CITAC news
Co-Operation on International Traceability in Analytical Chemistry
JANUARY 2001

Foreword by the chairman

Early every year is the time to find the CITAC Newsletter in the mailbox and 2001 is particularly significant in terms of laboratory work as the transition period from Guide 25 to ISO 17025 is in its "hot" stage. CITAC has developed for the chemical community documents regarding two central principles highlighted in ISO 17025: traceability and uncertainty.

For traceability a short document was issued that emphasizes the need of several standards and reference points, both chemical and physical, for a single type of measurement; the role of validation to move from a traceability of standards to a traceability of our own results; and, the obvious fact that the certification of reference materials comes through analogous measurements as practiced by laboratories. A consequence of this insight appears to be that the requirement of validations applies in more stringency to certification campaigns but is otherwise not dissimilar to the validations in general. It was through this reference to ordinary laboratory operations that the mode of achieving traceability of results became more visible to all of us involved in the casting of this paper. This is the place where I want to thank for all thoughtful input CITAC enjoyed along the way. In the meanwhile a joint group of CITAC and EURACHEM is trying to expand the view and perhaps enrich it with "practical examples".

For uncertainty, a joint effort of CITAC and EURACHEM produced a revised Guide on Uncertainty in Chemical Measurement. Here the practical experience with the 1st edition issued in 1995 led to the incorporation of more examples, both simpler and more sophisticated than original ones; to a more coherent presentation format of these examples in terms of tables and figures that is meant to be a model for implementation in the laboratories; and, to the enhanced reference to the links between results from validation studies and the estimation of measurement uncertainty.

These two guidelines have received a warm welcome by the community. You find information on their availability as well as that of our other two publications on accreditation on page 10 of this Newsletter.

For the current membership you find information at the end of the Newsletter. This is the place to thank all leaving member for their individual contributions:

Prof Mauricio Noguire Frota (INMETRO, Brazil), Dr Pan Xiu Rong (NRCCRM, China), Mr Scott Coates (AOAC International, USA) and Mr Peter Unger (A2LA, USA).

And I want to welcome officially all new members of CITAC who all add enormous experience and talent to our group: Dr Sandra Hart (AGAL, Australia), Dr Zhao Min (NRCCRM, China), Dr Ilya Kuselman (NPL, Israel), Dr Steve Ellison (LGC, UK), Dr Martin Milton (NPL, UK), Dr Al Pohland (AOAC International, USA) and Dr Warren Merkel (A2LA, USA).

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International approval for new Chemists' Measurement Uncertainty guide

The European analytical measurement forum EURACHEM, working in collaboration with its world-wide analogue CITAC, has approved the publication of the second edition of its internationally recognised guide *Quantifying Uncertainty in Analytical Measurement* (QUAM). With the increased emphasis on measurement uncertainty in the new Accreditation standard ISO 17025, this new EURACHEM/CITAC guide is a 'must have' for analytical laboratories accredited to ISO 17025 (formerly ISO Guide 25, or UKAS M10 in the UK).

The guide is an interpretation for analysts of the widely accepted ISO *Guide to the expression of Uncertainty in Measurement*. This second edition of this guide was prepared by the EURACHEM Measurement Uncertainty Working Group in collaboration with members of CITAC and AOAC International. It is a comprehensive revision of the first edition, with much important new material. Crucially, for analysts, it covers the estimation of measurement uncertainty form method validation data such as reproducibility and in-house precision studies, recovery tests on certified reference materials, and ruggedness/robustness studies. This is particularly important with the new ISO 17025 standard's acceptance of validation data as a source of uncertainty information.

The revision has taken comments and input from two major international workshops involving over 40 nations world-wide, as well as seeing wide circulation in draft form. With its CITAC input, the second edition is likely to see world-wide use and acceptance, and translations into at least three other languages are already under way (QUAM 1, the first edition, saw at least five European and East European translations and sold world-wide in it English edition).

There are many changes in the new edition. A new section on Analytical Measurement and Uncertainty covers the process of method validation and conduct of experimental studies to determine method performance and their relationship to uncertainty estimation, and notes the importance of traceability. The chapter on uncertainty estimation in the previous guide has been considerably expanded and split into four separate chapters, dealing with the four steps involved in estimating uncertainty. Step 1 deals with the specification of the measurand, Step 2 with identifying the uncertainty sources, Step 3, which has been considerably expanded to cover the use of existing method validation data, deals with quantifying the uncertainty and Step 4 covers the calculation of the combined uncertainty. The examples have been completely revised and new ones added; sections on dealing with concentration-dependent uncertainties and uncertainty near detection limits have been added, and the extensive tables on specific sources of uncertainty have been updated.

A printed copy will be available in due course from the EURACHEM secretariat and from LGC's Office of Reference Materials. The guide is available, in full, for free download in Adobe PDF format on the EURACHEM website as http://www.vtt.fi/ket/eurachem/quam2000-p1.pdf.

