CITAC news
Co-Operation on International Traceability in Analytical Chemistry
FEBRUARY 2000

Foreword by the Chairman

As you are becoming informed about CITAC activities, past and future, chemical laboratories are busy preparing themselves for the "new" requirements of ISO 17025 and are struggling with its enhanced status as an ISO Standard.

CITAC is eager to provide suitable interpretations on the renewed emphasis on uncertainty and traceability. Why renewed? Because these two essentials of good measurement science somewhat buried for a while in the hype around quality assurance of the ISO-9000 type; uncertainty and traceability have been around ever since a common system of units was established in the century before last.

In essence, traceability of a result encompasses the reference to one or more firm anchors that we call standards plus the reporting of a range in which we expect the true value to be. This expected closeness of our results to the true state of affairs we call uncertainty of measurement, and it cannot be any better than the uncertainty carried by the values of the standards. It is not frequently accentuated that possibly the best imaginable reference is a direct one, as this reduces the chain leading to the standards to just one single link. This direct link would be one without need of intermediate measurements of other people that unavoidably contribute their share to the uncertainty of our own measurement.

Without intermediate measurements we have the added benefit of a simple measurement infrastructure: reliable measurements on relevant standards, their distribution throughout the community of chemical laboratories and their judicious use in well validated analytical procedures.

So, while the official definition emphasizes the chain I suggest to concentrate more on the individual link. This brings us back to the careful and proper execution of analytical work that needs to be fully understood and validated. A detailed understanding helps us to avoid systematic deviation of results, a sound validation will provide us with the necessary raw data for the estimation of uncertainty and together they will lead to valid results, ... and valid results only can serve as a link in a traceability chain no matter how short the chain is.

The quality of individual measurements is more in line with the practice of analytical chemistry and the self-esteem (Selbstverständnis) of analytical scientists than the creation of chains and provides a well-founded rationale behind our pronounced validation efforts as no chain can be stronger than its weakest link.

The role of metrology institutes in chemistry then is one that centers around the provision of the required standards. Only in exceptional cases the most needed ones are pure substances as these generally can be supplied in plentitude by private enterprises.

Internally, CITAC has undertaken a relocation of its secretariat. After years of excellent service for our cause, LGC in London must be duly acknowledged for the outstanding job. As usual, such a level of support cannot be taken for granted on an institutionalized basis and I want to thank personally as well as in the name of all past-chairmen and on behalf of CITAC at large Dr. Ron Walker for his dedicated, able and prompt support at all times.

The secretariat has meanwhile been transferred to IRMM in Geel and is now run by Dr. Ioannis Papadakis. Feel free to contact him at all times, his coordinates are given on the last page of the Newsletter.

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University of Leoben, Austria

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ILAC

MUTUAL RECOGNITION ARRANGEMENT

Background

CITAC, as a Stakeholder Member of ILAC, (through the Laboratory Liaison Committee) has actively contributed to the development of the ILAC Arrangement. In April 1999 a small working group (which included a CITAC Member) was established to provide the framework and documentation to take this forward and the result of this work was presented to the ILAC General Assembly in Rio de Janeiro in October 1999. The General Assembly endorsed the general principles and decided that the draft documents, which have already been reviewed by the ILAC Membership in August – September 1999, should be further amended (as proposed by the ILAC Accreditation Policy Committee) and re-submitted to the Membership for a postal ballot to be concluded by 6 January 2000. These amended documents were put on the ILAC website (http://www.ilac.org) in November 1999 so that further input could be received from all ILAC Members and Stakeholders. The Arrangement Working Group at their (last) meeting on 17/18 February 2000 will consider these comments.

Hopefully this will culminate in the establishment of the formal ILAC Arrangement around mid 2000 and applications for Membership (of this Arrangement) could be accepted immediately thereafter.

Documents – ILAC Mutual Recognition Arrangement

The following five documents have been produced:

- *"Requirements for Evaluation of Accreditation Bodies" (P1);*
- *"Procedure for the Evaluation of Regional Corporation Bodies for the Purpose of Recognition" (P2);*
- *"Text of the Arrangement";*
- *"Terms of Reference and Composition of the Arrangement Management Committee";*
- *"Policy Statement".*

ILAC Organisation

The "Policy Statement" mentioned above sets out the objectives and principles of the operation of the ILAC Arrangement.

It creates an ILAC Arrangement Management Committee and Arrangement Council and the new proposed ILAC Organisation is shown below (Fig).

This structure will provide a (light) ILAC umbrella to oversee all future mutual recognition activities for accreditation bodies. The actual evaluation work will still be designated to a Regional Co-operation Body, eg EA, APLAC. The ILAC Management Committee will probably consist of the Vice-Chair of ILAC, the Chairman (or representatives) from each Regional Co-operation (currently EA and APLAC) and observers from Stakeholders (laboratory associations and other user bodies) and IAF. This Committee will be charged with the implementation and maintenance of the Arrangement and report to the Arrangement Council. Its duties will include:

- verification of proper conformance
- monitoring operations of regional co-operations and individual signatories
- identifying and recommending improvements
- maintaining a list of ILAC evaluation team leaders and members.

Confidence Building Activities between Regional Co-operations

The ILAC Memorandum of Understanding (MOU) was signed in September 1996 and this was the first step in the process towards an ILAC Arrangement.

