ISTITUTO SUPERIORE DI SANITÀ

6th Workshop

Proficiency Testing in Analytical Chemistry, Microbiology and Laboratory Medicine: Current Practice and Future Directions

Rome, Italy
October 5-7, 2008

ABSTRACT BOOK

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ISSN 0393-5620
ISTISAN Congressi
08/C8

Istituto Superiore di Sanità
6th Workshop. Proficiency Testing in Analytical Chemistry, Microbiology and Laboratory Medicine: Current Practice and Future Directions. Rome, Italy, October 5-7, 2008. Abstract book.
Edited by Brian Brookman, Antonio Menditto and Marina Patriarca
2008, xv, 102 p. ISTISAN Congressi 08/C8

Participation in Proficiency Testing (PT)/External Quality Assessment (EQA) Schemes is a key tool for analytical laboratories to demonstrate their competence to customers, accreditation bodies and regulators. This Workshop, the sixth organised by the Eurachem Proficiency Testing Working Group, addresses the status and needs of PT/EQA within the European Union and in developing areas of the world, enhancements in PT/EQA organisation and progresses in emerging fields as well as the end-user perspective.

Key words: Proficiency Testing, External quality assessment, Accreditation, Analytical laboratories

Istituto Superiore di Sanità
6° Workshop. Prove Valutative in Chimica Analitica, Microbiologia e Medicina di Laboratorio: Stato dell'Arte e Indirizzi Futuri. Roma, 5-7 ottobre 2008. Riassunti.
A cura di Brian Brookman, Antonio Menditto e Marina Patriarca
2008, xv, 102 p. ISTISAN Congressi 08/C8 (in inglese)

La partecipazione a programmi di prove valutative (Proficiency Testing, PT) o valutazione esterna di qualità (External Quality Assessment, EQA) è uno strumento cruciale che permette ai laboratori di prova di dimostrare la propria competenza a clienti, enti di accreditamento e organismi di controllo. In questo Workshop, il sesto di una serie organizzata dallo Eurachem Proficiency Testing Working Group, vengono discussi lo stato e le necessità di PT/EQA nell'Unione Europea e in aree del mondo in via di sviluppo, i miglioramenti nell'organizzazione di PT/EQA e i progressi in aree emergenti, dando anche spazio al punto di vista degli utenti finali.

Parole chiave: Prove valutative, Valutazione esterna della qualità, Accreditamento, Laboratori di prova

This event was supported by European Proficiency Testing Information System (EPTIS), International Laboratory Accreditation Cooperation, European Commission's Joint Research Centre Institute for Reference Materials and Measurements, LGC Standards. The following organisations provided support to delegates from developing countries: International Atomic Energy Agency, Physikalisch-Technische Bundesanstalt (Germany), United Nations Industrial Development Organization.

The skilled assistance of Mrs Valeria Patriarca for the preparation of this abstract book is gratefully acknowledged.

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Presidente dell'Istituto Superiore di Sanità e Direttore responsabile: *Enrico Garaci*
Registro della Stampa - Tribunale di Roma n. 131/88 del 1° marzo 1988

Redazione*: Paola De Castro, Egiziana Colletta* e *Patrizia Mochi*
La responsabilità dei dati scientifici e tecnici è dei singoli autori.

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HONOUR COMMITTEE

The event was organised by Eurachem, in cooperation with CITAC (Cooperation on International Traceability in Analytical Chemistry) and EQALM (European Committee for External Quality Assessment Programmes in Laboratory Medicine), under the auspices of Ministero del Lavoro, della Salute e delle Politiche Sociali (Italy), Ministero dell'Ambiente e della Tutela del Territorio e del Mare (Italy) and Provincia di Roma (Italy).
The Department of Public Veterinary Health and Food Safety of the Istituto Superiore di Sanità promoted and supported the organisation of the event in Rome (Italy). Members of the Honour Committee are listed below:

Kyriacos Tsimillis	Eurachem Chair, Pancyprian Union of Chemists, Nicosia, Cyprus
Steve Ellison	Eurachem Past Chair, LGC Limited, Teddington, United Kingdom
Ilya Kuselman	CITAC Chair, National Physical Laboratory of Israel, Jerusalem, Israel
Gunnar Nordin	EQALM Chair, Equalis AB, Uppsala, Sweden
Agostino Macrì	Director, Department of Public Veterinary Health and Food Safety, Istituto Superiore di Sanità, Rome, Italy

SCIENTIFIC COMMITTEE

The Eurachem Proficiency Testing Working Group aims to improve the organisation, and promote best practice, of Proficiency Testing (PT)/External Quality Assessment (EQA) in Europe, by creating a forum for discussion for organisers and users of PT/EQA Schemes and providing input into international activities related to PT/EQA. The organisation, with a triennial frequency, of international workshops on PT/EQA in analytical chemistry, microbiology and laboratory medicine, addressing current practice and future developments in PT/EQA, has been a key activity of the Working Group. The members of the Working Group are listed below:

Brian Brookman (Chair)	LGC Standards Proficiency Testing, Bury, United Kingdom
Ildi Ipoly	QualityConsult, Rome, Italy
Michael Koch	Universität Stuttgart, Stuttgart, Germany
Nineta Majcen	Metrology Institute of the Republic of Slovenia, Ljubljana, Slovenia
Irma Mäkinen	Finnish Environment Institute, Helsinki, Finland
Antonio Menditto	Istituto Superiore di Sanità, Rome, Italy
Miklos Naray	Hungarian Institute of Occupational Health, Budapest, Hungary
Tracey Noblett	LGC Standards Proficiency Testing, Bury, United Kingdom
Ender Okandan	Petroleum Research Laboratory, Ankara, Turkey
Ulf Örnemark	LGC Standards, Borås, Sweden
Marina Patriarca	Istituto Superiore di Sanità, Rome, Italy
Halina Polkowska-Motrenko	Institute of Nuclear Chemistry and Technology, Warsaw, Poland
Piotr Robouch	European Commission's Joint Research Centre, Institute for Reference Materials and Measurements, Geel, Belgium
Barry Tylee	Health & Safety Laboratory, Buxton, United Kingdom
Kees Van Putten	Ducares B.V., Zeist, The Netherlands

ORGANISING COMMITTEE

Several Italian organisations, including the public research institutes devoted to public health, metrology, environmental protection and innovative technologies, as well as the accreditation bodies and the standardisation body for chemical industry, have worked together to make this event possible and to promote the knowledge and practice of PT/EQA in Italy. The members of the Organising Committee are listed below:

Antonio Menditto (Chair)	Istituto Superiore di Sanità, Rome, Italy
Marina Patriarca (Chair)	Istituto Superiore di Sanità, Rome, Italy
Elena Amico di Meane	Istituto Nazionale di Ricerca Metrologica, Turin, Italy
Maria Belli	Istituto Superiore per la Protezione e la Ricerca Ambientale, Rome, Italy
Paolo Bianco	Sistema Nazionale per l'Accreditamento dei Laboratori, Federazione Italiana degli Enti di Accreditamento, Rome, Italy
Claudia Brunori	Ente per le Nuove Tecnologie, l'Energia e l'Ambiente, Rome, Italy
Carlo Corno	Associazione per l'Unificazione nel settore dell'Industria Chimica (UNICHIM), Milan, Italy
Enzo Ferrara	Istituto Nazionale di Ricerca Metrologica, Turin, Italy
Roberto Morabito	Ente per le Nuove Tecnologie, l'Energia e l'Ambiente, Rome, Italy
Mario Mosca	Servizio di Taratura in Italia, Federazione Italiana degli Enti di Accreditamento, Turin, Italy
Antonio Paoletti	Sistema Nazionale per l'Accreditamento dei Laboratori, Federazione Italiana degli Enti di Accreditamento, Rome, Italy
Giovanni Perego	Associazione per l'Unificazione nel settore dell'Industria Chimica (UNICHIM), Milan, Italy
Filippo Trifiletti	Sistema Nazionale per l'Accreditamento degli Organismi di Certificazione e Ispezione, Federazione Italiana degli Enti di Accreditamento Milan, Italy

TRAINERS AND SPEAKERS

Maria Belli — Head of the Environmental Metrology Laboratories at the Istituto Superiore per la Protezione e la Ricerca Ambientale, Rome, Italy (former Agenzia per la Protezione dell'Ambiente ed i Servizi Tecnici, APAT) - the Environmental Protection Agency of Italy. The laboratories produce matrix reference materials and organise inter-laboratory comparisons and Proficiency Testing in the field of environmental chemical/biological analysis. She represents the Italian Standardisation Body (UNI) at ISO/REMCO.

Magnus Holmgren — Quality Manager at the SP Technical Research Institute of Sweden and is also responsible for Engineering solid Mechanics. SP both organises and participates in Proficiency Tests. He is a member of the EA-Eurolab-Eurachem Permanent Liaison Group (PLG) and a member of the EA-Eurolab-Eurachem (EEE) Working Group on Proficiency Testing in Accreditation.

Michael Koch — Scientist (chemist) and lecturer at the Institute for Sanitary Engineering, Water Quality and Solid Waste Management of the Universität Stuttgart, Germany. He is the scientific director of AQS Baden-Wuerttemberg, a Proficiency Testing Scheme for water analysis in Germany. He is a member of the Eurachem Proficiency Testing Working Group, the board of EUROLAB-D, ISO CASCO Working Group 28 on the revision of ISO/IEC Guide 43-1 and 43-2 *i.e.* the development of ISO/IEC 17043, and some working groups for standardisation. He is involved in training and consultancy for quality assurance in analytical chemistry in developing countries and he is one of the editors of "Quality Assurance in Analytical Chemistry - Training and Teaching".

Ilya Kuselman — Scientific Director of the National Physical Laboratory of Israel. He is a specialist in metrology, quality and standardisation in chemistry and published about 170 papers and 9 patents in these fields. He is Chairman of "Cooperation on International Traceability in Analytical Chemistry" (CITAC), Regional Co-ordinator of the National Conference of Standard Laboratories (NCSL International), Member of the Interdivisional Working Party for Harmonization of Quality Assurance of the International Union of Pure and Applied Chemistry (IUPAC) and the Israeli representative at ISO/REMCO.

Ian Mann	Lead assessor at the Swiss Accreditation Service (SAS). He is active mainly in the medical, environmental and food analysis fields and also responsible within the SAS for the accreditation of PT providers. He is a member of a few international working groups including, EA-Eurolab-Eurachem (EEE) Working Group on Proficiency Testing in Accreditation, EA Interlaboratory Comparisons Working Group on testing and ISO CASCO Working Group 28 on the revision of ISO/IEC Guide 43-1 and 43-2 *i.e.* the development of ISO/IEC 17043. Ian is also leading a working group on the revision of the Eurachem guide "Selection, use and interpretation of Proficiency Testing (PT) Schemes".
Antonio Menditto	Senior scientist at the Department of Public Veterinary Health and Food Safety of Istituto Superiore di Sanità. He is active mainly in the food and medical analysis fields and a lead assessor for ISS ORL, the body involved in the accreditation of Italian laboratories in charge of official control of food products. He is Italian Ambassador of the TrainMiC (Training in metrology in chemistry) program, (IRMM, EC). He has been involved in the organisation and development of External Quality Assessment Schemes in clinical, environmental and occupational laboratory medicine and is a member of the Eurachem Proficiency Testing Working Group.
Ludwig Niewöhner	Head of the Gunshot Traces Unit at the Forensic Science Institute of the Bundeskriminalamt in Germany. He is a qualified examiner/expert for gunshot traces including shooting distance determination and gunshot residue analysis by SEM/EDX. Ludwig is a member of the Steering Committee of the ENFSI (European Network of Forensic Science Institutes) Expert Working Group "Firearms". He is also a technical advisor for gunshot residue analysis for the Association of Firearms and Tool Mark Examiners (AFTE). He is responsible for the performance of the ENFSI Proficiency Test on the identification of gunshot residue by SEM/EDX.
Piotr Robouch	Senior scientist at the Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre (JRC) of the European Commission. Presently he works at the Community Reference Laboratory for the authorisation of Feed Additives. Former consultant of the IMEP® program, he is member of the Eurachem Proficiency Testing Working Group, the EA-Eurolab-Eurachem (EEE) Proficiency Testing in Accreditation Working Group and the ISO CASCO Working Group 28 on the revision of ISO/IEC Guide 43-1 and 43-2 *i.e.*

	the development of ISO/IEC 17043. Experienced trainer, he was involved in the TrainMiC® program, related to the Training of Metrology in Chemistry throughout Europe.
Abdulghani Shakhashiro	Leader of the Reference Materials Group at the International Atomic Energy Agency's Laboratories in Seibersdorf, Austria. He is also responsible for conducting Proficiency Tests, such as ALMERA. As a quality manager of the Syrian Atomic Energy Commission, he founded the national Proficiency Test Scheme in Syria and managed the production of reference materials to fit the national needs. He has contributed to international workshops and training courses and published articles and reports related to Proficiency Tests and reference materials. He is actively involved in the implementation of IAEA technical cooperation projects in developing countries.
Tommy Šlapokas	Microbiologist at the National Food Administration in Sweden. He is responsible for microbiological method issues (ISO/CEN standardisation) and for the accredited Proficiency Test for drinking water (also partly food) at the governmental administration. Tommy is Chair of the microbiological water committee at the Swedish Standards Institute.
Philip Taylor	Responsible for activities in the area of metrology in chemistry, nuclear safeguards and radionuclide metrology at the Institute for Reference Materials and Measurements (IRMM) of the European Commission's Joint Research Centre. He is also the Operating Manager of the Community Reference Laboratory for Heavy Metals in Food and Feed. Philip has fostered the development of a Life Long Learning programme for metrology in chemistry (TrainMiC) which was developed in the framework of EU enlargement. This is combined with the provision of pan-EU interlaboratory comparisons for nuclear, environmental and food matrices.
Daniel W. Tholen	Owner of Dan Tholen Statistical Consulting in the USA. He is representing the Asia Pacific Laboratory Accreditation Cooperation (APLAC), Proficiency Testing Committee. He is responsible for the provision of consultation and training for a variety of professional organisations and accreditation bodies. He is an assessor for the A2LA PT accreditation and reference materials program and is the Convenor of the ISO CASCO Working Group 28 on the revision of ISO/IEC Guide 43-1 and 43-2 *i.e.* the development of ISO/IEC 17043.

Annette Thomas

Consultant Clinical Biochemist and Director of the Quality Laboratory at the Cardiff and Vale NHS Trust in the UK. She is also the Director of the Wales External Quality Assessment Scheme (WEQAS). She is responsible for the provision of support for Point of Care testing at one of the largest teaching hospitals in the UK and advises the Welsh Assembly Government on Point of Care issues. Annette is responsible for the Reference Laboratory which develops primary and/or secondary reference methods, and provides a European wide service to both manufacturers and EQA organisers in order to give stated, traceable, analyte values in calibrator, internal quality control and EQA materials.