Steve Ellison
LGC, UK

Note from the editor:
The CITAC members Pan Xiu Rong (NRCCRM, China), M. Salit (NIST, USA), A. Squirrell (NATA, Australia) and K. Yasuda (Hitachi Ltd, Japan) contributed on behalf of CITAC to the preparation of the above guide.

Eurachem/CITAC Uncertainty Guide in Swedish

The Eurachem/CITAC Guide, Quantifying Uncertainty in Analytical Measurement 2nd Ed., is available in printed form in Swedish. The decision to translate the Guide was taken by Eurachem Sweden already in the beginning of 1999. At that time, the Swedish accreditation body (SWEDAC) had just issued a policy document on measurement uncertainty. The policy stated that accredited laboratories should evaluate uncertainty according to the new international guidelines, e.g. those from ISO, Eurachem and EA. The work with implementing the new concepts should be done by the end of 2000. So far, Swedish laboratories had reported uncertainties mainly based on observed variation in control charts, i.e. an intermediate precision.

The release of the Swedish version in August 2000 was possible only due to an extensive communication with the Eurachem Uncertainty WG. By the end of last year, around 500 copies had been distributed. So far the Guide has been used in education internally at SP, in a training course for technical assessors and in three courses with participants mainly from water and industrial laboratories. Our, although limited, experience shows that the Guide can be used fairly easy when laboratories evaluate uncertainty from standardised Swedish methods. Those methods are often very detailed and contain large fragments that can be redistributed to comply with Steps 1-4 in the "process". We noted however that in many cases, the mathematical expression given in standards must be extended to enable correction for uncertainties associated with sampling and sample preparation. The most difficult part for the participants is still associated with mathematics and uncertainty propagation. Because control charts are very much used in Swedish laboratories, it is important that information from such can be used when uncertainty is evaluated. The concept demonstrated in the Guide for using cumulative precision components in the cause-and-effect diagrams will hence be very useful.

The Swedish Guide is available from SP as SP Report 2000:17, ISBN 91-7848-782-

Ulf Örnemark
SP, Sweden

Chemical Metrology in New Zealand

Why a chemical metrology programme in New Zealand?

A fundamental change has taken place in international trade relations in recent years. Shielding a nation's economy against imports, with protective duties and tariffs, is a model rapidly being phased out. Instead, international trade relies on internationally agreed standards for the quality of goods and services being traded, and mutually agreed systems to measure this quality. Examples of these developments are: activities on the international stage to harmonize national accreditation systems, the negotiation of the Mutual Recognition Arrangement (MRA) and the recently released international standard ISO/EEC 17025, which puts a far stronger emphasis on the demonstration of competence and comparability than its predecessor documents.

The Measurement Standards Laboratory of New Zealand (MSL) is New Zealand's National Metrology Institute. MSL has recognised the international demands on trading countries to maintain an appropriate measurement infrastructure and launched a new programme to develop a high-level measurement infrastructure for chemistry. The purpose of this programme is the establishment of a national reference system to provide national traceability and international comparability of chemical measurements.

What is the present situation in New Zealand?

New Zealand's economy is strongly dependent on exports, mainly food products, as well as metals, wood and wood products. A second cornerstone of New Zealand's economy is tourism. Therefore, special care is taken to ensure a clean environment, combined with an intensive and costly monitoring system. All these activities are underpinned by analytical chemical measurements and illustrate the importance of an appropriate chemical measurement infrastructure for New Zealand.

The measurement infrastructure in New Zealand presently consists of a number of autonomous organisations with specific and separate responsibilities. There is no formal structure although these organisations cooperate closely to ensure that the measurement system is coherent. MSL has the primary responsibility for the provision of physical measurement standards. Its activities cover the full spectrum of measurement science and technology activities from pure research, such as the measurement of fundamental constants, to applied technology, such as the commercialisation of measurement instrumentation. Calibration services provide the mechanism for the physical measurement standards maintained by MSL to be disseminated throughout New Zealand.

All these activities in the physical measurement area had no counterpart in the chemical measurements area until MSL launched its new program on chemical metrology a year ago.

What are the goals of the New Zealand chemical metrology programme?

The chemical metrology programme has to serve both, national needs to make reliable and comparable measurements, and to contribute to the international measurement system. Thus, the ultimate goal of the chemical metrology programme at MSL is the establishment of an infrastructure for chemical measurements based on conventional metrological principles. Furthermore, the programme will raise the awareness for chemical metrology and encourage a wider usage of reference materials, reference methods, and the participation in proficiency programmes. An important issue is education in chemical metrology. Therefore, MSL is offering extensive information on chemical metrology, and will encourage universities to include topics like uncertainty, traceability, reference materials and proficiency programmes into the curriculum.

A national measurement infrastructure in each country must take into account national needs and requirements in chemical metrology. Therefore, a careful and extensive collection of information and data has to be the basis for all decisions and activities. At present, MSL is collecting information in several ways. The core activity is a survey of more than 200 New Zealand testing laboratories. The collection is completed by extensive consultancy through experts in the main areas of testing activities, and personal contact with laboratories. Analysis of the information is to be completed by mid 2001, and used to plan a national chemical reference laboratory and establish a technical infrastructure for chemical metrology in New Zealand.