It was recognised that differences exist between the existing Regional Cooperations – EA and APLAC – which cover over 80% of the current ILAC Members – and that there was a continuing need for harmonisation of practices and procedures. Since that time, both regions have embarked on a number of confidence building activities and these were summarised at the Rio General Assembly. Mutual activity included attendance at meetings, joint evaluations of laboratory accreditation bodies in both regions, an audit of each Secretariat and general co-operation, particularly in relation to critical technical issues (eg proficiency testing, traceability and uncertainty policies etc). It was agreed that sufficient confidence in both systems had now been established to proceed with the ILAC Arrangement. This was a key step in the process.

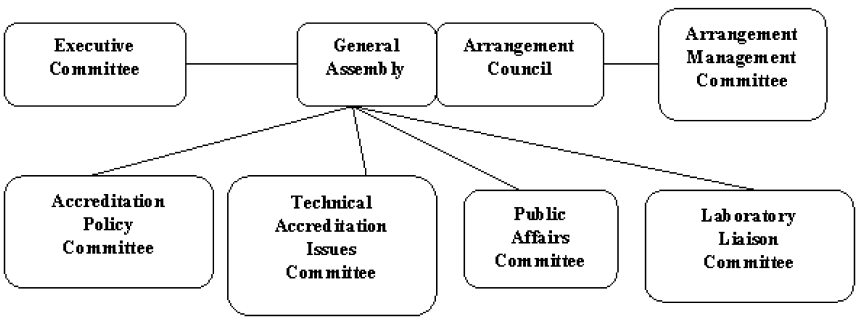
It is anticipated that both EA and APLAC will apply for membership of the ILAC Arrangement soon after its formal establishment and thus all existing EA MLA and APLAC MRA signatories will be incorporated. Applications from unaffiliated regions such as South Africa (SANAS) are also expected and in the future, other (developing) Regional Cooperations (such as IAAC and NACLA) should apply. By this means the network of formal recognition between accreditation bodies should be expanded to include over 30 economies worldwide and thus facilitate the "one stop" testing approval process (and thereby minimise technical barriers to trade).

Conclusions

I personally believe this is a major step forward (in the right direction). At a time where accreditation bodies and their customers (laboratories, governments, regulatory bodies, etc.) are demanding a more transparent demonstration of competence, it is essential that we provide the necessary "strengthening" of existing regional arrangements. This, together with the need for global future harmonisation, will be facilitated by the ILAC Arrangement and provide the technical framework to underpin existing and future government to government arrangements. In addition, the ILAC framework will provide an effective and efficient means of carrying out multi lateral evaluations (we cannot continue with the plethora of bilaterals and the excessive number of evaluation visits these require).

Such confidence can only come about by focussing on the key technical elements of our evaluations (and work carried out by the EA MLA Working Group chaired by Prof Mittmann will assist greatly here) so that we know that accreditation bodies who are signatories to the Arrangement, do indeed meet international requirements and thereby provide sufficient confidence in the test and calibration results produced by their accredited laboratories both between and within international borders.

Alan Squirrell
NATA, Australia



Reference Materials

IRMM Achievements in 1999

New

A new initiative started at the end of 1998 to make certificates and certification reports available on the Internet. Exactly one year later, seventy-four BCR or IRMM certificates and eight reports were available on the IRMM website under the heading "BCR Catalogue". This action will be continued systematically for all new and future CRM's.

The Internet BCR Catalogue

The IRMM catalogue of BCR Reference Materials is available on the Internet (http://www.irmm.jrc.be/mrm.html) and contains – compared to the 1999 BCR catalogue – the following new items:

New BCR CRMs

New BCR CRMs were certified in the environmental protection area, more specifically chlorophenols, chlorobenzenes, polychlorodibenzo-p-dioxins and polychlorodibenzofurans in industrial soils (BCR-529 and 530), polar pesticides in freeze-dried tap-water (BCR-606), in the food safety sector; aflatoxin M1 in chloroform (BCR RM 423) and saxitoxin derivate in lyophilised mussel (BCR-542 and 543) and in clinical chemistry; creatine kinase-2 (CK-MB Isoenzyme/BCR-608) and prostate specific antigen (BCR-613).

A further reference material certified for trace elements (BCR-664: glass) was also made available.

Renewal of BCR CRM

The popular BCR-185, trace elements in bovine liver, which became exhausted in 1999 was replaced by a new material prepared at IRMM (BCR-185R).

CRMs for Biotechnology

In the frame of a commercial agreement between Fluka Chemie A.G. and IRMM on the production and certification of reference materials for the identification of genetically modified organisms (GMO) by polymerase chain reaction (PCR) four CRMs of soya bean ("Roundup Ready") powder mixtures (IRMM-410) and four CRMs of maize ("Bt-176") powder mixtures (IRMM-411: 0, 0.1, 0.5, 1, 2.0 and 5% GMO) have been produced. These materials are jointly commercialized by Fluka Chemie A.G. and IRMM and have been tested in a large scale interlaboratory exercise organised in co-operation with the JRC's Institute for Health and Consumer Protection, Ispra (I). The materials are supplied with an IRMM certificate of validation.

Similar CRM's for "Bt-11" maize (IRMM-412) have recently been produced, containing 0, 1 and 2% Bt-11 maize.