PROGRAMME

Sunday, 5th October 2008

9.00-12.30 **TRAINING COURSE ON STATISTICS FOR PT/EQA SCHEMES**

Piotr Robouch
European Commission's Joint Research Centre, Institute for Reference Materials and Measurements, Geel, Belgium
Michael Koch
Universität Stuttgart, Germany
Ilya Kuselman
National Physical Laboratory of Israel

Objective:

- To understand the different statistical approaches used in PT/EQA

Topics:

- The jungle of standards and guidelines for the statistical treatment of PT data
- Assigned values and statistical methods for calculation, if consensus mean is used
- The Algorithm A in detail
- Z-score, z-score and colleagues - scoring procedures in PT and how to determine the standard deviation for proficiency assessment
- New guidance from IUPAC/CITAC on the "Implementation of PT Schemes for a limited number of participants and interpretation of their results"

14.00-17.30 **TRAINING COURSE ON SELECTION, USE AND INTERPRETATION OF PT/EQA SCHEMES**

Ian Mann
Swiss Accreditation Service, Switzerland
Antonio Menditto
Istituto Superiore di Sanità, Italy

Objective:

- To gain a better understanding of PT/EQA and to use and interpret the results of the PT/EQA Schemes as an improvement tool

Topics:

- Presentation of the revised EURACHEM guide on selection, use and interpretation of PT Schemes
- Selection of an appropriate PT/EQA provider and establishment of a PT/EQA plan
- Evaluation and use of PT/EQA results by the laboratory
- How to perform an efficient investigation following non conformance
- How to use PT/EQA as an improvement tool

Monday, 6th October 2008

Chair: **Brian Brookman**
LGC Standards, Teddington, United Kingdom

9.00 Welcome

9.15 *Frequency of PT/EQA Schemes and monitoring performance over time*
Annette Thomas
Quality Laboratory, Cardiff and Vale NHS Trust, Cardiff, United Kingdom

9.45 *Developments in Proficiency Testing within the European Union*
Philip Taylor
European Commission's Joint Research Centre Institute for Reference Materials and Measurements, Geel, Belgium

10.15 Coffee and Poster Session

11.00 *IAEA interlaboratory studies in service of analytical quality control of Member States' laboratories*
Abdulghani Shakhashiro
International Atomic Energy Agency, Wien, Austria

11.30 *The characteristics of a microbiological food and drinking water Proficiency Testing protocol in the Nordic countries*
Tommy Šlapokas
National Food Administration, Uppsala, Sweden

12.00 Poster Session

12.30 Lunch

14.00 Working Group discussions

15.45 Coffee and Poster Session

16.30 Presentation of conclusions of Working Groups

17.30 Adjournment

Tuesday, 7th October 2008

Chair: **Brian Brookman**

9.00 *Forensic PT: the ENFSI Proficiency Testing programme on identification of gunshot residue particles by SEM/EDX*
Ludwig Niewöhner
Forensic Science Institute of the Bundeskriminalamt, Wiesbaden, Germany

9.30 *ISO/IEC 17043: current status and proposed changes*
Daniel W. Tholen
American Association for Laboratory Accreditation (A2LA), Traverse City, Michigan, USA

10.00 Coffee and Poster Session

10.45 *PTs, heaven or hell for the laboratories? Laboratories' view on PT/EQA*
Magnus Holmgren
SP Technical Research Institute of Sweden, Borås, Sweden

11.15 *PT/EQA standards and guidelines: quality and reliability of test items*
Maria Belli
Istituto Superiore per la Protezione e la Ricerca Ambientale (Italian Environmental Protection Agency), Rome, Italy

11.45 Poster Session

12.30 Lunch

14.00 Working Group discussions

15.45 Coffee and Poster Session

16.30 Presentation of conclusions of Working Groups

17.30 Closing of the Workshop

PREFACE

This Workshop is the sixth of a series of international events, organised by the Eurachem Proficiency Testing Working Group, which addresses current practice and future directions of Proficiency Testing (PT) and external quality assessment (EQA) in analytical chemistry, microbiology and laboratory medicine. It is structured to include key-note lectures, discussions in working groups and poster sessions, to enable interactive participation and cross-fertilisation of ideas.

Special attention is given in this event to emerging issues such as the new standard for PT/EQA, the developments of PT/EQA in the European Union, and the status and needs of PT/EQA in developing areas of the world. In addition, enhancements in key areas of PT/EQA organisation, as well as developments in important and emerging fields, will be addressed. Last but not least, the end-user perspective of PT/EQA will be represented.

Several Italian organisations, including the public research institutes devoted to metrology, environmental protection, public health and innovative technologies, as well as accreditation and standardisation bodies, have worked together to make this event possible and to promote the knowledge and practice of PT/EQA in Italy.

Following the long tradition of Rome history in promoting standardisation, whilst also providing a bridge for cultures from different parts of the world to meet, this event will offer a forum for discussion to delegates from at least 47 different countries. Delegates, who were encouraged to contribute by presenting their experiences in the form of posters, have enthusiastically enriched this event with 74 contributions.

The 6th Workshop provides an excellent opportunity for both PT/EQA providers and end-users of PT/EQA, namely laboratories, accreditation bodies, regulators and the laboratories' customers, to come together and share their views.

Brian Brookman
Scientific Committee Chair

Antonio Menditto and Marina Patriarca
Organising Committee Chairs

Invited Lectures

FREQUENCY OF PT/EQA SCHEMES AND MONITORING PERFORMANCE OVER TIME

Annette Thomas
WEQAS, Cardiff and Vale NHS Trust, Cardiff, United Kingdom

The major function of Laboratory Medicine is the analytical measurement, in body fluids and tissues of individual patients, of those substances that are relevant for the understanding, prevention, diagnosis, monitoring and treatment of disease. This purpose requires many diverse procedures, all of which are associated with variation. The aim of Quality Assurance procedures is to ensure that procedures are in place to minimise these variations and to ensure that the quality of service is appropriate to maintain excellence in medical care; External Quality Assessment (EQA) is one of those procedures.

Frequency of EQA surveys vary greatly between organisations, countries, disciplines and analytes. Professional opinion related to the prevalence of a disease; the relative frequency of laboratory analysis; or a function of the analytical complexity of the investigation may determine them.

Recently a survey of EQALM members was undertaken to provide information on the current level of frequency of EQA participation across Europe. The data included the number of samples per round, the assignment of target values and the performance criteria used for each scheme.

For individual patient monitoring over time, using results from the same laboratory, the analytical variance is by far the major contributor to the performance characteristic. However, when general strategies such as goals for intervention for several centres are required, the bias becomes an essential characteristic. For example, the nine year Diabetes Control and Complications Trial (DCCT) study undertaken in Type 1 diabetic patients and the UK Prospective Diabetes Study (UKPDS) undertaken in Type 2 diabetic patients established the central role of HbA1c as the index for the long-term control of the glycaemic state. Specific global treatment goals were established based on HbA1c measurements. For HbA1c both strategies are therefore important factors.

Traditionally within Laboratory Medicine, each laboratory has participated in a specific EQA Scheme for every measurement technique it uses and for every analyte for each matrix (*e.g.* blood, urine or serum). EA and ILAC have recently recommended that participation in an EQA Scheme should be at a minimum frequency for each sub-discipline. A sub-discipline can include more than one measurement technique, property or product as long as equivalence and comparability can be demonstrated within the sub-discipline. To ascertain the views of EQA organisers on the concept of sub-disciplines, EQALM members were also asked to provide their views on a series of hypothetical scenarios.

DEVELOPMENTS IN PROFICIENCY TESTING WITHIN THE EUROPEAN UNION

Philip Taylor
Institute for Reference Materials and Measurements, European Commission's Joint Research Centre, Geel, Belgium

For many decades, people who measure have been comparing their results, particularly when data is exchanged across space or time, which is especially the case today. Such intercomparisons enable laboratories to demonstrate to themselves and to others that they are proficient in what they do. This is of course the reason why participation to Proficiency Testing (PT) is an important external quality assurance tool stipulated in ISO/IEC 17025.

Considering the vast domain of measurements in chemistry and bio-analysis, and considering the fact that accreditation according to ISO/IEC 17025 is a *quasi de facto* requirement for laboratories today, the need for appropriate PT Schemes continues to grow. The new European Legislative framework that strengthens the role of accreditation will only further emphasise this.

Such needs will create markets, one would argue. The number of PT Schemes has indeed grown over the past years. Are market forces alone sufficient to offer good quality PT Schemes in all areas, and what is good quality? Will we see only larger commercial PT providers surviving and providing pan-European or even global services? Will all laboratories in the various regions of Europe be able to acquire such PT services? Is there still then any need for a local aspect in PT? Is there a research dimension in PT and if so how does this get financed? And are customers and accreditation bodies making use of PT across Europe in a similar way? Is the potential of learning via PT sufficiently explored?

In brief, this contribution will try to analyse and reflect some trends in the PT world.

IAEA INTERLABORATORY STUDIES IN SERVICE OF ANALYTICAL QUALITY CONTROL OF MEMBER STATES' LABORATORIES

Abdulghani Shakhashiro, Umberto Sansone, Ales Fajgelj
IAEA Seibersdorf Laboratories, International Atomic Energy Agency, Wien, Austria

The International Atomic Energy Agency (IAEA) has been actively promoting the establishment of quality systems in Member States' laboratories through Technical Support programmes and analytical quality control services provided by the Agency's Laboratories Seibersdorf and Headquarters, and the IAEA Marine Environment Laboratories Monaco since many decades. The main IAEA activities in this area are:

- production, certification and distribution of reference materials characterised for radionuclides, trace elements, organic contaminants and stable isotopes;
- organisation of Proficiency Tests and interlaboratory comparisons;
- co-ordination of an international network of Analytical Laboratories for the Measurement of Environmental Radioactivity (ALMERA);
- provision of technical assistance through the Member States technical co-operation projects;
- provision of training and experts assistance.

This presentation will provide an overview of the IAEA continuous efforts in upgrading the approaches applied in production, characterisation and assignment of property values to reference materials used in the IAEA Proficiency Tests. Future plans for the reference materials production will also be presented.

A special emphasis will be given to the IAEA services provided to the developing countries, including the know how transfer in reference materials production and organisation of Proficiency Tests.

The paper will present the IAEA experiences, gained through several regional and interregional Technical Cooperation projects aimed to implement "Quality Assurance/Quality Control for Nuclear Analytical Techniques" in Latin America, Eastern Europe, Eastern Asia and Asia Pacific, where Proficiency Tests were included as an essential component to monitor the analytical performance of participating laboratories.

THE CHARACTERISTICS OF A MICROBIOLOGICAL FOOD AND DRINKING WATER PROFICIENCY TESTING PROTOCOL IN THE NORDIC COUNTRIES

Tommy Šlapokas
Microbiology Division, National Food Administration, Uppsala, Sweden

The National Food Administration (NFA) in Sweden is dealing with legislation, inspections, applied research, analyses etc. for food and drinking water (dw). Until the mid 90's NFA had to approve laboratories performing microbiological analyses in statutory sampling. NFA developed a PT program as one tool in the approval process. The start was 1981 for Swedish food laboratories and 1989 for DW laboratories. From 1988 NFA organised food PT and from 1992 DW PT for the Nordic countries. When accreditation took over laboratory approval in Sweden, NFA continued as PT provider because of their equipment, experience, routine, staff and feedback from laboratories. Nowadays NFA is providing test material for most EU-regulated parameters, and "PT-micro" is open for all countries.

The PT-micro work at NFA covers all phases from planning, test item production to evaluation and reporting. Information, application, result exchange and reports in pdf format are mainly handled by use of the website www.slv.se/absint.

Glass vials with matrix free, freeze-dried aliquots of culture mixtures of microorganisms, simulating real samples, are the test items used in both schemes. 2,500 items can be produced simultaneously and they have good stability and homogeneity.

The results are evaluated per sample and parameter. All results are compiled in an Appendix and are reported back in the same scale as they were originally recorded. That is natural numbers (counts/volume) in the DW scheme with low concentrations of organisms, but $\log_{10}$ (calculated counts/volume) in the food scheme with quite high concentrations and the use of dilutions. However, in calculations of statistics and z-scores the DW results are used square root transformed, while the original $\log_{10}$ scale is used for the food results. In both cases this implies decent normal distributions. "Robust" means and round-specific standard deviations are obtained after removal of outliers. Apparent false positive and false negative results are first removed without any test. z-scores are calculated from the robust values and visualised in a box-plot for every participant. Original result distributions are visualised in histograms. False negative results and outliers are specifically marked there, as well as together with false positive results in the Appendix.

Beneath the box-plot are the numbers of deviating results given in a table. The box-plot together with the table is indicating the participant performance.

Follow-up is the responsibility of the participants themselves. Extra test items can be ordered for free. Participants may contact NFA for discussions.

FORENSIC PT: THE ENFSI PROFICIENCY TESTING PROGRAMME ON IDENTFICATION OF GUNSHOT RESIDUE PARTICLES BY SEM/EDX

Ludwig Niewöhner (a), Jan Andrasko (b), Lawrence Gunaratnam (c)
(a) Forensic Science Institute of the Bundeskriminalamt, Wiesbaden, Germany
(b) National Laboratory of Forensic Science, Linköping, Sweden
(c) National Bureau of Investigation, Vantaa, Finland

Introduction. Within the framework of the ENFSI Working Group "Firearms" a Proficiency Testing programme on the detection and identification of gunshot residues (GSR) by SEM/EDX was set up and performed, with financial support from the European Union (JLS/2006/AGIS/041). The latest test was carried out in 2005/06 (GSR2005) and the results recently reported by Niewöhner and coworkers in the Journal of Forensic Sciences. The test material was designed by the Bundeskriminalamt and manufactured on order by an external company for SEM accessories. The participating laboratories were requested to determine the total number of PbSbBa containing particles on the test samples following their own laboratory specific methods of automated GSR particle search and detection by SEM/EDX.

Sample description. The test items for the Proficiency Test consisted of a set of completely identical samples as it is demanded within the ISO 5725 standard for the performance of Proficiency Tests. Therefore the samples were produced using a special, patent protected method (patent no. DE 199 32 357). Synthetic "GSR particles" of a known composition of lead, barium, and antimony and a size distribution of 0.5 to 2.4 µm were precipitated onto a silicon substrate of a size of 8 x 8 mm², and mounted on a SEM stub.

Evaluation of data. All participating laboratories were requested to analyse the received sample using their standard GSR examination routines on their SEM/EDX systems, and to report the results within 4 weeks to the organisation committee. Reports had to include the number of detected PbSbBa particles, their size and their exact position on the sample. Sample evaluation was performed by comparing the submitted data with the original data set from the sample production. An evaluation of the laboratory's performance to detect GSR particles by SEM/EDX was carried out using z-scores according to relevant IUPAC and EURACHEM guidelines. A comprehensive report on the results of the Proficiency Test was prepared and sent to all participants (also available from http://quodata.de). In 2008 a follow-up test was launched (GSR2008), which had been approved by ASCLD-LAB as GSR Proficiency Test (see: http://www.ascld-lab.org/legacy/ aslablegacyapprovedproviders.html).

ISO/IEC 17043: CURRENT STATUS AND PROPOSED CHANGES

Daniel W. Tholen
American Association for Laboratory Accreditation (A2LA), Traverse City, Michigan, USA

The revision of ISO/IEC Guide 43 is well underway. As of now the title is "ISO/IEC CD 17043 Conformity Assessment - General requirements for Proficiency Testing." The revision is being conducted by CASCO Working Group 28. The ballot on the Committee Draft was completed on 16 June, 2008 and it was successful, with 49 of the 72 CASCO P members voting for "approval for registration as a DIS in accordance with 2.5.6 of part 1 of the ISO/IEC Directives". 27 ballots included comments. There were no votes for disapproval and six abstentions. This is considered a very strong statement of broad support by the CASCO membership. There were over 500 comments submitted by the members and liaison organisations. The Working Group met on 3-5 September to resolve the comments and resolved to recommend that the document should advance as an ISO/IEC DIS. If CASCO agrees, the DIS will be distributed in December for a 5 month ballot and comment period.

There remains a concern about the document is the structure, which is not consistent with CASCO policy on common elements for CASCO standards. However, the WG agreed that it is more important for the document to be in harmony with ISO/IEC 17025 to the extent possible. Similarly, the document does not conform with the management system requirements in recently approved PAS 17005. The WG discussed this issue at length and agreed that to conform would require a significant delay in the approval of the document and make it not agree with 17025. Also, with the very strong ballot from CASCO members, there was no compelling reason for alignment. The issues on structure and alignment will be considered by the CASCO Chairman's Policy Committee (CPC) and CASCO Secretariat.

