uncertainties, the quality assurance of analytical measurement in chemistry, and also to promote the establishment of, and perfect an international chemical measurement system.

In recent years, China National Research Center of Certificated Reference Materials (NRCCRM) has engaged in the traceability study of reference materials and chemical measurement methods. Six national primary standards and nearly 50 national standards have been established, such as combustion heat, acidity, conductivity, viscosity, humidity and primary standard reagent, and more than 400 kinds of CRMs have been developed. We lay our emphasis on researching the applications of hig coulometry, frozen point method and isotope dilution mass spectrometry. We have taken part in more than 30 international comparisons of IDMS measurements, reference gases and primary standard reagents organized by CIPM. Most of them got good results and

some key comparison results came out on top. NRCCRM has received international recognition because of its accomplishments.

Now, we are starting to study and to improve China's measurement traceability system in chemistry, the comparison system about measurement methods and the values of quantities, the evaluation system of measurement uncertainty and the quality assurance system of analytical measurements. At the same time, NRCCRM conducts the works of developing reference materials and related measurement technology in the field of materials, energy resources, environmental protection, food and heath care, etc. The study of common technology about reference materials and their application researches are also undertaken.

In the beginning of this new century, the trend of economic globalization is becoming stronger. The scientific and technical development is advancing rapidly. As the largest developing country in the world, China's economy construction, development of science and technology as well as social development have entered into a new era after more than 20 year's reform. We will participate in the international activities with a more active and more open attitude. Let the world learn more about China and let China walk towards the world.

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