EC – IFCC Collaboration: CRMs for Clinical Chemistry

The development of reference methods, the validation and uniform calibration of commercially available test systems and the harmonisation of results obtained from different laboratories using commercially available immunoassays are essential for public health care. For reaching these goals, there is a strong need for having purified calibrators and matrix based CRMs available both for clinical laboratories and industry. For this reason, a collaboration agreement was signed in 1996 between the European Commission and the International Federation of Clinical Chemistry (IFCC) for the joint IRMM – IFCC production and certification of clinical reference materials.

Moreover, the in-vitro diagnostics directive, requiring test kits sold on the European market to be traceable to "reference systems of a higher order" even increases the importance of this collaboration. CRMs which may be used as part of the required "reference systems of a higher order" can be BCR, IRMM, NIST, or any other producer, provided they comply with the highest metrological principles.

Recent achievements include:

- The certification of a cortisol reference serum panel (IRMM/IFCC-451) was completed in 1999. It consists of 34 sera (80-750 nmol/L), certified using ID-GC/MS as a primary reference method applied in two versions in two independent laboratories.
- The certification of enzyme CRM's for GGT, LD, ALAT, CK-MB, ASAT, ALP and (-amylase at 37 °C, according to adapted IFCC methods is an ongoing project. The first 4 materials (IRMM/IFCC-452, 453, 454, 455) were released in January 2000.

IRMM – ISS Cooperation Agreement: CRMs for Antarctica

In the frame of a collaboration agreement between IRMM and the Istituto Superiore de Sanita (ISS), Rome (I), CRMs for Antarctica are produced at IRMM and certified in laboratory intercomparisons organised by ISS:

- **MURST-ISS-A1: Antarctic sediment certified for trace elements**, was finalised by ISS with the scientific and statistical assistance of IRMM's Reference Materials Unit; this CRM has been included in the IRMM catalogue of BCR CRMs and is available in 50 gram units.
- **MURST-ISS-A2: Antarctic krill** has been prepared starting from three catches made by Italian, American and Japanese Antarctic expeditions and is supplied in pre-weighed ready for use 0.5 gram samples **for single-shot analysis of trace elements**; the certification campaign has been completed and the material is now available in sets of 5 vials.

Jean Pauwels
IRMM, Belgium

Summary of what's new in the internet BCR catalogue as compared to 1999

CRM No	Designation
• BCR-185R	Trace elements in bovine liver (replacing CRM 185)
• BCR-529/530	Chlorophenols, chlorobenzenes, polychlorodibenzo-p-dioxins and polychlorodibenzofurans in industrial soils
• BCR-606	Polar pesticides in freeze-dried tap-water
• BCR-608	Creatine kinase-2 (CK-MB Isoenzyme)
• BCR-613	Prostate specific antigen (PSA)
• BCR-664	Trace elements in glass
• BCR RM 423	Aflatoxin M1 in chloroform
• BCR-542/543	Saxitoxin derivate in lyophilised mussel
• MURST-ISS-A2	Antartic Krill
• IRMM-410	Dried soya beans powder containing Genetically Modified Roundup ReadyTM Soya
• IRMM-411	Dried maize powder containing Genetically Modified Bt-176 Maize
• IRMM-412	Dried maize powder containing Genetically Modified Bt-11 Maize
• IRMM/IFCC-451	34 sera certified for their cortisol content (80-750 nmol/L)
• IRMM/IFCC-452	GGT certified according to an adapted IFCC reference method at 37 °C
• IRMM/IFCC-453	LD certified according to an adapted IFCC reference method at 37 °C
• IRMM/IFCC-454	ALAT certified according to an adapted IFCC reference method at 37 °C
• IRMM/IFCC-455	CK-MB certified according to an adapted IFCC reference method at 37 °C

BRAZIL

PROGRAM ON CHEMICAL METROLOGY

During the First Workshop on Metrology in Chemistry, organized by the National Institute of Metrology, Standardization and Industrial Quality (INMETRO) in November 1997, an effective Brazilian plan of action for the development of Metrology in Chemistry started. The purpose of this plan was to establish a national reference system for providing national traceability and international comparability of chemical measurements made in Brazil, focusing on identifying chemical measurement needs and priorities in Brazilian Industry, and those concerned with environmental, healthcare and food/nutritional decision making.

Activities under development since 1998:

Dissemination of metrological culture through education and awareness:

Looking for a continuous improvement of the metrological culture of our society, since 1998 many technical meetings, presentations and courses have been promoted by the national leading group with the support of industries, unions, technological centers, universities, associations and others. In addition, SIM (the Interamerican System for Metrology) and NIST (the National Institute of Standards and Technology) have developed and provided training courses in: Spectrochemical Methods, Organic Analytical Methods, Nuclear Analytical Methods, Classical Methods and Gas Metrology.

Identification of reference material needs to establish production priorities:

Getting information related to real needs in laboratories, through questionnaires, visits and workshops. Based on results obtained from these inquiries, coupled with existing resources, initial areas of focus will be gas mixtures, and food (milk). The program will be expanded to include other food products, petroleum and petrochemicals in the near future.

Participation in international intercomparison programs:

The participation in six intercomparison programs promoted by SIM – and coordinated by NIST – has been very important for the evaluation of measurement capabilities and practices within Brazil. Such participation will initially be very useful for us in identifying competent laboratories to form our network. In the longer term, Brazilian participation in SIM intercomparison exercise help form the basis for establishing traceability for our measurement results through linkage to key comparisons carried out under the auspices of the CCQM (Comité Consultatif Pour la Quantité de Matière).