The WG28 deliberations have generally been harmonious, with the only major area of disagreement being requirements for metrological traceability of assigned values, at least for some types of schemes (currently, "for PT Schemes in the areas of calibration"). Surprisingly, there were no significant concerns about this expressed in the comments from CASCO members. Other technical areas where significant changes have been agreed include restrictions on activities that can be subcontracted, requirements for calculating and reporting the uncertainty of assigned values, and the use of participants' estimated uncertainties. The presentation at the conference will address the status of the document following the CASCO member ballot and working group meeting, future plans, and the current status of the technical items mentioned above.

PTs, HEAVEN OR HELL FOR THE LABORATORIES? LABORATORIES' VIEW ON PT/EQA

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Inter-laboratory comparisons are by laboratories considered as good tools for improving quality and a useful help in education and training of staff. But laboratories are also facing some problems when participating in inter-comparisons.

There are different types of laboratories and they have different approaches to inter-laboratory comparisons in general and to PTs in particular. Laboratories in some technical field *e.g.* analytical chemistry, microbiology and medicine, are used to PTs. PTs are for this category of laboratories a commonly used tool in the quality assurance activities and it is often also mandatory for the laboratories to participate. On the other hand laboratories in other technical fields, *e.g.* mechanics and electricity, are less familiar with the concept of laboratory inter-comparisons and PTs in particular. As an illustration, a search in the database "EPTIS" for the testing field "analytical chemistry" and sets of products "agriculture", gave 67 hits while a search for the testing field "EMC" and sets of products "Electrical/Electronical components, devices and equipment" gave only 3 hits and a search with testing field "mechanics" and sets of products "metals" gave 20 hits but with only one major provider of the PT-Schemes. There are many technical fields, *e.g.* EMC or mechanics, where there are few or almost no regular PTs or inter-comparisons.

This is also influencing the way laboratories within different technical fields are looking on participation in inter-comparisons, whether the participation is voluntary or mandatory, requested by *e.g.* accreditation bodies or authorities. It is therefore important to take these differences between different technical fields into consideration when common documents with requirements (*e.g.* frequency) for participation inter-comparisons, especially PTs, are developed within the laboratory community.

One issue connected with participation in inter-comparisons which is very important for laboratories is the way successful participation is "paying off" in relation to the extent of the accreditation body's surveillance. It also important, that the accreditation bodies are handling non-successful participation in PTs in a pragmatic way. Laboratory inter-comparisons should first of all be seen as way to improve the quality of the participating laboratories' activities. For PTs on the other hand the importance of uniform and transparent handling of unsatisfactory results in Europe as well as in the entire world should be stressed.

The preliminary results from a European questionnaire concerning laboratories' view on inter-comparisons will be presented during the presentation.

PT/EQA STANDARDS AND GUIDELINES: QUALITY AND RELIABILITY OF TEST ITEMS

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Test items preparation and management are crucial points in PT/EQA organisation. Standards and international guidelines (ISO Guide 43-1:1997, draft ISO/IEC 17043, ILAC G13:2007, IUPAC harmonised protocol for PT of analytical chemistry laboratories) agree on the key characteristics for PT test items. Test materials should satisfy the following quality criteria: i) similarity to the materials routinely analysed (in respect of matrix composition, range of analyte concentrations, etc.); ii) sufficient homogeneity (all laboratories participating in the round should receive samples that are not significantly different in analyte concentration); iii) sufficient stability (test materials should not change significantly during the PT duration).

Sufficiently homogeneous and stable PT test items are used as reference to evaluate laboratory performances and to establish the comparability of measurements among a specific type of laboratories. ISO/REMCO defines reference material as "Material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement processes". In the view of this definition PT test items, in environmental chemistry are mainly reference materials.

In the last years, ISO/REMCO has prepared different guides for preparation of reference materials. The most relevant are ISO guide 34 "General requirements for the competence of reference material producers" and ISO guide 35 "Reference materials - General and statistical principles for certification" published respectively in 2000 and in 2006. ISO/REMCO Working Group 8 (convenor Steve Wood - United Kingdom) is drafting a new guide, now in the state of Committe Draft, aimed at the production of reference materials for precision control. This last guide can be very useful for PT test items preparation.

The last point regarding PT test items reliability is linked with assigned value traceability. Generally, if all participants calibrate their equipment with traceable measurement standards or all use a standardized method for method dependent measurements, the consensus values will be traceable to the International System or to the selected method. The use of an external traceable assigned value, instead of a consensus value, improves the reliability of the PT test item, because it links the measurements carried out by the field laboratories to the highest metrological level. The general adoption of PT test items with traceable values is still under discussion, because the cost of such schemes, using certified reference materials, could be very high.

The paper discusses the important steps that are necessary to assure the quality and the reliability of PT test items. Additionally, the paper covers some ISPRA (former APAT) experiences on preparation of environmental reference materials for PT organisation.

Poster Contributions

PROFICIENCY TESTING INCORPORATING METROLOGICALLY TRACEABLE REFERENCE VALUES

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Proficiency Tests (PTs) conducted by the National Measurement Institute, Australia (NMIA) have used metrologically traceable reference values and associated uncertainties as assigned values. Examples including pesticide residues in fruit and vegetables and extractible metals in soil will be presented. Laboratories from Australia, South East Asia and South America participated in these PTs.

Mass spectrometry was used to make reference measurements. The measurement methods had been validated and their accuracy demonstrated over the course of NMIA's participation in key comparisons coordinated by the Consultative Committee for Amount of Substance - Metrology in Chemistry (CCQM). Thus the PTs served as a means of disseminating the NMIA's high level measurement capability to working laboratories in Australia.

Reference values were used in PTs for pesticide residues in fruit and vegetable. Here there was no consensus amongst the participating laboratories upon which an assigned value could be based and so a reference value provided a sound basis for the assessment of laboratory performance using z-scores and E_n-numbers. This illustrated the usefulness of a traceable reference value for difficult analytes.

The case of extractible metals presented a difficulty. This was an empirical measurement, where the method of extraction defined the measurand. It was possible to make measurements traceable only to the particular method and acid mixture used to extract the metal. However, the results of the study demonstrated that the majority of participants were using methods providing equivalent results.

The extent to which participation in PT provides a means for laboratories to demonstrate the traceability of their measurement results will be discussed and a traceability chain, from routine measurements to the International System of units, will be presented.

CRITERIA TO DEFINE THE SD FOR PROFICIENCY ASSESSMENT OF DETERMINATIONS OF ESSENTIAL TRACE ELEMENTS IN SERUM: COMPARISON OF Z-SCORES BASED ON THE HORWITZ FUNCTION OR BIOLOGICAL VARIABILITY

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A critical issue in the organisation of Proficiency Testing/External Quality Assessment Schemes (PT/EQAS) is the definition of criteria against which the performance of individual laboratories should be judged. Previous work by this group (Network of Organisers of EQAS for Occupational and Environmental Laboratory Medicine (www.occupational-environmental-laboratory.com) and within the European Project CoEPT has shown that different criteria are used even in the same field of application by different scheme organisers. Therefore the performance of the same laboratory could be judged differently in different schemes. For this reason, the Network Members collaborate to define common minimum acceptable levels of performance, taking into account both clinical and analytical issues, and have already agreed limits for some measurands. In the pursuit for a sound basis for predictable analytical variability, the possibility to apply the Horwitz function, originally developed using data from interlaboratory method performance studies for food analysis, has been discussed and compared with previously agreed limits. Sets of results obtained from the distribution of test materials in the Network schemes (for Cu, Se or Zn in serum) were plotted against quality specifications derived from the Horwitz function or available information on biological variability. Test materials were grouped by concentration intervals and the performance of participants compared over the same range of concentration using either of the two criteria. In addition, the distribution of the standard deviations obtained over several trials in different EQAS and countries, in the same period of time, was plotted against both criteria.

Neither the Horwitz or the Fraser criteria were fulfilled by the majority of participants. Except for Se, Horwitz criteria suggested a more severe evaluation than Fraser desirable criteria, the latter being very severe as regard the participant analytical variability. In

general, the distribution of standard deviation for these tests did not follow the same proposed law as in food analysis. Therefore, care should be taken before applying criteria derived from other sources to the clinical field. Collaboration among organisers of EQAS in the same field is essential to highlight potential problems, to share experience and develop adequate performance goals.

PROFICIENCY TESTING PROGRAMS ORGANISED BY THE DEPARTMENT OF SCIENCE SERVICE, THAILAND

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The Proficiency Testing Section, Bureau of Laboratory Accreditation, Department of Science Service (DSS) is responsible for organizing Proficiency Testing (PT) programs.

The programs include several fields such as food chemistry, food microbiology and environmental field. For the year 2008, the DSS offers:

- aerobic plate count in starch;
- moisture, polarization, colour and reducing sugars in sugar;
- moisture, protein and ash in flour;
- moisture, protein, fat, crude fiber and ash in feeding stuffs;
- Ca, Fe, K, Mg , Na, P and Zn in feeding stuffs;
- heavy metals in water;
- Total Dissolved Solids (TDS) and Suspended Solids (SS) in water;
- pH value of water;
- chemical Oxygen Demand of water.

The participants are mostly from within Thailand. However, we have offered PT programmes to a number of laboratories in Pakistan since both countries have signed a Memorandum Of Understanding (MOU) for scientific and technological cooperation.

Sample preparations are organized by the DSS and checked for homogeneity. Samples are sent to the participants by express mail. The assigned value for test material used in the PT Scheme is the robust average. The performance of each laboratory is with z-scores, using the robust standard deviation of the results reported by all the participants in the PT program. The robust mean and robust standard deviation are calculated using Algorithm A from ISO 13528:2005.

PROFICIENCY TESTING FOR ECOTOXICITY EVALUATION OF WASTE

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In this paper, the Italian results of a project of the German Minister of Environment (BMU), entitled: "Evaluation of a biotest battery for the ecotoxicological characterization of waste and waste eluates by interlaboratory test", are presented. The aim of the project was the evaluation of a biotest battery, which can be used to assess the ecotoxicity of waste material and eluates.

In the European Waste List 2001/1118/EC, 170 waste types are defined as "mirror entries", which differentiates between the waste material with or without hazardous compounds. In the Directive on Hazardous Waste 91/689/EEC 14 criteria for the characterization of wastes as hazardous are listed. The criteria H14 "ecotoxic" can be determined by the use of biological test methods.

Sixty seven laboratories from different countries participated in the interlaboratory study. Among them there were five Italian laboratories: the laboratory of Environmental Metrology of Italian Environment Protection and Technical Services Agency (APAT, now ISPRA) and chemical and ecotoxicological laboratories of Regional Agencies of Environmental Protection (Umbria, Marche, Toscana, Piemonte).

Three waste samples, prepared by the German Federal Institute for Materials Research and Testing (BAM), were chosen. The toxicity of the test samples ranged from moderate to weak effects in the biotest. The samples were solid, dry, ground (4 mm particle size), without any volatile compounds. They were: a Municipal Waste Incineration Ash, a contaminated wood, a gaswork soil.

The biotests were performed on solid waste and/or their leachates using a basic bioassays battery complemented voluntarily with additional test systems. All the Italian laboratories have carried out the leaching test following UNI EN 12457-2, and the analytical characterization of the eluates (pH, conductivity, TOC, metals like Cu, Cr, Ni).

The following biotests systems were performed on eluates:

- determination of the inhibitory effect on the light emission of *Vibrio fischeri*;
- freshwater algal growth inhibition test with *Pseudokirchneriella subcapitata*;
- determination of the inhibition of the mobility of *Daphnia magna* Strauss-Acute toxicity test.

Each laboratory investigated every samples in a range-finding-test for the basic test battery to define the concentrations of the definitive ecotoxicity tests in order to estimate the relative EC50 (95% confidence interval). The Italian final results respect to the overall ones of the European interlaboratory study are presented and a graphical evaluation of the good Italian performance respect to the consensus values is shown.

MEASUREMENT OF PAHs IN ENVIRONMENTAL MATRICES: RESULTS FROM AN INTERCOMPARISON EXERCISE ON THE DIFFERENT STEPS OF THE MEASUREMENT PROCEDURE

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Proficiency Testing is becoming a feature of laboratory accreditation and the generated results are used to assess the technical competence of the participating laboratories. The Italian National Environmental Protection Agency (APAT) plays an important role in order to support the Regional laboratories to improve the quality of their analytical measurements. APAT's mission is tailored to assure the quality and comparability of the analytical data across Italy and to achieve harmonization of the analytical approach to the environmental issues. Within this framework an intercomparison exercise on PAHs measurement on environmental samples was carried out.

The measurement of PAHs in environmental matrices represents constantly a challenging issue within the environmental laboratories. A wide set of measurement methods are commonly used and differ from each other regarding the extraction phase, the purification and the instrumental measurement. All these phases of the measurement process affect the quality of the measurement results and represent a source of variability among the laboratories.

The aim of the intercomparison was to assess the performance of the laboratories for the measurement procedure, separating the different steps (from the extraction to the instrumental measurement). To this aim the APAT's Environmental Metrology Unit produced and distributed to 59 laboratories two matrix reference materials: i) a polluted soil (APAT RM014); ii) an extract (n-exane) reference material of the same polluted soil (APAT RM017). In addition, in order to evaluate the measurement performance without any possible matrix interferences, a vial containing a "blind" PAHs mixture standard stock solution was delivered to the laboratories. This approach allowed the variability due to the different steps of the measurement procedure to be studied separately, and enabled an appreciation of their relative weighting in respect of the complete measurement process. The reference materials produced by APAT were characterized according to ISO Guide 35 and ISO 13528:2005 by expert laboratories and by consensus value of the laboratories participating in the intercomparison. The PAHs standard stock solution was characterized by the producer. Homogeneity and stability tests were performed on the reference materials. The laboratories results were assessed according to ISO 13528:2005. The paper illustrates the results of the intercomparison exercise.

THE 40 YEARS OF THE QUALCODUNA PROFICIENCY TESTING SCHEME - PAST EXPERIENCES AND FUTURE CHALLANGES

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Proficiency Testing Schemes (PTS) are regarded as a crucial element of the quality assurance and control system of analytical results. In Hungary, the first regular intercomparison exercise in the water sector began as early as 1969, with the participation of the 12 analytical laboratories of the departmental Water Management Directorates. This poster aims to present the scheme for a wider audience, as well as to highlight lessons learnt during the exercises and present future challenges.

Organised by VITUKI, these exercises initially covered three analytes in synthetic samples. Today and some 120 distributions later, the scheme covers about 150 laboratories from all segments of the analytical sector in Hungary, periodically testing their proficiency in predominantly natural samples (surface, drinking and wastewater, sewage sludge) for over a hundred determinands (general parameters, nutrients, anions, metals, organic pollutants, hydrobiological and group parameters, radioactivity).

The regularity and longevity of the scheme enabled the observation of longitudinal trends. Our strategy is to distribute synthetic samples first, and convert to real samples later with gradually lower concentration ranges as the overall analytical performance improves. This approach proved to be, from the very beginning, a valuable tool to find the causes of and subsequently to reduce and eliminate analytical errors in everyday laboratory practice.

The programme follows the Youden-pair experimental design, *i.e.* two samples of identical matrix and with a small (and known) concentration difference are sent out for each determinand. This not only provides the opportunity to easily visualize the overall performance on the so-called Youden-plot, but also allows for the type of error being assessed at even individual laboratory level (systematic or random). The assigned value is determined as the median of participants, while performance evaluation is based on comparison of percentage bias against pre-set, fit-for-purpose criteria that are derived from water industry consensus guidelines, analytical experience and standard deviation of previous datasets.

Challenges ahead are nonetheless numerous: new analytes, lower concentration levels are required by ever more stringent regulations; a plethora of guidance documents and standards are being published/updated or have been so recently, intending to prescribe and harmonise best practice (recommendations on robust statistics, prevalence of z-score in performance evaluation, increasing emphasis on accreditation etc.) VITUKI is committed to provide a quality PT Scheme in the future, as shown by our participation in the European PT harmonisation process in support of the Water Framework Directive, which opens the door for further refinement and improvement of our scheme.