New Zealand is a small country and has limited resources. Accordingly, the international activities to demonstrate competence (key comparisons) have to be harmonised with the national activities. So far, it is not clear in which area of chemical testing, work will be started first. However, instrumentation, equipment and education of staff of the future national chemical reference laboratory have to satisfy both activities. This means that key comparisons and work in regard to the infrastructure, e.g. the development of reference materials or the organisation of proficiency programmes, have to be located in similar areas of chemical analysis.

It is MSL's intention to start as soon as possible with practical laboratory work, and the establishment of a virtual laboratory will be the short-term strategy. A network will be set up based on the already existing competence and excellence of established laboratories. The research activities will be coordinated and connected by a secretariat located at MSL. The secretariat will, also, provide other administrative activities like representation, marketing and promotion, and advisory, educational and training services. MSL hopes eventually to have its own laboratory facilities to supplement the capabilities of the virtual laboratory or if necessary partially replace them.

The MSL chemical metrology programme will be complementary to other international programmes. Accordingly, the provision of New Zealand laboratories with already available data and information is one of the first goals of the chemical metrology programme. Several databases have already been built up for reference materials, proficiency programmes, literature and a collection of organisations dealing with chemical metrology. These soon will be available via MSL's web-page [http://www.irl.cri.nz/msl/].

Because of the international background of the programme MSL is seeking cooperation and strategic collaboration with other NMIs and organisations. A first cooperation, to exchange knowledge and experience, is going be established with MSL's Australian counterpart, National Analytical Reference Laboratory (NARL). This, also, is the background of a binational forum, the Australian - New Zealand Chemical Standards Cooperation (ANZ CSC), established to exchange information on chemical standards and to help to coordinate chemical standards work within the contributing organisations.

The national traceability scheme is considerably influenced by the accreditation system; the requirements of the national accreditation authority determine the effort laboratories put into traceability. Regular contact and close cooperation with the local accreditation authority International Accreditation New Zealand (IANZ) has therefore been established.

Conclusions

In contrast to physical metrology, chemical metrologists have just begun to tackle the problems of establishing an international system to achieve traceability and comparability of measurement results. There is a huge number of reference materials lacking and unsolved problems. New Zealand's contribution to fill that gap can only be limited and cover small, well-defined areas. Nevertheless, New Zealand is seeking integration and involvement in the international metrological community and is willing to contribute as much knowledge as possible. Accordingly, MSL invites other organisations for collaboration and cooperation to exchange understanding and to learn from each other's experience of building a chemical metrology structure.

Marcus Krapp
MSL, New Zealand

Important step towards international comparability and recognition of chemical measurement results: the CIPM Mutual Recognition Arrangement

With the globalisation of trade and industry continuing, reliability of and confidence in measurement results are of increasing importance, also in the field of chemical analysis. Measurement results are directly and indirectly involved in almost every aspect of daily life and play an essential role in border-crossing exchanges of goods and services. As an important prerequisite for confidence in measurement results the International Committee of Weights and Measures (CIPM) of the Metre Convention has drawn up the Mutual Recognition Arrangement (MRA) for national measurement standards and measurement and calibration certificates issued by national metrology institutes (NMIs). This CIPM-MRA has been signed by the great majority of the NMIs of the Metre Convention member states and put into force by the 1999 General Assembly of the Metre Convention. Its central aim is to extend and consolidate worldwide confidence in measurement results and to provide governments and other parties with a safe technical foundation for wider arrangements related to international trade, commerce and regulatory affairs. The field of chemical measurements is included in the MRA through the CIPM Consultative Committee for amount of substance (CCQM).

Confidence and hence mutual recognition is based on statements on the equivalence of national measurement standards which in turn are based on the results of so-called key comparisons, which are carefully selected to be representative of the subject field in question and carried out by the NMIs including national chemical institutes. The degree of equivalence is the difference between a participant's result and a reference value which is finally adopted by the responsible Consultative Committee (CC) as the result of the exercise, including the overall uncertainty of this difference.

Recognition of calibration certificates issued by national institutes for calibrations carried out using the national measurement standards requires additional evidence of competence, for example in the form of a third-party accreditation according to the ISO IEC Standard 17025 or a peer review. Two kinds of key comparisons are distinguished:

- CIPM key comparisons which are carried out by the CCs and in which the most experienced national laboratories (and laboratories nominated by NMIs) take part.
- RMO key comparisons which are carried out by the **R**egional **M**etrology **O**rganizations (like EUROMET, SIM, APMP, …), including one or more laboratories which participate in the corresponding CIPM key comparison and can, therefore, act as disseminators.

In this way all national laboratories responsible for establishing traceability to national standards and interested to join the MRA can take part in key comparisons and ascertain their degree of equivalence with others, with a (relative) minimum of effort and outlay.

As a result, the national references and the measurement capabilities of the national institutes will reach a higher level of mutual recognition and trustworthiness. Whereas this process is mainly an extension and consolidation of mutual recognition already existing in metrology in general, it is the first attempt at establishing a globally recognised reference framework for chemical measurements.