Standardization:

In Brazil the committee of the Brazilian Technical Standards Association (ABNT) is working to assure Brazilian conformity to all relevant ISO Guides concerning the production and distribution of Certified Reference Materials.

Insertion of Metrology in Chemistry matters into University disciplines:

Professors from different universities, under the coordination of the Brazilian Subcommittee of Metrology in Chemistry and the Group of Six Public Chemistry Courses of São Paulo State, started last year to develop pilot courses at the university integrating subjects related to chemical metrology like: uncertainty, traceability, reference materials, intercomparisons and others.

Coordination of the ad hoc Group for Reference Materials of Mercosur under the agreement "Asociación Estrategica entre los Institutos de Tecnologia Industrial del Mercosur".

Interaction with international organizations:

INMETRO has been participating of the SIM Chemical Metrology Working Group and have begun interactions with the Organic, Inorganic and Gas Analysis Working Groups within CCQM. In addition, technical interactions are being established with NIST, NPL, NRCCRM, NMi, LGC and others.

Specific Activities planned for 2000:

Implementation of chemical metrology program in Brazil with two independent, but closely related objectives:

- establishing a national network of competent laboratories with the capability to perform analytical measurements of the highest metrological order
- establishing formal agreements and strategic collaborations with other NMI's within SIM and other metrological regions
- begin utilizing this new virtual laboratory in the production of CRMs

Development of a national program for gases with the purpose of:

- development and dissemination of primary gas mixtures standards
- establishing bi and multilateral collaborations in the area of gas mixture standards
- providing quality assurance services for commercial production of high-quality traceable gas mixture standard in Brazil

Establishing clear priorities for expansion of the Brazilian Metrology Program. At this moment the highest priority areas for expansion appear to be reference materials for products such as petroleum, petrochemicals, food, clinical and health products.

Final remarks:

Many activities described above must be established by October 2000, when we will convene an international workshop on metrology in chemistry. This workshop is intended to be a forum to discuss both chemical metrology-related developments in Brazil and – worldwide activities and advances with foreign experts. For further information, please contact: vponcano@ipt.br

Vera M. L.Ponçano A. Silva
IPT / INMETRO, Brazil

Report: CITAC Quantifying the Commercial Reference 11 March 1999,

Introduction

CITAC continued its close association with the Pittsburgh conference by holding a symposium on reference materials (RMs) at PITTCON '99. Bernard King, from the Laboratory of the Government Chemist in the UK chaired the session and introduced the theme. He reminded delegates of the importance of RMs to analytical quality, highlighting their role in calibration, validation, estimating measurement uncertainty and day-to-day QA. He claimed that whilst pressure was rightly being put on analysts to use RMs, there was as yet no comparable pressure on RM producers to provide transparent evidence of the quality of their materials. He predicted, however, that such pressures were necessary and could soon be expected. The aim of the symposium was to explore developments in metrology in chemistry in different parts of the world, directed at linking commercial RMs to national and international standards. In addition, the links between these developments and developments in the field of accreditation of RM producers would be explored.

Measurement Uncertainty

The first presentation addressed the evaluation of the uncertainty of certified property values. This was given by Wolfhard Wegscheider, Chair of CITAC, a member of the joint CITAC EURACHEM Working Group on measurement uncertainty and a Professor at Leoben University in Austria. He reviewed developments in Europe and in the USA and gave some views about emerging strategies.

Forensic Ethanol Standards

In 1997, CITAC carried out an international interlaboratory study of ethanol in water standards produced by standards laboratories around the world. These standards (RMs) are used by forensic laboratories for the calibration of measurements associated with drinking and driving prosecutions. A report of this study was given by the project co-ordinator, Bernard King, with the aim of illustrating how the equivalence of national standards can be evaluated. Such studies do not establish traceability, but do provide independent verification of traceability claims. They also facilitate links between high level, metrologically prepared RMs and more working level RMs, and provide transparency of the quality of the certification data.

Providing Links between National Standards and Commercial RMs

Willie May, head of the Analytical Chemistry Division, National Standards & Technology Institute, described the role that NIST has in providing the traceability link in the production

Symposium PITTCON '99 Reliability of Materials Orlando, USA

of commercial reference materials in the USA. The speaker noted that although NIST has met the reference materials needs of US industry and commerce for nearly 100 years, the chemical measurement 'universe' was extremely diverse, and it was becoming increasingly difficult to maintain this position. With the fast pace of technological change and greater measurement needs, the demand for additional quantities and specific varieties of reference materials has mushroomed. NIST does not have the resources to provide SRMs to meet all these needs.

Accreditation of RM Producers

An important mechanism for demonstrating the quality of a laboratory is peer group accreditation. Accreditation has been used to assure the quality of calibration and test laboratories for many years and work is now nearing completion on the establishment of criteria for the accreditation of RM producers. Peter Unger, President of the American Association for Laboratory Accreditation, plans to offer such accreditation and has contributed to the production of international guides describing the requirements for demonstrating the competence of RM producers.

The needs of the commercial RM Producers

Chuck Wibby, vice-president of Environmental Resource Associates, Colorado, completed the presentations by looking at the needs of secondary/commercial reference material producers in the market place. To meet this need, the Chemical Reference Materials Manufacturers Association (CRMMA) was founded in 1995 in order to promote a free and open market, ensure better communications with the US government and to encourage the production and marketing of high quality materials. CRMMA now has 20 members including NIST, A2LA and NMi.