AN INVESTIGATION INTO *ESCHERICHIA COLI* METHODOLOGY USING PROFICIENCY TESTING SCHEME DATA

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Introduction. Proficiency Testing is a powerful tool. Used appropriately, it can bring significant benefits to laboratories. The primary aim of most PT Schemes is to provide the infrastructure for a laboratory to monitor and improve the quality of its routine analytical measurements. PT should be used as a laboratory aid and its results assessed over a period of time, to uncover any trends. It is not uncommon for participants in a PT Scheme to contact the provider to request information on method performance for a particular test parameter. Using the data from a PT Scheme can be of value when assessing methodology, due to the high number of participants performing the same analysis. *Escherichia coli* is found in human and animal intestines, therefore its presence in food is believed to indicate faecal contamination. However this organism is also widespread throughout the environment. The presence of *Escherichia coli* in food may be faecal or environmental in origin. Historically it is used as an "indicator" organism, to indicate whether more serious pathogens are likely to be present. A number of methods may be employed when testing for *Escherichia coli*, the current standard ISO 16649-2:2001, recommends the use of tryptone-bile-glucuronic medium (TBX) with incubation for 18-24 h at 44±1˚C. If the presence of stressed cells is suspected then an initial incubation period of 4 hr at 37˚C should be considered. Other methods commonly used include Petrifilm and most probable number.

Method.

- Collation of historical data from the LGC Standards test material QM16F over a number of distributions;
- calculation of the percentage of participants using each method;
- calculation of the median of results for each method and comparing to the assigned value for that test parameter;
- assessment of any trends.

Results.

- The percentage of participants employing each method. Only commonly used methods are included, all others are grouped under "other". The only difficulty is that participants do not always record their chosen method;
- the comparison of the median of results for each particular method with the assigned value over a period of time.

Conclusion.

- Does the method employed for the enumeration of *Escherichia coli* affect performance?
- Are there any significant trends?

THE VIRTUAL INSTITUTE OF REFERENCE MATERIALS BACK IN FULL SWING: A MEETING PLACE ALSO FOR PT PROVIDERS AND ANALYTICAL LABORATORIES

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The Virtual Institute for Reference Materials (VIRM) is the meeting place for the Reference Material (RM) community. The VIRM innovative character consists in its virtual structure, combining a minimal logistic infrastructure with the maximal effectiveness of a World-wide ICT network. VIRM was originally established as a legally independent not-for-profit association with legal seat in Luxembourg and with 18 founding members. In January 2007, the General Assembly of VIRM unanimously decided for the liquidation of the VIRM asbl and approved the transfer of its assets to QualityConsult (www.aqc.it), an Italian non-for-profit association acting mainly as PT provider. On 30 November 2007 VIRM was officially liquidated and thus incorporated into QualityConsult. After a reorganization period, starting from 1st July 2008 the VIRM is back in full swing. QualityConsult has sought a strategic alliance with Mermayde (Bergen, NL) for the maintenance and further development of VIRM.

VIRM offers a broad range of useful tools in the field of RMs and Quality Control. Its website is the key link between the RMs stakeholders. It offers extensive search facilities (people, projects, RMs etc.) and is a central place for dissemination of information (www.VIRM.net). Among the other features, it is worth mentioning the searchable database of RMs. The VIRM RM database is a powerful tool currently containing about 4,500 among certified and not-certified RMs (24,500 records) from worldwide RMs producers in multiple fields.

The participation in Proficiency Testing Schemes (PTS) is a necessary tool for external quality control. One of the main keys for the organization of a successful PT is the availability of high quality test material covering as much as possible the request from the analytical laboratories. According to a survey carried out within the EAQC-WISE project, financed by EC, the majority of European PT providers utilizes non certified RMs as test samples. However, if the information related to the availability of certified RMs is scarce, not considering the 4-5 worldwide big producers (NIST, BCR, NRCC, etc.), the information related to the availability of not certified RMs is almost absent. The VIRM database offers a valuable and unique tool for PTs providers for checking the existence and the availability of not certified RMs worldwide and for being linked directly to the producers.

Furthermore, being QualityConsult a PT provider that, by the way, entered in contact with its main producer of test samples througn the VIRM database, the new management of VIRM planned to open on the VIRM website, before the end of 2008, a section fully dedicated to PTs, where, among other, PTs providers may advertise their schemes and customers find the scheme more suitable for their aim.

PROFICIENCY TESTING AS A TOOL IN THE QUALITY CONTROL OF THE MEASUREMENT RESULTS OF CHEMICAL LABORATORIES OF SLOVENIAN MANUFACTURERS OF PRECIOUS METAL ARTICLES

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The Department for Chemical Measurements (SKM), at the Metrology Institute of the Republic of Slovenia (MIRS), is a national institution responsible for chemical testing and conformity assessment of precious metal articles. The activities of SKM, which is the holder of the national standard for the SI unit mol, are mainly focused on the execution of the measurements of the mass fraction of precious metals in precious metal alloys at the highest metrological level in the country. Therefore a Proficiency Testing Scheme is organised by SKM each year in order to examine and assure the appropriate performance of the measurements of the mass fraction of precious metals in all those jewellery producer's laboratories in Slovenia, which have been authorised by MIRS to perform chemical analysis of gold and silver articles.

The organisation of MIRS-SKM Proficiency Testing Scheme using gold and silver wire as test materials, containing 585‰ of gold and 925‰ of silver, respectively, is presented. The measurement procedures via which the reference value of the mass fraction of gold or silver in the alloy was assigned to each test material is reported and the homogeneity study performed on both test materials is explained. The results obtained in MIRS-SKM Proficiency Testing Schemes in recent years are shown. A particular emphasis is given to the description of the situation when a measurement result of the participating laboratory was not appropriate, since the observed deviation of particular measurement result from reference value was larger than it would be acceptable according to the evaluated measurement uncertainty. The approach used for the investigation of possible sources of inaccurate measurement results is described, including the presentation of examples of selected chemical laboratories with detected non compliances and consequent corrective actions. The role of PT in such process is identified and explained. The involvement of PT enables not only the confirmation of appropriate performance of the measurements but also serves as a tool for external quality assessment. The presented study demonstrates that via a described approach an appropriate execution of the measurement of the mass fraction of precious metals is obtained in Slovenia.

THE EUROPEAN NETWORK OF PT PROVIDERS IN SUPPORT OF THE IMPLEMENTATION OF THE WATER FRAMEWORK DIRECTIVE

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The implementation of the Water Framework Directive (WFD) requires the design of monitoring programmes for all Member States, based on chemical, biological and ecological measurements. Monitoring data are the basis for regulatory decisions and measures required to achieve WFD environmental objectives, and need to be reliable and comparable. In order to achieve this, appropriate analytical quality assurance and control (QA/QC) have to be established across all EU Member States.

The pending QA/QC Commission Directive requires that laboratories demonstrate their competence by participation in Proficiency Testing programmes covering all measurands at levels of concentrations that are representative of chemical monitoring programmes carried out under the WFD. Unfortunately, there are currently several options to organise PTs and to evaluate laboratories analytical performances. Depending on the approach applied by the PT provider, the same results could in theory be judged to be satisfactory in one PT Scheme and not satisfactory in another. This raises the need for a harmonised approach to PTs in support of the WFD.

To cope with this situation, the birth of a "Network of PT providers to support implementation of the Water Framework Directive (PT-WFD)" was proposed and facilitated (within the frame of the EAQC-WISE project). The official birth of the network will take place in Rome on 8th October 2008. The self-committed Network is composed by PT providers experienced with PT Schemes related to WFD monitoring analyses. External experts in the PT field shall act as external advisors of the Network and their presence will assure also an external scientific control of the Network operation.

The objective of the Network is to provide harmonised PT Schemes meeting the specific requirements of the WFD (in terms of analytes, matrices, and concentration levels), based on high quality criteria and organized in a harmonised and comparable way. In order to ensure consistent performance assessment, the PT providers adhering to the Network will follow common rules in terms of test material typology, schemes organization/setup, data elaboration and evaluation. PT Schemes run under the Network will operate in accordance with the requirements of ISO/IEC Guide 43 and ILAC G13 and the results will be evaluated on the basis of the internationally recognised scoring systems (ISO 13528). Besides guaranteeing a harmonized laboratory performance evaluation that is fit for purpose for WFD implementation, the Network of PT providers will also allow the development of productive synergies among PTs providers aiming to find and apply feasible solutions for new developments and gaps in the spectrum of needed PT Schemes. The added value of creating such a Network lies in the potential to organize PT Schemes

for all parameters on the Priority Substance list, including "difficult" parameters, and to organize PT Schemes at European level for analytes that are analysed only by few laboratories in each country.

EVALUATION OF THE PERFORMANCE OF EUROPEAN ANALYTICAL LABORATORIES IN WFD MONITORING ANALYSIS BASED ON THE RESULTS OF THE PROFICIENCY TESTING SCHEMES ORGANIZED WITHIN THE FRAME OF THE SWIFT-WFD PROJECT (2004-2006)

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With the commencement of Water Framework Directive (WFD) monitoring in December 2006 the EU member states had to finalise their monitoring programmes. The comparability of monitoring data obtained in different European Countries is without doubt a key issue for the successful implementation of WFD.

Within this frame, the project "Screening method for Water data InFormation in support of the Water Framework Directive - SWIFT-WFD" was funded by the European Commission (contract no: SSPI-CT-2003-502492, January 2004 - March 2007). The main objective of the project was to support the successful implementation of the WFD, closely depending on the quality of monitoring data and its comparability from river basin to river basin. This objective required primarily, but among others, the development, validation and dissemination of rapid, affordable and user friendly measurement techniques, especially devoted to the analysis of the priority substances listed in the WFD, and the evaluation of the reliability and the comparability of the data produced by these methods all over Europe.

Within the project selected water matrix Reference Materials (RMs) were produced and used for the organisation of three campaigns of Proficiency Testing Schemes (PTS) at international scale, primarily addressed to monitoring laboratories. The long-term objective was to provide an on-going PT activity in support of all laboratories involved in monitoring activities for the implementation and follow-up of the WFD. The 3 SWIFT-WFD PTs focused on the assessment of laboratory performance in both inorganic (major components and trace elements) and organic (PAHs and pesticides) analytes at natural and fortified concentration levels. The results of the 3 PTs were evaluated to provide a basis of information about the performance and thus the capacities of European analytical laboratories to comply with the requirements set out in the WFD.

All the activities related to the PTs were carried out within the frame of the SWIFT-WFD and coordinated by ENEA (IT). Organization, data elaboration and evaluation were conducted by QualityConsult (IT) according to the relevant international standards and guides on PTs management *e.g.*: ISO/IEC Guide 43-1:1997, ILAC G13:2000 and ISO Guide 13528:2005.

Overall, a total of 94 selected laboratories (both applying classical and screening/emerging test methods) from 21 European Countries participated in the 3 SWIFT-WFD PTS. A total number of 5255 sets of results were received and processed in the statistical evaluation of performances.

The outcome of the SWIFT-WFD PTS provides a valuable tool to evaluate both the state of the art of water monitoring analyses and the general performance evaluation of monitoring laboratories in European member states.

AN INTER LABORATORY COMPARISON TO TEST THE PERFORMANCE OF LABORATORIES IN APPLYING THE NEW EUROPEAN STANDARD EN 14902 FOR THE DETERMINATION OF As, Cd, Ni AND Pb IN THE PM_{10} FRACTION OF AIRBORNE SUSPENDED PARTICULATE MATTER BY ETAAS AND ICP- MS

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WASP (Workplace Analysis Scheme for Proficiency) and EnACT (Environmental Analytical Chemical Testing) are Proficiency Testing programmes for the analysis of air monitoring samples and are administered by the Health and Safety Laboratory. The development of the new EN 14902 (BS EN 14902:2005) -measurement standard for sampling and measurement of metals in ambient air particles- has highlighted the need for new filter-based quality control materials to demonstrate comparability in measurements undertaken by European member states. This poster will present details on how HSL has modified a multiport sampling system to generate large batches of near identical ambient air filters for possible use in Proficiency Testing programmes. Details on how the filters were characterised using independent analytical techniques (ICP-MS, INAA, PIXE and TXRF) to produce target values will be presented as will the subsequent results from an inter laboratory comparison involving 24 laboratories from 12 countries (10 EU States plus Canada and USA).

UNCERTAINTY OF CONSENSUS VALUES FOR THE CONCENTRATION OF CADMIUM AND LEAD IN BLOOD: AN ASSESSMENT

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An important issue in External Quality Assessment (EQA) programmes, is the assignment of values to EQA materials and the estimate of their uncertainty, since these characteristics of EQA materials have an impact on the evaluation of the performance of participants. Guidance on this issue [ISO guide 43-1] indicates different methods for the assignment of values and their uncertainties, namely formulation, determination by reference method, consensus of expert laboratories and overall consensus of the results reported by participants. In addition, it is recommended that the standard uncertainty of assigned values to be less than one third of the standard deviation for performance assessment used for calculating z-scores.

In practice, owing to the complexity of most chemical analysis, the scarcity of reference methods and materials and the associated costs, assignment of values by consensus is still the most common practice in EQAS. Uncertainties of values assigned by consensus of the results of all participants in an EQA round can be evaluated by means of Algorithm A [ISO 13528]. It is expected that, in situations where considerable experience exists and established analytical techniques are available, consensus values are a reliable estimate of the measurand value and the application of Algorithm A can provide a reliable estimate of the uncertainty of consensus values.

The determination of trace elements in biological fluids is a specialised field within laboratory medicine, performed by a limited number of laboratories, but mainly by well-established atomic spectrometry techniques. As the organisers of the Italian national EQAS for trace elements in biological fluids (METOS), we were interested in evaluating the consistency and validity of such approach for the determination of consensus values and their uncertainties in this specific field. Data sets for the concentration of Cd or Pb in blood, collected in 14 consecutive EQA rounds (each comprising three EQA samples) over the period 2004-2008, were considered. Consensus values were chosen as the median of the results provided by the participants. The uncertainty of each consensus value was determined by Algorithm A. We assessed the consistency of uncertainty values calculated from different sets of data, obtained at different times, for different levels of concentrations and with a variable number of participants, and verified the suitability of such estimates with respect to the evaluation of laboratory performances based on standard deviation for performance assessment derived from both clinical and analytical considerations. Additional information was obtained from data sets referring to separate distributions of the same EQA material.

EXPERIENCES OF THE COMMUNITY REFERENCE LABORATORY FOR CHEMICAL ELEMENTS IN FOOD OF ANIMAL ORIGIN IN THE PREPARATION OF TEST MATERIALS FOR PROFICIENCY TESTING

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One of the activities of the Community Reference Laboratory for chemical elements in food of animal origin at the Istituto Superiore di Sanità (CRL-ISS) is the organization of Proficiency Testing for the National Reference Laboratories of the European Member States. These laboratories are often called to solve analytical controversies regarding the determination of chemical elements at concentrations close to the "maximum permitted levels". Thus, taking into account this aspect, and in consideration of the difficulty encountered in finding suitable reference materials in the market, since 2006 the CRL-ISS has personally taken care of the preparation of some matrices for PTs, namely milk, fish and meat, with the exception of the first fish sample. The chemical elements considered in the preparation of materials were arsenic, cadmium, mercury (fish) and lead; they were spiked, when necessary, to the various matrices at concentrations of interest such as those indicated in the Commission Regulation (EC) 1881/2006.