A central problem of the practical implementation of the MRA in the field of chemistry is the selection of the key comparisons which are to form the technical basis. These comparisons must be selected so that they are representative of the vast variety of chemical measurement tasks and at the same time restricted in number. It is obvious that it will not be possible to create a complete set of key comparisons covering everything in chemical analysis. It is important that the major fields with high traceability requirements are covered and that the number of key comparisons is kept at a manageable size.

The CCQM has identified the following priority areas according to the worldwide demand for traceability of chemical measurements:

- Health care
- Food
- Environment
- Advanced materials
- Commodities
- Forensics.

Biotechnology is appearing at the horizon.
CIPM key comparisons, mostly preceded by so-called pilot studies, have so far been carried out in the following subfields of the above priority areas:

- Diagnostic markers in clinical chemistry
- Pesticides in natural matrix
- Toxic elements in natural and drinking water
- Toxic elements in sediments
- Automobile exhaust emission surveillance
- Air quality surveillance
- Calibration solutions for elemental solutions
- pH measurement
- Breath alcohol analysis for drink-and-driving legislation,

representing a total of about 10 key comparisons. The list of key comparisons is now being extended and completed. It can be expected that it will finally contain more than 50 key comparisons which will be carried out periodically (every three to five years) in order to establish and maintain global comparability in the most important areas of chemical analysis.

In several areas, the CIPM key comparisons are now followed by RMO key comparisons.

The results obtained so far in key comparisons show that there is already a group of national laboratories which are capable of establishing an international reference framework for chemical measurements with sufficient accuracy, even if complicated matrices are involved, although a lot of difficulties must still be overcome.

The results of all key comparisons are collected in appendix B to the MRA and are publicly accessible via the Internet in a database maintained at the Bureau International des Poids et Mesures (BIPM) at www.bipm.fr. The database contains the key comparison reference value (KCRV) of each key comparison, established by the responsible CC, as well as its uncertainty, the degrees of equivalence of the participants with respect to the KCRV (difference and uncertainty of the difference), and the mutual degrees of equivalence of the participants.

Appendix C to the MRA specifies the calibration and measurement capabilities (CMSs) which the national institutes provide to customers, either directly or indirectly, via an appropriate dissemination mechanism. The entries in the database, (accessible via the Internet at a later date), are closely linked to appendix B which, in addition to the key comparisons, contains results of other categories of comparisons. After appendix B has been filled up with results of comparison measurements, at least a large fraction of the entries in appendix C will be supported by "hard facts" necessary to underpin the trustworthiness of the CMC claims. For those entries for which no link exists to appendix B, other evidence of the provider's competence as mentioned already will be requested by the RMOs and the Joint Committee of the RMOs and the BIPM (JCRB) that assess the entries before approval is given to include them into appendix C.

Appendices B and C to the MRA and the information contained in them form a transparent system which reflects the metrological capabilities and the degrees of equivalence of the national institutes. It is accessible to all interested parties, e.g. accreditation bodies, regulatory authorities or trade partners, that want to check the credibility of traceability claims of measurement results which are important in order to judge the quality of goods and services. The CIPM-MRA is an important step towards international comparability and recognition of measurement results, but it is obvious that comparability and mutual recognition are required not only at the national standards level but for the entire dissemination structures in order that the CIPM-MRA can be fully utilised. This holds in particular for chemical measurements.

First experience with appendices B and C to the MRA has already been gained in the gas analysis sector of chemical analysis. The results of the first CCQM key comparison have been entered into appendix B in database. The RMOs have reviewed the submitted CMC entries of their members and forwarded those to the JCRB for approval and inclusion in appendix C, which have been declared reliable. Within EUROMET, for example, the subject field METCHEM (formerly Amount of Substance), and in this subject field the working group for gas analysis carried out this task. Two pre-reviewers were appointed by the METCHEM rapporteur for every CMC provider, and their findings were discussed at a meeting chaired by the working group convener. Considerable revisions were necessary before the entries ((230) could be recommended as reliable to the EUROMET Chairman who then forwarded them to the JCRB. Mutual checks among the RMOs will follow.

The next step will be submission of the CMC entries for the other subfields of chemical analysis: inorganic, organic and electrochemical analysis. The deadline for submission to the JCRB is 25 May 2001.

After conclusion of the approval procedure a complete set of mutually recognised calibration and measurement capabilities of those national institutes, which provide the references for the traceability of chemical measurements and are signatories to the CIPM-MRA, will be available for the first time.

Wolfgang Richter
PTB, Germany

International Laboratory Accreditation Cooperation MRA

In the last CITAC News (February 2000) I presented an overview of the developments in relation to an ILAC Mutual Recognition Arrangement. I can now report that this Arrangement actually comes into force on 31 January 2001 and am pleased that CITAC, as a stakeholder member of ILAC, was able to make an active contribution. An edited revised-version of the 2 November 2000 media release is attached.