Summarizing

In summary, Bernard King concluded that there were developments on a number of fronts and that collectively they had the makings of a robust system. Although there was clearly more work to be done in developing and applying the various mechanisms, he was hopeful that when a similar symposium was held in a few years time there would be significant progress to report.

A full report of the symposium would be published in one of the forthcoming issues of Accreditation and Quality Assurance journal. The CITAC Secretariat can supply more information.

B. King & R. Walker
LGC, United Kingdom

Singapore Productivity and Standards Board

CHEMICAL METROLOGY – THE SINGAPORE WAY

The Singapore Government recognises the importance of ensuring traceability of chemical measurements in our predominantly trade-based economy. Being the custodian of national measurement standards, the Singapore Productivity and Standards Board (PSB) has therefore been tasked with the management of the Chemical Metrology Programme as part of Phase IV National Metrology Upgrading activities.

To achieve the Programme's aims of ensuring traceability and hence, comparability for chemical measurements performed within the country, three main activities have been put in place:

- To encourage a wider usage of certified reference materials for appropriate processes. PSB shall support the industries by the development of pertinent certified reference materials in addition to providing advisory services on certified reference materials.
- To organise and promote regular participation in proficiency testing (PT) schemes. PSB has undertaken to co-ordinate PT schemes which are tailored to local industries.
- To heighten awareness of the need for traceability in chemical measurements by conducting public seminars and courses on related topics.

Since inception of the Programme two years ago, PSB has organised seven PT schemes on numerous analytes in water, paint and food. In order to cater to industries performing measurements at trace levels, a PT scheme is in the pipeline for correlation of measurands at sub-parts per billion levels. Similarly, plans are underway to develop a range of single-substance low-level certified reference materials for the electronics and related industries. A series of workshops and seminars focussing on uncertainty measurements and quality issues in chemical analysis will also be offered in the coming year.

PSB has taken a proactive approach and is working closely with several parties to broadcast the concepts of chemical metrology. Regular contact with the local accreditation authority, Singapore Accreditation Council-Singapore Laboratory Accreditation Scheme (SAC-SINGLAS), ensure that chemical metrology activities and facilities are made known to accredited laboratories in Singapore. Academia and industry are also consulted for charting of direction in the Chemical Metrology Programme. By so doing, it is hoped that in time to come, all chemical analysts will have an awareness for chemical metrology and subsequently, perform traceable chemical measurements in their laboratory applications.

For further information, please visit our website at:
http://www.psb.gov.sg/technical/chemical
or fax your enquiry to (065)-7794359.

Delia Lai
PSB, Singapore

FAO, IAEA, AOAC. INT./IUPAC INTERNATIONAL WORKSHOP

PRINCIPLES AND PRACTICES OF METHOD WALIDATION

4-6 NOVEMBER 1999, BUDAPEST, HUNGARY

The International Workshop on "Principles and Practices of Method Validation" took place between 4 and 6 November 1999 in Budapest, Hungary. This workshop was organised jointly by the FAO/IAEA Training and Reference Centre for Food and Pesticide Control, AOAC Int. and the IUPAC Interdivisional Working Party on Harmonisation of Quality Assurance Schemes for Analytical Laboratories. It resulted from the internationally recognised fact that the full method validation carried out through interlaboratory method performance study is a very expensive and limited exercise. It is impossible to organise interlaboratory studies for all analytical methods in use for determination of analytes in various analyte/matrix combinations. A formal basis for the workshop organisation were:

- recommendations of the FAO/IAEA Consultants' Meeting on Validation of Analytical Methods for Food Control", IAEA Vienna, 1997,
- IUPAC project 5/97/8 "Protocol for In-house Method Validation; and
- IUPAC project 5/2/99 "Preparation and Harmonisation of Internationally Harmonised Guidelines for In-house Method Validation".

In all three cases the in-house method validation (single laboratory method validation) is scientifically/technically presented as an alternative to the current internationally accepted method validation practice, namely interlaboratory method performance studies. It is described in the IUPAC, AOAC Int. and ISO in 1988 developed guidance.[1,2] In this respect, the present workshop might be seen as an important event, actually discussing and establishing technical guidelines to be followed within a single laboratory performing method validation. The process required to elaborate all technical details and to change the "philosophy" and consequently the international legislation may take some years. In this process the present workshop was an important milestone.

The aim of the workshop was to bring together scientists and representatives of different agencies, governments, standardisation organisations and accreditation bodies involved in method validation in general or in the acceptance of analytical methods for legislative purposes. Over 130 participants from 35 countries attended the workshop. International organisations, i.e. AOAC Int., CITAC, EURACHEM, FAO, IAEA, IUPAC, etc. were also represented.

The first day of the workshop was dedicated to presentations (lectures and posters), while on the second day two draft documents were introduced and explained:

- IUPAC Harmonised Guidelines for the In-house Validation of Methods of Analysis (Technical Report), prepared by R. Wood, M. Thompson and S. Ellison.

The basic concept of the document and the approach for in-house method validation based on the evaluation of uncertainty sources associated with each specific analytical method were largely accepted and after revision the document will be sent to IUPAC, AOAC Int., ISO, EURACHEM, and CITAC for endorsement. Its publication in the Journal of Pure and Applied Chemistry is expected at the end of the year 2000. As already mentioned, this will not be the end of the complete process. The adoption of this new approach in the laboratories and its acceptance by legislative authorities will require some more time.