The preparation of the test materials from commercial foods required specific procedures according to the matrix considered. More in detail, milk samples were sent in freeze-dried form the first time, and in liquid form the second time. For this, partially skimmed milk was spiked with suitable amounts of arsenic, cadmium and lead, and frozen-dried or sterilized in autoclave, respectively. The preparation of test materials from meat, either bovine or porcine, required a more complex procedure including several steps of homogenization, spiking of As, Cd, Pb, freeze-drying, grinding, sieving, quartering and final γ-irradiation. As for fish, in consideration of the natural presence of mercury, our aim was to obtain a suitable concentration of this element in the freeze-dried material due to the endogenous content of mercury, without any spiking. For this, two different species of fish, namely swordfish and hake were mixed. At first, the two different materials were homogenized separately and then together. For As and Cd, the endogenous content of the elements was maintained as well; instead, for lead, a suitable amount of element was spiked so as to obtain a final concentration in the freeze-dried material below the maximum level allowed.

In general, the homogeneity of the test materials was assessed by means of the statistical test "sufficient homogeneity" described in the International Harmonized Protocol for PTs. The number of the bottles to be tested for homogeneity was obtained according to the formula $\sqrt{n}$ -1. The Cochran's test, applied to identify the outliers, did not show any value

to be eliminated. The homogeneity was estimated by calculating the between-sample variance and by comparing it with a critical value. In general, the stability test showed that all materials were fit for purpose.

BEST PERFORMANCE OF A LABORATORY TAKING PART IN INTERNATIONAL PROFICIENCY TESTING EXERCISES

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Participation in Proficiency Testing (PT) or Inter-Laboratory Comparison (ILC) exercises is required by the EN ISO/IEC Standard 17025:2005 for checking or proving the laboratory performance. The results from international PT/ILC exercises are also used in another major application, namely certification of reference materials, *e.g.* EURONORM CRMs.

In the end, a careful evaluation of the experimental data obtained in international PT or round -robin exercises must be performed by the organiser using statistical criteria, in order to assure the lowest measurement uncertainty at each element level.

Usually, in international PT exercises the participating laboratories may employ different analytical techniques. There are some advantages in that case because one may get a more complete element composition than that obtained by an unique analytical technique, and the "Analysis report" relies on combined results. Obviously, each participating laboratory should have validated its method for the investigated material, thus ensuring that there is no bias between different techniques.

An example is the elemental composition of the Stainless Steel ECRM 379-1, established by the KIMAB Institute (Sweden). The initial results, obtained by different atomic techniques (X-Ray Fluorescence, Optical Emission Spectrometry and Glow Discharge Source) from eighteen laboratories participating in the international PT exercise (2004-2005) organised by KIMAB, were later completed with the Antimony concentration obtained by the nuclear Instrumental Neutron Activation Analysis (INAA) technique; the Sb concentration was determined as the mean value of the results of five INAA labs. from four European Union Member countries on samples from the same Stainless Steel material, within the framework of the first ILC exercise of the mini-European INAA network (INAA-Net) built in 2005.

Participation in PT/ILC exercises obliges any laboratory to improve its performance. One way is to extend the type of techniques employed by adding a new, alternative one, different from the analytical method currently used in the laboratory. The main advantages are: completion of the element composition, elimination of bias and results with small measurement uncertainty.

The Analysis Report based on internal Inter-Technique-Comparison can provide the best performance, if established by the criteria used in international PT/ILC Schemes.

This work presents the results of a combined analysis on a Stainless Steel CRM sample carried out by INAA and OES, as our best analytical solution in view of participation in PT experiments for Stainless Steel certification.

PROFICIENCY TESTING SCHEME FOR CHEMICAL ANALYSIS OF WATER IN AFRICA

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Introduction. The Southern African Development Community Association of Water Testing Laboratories (SADCWaterLab), a union of laboratories with common interests, was established by the Southern African Development Community Cooperation in Measurement Traceability (SADCMET) to carry out a water Proficiency Testing (PT) Scheme and to facilitate collaboration among participating laboratories. The schemes were organised with the financial support of the Physikalisch-Technische Bundesanstalt (Germany), which is gratefully acknowledged. More than 40 water testing laboratories participate in the scheme, which aims to ensure that the water used in the region meets acceptable chemical limits and thus is safe for human consumption.

Purpose. Access to proper potable water is a human right. The improvement of water supply systems is crucial:

- in the fight against worldwide poverty;
- to reduce the potential for conflicts;
- to strengthen international security.

To ensure the quality of water, chemical analysis in local laboratories is necessary.

With the help of the National Metrology Institute of Germany (PTB), a PT Scheme for chemical water analysis was instituted for the SADC and EAC regions, directed by the SADCWaterLab Association. This PT system offers the opportunity for all interested laboratories to participate in a regional, affordable PT Scheme to improve and demonstrate their competence to customers, authorities and accreditation bodies.

Parameters. The following analytes are included in the PT Scheme:

- anions: chloride, fluoride, nitrate, phosphate and sulphate;
- cations:·calcium, magnesium, potassium and sodium;
- minor elements: iron and manganese;
- trace elements: aluminium, arsenic, cadmium, chromium, copper, lead, nickel and zinc.

Samples. The PT samples are prepared based on pure water by spiking with relevant chemicals. This enables to derive the assigned value directly from the formulation with a very low uncertainty.

Evaluation and assessment. The assessment of performance is based on z-scores, using the robust standard deviation of the data set as the standard deviation for proficiency assessment, provided it is lower than the fitness-for-purpose value agreed on between participants. Where the calculated value is higher, the fitness-for-purpose value is used. A method specific evaluation is made, and help is provided for laboratories, which need corrective actions.

Experiences. The quality of the laboratories is very much varying. There are some very good ones, but other have to struggle much with difficulties arising from the lack of equipment, chemicals or training for the staff. SADCWaterLab gives them the opportunity for networking and mutual help, which will improve the quality of the laboratories in the future.

MICROBIOLOGICAL PARAMETERS IN WATER QUALITY CONTROL. PROFICIENCY TESTINGS ORGANIZED BY UNICHIM AND ISTITUTO SUPERIORE DI SANITÀ

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Since many years, UNICHIM organises a number of Proficiency Testing (PT) for their members, concerning the control of the quality of water, foods, as well as of the pollution in soils, sediments and wastes. PT's are managed in conformity to the requirements of the ISO/IEC Guide 43-1 and ILAC Guidelines G-13. At the present time, from 150 to more than 250 laboratories are participating to each run.

The present contribution deals with the determination of the microbiological parameters required by Italian legislation (Total bacterial count, Total Coliforms count, *Escherichia coli, Enterococcus, Pseudomonas aeruginosa, Clostridium perfringens*). For most of the above parameters, the distribution of the results reported by the participant laboratories was found to be reasonably described by the negative binomial model with a overdispersion coefficient of ca. 0.30. Evaluation of individual laboratory performance is actually done, based on the above model, by using an assigned value corresponding to the robust average of the results obtained by a group of expert laboratories.

An important issue has to be remarked, coming from the comparison of results obtained by applying the Most Probable Numbers (MPN) and Membrane Filtration (MF) techniques in the determination of *Escherichia coli* and Total coliforms. For all the runs performed, the robust average of MPN results was found to be 20-30% higher than the corresponding robust average of MF results, the significancy of the difference between the two groups of results being confirmed by the Maximum Likelyhood Functional Relationship (MLFR) test.

PROFICIENCY TESTING SCHEME AS ONE TOOL FOR IMPROVEMENT OF THE QUALITY OF THE MEASUREMENTS

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The aim of the present paper is to evaluate some results of an interlaboratory study of metals in water matrix. In an effort to improve laboratories performing metals, anions and organic analysis in water, the SENAI-CETIND (Brazilian Industrial Learning Service - Industrial Technological Center) has implemented a Proficiency Testing Scheme program since 2001. Proficiency Testing (PT) is a means of assessing the ability of laboratories to competently perform specific tests and/or measurements. Its supplements a laboratory`s own internal quality control procedure by providing an additional external audit of their testing capability and provides laboratories with a sound basis for continuous improvement. The conduction of our external quality assurance system is based on the requirements of ISO Guide 43 and thus also corresponds to the international criteria for Proficiency Testing in chemical laboratories. Currently about 45 laboratories are participating, per round.

The water samples were prepared by gravimetric formulation. They consist of synthetic samples, which are assembled from ultrapure water, pure reagents and pure standard chemicals from NIST (National Institute of Standard & Technology). The estimation of the assigned value was obtained by the consensus value among participants using the robust statistics (median), which is less vulnerable to outliers.

Two levels of a test material containing metals were prepared. Reference concentrations were calculated by combination from the gravimetric data of sample preparation and results obtained from validated methods usually used to measure the analytes. In each series correctness of the reference values, homogeneity and stability of the PT samples were checked.

The participants were evaluated by three methodologies: z-score, E_n numbers and confidence ellipse plot.

In this Proficiency Testing most laboratories obtained good results regarding accuracy (z-score and ellipse). For all analytes: Ag, As, Ca, Cd, Co, Cr, Cu, Fe, Hg, Mg, Mn, Na, Ni, Pb, Se e Zn the percentage of satisfactory results was superior to 72%.

The percentage of satisfactory results as *Normalized Error,* was superior to 50%. Concerning to Na, As e Hg this percentage was 40, 46 and 45%, respectively. This shows that many of the laboratories are calculating the uncertainty of the measurements in a proper way. However, it was observed that the standard deviations of the measurements are not being taken in account in the calculations of the uncertainty of some laboratories.

This Proficiency Testing program must continue to be free of charge. It has to be considered as an education tool.

APPLICATION OF INTERLABORATORY STUDIES: WATER TOXICITY DETERMINATIONS WITH *DAPHNIA MAGNA*

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Toxicity tests are important for assessing the impact of chemicals on aquatic ecosystems because they indicate toxic effects of complex chemical mixtures. Several methods have been developed for rapid assessment of the dangerous biological effects on samples. Theses methods can be important improvements for the comprehensive environmental monitoring and complementary tools for implementing the new environmental directives and regulations in Europe.

However, toxicity tests using different species have shown different sensitivities and endpoints and only a limit number of tests have been standardized and validated. In order to verify the quality of biological measurements, to estimate the analytical precision of different participating laboratories using the same test, and to determine if statistically significant differences exist between results of participating laboratories, different interlaboratory exercises have been carried out.

Acute toxicity tests with *Daphnia magna* are widely used and has many advantages, such as high sensitivity and short reproductive cycle. In order to verify the quality of such tests the National Institute of Chemistry, organized an interlaboratory comparison. Several interlaboratory trials named "ILC-Waste Water" (ILC-WW) were organized in the last seven years. Toxicity with the *Daphnia magna*- mobility inhibition test (ISO 6341) was included in eight rounds and about twenty laboratories, mostly from Slovenia, took part in each study. The toxic potentials of samples were estimated by using standard bioassay organisms which were exposed to a known concentration of the sample for a specified time (24 hours) and conditions. The toxicity of the sample is generally expressed as the concentration that affects 50% of the exposed organisms and is defined by the term EC50.

Two samples (T1 and T2) at two different concentration levels were prepared for the acute toxicity test. For sample T1 the 24h EC50 was expected between 10-80 vol.%, and for the T2 between 0.1-10 vol.%. The samples (simulated wastewater) were carefully prepared and their homogeneity and stability were verified.

The purpose of the scheme was to enable participants to check their day-to day performance. The results of the toxicity tests were very good. The coefficient of variation of the participants results decreased for sample T1 from 39.6% in the first comparison to 14.5% in the ILC WW 6, and for sample T2 from 62.9% to 30.2% in the ILC WW 12, respectively.

MANAGEMENT OF A PROFICIENCY TESTING FOR ANIMAL NUTRITION LABORATORIES

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An interlaboratory program has been conducted for the comparison of results provided by laboratories that perform feed analyses of animal nutrition carried out by the Embrapa Cattle-Southeast, a Brazilian Agricultural Research Organization. The program structure and normalization are accomplished in accordance with the ABNT ISO/IEC GUIA 43 rules and with the harmonized international protocol of proficiency assays in analytical laboratories. All steps, from data acquisition to furnished the results, are controlled by a website, by using a dedicate program especially developed to bank of data control. Nowadays, 44 laboratories, provided from Universities, governmental research centers, and private companies, representing all the Brazilian regions, have been participating of the program. The essays foreseen by the program are the normally carried out by animal nutrition laboratories in animal feeds and mineral supplements, such as determination of dry matter, *in vitro* digestibility, neutral detergent fiber, acid detergent fiber, crude protein, ether extract, lignin, ash and macro- and micronutrients (Ca, P, Mg, K, S, Cu, Fe, Mn, Zn, and Na). Four turns of the program is yearly performed, each two months, and each parcel is composed by three different kinds of animal feed and mineral supplements. The statistical project employed to evaluate the laboratories scores is based on the rules recommended by the ABNT ISO/IEC GUIA 43-1, and the score z is adopted. For the values designates evaluation, median and robust standard deviation, the consensus values based on the results provided by the participants are considered. Another proposition is to prepare and validate non certified reference materials that could be used as internal quality control by the participants of the program. This work presents the experience in coordinating the intercomparison exercise.

PRELIMINARY REPORT ON THE ORGANISATION OF A PROFICIENCY TESTING PROGRAM IN FOOD MICROBIOLOGY AS A TOOL TO GUARANTEE THE EQUIVALENCE OF THE OFFICIAL CONTROL SYSTEM

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After *Listeria monocytogenes* was isolated in products imported from Italy, in 2002 the Italian Ministry of Health developed an integrated approach to assure the equivalence between the Italian and the US official control systems. An integral part of the ongoing program is laboratory proficiency-testing; its aim is monitoring the technical skills of the official laboratories carrying out microbiological tests for *Salmonella spp*. and *Listeria monocytogenes* on meat products to be exported in the USA.

This paper describes the protocol for production and control of the batches of matrix samples contaminated with *Listeria monocytogenes* and *Salmonella spp*. (target microrganisms) used in the interlaboratory study. The batches were made of a set of 35 samples; some of which contaminated with the target microrganisms at two different concentration levels (L1=10-50 CFU/g; L2=300-1,000 CFU/g). Negative samples were contaminated with a pool of non-target bacteria (10,000 CFU/ml). After preparation, all samples were stored frozen until their shipment to the participating laboratories.

On the basis of the results provided on 35 samples, it is possible to assess the laboratory technical skills to provide correct results with a 89.1-100% probability level (p=0.95).

Each batch was tested for stability and homogeneity and the results were statistically assessed. For each level of contamination L1 and L2, homogeneity was evaluated before freezing by means of microbial count on 20 samples; after freezing, microbial counts on 20 samples were performed at fixed intervals (4, 8 and 16 days). Stability was evaluated by means of microbial counts on 3 samples at 0, 2, 4, 6, 8, 10, 12, 14, 16, 26, 36 and 46 days.

The results of homogeneity tests on L1 samples were analysed by means of the Cochran dispersion index, while homogeneity of L2 samples was evaluated by means of the Kolmogorov-Smirnov test, using k and p values.

The optimal stability point was identified by means of regression analysis, *i.e.* samples were considered stable when the slope of the regression line was not statistically different from zero (CI 95%).

Each participating laboratory received a batch of samples, stored in dry ice, within 24 hours from shipment. Management of the interlaboratory trials was achieved by means of a Website, where participants were allowed password protected access in order to subscribe, send their results and download protocols.

The results of the homogeneity and stability tests carried out in 2006-2008 during six interlaboratory trials confirmed the reproducibility of the protocol for the preparation of the sample matrix.

INTEGRATION OF TARGET MEASUREMENT UNCERTAINTIES IN PROFICIENCY TESTING SCHEMES: A CASE STUDY FOR WATER ANALYSIS IN SLOVENIA

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Generation of analytical data in the environmental sector, which are "fit for purpose" is designated as "high" priority in the EU. To ensure reliable and comparable chemical measurements, it is necessary to have unified systems on international and national levels which will enable chemical analysts to attain and demonstrate the comparability and traceability of their measurements. To achieve this goal, the following tasks must be established: use of validated methods, procedures for uncertainty evaluation, traceability establishement, and implementation of quality assurance/quality control system. Proficiency Testing as external quality assesment plays role of independent evidence of laboratories' performance. To enable laboratories to fulfill requirements stated in legislation, methodology for evaluation of laboratories' performance in Proficiency Testing Schemes shoud incorporate principles of measurement results which are fit for intended use and incorporate the target measurement uncertainty.