Media Release

Signing of ILAC International Mutual Recognition Arrangement to Enhance Trade

An international arrangement, signed in Washington, DC, on 2 November 2000, will enhance the acceptance of technical data accompanying goods crossing national borders. The Arrangement, which involves 37 member bodies from 28 economies represented at the General Assembly of the International Laboratory Accreditation Cooperation (ILAC), means that goods tested in one country by a laboratory that is accredited under a signatory to the Arrangement, will be accepted by other signatories. This is a major step towards reducing or eliminating the need for re-testing of the goods by the importing country.

The Arrangement enters into force from 31 January 2001.

Belinda Collins, Chair of ILAC, noted the significance of the signing, "For many years, the retesting of goods by an importing country has been considered as a major technical barrier to trade. The World Trade Organization (WTO) identified such technical barriers as a major concern to world trade since the mid-1970s. Such barriers can not only add significant cost to goods entering a country, but can also delay, and in some cases prevent, the goods being accepted by foreign markets."

Dr. Collins further explained that "ILAC has been working towards overcoming these technical barriers for the last two decades by encouraging the development of regional recognition arrangements culminating in today's global recognition arrangement among representative bodies in each country. This will facilitate the acceptance of goods already tested by an accredited laboratory. Thus, goods tested in one country should enjoy easier access to foreign markets participating in the Arrangement."

The key to the Arrangement is the developing network of accredited testing and calibration facilities around the globe that are evaluated and recognized as being competent by specific authorities, known as laboratory accreditation bodies. These bodies are located in many economies and many of them participate in ILAC.

A cornerstone of the new Arrangement is the utilization of existing or developing regional arrangements established in the Americas, the Asia Pacific region, Europe and Southern Africa. The bodies participating in these regional arrangements are responsible for maintaining the necessary confidence in accreditation bodies from their region that are signatories to the new ILAC Arrangement.

Mike Peet, Chair of the ILAC committee that developed the new Arrangement, explained the basis for the Arrangement's implementation by the international community: "Now that the Arrangement is in place, the next crucial step is for governments to take advantage of this Arrangement by using it to further develop or enhance trade agreements."

"There is now a firm foundation in place for manufacturers and exporters that have their goods tested by accredited laboratories to enjoy greater market access, less costs associated with re-testing, and overall greater competitiveness in global markets", he explained.

Established in 1977, ILAC is the peak international forum for the harmonization of laboratory accreditation procedures as a means of reducing technical barriers to trade, and the promotion of laboratory accreditation as a mechanism to enhance confidence in testing and calibration facilities, both domestically and internationally.

The following economies will participate in the Arrangement:

Australia
Belgium
Brazil
Canada
People's Republic of China
Czech Republic
Denmark
Finland
France
Germany
Hong Kong, China
India
Ireland
Italy
Japan
Republic of Korea
The Netherlands
New Zealand
Norway
Singapore
South Africa
Spain
Sweden
Switzerland
Chinese Taipei
United Kingdom
United States of America
Vietnam

For further details on the ILAC Arrangement please contact the ILAC Secretariat on ph: +612 9736 8374, fax: +612 9736 8373 or e-mail; ilac@nata.asn.au or visit the ILAC website at http://www.ilac.org.

Allan Squirrell
NATA, Australia

The Role of Reference Standards in the Sydney 2000 Olympic Games Drug Testing Program

Planning for the mammoth analytical chemistry enterprise that forms an integral part of every modern Olympiad began with the Sydney bid submitted in 1994, for the 2000 Games. The Host City is responsible for providing the drug-testing program needed to detect a wide range of performance enhancing drugs in compliance with the rules of the International Olympic Committee (IOC). The Sydney Organising Committee for the Olympic Games (SOCOG) was able to take advantage of the existing infrastructure for sports drug testing in Australia. The Australian Sports Drug Testing Laboratory (ASDTL), part of the Australian Government Analytical Laboratories (AGAL), obtained IOC accreditation in 1990 and together with the Australian Sports Drug Agency is the lynchpin of Australia's fight against drugs in sport.

Planning began in earnest in 1996 to enable ASDTL to expand to meet the demands expected of it in September 2000. Typically ASDTL processes approximately 6000 samples per year with a 10 to 14 day turnaround (reporting) time. However during the Olympic Games some 2000 samples were expected with results required in 24 to 48 hours. This number was ultimately increased, at the last minute, to include a further 700 out-of-competition tests and 300 blood tests for erythropoietin (EPO). The laboratory was thus required to compress a good part of a single year's work into two weeks of intensive, heavily scrutinised effort.

Each urine sample submitted for testing undergoes screening for a range of drugs including stimulants, narcotics, anabolic agents including steroids, diuretics, peptide hormones, and some sports -blockers in accordance with the IOC list of banned substances. Each screen uses different extraction processes to concentrate and purify the urine with a final measurement step undertaken using gas chromatography – mass spectrometry wherever possible. In order to cater for the large number of samples in such a short time it was obviously necessary to massively expand the laboratory both in physical size, number of instruments and number of trained staff. The size of this increase can be seen in **Fig 1**, which shows the extent of the capacity. Staff numbers increased from 22 to 70 by recruiting staff from within the parent organisation and recruiting willing volunteers. Thirteen international scientists and colleagues joined the team.