- The "Practical Procedures to Validate Method Performance and Results of Analysis of Pesticide and Veterinary Drug Residues, and Trace Organic Contaminants in Food", a discussion document, prepared by A. Ambrus, FAO/IAEA Training and Reference Centre for Food and Pesticide Control

'Practical procedure' is under further elaboration by the experts group of the AOAC Int., FAO/IAEA IUPAC. Publication of the document is foreseen for the first half of the year 2000. It will be further discussed by the Codex Committees on Veterinary Drug Residues in Food and Pesticide Residues at their next meeting.

Both documents are intended to provide useful guidance to those involved in, or who assess the results of, method validation. This includes analysts, regulators and accreditors in national, regional and international organizations. (Both documents are available upon request.)

The third day of the workshop was dedicated to general discussion regarding the quality requirements to be met when analytical methods are validated and specifically to comments and recommendations regarding the two draft documents presented. From logistical reasons, a discussion was mainly oriented to the methods applied for pesticide, veterinary drug residues and trace organic contaminants in food. These are the fields were the general guidelines for method validation cannot be readily used and specific guidelines are required to accommodate the specific requirements and limitations of trace analytical methods. Further, the lack of collaborative studies severely hampered the progress in elaboration of Codex Standards for veterinary drug residues, and a practical guidance document is urgently required for performing method validation in a single laboratory. Nevertheless, single laboratory method validation approach is important also for all other analytical methods. In this respect specific guidance on (minimum) quality criteria and other requirements will need to be prepared.

The following topics were discussed in relation to method validation:

- use of collaboratively studied methods;

proficiency testing;

- uncertainty of analytical measurement; and;
- role of LOD/LOQ.

Proceedings of the workshop, including most of the presentations (lectures and posters) will be published in a special proceedings series book by the Royal Society of Chemistry, Cambridge, UK within the next half a year and this may already be ordered.

The workshop was locally organised by the Plant Health and Soil Conservation Station of Budapest in a very nice environment of the Hungarian Academy of Sciences and has resulted in three very intensive and productive days.

Dr. Ales Fajgelj
Chairman, IUPAC Interdivisional Working Party on Harmonisation of Quality Assurance Schemes for Analytical Laboratories IAEA, Agency's Laboratories Seibersdorf A-2444 Seibersdorf, Austria

Dr. Árpád Ambrus
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[1] Horwitz W, *Pure & Appl. Chem.,* Vol 60, pp. 855-867, 1988
[2] Pocklington WD, *Pure & Appl. Chem.,* Vol 62, pp. 149-162, 1990

CITAC 2000 PITTCON Symposium

IMPACT AND IMPLICATIONS OF ISO 17025 ON CHEMICAL MEASUREMENT COMMUNITIES

The Fairmont Hotel New Orleans – Wednesday, March 15, 2000

The new Laboratory Accreditation Standard, ISO 17025 is expected to be implemented in year 2000. When implemented, it will set new requirements for method validation, traceability and measurement uncertainty. Its impact on Chemical Testing Laboratories and their customers will depend on the degree of rigor and the even-handedness with which the new measurements are implemented. The symposium will provide an opportunity to discuss and debate these new requirements and their potential benefits.

The impact on laboratories, accreditation bodies, commercial reference materials and PT providers as well as National Metrology Institutes/Standards Laboratories will be also be discussed.

Scientific Program

09:00	Opening Remarks	W. E. May
09:10	ISO 17025: What It Is, What It Is Not, and Who Benefits	L. Neumann
09:50	Some Views from Chemical testing Laboratories	R. Robertson
10:30	*Break*	
10:50	Strategies for Implementing ISO 17025	P. Unger
11:30	A Pragmatic Approach to ISO 17025	B. King
12:10	*Lunch*	
01:00	Assuring the Quality of RMs and PT Schemes	R. Parris
14:00	Demonstrating the Equivalence of National Measurement capabilities	R. Kaarls
14:40	*Break*	
15:00	How Commercial RM Producers and PT Providers Can Help	C. Wibby
15:40	Open Discussion concerning: • Areas of special need • Action plan for next two years • Longer term needs	T. E. Gills
16:45	Closing Remarks	W. Wegscheider

INTERLABORATORY COMPARISON

International Measurement Evaluation Programme (IMEP); prospects at the start of the 21st century

The International Measurement Evaluation Programme (IMEP) was established in 1988, in order to shed light on the actual state of practice in chemical amount measurements. The programme is operated by IRMM (Institute for Reference Materials and Measurements), a research institute of the European Commission's Joint Research Centre.

IMEP is one of the few interlaboratory comparison programmes worldwide that are not based on consensus values as reference values (i.e. derived from the participants' results). IMEP participants can compare their results to a "reference range" which is tracable as far as possible to the SI. The establishment of metrological reference ranges is done in collaboration with measurement laboratories around the world which have a demonstrated record of experience in measurement and uncertainty evaluation in the application concerned. IMEP is open to all laboratories and full confidentiality is guaranteed with respect to the link between results and the participants' identity. Until today IMEP rounds have been focusing on (trace) elements in serum, natural and synthetic water, polyethylene, car-catalysts and on carbon and oxygen isotope amount ratios in carbon dioxide.