Proficiency Testing Scheme, organized by the National Institute of Chemistry, Ljubljana, Slovenia, was designed specifically to support EU Drinking Water Directive (98/83/EC). Methodology for scoring system, which takes into account "fitness for purpose" based standard deviation for proficiency assessment, was developed and applied. A description of the scheme is given: preparation of test material, homogeneity and stability testing, reference measurements for establishing independent reference value with stated traceability, and setting performance criteria for the participants. Modified zeta-score by application of target uncertainty was applied in way that it fulfilled requirements defined in the Drinking Water Directive. Results are reported for nitrate in water as illustration. The presented approach can also be applied in other spheres of measurements.

WASTEWATER PROFICIENCY TESTING: A PORTUGUESE CASE

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RELACRE in partnership with the Laboratory of Environmental Analysis and Quality Control of INETI organized in 2007 an interlaboratory test using a real domestic effluent that underwent the preliminary treatment.

The wastewater sample was sampled and continuously recirculated under nitrogen atmosphere in a 120 L closed plastic container, by external pumping. Sub-samples were obtained and properly preserved until distribution.

Preliminary tests were performed to assess the homogeneity for 13 parameters: pH, Total Suspended Solids (TSS), biochemical oxygen demand after 5 days (BOD_5), Chemical Oxygen Demand (COD), phosphorous, ammonium nitrogen, Kjeldahl nitrogen and metals (Mn, Cu, Cd, Pb, Fe and Cr). For metals, the sample was spiked since its original content was lower than the desired target values. The homogeneity tests failed for BOD_5, CQO and TSS due to the heterogeneity of suspension matter, in our view, and these three parameters were not considered for distribution.

Measurements made by INETI were traceable to reference materials, and measurement uncertainties were estimated using component by component approach ($N\text{-}NH_4$ and N-Kjeldahl) and intralaboratory approach based on validation and quality control data (pH, total P , Mn, Cu, Cd, Pb, Fe and Cr).

The assigned values were established either by using the target values provided by INETI (N-Kjeldahl, $N\text{-}NH_4$ and Cu) or consensus values from participants (pH, total P, Pb, Fe, Mn. Cd and Cr) - consensus values were obtained using robust statistics recommended by ISO 13528:2005. Legal performance criteria requested by Portuguese legislation (Decreto-Lei Nº 236/98) was used to obtain the assigned standard deviation.

Performance of participating laboratories was assessed using *z*-scores, and totally satisfactory results were obtained for pH and Pb (100%); for the other parameters, mostly satisfactory results were observed: N - Kjeldahl (95%); Cu, Cd and Mn (88-87%); Cr and N-NH4 (76-75%); total P and Fe (70-67%). The normalized error was used as an additional evaluation tool for those participants reporting uncertainties.

In this work we report and assess the results of this Proficiency Test, where 32 laboratories participated.

THE ORGANISATION FOR THE PROHIBITION OF CHEMICAL WEAPONS OFFICIAL PROFICIENCY TESTS: CURRENT STATUS AND FUTURE DIRECTIONS

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The Organisation for the Prohibition of Chemical Weapons (OPCW) has been organising and conducting Official Proficiency Tests (PT) since 1996 in accordance with ILAC-G13 to certify laboratories for the analysis of authentic samples under the provision of Chemical Weapons Convention.

The tests are part of a mechanism to ensure that there are laboratories that have proven competence in the analysis of chemicals related to the Convention. Laboratories that have successfully competed the tests are designated by the Director General of the OCPW for analysis of samples. To maintain the Designated Laboratory status, a laboratory must take and pass at least one of the two Proficiency Tests offered per calendar year.

Unlike many Proficiency Tests, the OPCW Proficiency Tests are qualitative, that is the laboratories must determine if any of a very large set (essentially infinite) of chemicals relevant to the Convention are present in the samples. The tests are organized with the assistance of two laboratories, one preparing the test samples, and the other evaluating the test results. The poster provides an overview of the current status of these PTs and outlines salient features regarding procedure and scope of tests, selection of assisting laboratories, scoring and performance rating of participating labs. The emerging issues and future directions are also discussed.

PERFORMANCE OF MM ESTIMATORS ON MULTI-MODAL DATA: A POSSIBLE ALTERNATIVE TO KERNEL DENSITY AND MIXTURE MODELLING METHODS

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The presence of substantial sub-populations in PT data can sometimes lead to multimodality or to other substantial asymmetry. Multimodality and strong skew both compromise the estimation of consensus values and the subsequent interpretation of PT data. Because of the comparatively large proportions of data involved and the asymmetry, traditional robust estimates which rely on symmetry (such the median and the Huber estimates described in ISO 13528) can be appreciably biased. Although one extreme way to treat this problem is to abandon the round, this is undesirable if there are reasons to believe that the majority are performing properly and that the skew or multimodality arise from real and undesirable differences in execution of test methods. The present IUPAC Harmonised protocol therefore includes guidance on identifying the major mode and assigning a value and uncertainty based on the major mode alone.

The IUPAC guidance relies on kernel density estimation and bootstrapping, which can be effective but is comparatively complex. In this paper, the behaviour of "MM-estimators" on such data is examined. MM-estimators are robust estimators with high efficiency and high breakdown point, and rely on the use of a redescending characteristic function which allocates extremely low or zero weights to extreme points. It is shown that use of such estimators with deliberately reduced bandwidth (and therefore somewhat reduced efficiency) can provide estimates of major mode location which are similar to kernel density modes and less biased by the minor modes than the median or other M-estimates (such as the Huber estimate) described in ISO 13528. They therefore offer a possible alternative approach to the treatment of data containing a substantial biased sub-population.

PROFICIENCY TESTING ACTIVITY AT AFSSA FOUGERES COMMUNITY REFERENCE LABORATORY FOR ANTIMICROBIAL RESIDUES IN FOOD

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Monitoring of veterinary drug residues in the European Union is based on the Council Directive 96/23/EC. One of the duties of the Community Reference Laboratory (CRL) is to organize Proficiency Testing for the analysis of antibiotic residues in food from animal origin. Proficiency Testing is intended to allow the participants to test their routine methods and to assess their competence on this specific analysis. Laboratories authorized to perform official residue controls in accordance with the Council Directive 96/23/EC must be integrated in an internationally recognized external quality assurance and accreditation system.

The CRL is accredited according to ISO/IEC 17025 by COFRAC, the French accreditation body and intends to apply to COFRAC also for the accreditation of its Proficiency Testing activities, according to the document LABCIL REF 02 rev02 of September 2007: "Organizers of Proficiency Tests - requirements for the accreditation". This document is made of two parts: management requirements, from ISO/IEC 17025, and technical requirements, from the ISO Guide 43-1.

In order to fulfil each requirement of this document the CRL decided to develop two documents: a quality manual and a quality plan. The conclusions of this work will be presented, describing how the CRL plans to satisfy the requirements for accreditation and deal with critical activities such as: the preparation of suitable materials; homogeneity and stability testing; coding and shipping of test samples; calculation of the assigned value; evaluation of laboratories performance by means of z-scores and choice of the standard deviation for proficiency assessment.

ROLE OF PROFICIENCY TESTING AND EXTERNAL QUALITY ASSESSMENT IN BUILDING A QUALITY ASSURANCE SYSTEM IN REGULATORY TESTING LABORATORIES: AUDITORS' EXPERIENCE

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The laboratories at Istituto Superiore di Sanità (the leading technical and scientific public body of Italian National Health Service) carry out analytical tests on drugs, medical devices, food, water or other types of materials. In this work, the authors describe their experience gained as quality assurance auditors of those testing laboratories, as well as of similar national and international structures, and highlight some of the most important points for effective management of quality assurance systems that regulate control and experimental activities.

Quality assurance systems, whether they are applied to testing laboratories or more in general to regulatory service providers, should be accompanied by the following key aspects:

- role of human factor (technical/scientific staff and management): all those participating in the structure activities significantly affect the quality of services provided, whatever their field of activity in the structure;
- role of a systematic approach: this is a powerful tool for effectively and efficiently implementing all instruments of quality (planning, implementation, verification, reaction), and it results into optimizing resources and achieving the main objective: the stakeholders satisfaction to the quality of service provided. In particular, in the case of testing laboratories for drugs, vaccines or medical devices, stakeholders are normally the Competent Authority, while contractors may also be private bodies according to applicable laws;
- role of Proficiency Testing and External Quality Assessment: they are instrumental in driving the efficacy of quality assurance systems in networked laboratory activities.

Examining data collected during approximately 300 audits, it is evident that a coordinated effort of all components is always needed to achieve an efficient quality assurance system management in control and experimental activities. It is also evident that active and continuous participation of technical/scientific personnel to Proficiency Testing and External Quality Assessment is a critical asset for the structure, as shown by testing laboratories that are routinely involved in such exercises.

It can be concluded that a quality policy that requires systematic approach to quality management and foresees Proficiency Testing and External Quality Assessment programs in the analytical laboratories is one of the most important issues for an efficient quality assurance system in control and experimental activities.

The final outcome is to keep the system under control and within pre-established parameters as well as structure reliability. This in turn contributes to facilitate bilateral-multilateral recognition and agreement procedures with public and private bodies, providing clear advantages in terms of both economics and image.

HAS PROFICIENCY TESTING ACTUALLY IMPROVED LABORATORY PERFORMANCE OVER TIME?

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Introduction. Proficiency Testing Schemes are available and open to laboratories in a wide range of industries and are designed to promote quality and comparability in the measurement of a vast range of matrices and test methods. One important aspect of their design is the monitoring and improvement of the quality of a laboratory's capability to perform analysis over prolonged periods of time. Information obtained in this way can assist in the evaluation of methods, instrumentation, the education of laboratory staff and demonstrate to third parties the quality of data produced. Theoretically, long term involvement in Proficiency Testing allows participating laboratories to analyse trends in their performance over time which subsequently allows them to identify potential concerns and failures within their systems and the constant cycle of 'test, evaluate and assess' should ensure that quality is continuously reviewed and improved.

Methods. Historical data from various Proficiency Testing Schemes was statistically analysed to demonstrate where involvement in third party testing, over a prolonged period of time, has improved the performance of those laboratories taking part.

Various approaches have been used to identify and quantify these improvements. These include:

- a review of improvements over time demonstrated by evaluating the relative percentages of laboratories successfully achieving satisfactory z-scores;
- a summary of RSD's for defined methods over time;
- a review of improvements in the coefficient of variation (%CV) over time;
- a review of laboratory performance indicating trends over a prolonged period of time;
- an example indicating where Proficiency Testing data has been used as part of the validation of the introduction of a new European reference method.

Results. Results covering a variety of food and beverage industries have been collated and in all cases it has been demonstrated that long term participation in Proficiency Testing has improved the quality of the laboratory testing. The data analysed covers the dairy, non alcoholic beverage and alcohol brewing industries and in each case an increase in results being classified as satisfactory that clearly demonstrated over the period of the investigation. In addition, reductions in %CV and the standard deviation of results, returned by participating laboratories, indicate long term improvements have been made.

Conclusions. Statistics, analysed from a variety of Proficiency Testing Schemes, demonstrate that consistent improvements in performance have been attained by using the analytical tool of Proficiency Testing.

PROFICIENCY TESTING RESULTS OF EU NATIONAL REFERENCE LABORATORIES RELATED TO THE DETERMINATION OF CHEMICAL ELEMENTS IN MILK, MEAT AND FISH

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From the end of 2005 to 2007, the Community Reference Laboratory for chemical elements in food of animal origin at Istituto Superiore di Sanità (CRL-ISS) has organized six rounds of Proficiency Testing for the National Reference Laboratories (NRLs) of the EU Member States regarding the determination of some chemical elements (As, Cd, Hg, Pb) in food of animal origin. During the exercise, the number of participants (one for each Member State) varied due to the entry of new States in the Union. The matrices taken into account were milk, meat (bovine, porcine) and fish. The test materials used for PTs, containing the elements at concentrations close to the "maximum permitted levels" (Cd, Hg, Pb), were prepared in house, with the exception of the first fish sample. In the whole, the NRLs analysed two samples of fish, four samples of milk, two samples of bovine muscle and one sample of porcine meat, mainly using methods based on the ETA-AAS and ICP-MS.

Among the various methods proposed for the scoring of results, we chose the z-score approach. In our scheme, the assigned value for each test material was derived from the robust mean of results (Huber H15); taking into account the restricted number of participants (n=21-25), the factors which can compromise robust estimates (*e.g.* different analytical methods) were also considered. In general, as for the standard deviation for Proficiency Testing assessment (σp), we used a value derived from the Horwitz equation for element concentrations >120 μg/kg, and the more stringent Thompson equation for lower concentrations.

Excluding lead, there is a high percentage of z-scores less than or equal to 2, specifically for Cd and Hg which showed a percentage of values higher than 95% in the satisfactory class; moreover, the frequency of z-scores less than 1 resulted quite high inside the class lower than 2 for all elements. As for arsenic, about 4.7% of z-score values resulted in the range $2<Z\leq3$, and about 4% were >3. However, the frequency of these values was higher at high concentrations usually found in fish. Thus, if we exclude the fish matrix, the percentage of satisfactory z-scores increases up to 96%. With regard to lead, the results showed that the analysis of this element seems to be still the most difficult one; however, the outcome of the last year of PTs was quite satisfactory, with a decrease from 9% to 2% in the unsatisfactory class. As for mercury, the frequency of z-scores in the range from 2 to 3 resulted very low, and this could be linked not only to the availability of simple techniques but also to a greater frequency of incurred samples at measurable concentrations, often close to the maximum permitted limit.

The results derived from these PTs constitute a useful basis to define a value of σ_p so as to match the optimal requirements of the NRLs.

MONITORING OF PT/EQA PERFORMANCE OVER TIME IN AN END-USER, MULTI-SITE LABORATORY WORKING IN THE FIELD OF FOOD MICROBIOLOGY

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To report a satisfactory result in Proficiency Tests is not the final objective of a laboratory. A more critical issue is managing the results obtained by the participants inside the laboratory on each sample, and on the samples over time.

A correct aggregation of the results can give both useful information about each lab-technician, and, over time, information about the quality of the media and the reagents used, about the management of apparatus etc.

We are a multi-site public laboratory (6 laboratories in different areas of Puglia and Basilicata) performing the official microbiological control of food samples. Twice a year, we carry out an external PT/EQA on every analytical method. Currently, each lab-technician performs the test twice on each PT-sample. For each sample, we need to evaluate about 24 results for which we need appropriate tools.

Bearing in mind the graphic prepared by CEFAS for evaluating the results of the Shellfish Scheme (Summary Results - EQA for Food - Shellfish Scheme Health-Protection-Agency CEFAS), we constructed an electronic control chart for the quantitative methods: the samples examined over time are put on the x axis and the value of z-score established by PT providers on the y axis. The data from each lab-technician is arranged as a series. The higher and lower acceptable values are put as ± twice the value of the z-score, while higher and lower critical limits as +/- three times the value of the z-score.

Through the Control chart it is possible to evaluate easily the performance of each technician and of each sub-laboratory; any individual deviation can soon be recognized as a true casual mistake and the tendency of a single technician to go out of acceptable and/or critical values is quickly recognized. The Control chart also helps to recognize if the lack of quality occurs in one, more or all sub-laboratories. One can quickly gather the indications on what are the possible causes: for example, given that the cultural media are centrally produced and later on distributed to all, a wrong result obtained by all the technicians highlights a likely problem with the medium used, while if all of the technicians of the same sub-laboratory give the wrong results, it is probable that the equipment used is out of control.