Figure 1

Instrument	Number required	Screen
GCMS	8	Steroids
GCMS	4	Diuretics
GCMS	4	Narcotics
GCMS	1	Endogenous steroids
GCMS	2	Stimulants
GCMS	1	Confirmation
GC-NPD	4	Stimulants
HPLC	4	Diuretics
HRMS	4	Steroids
CIR	2	Endogenous steroids
LCMS	1	Diuretics confirmations
MSMS	2	Confirmation
Automated SPE	5	Sample preparation
Sample Dispensing units	3	Sample preparation
Blood EPO	2	Blood Parameters
Blood EPO	1	EPO, urine EPO, HCG
Blood EPO	1	sTfr
Urine EPO	1	Camera
TOTAL	**50**	

Figure 2

Standard Type	Number of Reference Materials Produced by NARL
Deuterated Steroid	16
Steroid metabolite	36
Steroid Conjugate (glucuronide/sulphate)	32

While it was important to expand the size of the laboratory to cater for such enormous increase in effort it was also important to ensure it was done with full maintenance of quality of measurement. This was assisted by the availability of reference materials for use in the analytical process. This allowed all aspects of the processes to be monitored using deuterated standards and to ensure only valid results were reported.

When a banned drug is detected using an initial screening protocol its presence must be confirmed using gas chromatography - mass spectrometry. This entails the direct comparison of retention time and mass spectral information obtained when the sample and a clean urine spiked with the drug are run in the same batch. For many drugs which are excreted unchanged there is less problem in obtaining a standard whose identity is well characterised. However many anabolic steroids undergo extensive metabolism and the only source of these metabolites has frequently been a reference urine from volunteers who have taken the drug. It was obviously preferable to have pure standards of the major metabolites available to confirm doping cases. In 1996 a program was initiated by ASDTL to have the

Curator of Standards Section of AGAL begin the synthesis, purification, characterisation, and certification of a number of steroid metabolites. The aim was to produce the compounds in sufficient quantity so that they could be made available as certified reference standards to all those involved in the fight against doping. This involved both in-house synthesis and the use of external contractors. In 1997 the National Analytical Reference Laboratory (NARL) was established within AGAL. The Curator of Standards section then became an integral part of the Pure Substance Reference Materials Team (PSRMT) of NARL and the steroid synthesis program became one of NARL's major activities. As well as preparing certified standards for the metabolites, stable isotopically labelled compounds were prepared which could be used as surrogates in every sample. In this way the many analytical processes involved could be monitored to ensure that the entire process was performing effectively. For example in the steroid screen six deuterated analogues are added to each sample to check various aspects of the analysis. This allows processes such as enzyme hydrolysis, recovery, retention time and GC column suitability to be monitored for every sample.

NARL has taken the reference materials program further by obtaining accreditation under ILAC G12:2000 Guidelines for the Requirements for the Competence of Reference Material Production from the Australian accreditation body NATA. This was the first such accreditation obtained internationally for reference material production. Under this accreditation the extensive number of standards shown in **Fig 2** have been prepared and are being offered to anti-doping laboratories for use in their testing programs.

During the 2000 Games the staff from NARL and AGAL's Research and Development section worked with ASDTL so that a pool of experts were deployed to ensure that the analytical work was performed to the required standards.

Thanks to extensive planning and the magnificent efforts of all AGAL staff the Olympic drug testing went smoothly. The Sydney 2000 Olympic Games were declared to be the best ever and those involved in the drug testing can feel partly responsible for the accolade. The analytical reference standards provided by NARL made a significant contribution to this achievement. The collaboration serves as a shining example of how a reference laboratory (NARL) and a working laboratory (eg ASDTL) can together produce an optimum outcome for the stakeholders they serve.

G. Trout, R. Kazlauskas and S. Westwood
AGAL, Australia

CITAC position paper on: Traceability in Chemical Measurement

During the 11th CITAC meeting (11th November 1999, Tsukuba, Japan), CITAC members considering the enhanced importance of traceability in ISO 17025 agreed to prepare a paper concerning traceability in analytical chemistry. The drafting group (consisted of 5 CITAC members) worked in close interaction with all CITAC members and it was possible to produce a draft document by February 2000. The draft document was extensively discussed in the 12th CITAC meeting (14th March 2000, New Orleans, USA) and it was accordingly endorsed as the "*CITAC position paper on Traceability in Chemical Measurement*". The full paper is published in the Accreditation and Quality Assurance journal (Volume 5, Number 9, September 2000, pages 388-389), the ILAC news (No 17, September 2000, pages 21-22) and the BAM bulletin (No 23, Autumn 2000, pages 22-23), whereas it was communicated to interested international bodies all over the world such as CCQM, EURACHEM, EEE-PT-WG and others. The paper can be downloaded in the CITAC website (http://www.vtt.fi/ket/citac/) and the executive summary follows.

Executive Summary

Traceability is a key element in the mutual recognition of testing results. This explains the renewed emphasis on this topic particularly in ISO 17025.