IMEP runs under the auspices and is supported by IUPAC, EURACHEM, EUROMET and CITAC. In addition the past few years IMEP developed stronger links with accreditation and metrology. Four rounds have served as pilot projects in the framework of the collaboration between IRMM and European Accreditation (EA). Moreover, on a number of occasions certified test samples of IMEP rounds were also used in key comparisons (e.g. IMEP-9) or pilot studies (e.g. IMEP-14) of CCQM. In this way degree of equivalence of measurements results from top level (national metrology institutes) to bottom (field laboratories) could be established.

One of the IMEP rounds finalised during 1999 was IMEP-11, trace elements in car exhaust catalyst. This round supported needs of the automobile and recycling industry. The round addressed the elements Pt, Zr, Ce and Hf in a car exhaust catalyst and 36 laboratories from 16 countries participated. Figure 1 presents the participants' results for Pt in this matrix, grouped according to the self-declared degree of experience for this type of analysis.

Table 1 presents the IMEP rounds, which IRMM will launch in the near future. More detailed information and registration documents will be sent to the interested people as soon as practical details are available.

Figure 1. IMEP-11 participants' results for Pt in a car exhaust catalyst. The results are grouped according to the self-declared degree of experience of the laboratory for this type of analysis.

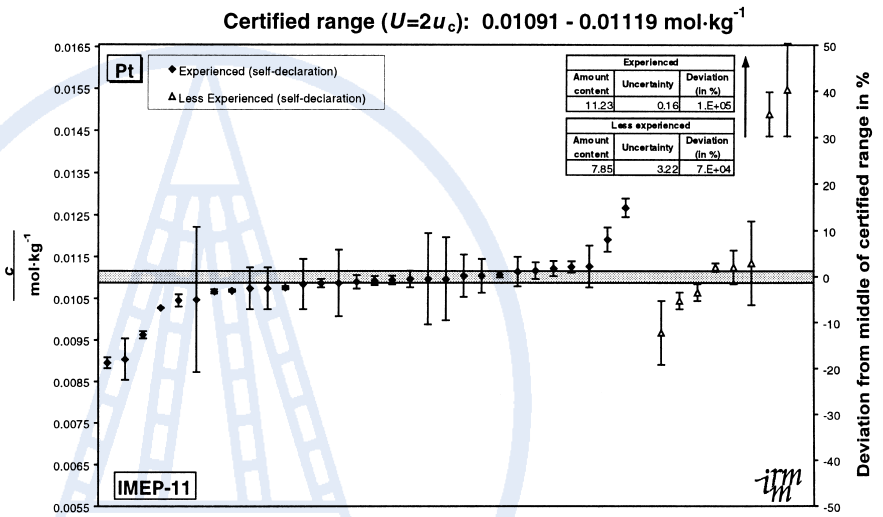

Table 1: IMEP rounds planned to be launched in the near future

IMEP Round	Title	Timing
IMEP 12	Trace elements in water	2000-2001
IMEP 15	$^{10}B/^{11}B$ isotope ratios in water	2000-2001
IMEP 16	Pb content and Pb isotopic composition in Wine	2000-2001
IMEP 17	Trace and minor constituents in human serum	2001-2002
IMEP-18	S in fuel	2001-2002
IMEP-19	Cd in Rice	2001-2002

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PRACTISIONERS' REPORT

ESTABLISHING TRACEABILITY SYSTEM YIELD TO FRUITFUL RESULTS IN CLINICAL CHEMISTRY

The traceability measurement system for sodium (Na), potassium (K) and chloride (Cl) concentration measurements in serum was a build-up based on rationality of chemical analysis in Japan (Fig.1). The novel certified serum reference material (CRM) was mainly used in evaluation, correction and quality assurance (QA) of uncertainty from measured values in routine analysis by using ion selective electrodes (ISE) method.

In Japan an external quality assessment (EQA) program sponsored by Japan Medical Association (JMA) has been performed. The total number of participating laboratories approximates 3,000 per year. Fig. 2 shows the progress on the coefficients of variation (CV) with the passage of time for Na, K and Cl measured values as determined by the ISE method at all laboratories. It is clear that the introduction of the secondary serum CRM (in 1987) drastically improved CV values. In 1998, CV was 0.8% for Na, 1.2% for K and 1.4% for Cl, maintaining CV limits.

Since the uncertainty of measured values is not observed in this survey, it must be confirmed by a different procedure. The proficiency testing (PT) was started as a part of an EQA for Na, K and Cl measurements using the ISE method according to the protocol for PT by Japan Society of Clinical Chemistry (JSCC) on each Area Committee in Japan.

The frozen QA samples are distributed to participating laboratories. Immediately after delivery to each laboratory, the samples are measured (5 replicates) and their data reported. Such data are collected to obtain a bias (B), 95% confidence limits of the mean value and the measurement uncertainty (Cm). These results are then compared with allowance limits to evaluate commutability.

In a PT for K organised by Tsukuba University and Ibaraki Association of Medical Technologists in Ibaraki area in 1998, a target value of K (for high level) was 4.96 ± 0.01 mmol/L. In this study, 65 out of 66 participating laboratories satisfied the Cm allowance limit.

In this way, an evaluation system establishing target values and obtaining the difference from the target values has been carried out for each laboratory per area throughout Japan. Uncertainty of measured values for Na, K and Cl are secured in this way in Japan.