A WORLD-WIDE PT INFORMATION SYSTEM: INTRODUCTION TO EPTIS

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EPTIS (www.eptis.bam.de) is an internet based information system on Proficiency Testing Schemes (PTS). Today more than 900 PTS from 22 countries are listed. For each scheme key information concerning *e.g.*:

- testing fields;
- test items;
- tested properties;
- frequency of performance;
- participation fees;
- accreditation status;
- contact details of the PT provider,

is provided for a first selection. Additionally, further information is available with regard to *e.g.* the quality system of the PT provider and the statistical protocol used for the evaluation of the results. The questionnaire which is used to collect the respective information was developed on the basis of ISO/IEC Guide 43 in order to enable EPTIS users to make their selection on a sound basis.

Background. EPTIS is available in the internet since 2000. It was developed within a project sponsored by the European Commission by 16 European partner organisations and started with approx. 500 registered PTS. Meanwhile EPTIS has grown considerably, both in terms of schemes and country participation. At present the EPTIS consortium consists of 32 partners from Europe, the Americas and Australia but not all of them have already national PTS listed in the system. Since its start also the number of database inquiries has been steadily increasing: on the average more than 2,000 individuals are using the database each month. Additionally the EPTIS secretariat is increasingly being contacted by e-mail in particular by users from developing countries who need advice. This reflects the growing importance of Proficiency Testing within the accreditation process.

New software version. Recently a new EPTIS version has been launched after having reprogrammed the query tool for the database. Among others our aim was to develop easier access to the existing data in a more user friendly manner. The major advantage is that the new programme allows for a full-text search. Moreover, a general help function (help button) and additionally a context-sensitive help function have also been implemented. The web design was also changed in order to provide barrier-free access for handicapped persons.

Future development. A major task for the future will be giving the PT providers the opportunity to register and update their schemes themselves in a controlled process with the involvement of the national coordinators. This should help to facilitate the task of the latter.

THE PROFICIENCY TESTING IN FOOD MICROBIOLOGY "AQUA" OF ISTITUTO ZOOPROFILATTICO SPERIMENTALE DELLE VENEZIE: AN EXAMPLE OF HOMOGENEITY AND STABILITY OF TEST SAMPLE AND Z-SCORE CALCULATION

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The ISO/IEC Standard 17025:2005 requires laboratories to demonstrate their competence and the quality of their results and emphasises the importance of interlaboratory comparisons as an independent means to demonstrate results' reliability. To satisfy these requirements, in 1999 the Food Microbiology laboratory of the Istituto Zooprofilattico Sperimentale delle Venezie (IZSVe) started an interlaboratory comparison programme for food microbiological tests, known as "AQUA - interlaboratory comparisons for QUAlity Assurance", integrated in 2006 with quantitative tests.

In this paper the methods used to set up Proficiency Testing Schemes for quantitative tests are presented, including production of test samples, statistical methods for estimating their homogeneity and stability, data processing and data evaluation. Contaminated test samples (lyophilised food matrices) are produced using certified *American Type Culture Collection* (ATCC) bacterial strains. Homogeneity is controlled before each interlaboratory trial and stability is checked during the whole period allowed for testing. For homogeneity and stability tests m=10 and n=5 units, respectively, are randomly selected from the original stock and two portions of each sample are tested.

After removal of outliers according to the Cochran's test, assessment of sufficient homogeneity and stability are carried out according to the IUPAC Harmonised Protocol (2006). Test samples, one bottle for microorganism, are sent refrigerated, to the participants, who analyse them (more repetitions for more operators) and report their results to IZSVe through e-mail or fax. The data are usually transformed in log-data to obtain a normal distribution. Outliers, identified by the Box plot and the Grubb's test are removed The normality of the distribution is verified by the Shapiro-Francia's test. For the evaluation of laboratory performance, z-scores are calculated as follows and evaluated according to the criteria reported given in the ISO Guide 43-1:1997:

$$Z - score = \frac{(x - X_a)}{\sigma_p} \text{ where}$$

x= observed result (log); X_a= assigned value (median value of data without outliers) (log); σ_p= target standard deviation for proficiency assessment defined on the basis of previous data.

This scheme for the analysis of quantitative data is in place only since 2006, so there are not sufficient data to draw a trend of the percentage of successes, also because differences

in performance can arise from the variability of the quantities of microorganism chosen in each trial and the variable number and type of participants (on average: 35-40) in each trail. However, the analysis of the results of the same laboratory over several trials by means of control charts allows to evaluate the trend of the laboratory and to verify that the results are in statistical quality control.

CONSIDERATION CONCERNING INTERLABORATORY TEST FOR CEMENT ORGANIZED BY THE TESTING LABORATORY OF CEPROCIM S.A. IN THE LAST 20 YEARS

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CEPROCIM S.A. laboratory has organised yearly, since 1988, Interlaboratory Test for cement, with over 40 laboratories representing Research institutes, Cement plants, Pre cast units, Hydro plant construction companies, Industrial construction companies, Building companies etc. from nine countries - Romania, Republic of Moldova, Croatia, Serbia and Montenegro, Macedonia, Bulgaria, Hungary, Ukraine and Lebanon.

The subject of the Interlaboratory Test for cement were 37 laboratory test: twelve chemical determinations (Loss on ignition, SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, Free CaO, Insoluble residue, Na_2O, K_2O, Cl^-), seven physical tests (Residue on the 90 μm sieve, Density, Specific surface area, Standard consistency, Initial setting time, Final setting time, and Soundness) and nine mechanical tests (Weight at demoulding; Flexural strength at 1, 2, 7, and 28 days; and Compressive strength at 1, 2, 7, and 28 days) carried out with two types of sand - from CEPROCIM and from each participant laboratory.

An analysis of the efficiency of Interlaboratory Test for cement made for the last 20 years generally shows a decreasing trend for the coefficient of variation (CV) of both repeatability and reproducibility. Between 1988-1995 when the old Romanian standards were used, CV of chemical, physical and mechanical determinations present constant or decreasing values in time. Since 1995 and up to 2008 when the European standards of method EN 196 series were applied, after a critical period of transition, CV of chemical, physical and mechanical determinations continuously decrease.

The results strongly prove that Interlaboratory test is one of the most efficient tools to establish a common language for all the participant laboratories doing the cement testing procedures.

THE NPL ENVIRONMENTAL RADIOACTIVITY PROFICIENCY TEST EXERCISE

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Since the late 1980s, the National Physical Laboratory (NPL) has organised 13 Proficiency Test exercises for a range of radionuclides including fission products and activation products of both reactor construction materials and nuclear fuel. The exercises were designed to identify analytical problems, to support UKAS accreditation and to provide a regular forum for discussion and technology transfer in this area. The exercises have been run approximately once every eighteen months by NPL. In the early exercises, the participants almost exclusively originated from the UK measurement community, while in the later exercises more overseas laboratories took part. In the most recent Proficiency Test exercise (2007), more than half of the 64 participants represented overseas laboratories.

The range of sample types available for analysis has been mainly aqueous. In the 2007 exercise, eight samples were available for analysis (including a solid material; neutron activated concrete powder) containing in total 28 alpha-, beta- and gamma-emitting nuclides at several concentration levels. Three tests were used to assess the performance: the zeta test, the *z*-test and the relative uncertainty outlier test. A result was only classified as 'in agreement' when all three tests were passed. The data was graphically presentated as several plots: i) deviation plots; ii) zeta score plots; iii) relative uncertainty plots; iv) "Kiri plots" in which the squared ratio between the laboratory uncertainty u_L and the standard uncertainty for proficiency assessment σ_p is plotted against the *z*-scores.

This paper reports the execution of the 2007 exercise carried out by NPL to assess the performance of the participating laboratories. The main part of the exercise was to identify and/or traceably quantify the activity levels of radionuclides present in the samples. The certified activity values of all nuclides were traceable to national standards of radioactivity. This traceability to national standards in turn provides traceability at an international level to the ultimate reference point of all measurements, the SI reference value maintained by the Bureau International des Poids et Mesures (BIPM).

PROFICIENCY TESTING SCHEMES AS TOOLS FOR THE SUCCESSFUL IMPLEMENTATION OF THE WATER FRAMEWORK DIRECTIVE AT EUROPEAN LEVEL: THE TAQC-WFD EXPERIENCE

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The reliability (Quality Control) and comparability of measurements is a key issue and requires not only the availability of suitable Quality Control (QC) tools, but also their correct implementation and use in a harmonized way both within and beyond Community level.

In 2000 the European Commission has approved the Water Framework Directive (WFD) (Directive 2000/60/EC), and subsequently funded several projects with the main objective of providing support to the successful implementation of this WFD. The success of the WFD implementation in analytical laboratories does not depend only on the improvement of the already existing laboratory staff, but also on creating a "correct" culture in the younger generation of scientists for the appreciation and future implementation of the directive. This latter was the primary aim of the TAQC-WFD project financed by the European Commission under contract nr MSCF-CT-2005-029922 from 01/02/2006 to 31/01/2008.

The TAQC-WFD consortium included 5 partners: QualityConsult (IT, Coordinator), University of Malta (MT), Corvinus University of Budapest (HU), University of Warsaw (PL) and Mermayde (NL). The TAQC-WFD series of events comprised five training events using a similar format organised in different regions of Europe to allow the participation of young researchers. The in total 139 participants came from 32 countries (Europe and 3rd countries).

The courses consisted of lecturing by recognised experts and practical exercises. These practical exercises consisted of two components: computer assisted training during the course (uncertainty calculations, preparation of control charts, PT evaluation) and follow up exercises in the home institutions (participation in a Proficiency Testing (PT) Scheme; setting up and maintaining Control Charts).

The TAQC-WFD Proficiency Test exercises were organized on matrix test materials relevant to the WFD:

- TAQC-1 - trace elements in river sediment;
- TAQC-2 - poly-aromatic hydrocarbons in river sediment;
- TAQC-3 - major components in spring water.

All these PT exercises had a significant number of participants. The evaluation reports of the TAQC-WFD PTs provided a very good tool to measure the state-of-the-art performance of young researchers in Europe and its vicinity, especially in comparison with similar reports produced on the performance of routine laboratories of the field.

The results of both follow-up exercises were discussed in a technical meeting, organized as 6th event of the series, under the guidance of experts in the field.

The poster will discuss the results of the Proficiency Testing exercises in detail.

A PROFICIENCY TESTING SCHEME TO DETECT VIRAL FISH DISEASES

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A Proficiency Testing Scheme has been provided by the European Community Reference Laboratory (CRL) for Fish Diseases every year since 1996. The test is provided to all European National Reference Laboratories (NRLs) that are obliged to participate. As the test is also provided to a limited number of non-European NRLs, the total number of participants is about 35.

The test is primarily designed to assess the ability of participating laboratories to identify and quantify the notifiable non-exotic fish pathogenic viruses: Viral Haemorrhagic Septicaemia Virus (VHSV), Infectious Haematopoietic Necrosis Virus (IHNV) and Spring Viraemia of Carp Virus (SVCV), but also to assess their ability to differentiate other fish viruses such as infectious pancreatic necrosis virus, perch rhabdovirus etc.

Five coded ampoules are provided to participants containing lyophilised supernatant from infected cell cultures. Participating laboratories are asked to identify viruses and to perform a titration of viruses in order to assess cell susceptibility towards virus infection. Participants are asked to reply within 8 weeks of receiving the test. The CRL collect the data and provide statistical and graphic pictures of the performance of the individual laboratory relative to other participants.

The Proficiency Test has been used for additional purposes, *e.g.* to test the ability of participants to identify double infections, to assess cell line susceptibility between laboratories, to test the ability of laboratories to genotype virus isolates, and to analyse the inter-laboratory quality of sequencing results. In this poster, we present results and experiences obtained from this Proficiency Test.

DESCRIPTION OF CONCENTRATION DEPENDENCE OF STANDARD DEVIATIONS IN DRINKING WATER PTS USING THE VARIANCE FUNCTION FROM ISO/TS 20612

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Introduction. In the past there were many attempts to describe the concentration dependence of standard deviations in interlaboratory comparisons, the Horwitz model being the most popular general model: The fact that this model does not distinguish between different parameters and/or analytical techniques has been criticised in the past.

ISO 5725-2 (clause 7.5.2) also describes possible functional relationships between precision and the mean level, lg sR=c + d lg m (or sR=c md) being one of these, where c and d are empirical parameters of the function and m is the content of analyte as mass fraction.

In ISO/TS 20612 a variance function sR=exp(theta0 + theta1 ln m) is recommended to calculate the standard deviation for proficiency assessment where the standard deviation can be expected to follow such a model (theta0 and theta1 are again empirical parameters). This approach is equivalent with the ISO 5725-2 approach with c=exp(theta0) and d=theta1 and the Horwitz model is a special case of these functions, where c=0.02 and d=0.8495.

Drinking water PTs. Data from more than 60 PT rounds organized by four different PT provider were used to calculate variance functions for 90 different parameters. These coefficients are presented and where possible (*i.e.* where it was possible to express the analyte content as a mass fraction) compared with the classical Horwitz function. In all cases the standard deviations for drinking water analysis were far below the values calculated using the Horwitz model, which originally was developed for food analysis.

These parameter specific variance functions may be used for estimation of the concentration dependence of measurement uncertainties and also for plausibility checks of measurement uncertainties estimated in individual laboratories. Finally, of course, they give an indication about the quality of drinking water labs in Germany in total.

TRACEABLE REFERENCE VALUES FOR DRINKING WATER PROFICIENCY TESTING

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Introduction. In the analysis of environmental samples it is difficult to ensure the traceability of the measurements to SI units, because calibration is normally done using matrix-free calibrants, but the matrix is expected to influence the determination. One possibility to solve this problem is the use of certified reference materials. But often those reference materials are not readily available. The idea was therefore to use existing Proficiency Testing laboratory intercomparisons for this purpose by spiking real matrices (drinking water, groundwater or wastewater) with the chemical species to be analysed. To realise traceable reference values, an uncertainty budget can be calculated for the added amount of measurand. The mass concentration of the measurands in the original matrix can be calculated from the consensus means of the participants' data and the added amount in a standard addition like-method.

Reference values. To ensure traceability of reference values, the procedure of sample production (stock solutions, dilutions, sample lot) must be done on a gravimetrical basis with chemicals of highest possible purity. The mass of the final lot is determined by measuring the density of the solution. All is done with calibrated balances and thermometers which ensures the traceability.

The consensus means are obtained from the participants results using robust statistical methods and the uncertainties are calculated according to ISO 13528. The uncertainties of the spikes were calculated by establishing a fully uncertainty budget. The matrix concentration and its uncertainty were calculated using generalized least-square regression using the computer programm B_LEAST. The final reference values for the mass concentration were calculated from the matrix mass concentration and the mass concentrations resulting from the spikes. The uncertainties of these reference values were calculated from the combination of the uncertainties of the matrix value and the spikes.

Consensus means and reference values of As and Sb - an example. The mentioned procedure above was applied in an AQS Baden-Württemberg PT for elements in drinking water in 2007. The resulting uncertainties of the reference values were in the range between 1.1% (for the highest level of As) and 9.6% (for the lowest level of Sb). It was observed that especially for higher mass concentrations, the uncertainties of the reference values were much lower than those of the consensus values.

Conclusions. The procedure for calculating reference values without reference measurements seems to be a promising tool to introduce traceability into routine analytical chemistry and will be presented. The most serious problem that is still to be solved, is the traceability of the purity of the spiking substances used. The presentation will also give an overview of experiences from further PTs.

USE OF VARIANCE FUNCTIONS FOR ASSESSMENT IN PROFICIENCY TESTING

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Problem. In PTs often several samples are distributed that are of the same type, but differ in concentration. If a sample-by-sample evaluation is performed there will be fluctuations in the variances estimated from PT data. If these standard deviations are used for proficiency assessment, a participant is punished for being in a data set where the estimated standard deviation is low because of random variations. If he is in a data set with a high standard deviation (again due to random effects) it will be easier to pass the PT.