For chemical measurements this involves the need for stated references and a clear uncertainty statement, which should be derived from an uncertainty budget with due regard to the fact that several references, such as amount of substance, mass, volume, time, temperature are generally involved in a single analytical procedure contributing distinct, but different portions to the overall uncertainty.

This uncertainty budget must not only take into account the uncertainties of all the references used in connection with the analytical procedure, but also the uncertainties from the operation of the laboratory procedure as documented in the validation report. The uncertainty from the measurement procedure is frequently much larger than the uncertainties carried by the references.

I. Papadakis
EC-JRC-IRMM
CITAC Secretary

NIST Centennial Celebration at PITTCON 2001

The National Institute of Standards and Technology (NIST) will reach a historical milestone in the year 2001 when we celebrate the 100th anniversary of our founding in 1901 as the National Bureau of Standards. The general theme for all of the year-long NIST Centennial celebration activities is "NIST at 100 -Foundations for Progress." The official series of activities will commence during the week of March 5 at the NIST Gaithersburg campus (http://centennial.nist.gov/summary.html). PITTCON 2001 will also include a two-day symposium entitled "NIST Contributions to Chemical Measurement Science and Technology: A 100-Year Perspective." The symposium at PITTCON will be comprised of four half-day plenary sessions plus a poster session:

- **Sunday, March 4** (1:30 – 5:00 PM)
  NIST Activities in Chemical Measurement Science: A Historical Perspective
- **Monday, March 5** (8:30 AM – Noon)
  Impact of NIST Chemical Measurement and Standards Programs: Customer Views
- **Monday, March 5** (1:30 – 5:00 PM)
  Impact of NIST Activities in Chemical Measurement Science: International Perspective
- **Monday, March 5** (5:30 – 7:00 PM)
  Mixer/Poster Session: "NIST Activities in Chemical Measurement Science and Their Impact on U.S. Industrial Competitiveness, Trade, and Environmental and Health-Related Decision Making"
- **Tuesday, March 6** (8:30 AM – Noon)
  Looking Ahead: Chemical Measurement Methods, Standards and Technology Needs for the Future in
  - Biotechnology
  - Health care
  - Manufacturing
  - Micro- and Nano-technology
  - Agriculture and food

Throughout these five events, invited speakers from around the world will discuss and illustrate NIST's past, present, and *projected future* research and service programs in chemical measurement science and their impact on industrial productivity and competitiveness, equity in trade, and environmental and health-related decision making.

NIST also will have expanded exhibit space for 2001. The exhibit will feature illustrations of our contributions to measurement science during its first 100 years in addition to our Standard Reference Materials, Standard Reference Data and related activities.

Please come and share in our 100-year celebration at PITTCON 2001.

Willie May
NIST, USA

The Eurachem/EQALM Workshop on Proficiency Testing, Borås, Sweden, 24 to 26 September 2000

Introduction

The 3rd Eurachem/EQALM Workshop "Proficiency Testing in Analytical Chemistry, Microbiology and Laboratory Medicine" was held at the Grand Hotel, Borås, Sweden between 24 and 26 September 2000. The workshop was organised by SP, the Swedish National Testing and Research Institute, and sponsored by LGC, IRMM, Kiwa NV and Bayer AG. The planning of the workshop, including the programme and content, was carried out by the Eurachem Proficiency Testing Mirror Group, which is chaired by Nick Boley of LGC. The workshop was primarily aimed at European Proficiency Testing organisers, participants and accreditation bodies but in reality there were over 130 delegates, from 26 countries in Europe, Asia, The Americas and Australasia.

Objectives

The main objectives of the workshop were as follows:

- To review developments in proficiency testing in the last 5 years
- To consider important potential developments in the future for proficiency testing
- To bring together the analytical chemistry, microbiology and laboratory medicine sectors to enable these sectors to learn from each other
- To promote recent initiatives in international aspects of PT

The organising committee also wished to stimulate discussion and involvement of all delegates in a friendly and relatively informal atmosphere.

Workshop Program

On Sunday 24th September the Workshop started with an introductory training course on a number of issues that would be covered in greater detail during the workshop. This was designed to introduce topics, in an uncontroversial format, too participants that may have limited experience or as a precursory warm up exercise to the following two days. Initially 50 participants had registered for the training course, which rose to nearly 80 attendees on the day. The main workshop commenced at 8:30am on the 25 September. The Chairman, Nick Boley of LGC, outlined the following two day programme, and defined the objectives of the workshop, before handing over the Claes Bankvall, President of SP to formally welcomed all delegates to Borås and the workshop. Ulf Örnemark of SP then gave a short address on behalf of the organising committee, followed by Ed de Leer of NMi, and the Chair of Eurachem, who welcomed all delegates on behalf of Eurachem.