Figure 1. Accuracy-based Measurement System for Na, K and Cl Concentration Measurement in Serum

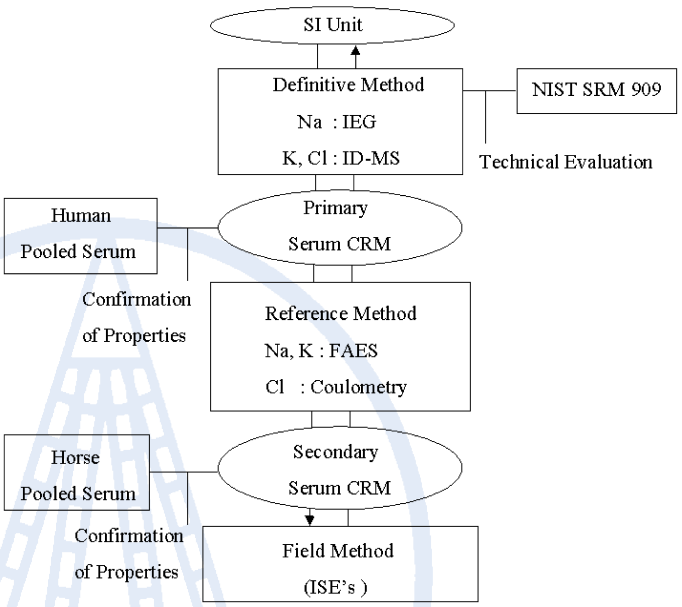
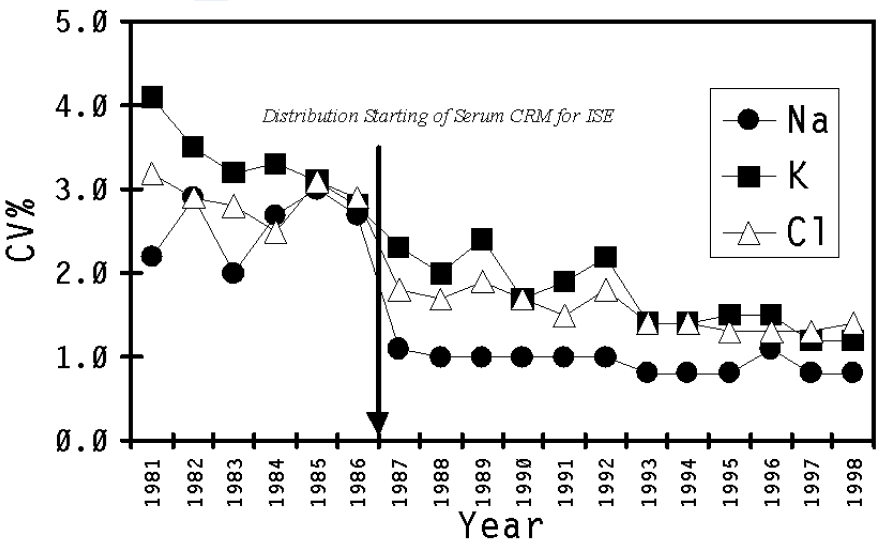


Figure 2. Progress of CV Value for Na, K and Cl Measurement by ISE Method in EQA of Japan Medical Association



CITAC '99 Japan Symposium

PRACTICAL REALISATION OF METROLOGY FOR THE 21ST CENTURY

Tsukuba, Japan, 9-11 November 1999

The CITAC '99 Japan Symposium was part of CITAC's policy to organise special events for the development and promotion of the concepts and ideas, which CITAC is concerned about.

The Symposium was organised by the Japanese members of CITAC Working Group in collaboration with the Japan Society for Analytical Chemistry (JSAC) and the National Institute of Materials and Chemical Research (NIMC) at the Tsukuba International Congress Center. On the third day of the Symposium the CITAC working group meeting took place in the same venue.

Approximately 300 scientists actively participated in the Symposium, the majority were Japanese but also scientist attended from other Asian, European, Australian, South and North American countries. Participants from industry, governmental organisations, national metrology institutes, international organisations, universities and research establishments were present, contributing to the high scientific level of the event.

The scientific programme of the Symposium consisted of three theoretical sessions, four workshops and two poster sessions. Two of the theoretical sessions were dealing with "Reference Materials" presenting the North American and European approaches in the first session and the Japanese approaches in the second. The third theoretical session was treating "Traceability in Chemical Measurements" where key contribution for the development of these concept were presented.

The four workshops consisted of theoretical introductions (three lectures in each workshop) followed by discussion with the audience dealing with very important and interesting subjects. In the "Laboratory Accreditation" workshop the Australian, North American and Japanese approaches were presented whereas the European, Japanese and North American approaches introduced the "Quantification of Uncertainty" workshop. The "Proficiency Testing" workshop featured Australian, Japanese and European approaches and the last workshop on "Education and Training" a European, a Japanese and another Asian approach were presented. All four workshops attracted the full attention of the Symposium participants and resulted in very fruitful discussions during and after the workshops.

The two poster sessions were very impressive. Mainly Japanese scientists, whilst covering a large variety of chemistry subjects and contributing to the high scientific level of the symposium, presented more than 100 posters.

The meeting of the CITAC working group members was held on the third day of the symposium. The major outcome of the meeting was the decision to proceed quickly with a new CITAC "position paper" on "traceability" which will be prepared from a drafting committee before PITTCON 2000 where it will be revised and voted on by CITAC members. Moreover CITAC decided to organise a workshop concerning the implementation of ISO 17025 during PITTCON 2000 (more details elsewhere in this issue).

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CITAC '99 Japan symposium, in session

CITAC working group meeting participants, Tsukuba, November '99

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