The morning was occupied by 4 excellent keynote talks from:

- Alan Squirrell of NATA (Australia) on the Accreditation of PT Schemes
- Jytte Molin Christensen of NIOH (Denmark) on proficiency testing ion the Occupational Hygiene sector
- Keith Jewell of CCFRA (UK) on the problems facing proficiency testing in microbiology, and
- Jean-Claude Libeer of IHE (Belgium) on his experiences in External Quality Assurance in laboratory medicine

All talks were well received with a number of interesting questions and comments. The Chairman then introduced the Working Groups then briefed the delegates on their tasks. Seven Working Groups had been planned, each covering a different topic:

- Aspects of Proficiency Testing and Accreditation
- Proficiency Testing in Analytical Chemistry
- Proficiency Testing in Microbiology
- Proficiency Testing in Laboratory Medicine
- Measurement Uncertainty in Proficiency Testing
- International Harmonisation of Proficiency Testing
- Proficiency Testing's role in Chemical Metrology

Each Working Group was lead by a Chair and aided by an assistant chair. Preformulated questions, prepared by the Workshop chairman, were given to the Working Groups to aid their discussions. The composition of the Working Groups was approximately 70% delegates who had expertise or experience in the main topic of the group, and the remainder from other areas to provide a different view and promote cross-fertilisation.

All Working Groups had very lively discussions with a high level of participation, and the reports presented to plenary were of a high standard, and raised some interesting issues.

The second day started with a further four excellent keynote talks from:

- Leopoldo Cortez of IPQ (Portugal) on the work of the EEE Proficiency Testing Working Group
- Manfred Golze of BAM (Germany) on the EPTIS European on-line database of PT schemes
- Paul de Bièvre of IRMM (European Commission) on the Establishment of Acceptability Criteria for uncertainty in PT, and
- Adriaan van der Veen of NMi (The Netherands) on Uncertainty Evaluation on Proficiency Testing

The Working Groups then reconvened to discuss a further set of questions and topics prepared by the Workshop Chairman. The lively discussions continued and the reports from each Working Group were again of a high standard, promoting some searching questions.

The Workshop concluded with a closing summary from the Chairman, where he outlined many of the issues that had been highlighted during the two days, followed by a closing address by Jean-Claude Libeer on behalf of EQALM.

The workshop was extremely successful, meeting the objectives set at the outset. Many delegates went out of their way to express their thanks to the Chairman and the organisers for the quality and conduct of the workshop, and many more stated that it had been a very enjoyable experience from which they had gained a great deal of knowledge and understanding, as well as making some good professional contacts. Members of the Eurachem Executive Committee stated that the workshop was one of the most successful held

under their auspices. In response to many delegates, the issues raised by the Working Groups, and the Eurachem Executive Committee, it was decided that a follow-up workshop be organised in September 2002 to address many of the issues raised and build on the momentum of the workshop.

The issues raised at the workshop were summarised as follows:

- There is a need to harmonise the way PT schemes operate, and are used, but there is no desire for a uniform approach
- The issues of Measurement Uncertainty and traceability of Reference Values will become more important in the next few years, but not in all PT schemes
- There are some differences between PT in the analytical and clinical chemistry sectors, but many more similarities
- Microbiology is a special case!
- Accreditation or other recognition of PT scheme providers is developing rapidly throughout the world
- PT scheme must be fit-for-purpose
- More use must be made of electronic data entry, handling, reporting, with greater use of e-mail and the internet
- PT still has a large educational element
- The independence and impartiality of PT schemes from accreditation bodies and regulators is important

The Eurachem PT Mirror Group discussed the workshop at their meeting the following day and it was agreed that there was too much emphasis on the metrological aspects of PT from a largely theoretical or academic perspective, with insufficient thought given to the views of participating laboratories, to whom the PT providers must listen as part of their business relationship with their customers. These two strands can be in conflict, and the PT provider tends to get caught in the middle. For the next workshop, it was agreed that this balance needs to be redressed to some extent, and that more emphasis be placed on laboratories, their customers, accreditation bodies and regulators, in the use and application of PT.

The proceedings of the workshop, including the keynote talks, Working Group reports, Chairman's summary and the many papers and posters submitted, will be published in a special double edition of AQUAL in April 2001.

Steve Evans
LGC, UK

Avaliability of CITAC documents

In the past six years CITAC produced a number of documents (guides and papers) in the service of the chemical measurement community. Several colleagues requested on a number of occasions information about these documents. We are pleased to announce that all the CITAC documents are accessible for download or order via Internet. The table below summarises this information.

TITLE OF DOCUMENT	WEBSITE ADDRESS
CITAC guide 3 (revised, 2000): *Quantifying Uncertainty in Analytical Measurement, The Second Edition*	http://www.vtt.fi/ket/eurachem/quam2000-p1.pdf (download free of charge)
CITAC position paper (1999): Traceability in Chemical Measurement	http://www.vtt.fi/ket/citac/traceability.pdf (download free of charge)
CITAC Guide 2 (1998): *Quality Assurance for Research and Development and Non-routine Analysis*	http://www.vtt.fi/ket/citac/rdguide.pdf (download free of charge)
CITAC Guide 1 (1995): *International Guide to Quality in Analytical Chemistry: An Aid to Accreditation*	http://www.rsc.org/cgi-bin/fx.exe (place an order)

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