Solution. According to ISO 13528 (clause 6.4) a model may be used to derive the standard deviation for proficiency assessment. ISO/TS 20612 describes the calculation of a variance function from PT data, if similar samples are used that differ in the concentration of the analyte only. In these cases we can assume that there are no steps or discontinuities in the concentration dependence of the variance. The use of such a model instead of the standard deviations estimated from participant's data on a sample-by-sample basis reduces injustice in the assessment of participants.

Statistical modelling. Normally distributed data sets with 3,000 data were used to repeatedly estimate standard deviations on the basis of a sample of 40 data out these data sets. This is done 100 times. The variation in these estimates explains that the variation between standard deviations in the PT round probably is due to random effects.

IMPLEMENTATION OF PROFICIENCY TESTING SCHEMES FOR A LIMITED NUMBER OF PARTICIPANTS AND INTERPRETATION OF THE PT RESULTS

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A metrological background for implementation of Proficiency Testing (PT) Schemes for a limited number N of laboratories-participants (less then 20-30) is discussed. It is shown that such schemes should be based on use of certified reference materials with traceable property values, as test items.

The discussion considers effect of a limited population of PT participants N_p on statistical parameters of the PT results for a given sample of N responses from this population. When N_p is finite and the sample fraction N/N_p is not negligible, a correction to the statistical parameters may be necessary.

Scores suitable for laboratory performance assessment in such PT Schemes are compared. A possibility to assess collective performance (of PT participants as a group) by comparison of the PT consensus mean or median with the certified value is analyzed also.

It is shown that achieving quality of PT results in the framework of the concept "tested once, accepted everywhere" requires both metrological comparability and compatibility of the results.

DESIGN OF PROFICIENCY TESTS PROVIDED BY COMMUNITY REFERENCE LABORATORIES FOR PAHs AND MYCOTOXINS TO NATIONAL REFERENCE LABORATORIES

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The Community Reference Laboratories (CRL) for Mycotoxins and PAHs organise within the frame of Regulation (EC) 882/2004, organise on a regular basis comparative testing for National Reference Laboratories (NRLs). These tests are planned and conducted on the basis of the IUPAC-AOAC International Harmonised Protocol for the Proficiency Testing (PT) of Analytical Chemical Laboratories.

Participation in the PTs organised by the CRLs is mandatory for NRLs free of charge. The selection of food matrices for the forthcoming PT is agreed with the Directorate General for Health and Consumer Protection (DG SANCO) and NRLs. The PT includes at least one test sample; it might also include additional test solutions, to verify correctness and influence of the calibration, and/or blank material to be spiked for recovery calculation. Besides z-scores, also potential sources for deviations will be evaluated and reported back to the participants.

PTs are designed in such a way to work as a training for the network, with increasing complexity in following years (*e.g.* the sample could be a standard mixture the first year, a spiked matrix the second, a naturally contaminated matrix the third, etc.).

Details of the method in use in each NRL are reported together with the PT results.

NRLs are requested to provide method's performance criteria (LOD, LOQ Recovery) and standard uncertainty plus coverage factor, in compliance with Commission Regulation (EC) No 333/2007 and 401/2006. Precision of the methods used by the individual laboratories participating in the PT is evaluated through replicate measurements of test materials, which might also be carried out in different analytical sessions. The number of significant figures reported shall be checked for legislation compliance as well.

The outcome of the PT is discussed in a workshop with NRLs and DG SANCO. During the workshop, training on possible critical steps of methods is provided together with training on general issues (*e.g.* uncertainty of measurements).

Examples of PT designs applied and of root cause analysis elaborated for unsatisfactorily performing laboratories, like influence of calibration or recovery calculation on the results reported, possible method problems revealed through the comparison of deviations obtained for a standard mixture and for a sample, are discussed in this expert network.

PROFICIENCY TESTING SCHEME FOR MICROBIOLOGICAL ANALYSIS OF WATER IN AFRICA

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Introduction. The water Microbiology Proficiency Testing Scheme is a new development in the schemes that have been operating in the region. This scheme and the ones before it have all been supported by the Physikalisch-Technische Bundesanstalt (PTB) in Germany. However, since PTB cannot run two water schemes concurrently in both East Africa Community and SADC countries, the water Microbiology testing scheme is being prepared by the Uganda National Bureau of Standards for both the EAC and SADC regions. The scheme is taking off in July 2008 and 22 laboratories have expressed interest to participate. The laboratories are from 9 countries namely: Namibia, Zambia, Mauritius, Malawi, Ethiopia, Kenya, Rwanda, Tanzania and Uganda.

Purpose. To ensure that quality of drinking water meets both national and other regional standard specifications, microbiological analysis in laboratories is a necessary preresiquite. With the help of PTB, a PT Scheme for chemical water analysis was instituted for the SADC and EAC regions, directed by the SADC Water Lab Association. This PT system offers the opportunity for all interested laboratories to participate in a regional, affordable PT Scheme to improve and demonstrate their quality to customers, authorities and accreditation bodies. However, there was no arrangement for microbiological analysis for water then.

Parameter for analysis. The water samples will be analyzed for *Escherichia coli* as the main indicator organism for fecal contamination and Total plate counts.

Samples. The PT samples are prepared by spiking with a known concentration of Escherichia coli in a final volume of 100 mL transport medium and for total plate count in a final volume of 10 mL transport medium. The samples will be transported under cool ice packs in a card board box and are expected to reach the furthest destination latest by two weeks.

Evaluation and assessment. The assessment of the values is based on z-scores, using the robust standard deviation of the data set as standard deviation for proficiency assessment, provided it is lower than the fitness-for-purpose value agreed on between participants. Where the calculated value is higher, the fitness-for-purpose value is used. A method specific evaluation is made and help is provided for laboratories with need for corrective actions.

Experiences. The laboratory has run 3 trial runs and experienced some problems especially in preparing a stable bacterial solution. Recently two concentrated bacterial suspensions were prepared that were sufficiently stable during a 10 day period. This kind of stability should be adequate for the performance of a microbiology PT. That is because low temperatures needed during shipping can probably be maintained for up to two days only. Therefore participants should analyze the samples as soon as they receive them.

A large batch sample of *Escherichia coli* was prepared with a final concentration of 12 CFU/100 ml. Homogeneity of the batch was checked by performing 15 times 100 ml filtrations and considered adequate.

The first PT run for the participating laboratories is scheduled for August 2008.

MICROBIOLOGY PROFICIENCY TESTING FOR DRINKING WATER IN GERMANY

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The German drinking water directive requests laboratories analyzing drinking water to be accredited, implement an internal quality control system and take part in Proficiency Testing Schemes (PTS) as external quality control. For certain microbiological parameters the German Federal Environmental Agency (UBA) recommends passing of Proficiency Testing Schemes twice a year. Political demand is that the PT provider delivers samples closely resembling natural drinking water. The Health Department of Lower Saxony, NLGA, Aurich, has been offering microbial drinking water PT Schemes since 1995. Meeting the political demand a system using spiked water/mineral medium as reference material was established rather than using dried or freeze-dried material. Today more than 600 German laboratories and 36 laboratories from other European countries take part in schemes for the parameters *E. coli*/*coliform bacteria*, *Total plate counts*, *Pseudomonas aeruginosa*, *Legionella spec*., *Enterococci*, *Clostridium perfringens* and the parameters of the European bathing water directive with a mean of roughly 400 participants at a time.

Usually evaluation and statistics follow the German standard (DIN 38402-45) for chemical PT Schemes. In order to be able to use this kind of statistics samples have to be of high quality concerning bacterial distribution and stability. Internal quality control in our lab as well as analysis of participants results regularly demonstrate that these conditions are met.

In addition to serving as external control for each participant the data collected from the PT give valuable insight into problems arising from interaction between filter material and agar plates. Especially *Clostridium perfringens* and *Legionella spec*. show high variability in results depending on the material used.

A NEW MATERIAL FOR EXTERNAL QUALITY ASSESSMENT OF BLOOD GASES, ESPECIALLY PO_2

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EQUALIS (External Quality Assurance in Laboratory Medicine in Sweden) has previously for External Quality Assurance (EQA) of blood gas analysis used two different sample materials, one with aqueous matrix and one with a matrix of haemoglobin of bovine origin (EQUIL). The aqueous material is tonometered (equilibrated with certified gases) by the manufacturer and transfered into glass vials which are sealed. The haemoglobin material must be stored in an oxygen free environment to prevent the oxidation of haemoglobin to methaemoglobin (MetHg). Tonometry must then be carried out at the respective laboratory directly before use.

As previously reported by Larsson, Sandhagen and Kallner in 1999, the aqueous material is, however, not suitable for EQA of pO_2, since water does not have any buffering capacity for oxygen. Thus the reproducibility (CV) for aqueous material for pO_2 was between 15 and 25% for hospitals in Sweden for various EQA mailings over time. pO_2 has therefore been excluded in the aqueous EQA program.

The reproducibility for the material with haemoglobin matrix, after local tonometry, has varied between 2 and 4%. This program requires, however, that EQUALIS in addition to the vial with the material must also distribute a small gas cylinder with, for the laboratory, unknown gas fractions. This has been a fairly expensive program because of the extra cost of gas cylinders and that the participants have to invest in special equipment to be able to tonometer the material. As these procedures are expensive, smaller laboratories have chosen to not participate in EQA for pO_2. EQUALIS has therefore from January 2008 tested Tonometrol, a new material with haemoglobin from EUROTROL (Ede, the Netherlands). This material is stabilised chemically and tonometered at the factory and is stable several months at -18°C. The reproducibility for pO_2 during the trial period for Tonometrol is not fully in line with that from the haemoglobin material which is tonometered locally prior to use, although it is much better than that of aqueous materials, especially at the lower levels of oxygen tension where the critical decision limits are.

Our conclusion is that the reproducibility for Tonometrol is acceptable in view of the benefits of simpler and cheaper procedures, that makes it possible for all laboratories to participate in the external quality assessment of pO_2, one of the most important analytes in the intensive care.

PROFICIENCY TESTING FOR MEASUREMENT OF RADON (^{222}Rn) IN DRINKING WATER

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The aim of this paper is to present problems, which were found in carrying out the Proficiency Test for determination of radon (^{222}Rn). The Finnish Environment Institute (SYKE) in collaboration with the Radiation and Nuclear Safety Authority (STUK) carried out the Proficiency Test for measurement of radon in water pumped from two drilled wells in November 2007.

In total, 22 Finnish local food and environmental or private laboratories participated in the Proficiency Test. STUK has supplied the regional laboratories with RADEK MKGB-01 equipment based on gamma spectrometry. Considering radiation protection, the most important radioactive substance in water is radon, which occurs especially in water of drilled wells. When using water, radon is released from water into air, exposing the lungs to radiation. Approximately 20,000 Finns use water from wells drilled in bedrock, which exceed the radon concentration limit of 1,000 Bq l^{-1} recommended for private wells.

Because radon releases easily into air, the samples for determination of radon were pumped from two drilled wells directly to the one-litre glass bottles. Aliquot samples of 10 ml for measurement with liquid scintillation counting were taken, too. In general, radon releases from rock cracks into ground water irregularly. Though water was let to run for a half hour before sampling the concentration of radon increased during sampling with the result, that each participant received an individual sample. Therefore the results of each participant were compared with the results measured by STUK using the liquid scintillation counting, which was used as the reference method in this Proficiency Test. The measurement uncertainty of the reference method was 3.7%.

The results reported by the participants were generally smaller than the results measured by STUK. The mean difference between the participant's result and the result measured by STUK was -7.4% (Sample 1) and -6.2% (Sample 2). The comparison of the results obtained with liquid scintillation counting and with the RADEK equipment was carried out at STUK.

The results obtained with the RADEK equipment were somewhat lower than the results obtained with the liquid scintillation counting. For the RADEK MKGB-01 equipment the major error source was pretreatment of samples.

In estimating laboratory performance 73% (Sample 1) and 82% (Sample 2) from the participants' results were satisfactory deviating less than ±10% from the value measured by STUK using the liquid scintillation counting.

AN EXPERIMENTAL EXTERNAL QUALITY ASSESSMENT PROGRAMME RELATED TO THE MONITORING PLAN AGAINST AEDES ALBOPTICUS (TIGER MOSQUITO) IN EMILIA ROMAGNA (ITALY)

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Introduction. Following the epidemic disease caused by the Chikungunya virus detected in the Provinces of Romagna (Italy) during 2007, a specific monitoring-plan against the bug-vector *Aedes albopticus* was set up by the health authorities of the Emilia-Romagna Region. The monitoring plan was based on standard traps consisting of black containers filled with approximately half-litre of water. A specific substratum of deposition for the eggs (masonite) was fixed inside the containers. ARPA was recognized professionally qualified for the reading of the substrata, in consideration of the experience acquired in this activity during the past season. The analytical method consisted in the simple enumeration of the eggs spawned on the substratum, using an optic microscope. Even if the technique was not particularly sophisticated, in order to guarantee data comparability, a control circuit among the seven laboratories involved in the project, was organised.

Materials and methods. Five ARPA centres and two university labs were involved, for a total of 14 operators. The goal of the circuit was to reproduce, as much as possible, natural environmental conditions for *Aedes albopticus'* eggs: whole, opened and, dehydrated. So, 10 substrata, coming from traps belonging to a controlled breeding of Culicidi, were analyzed. The same microscope, making a single reading for every substratum, was used by all the analysts. Analytical data were statistically elaborated with the "XLSTAT" program.

Results. In order to check if the performances provided by the different laboratories were significantly different, ANOVA analysis was performed. Such orientation is referable to: i) the lack of experimental data to justify the use of a z-score test; ii) the lack of a true-value (mean of "population") to compare the readings with.

Environmental data showed a sufficient alignment among laboratories performances, also graphically visible due to the absence of relevant peaks. Moreover the software makes a grouping of the variables "laboratory" filling them in the same cluster "A", underlining the concept that there wasn't significant reading variance among reading performances.

Conclusions. According to the observations, we can assert that the enumeration of *Aedes albopticus'* eggs in the different labs was not statistically different (95% confidence level); in addition, we can also say that the performance of the laboratories involved in the regional project of *Aedes albopticus'* surveillance provided aligned, homogeneous and comparable analytical data.

PROFICIENCY TESTING FOR ANALYTICAL CHEMISTRY LABORATORIES IN THE EAST AFRICAN COMMUNITY COUNTRIES

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Introduction. To help local laboratories in both the public and private sector improve the correctness of their results and boost customer confidence, Proficiency Testing (PT) Schemes are being organized by the Bureaus of Standards in the EAC countries Tanzania, Uganda and Kenya, under the Standardization, Quality Assurance, Metrology, & Testing (SQMT) Protocol of the EAC, with financial and technical support from the Physikalisch-Technische Bundesanstalt (PTB) in Germany. The PT Schemes are scientifically guided by the Universität Stuttgart of Germany. Scope of the schemes: TBS is running a scheme on the analysis of Edible Salt, which is analyzed for sodium chloride, magnesium, calcium, iodine and moisture content. 29 laboratories, mainly from EAC countries, participated in the 2007 round. KEBS is providing interlaboratory comparisons for the analysis of wheat flour. The participants are invited to analyze the samples for moisture, crude protein, total ash, acidity and fat. In 2007 25 laboratories from EAC countries delivered results. UNBS started with a PT Scheme for the analysis of water, with 33 laboratories participating in 2007. Due to a closer cooperation with the PT programme under the umbrella of the Southern African Development Community Cooperation in Measurement Traceability (SADCMET), it was decided to change to PT Scheme for the analysis of Edible Vegetable Oil, which will be provided for the first time in 2008. This will broaden the scope of PT Schemes in sub-Saharan African. The Edible Oil will be analyzed for peroxide value, acid value, moisture and volatiles, iodine value, refractive index, relative density, copper, iron, and nickel. Design of the schemes: Natural samples are used as PT samples. Homogeneity and stability of the samples are tested according to the relevant standards. The consensus means from the participants results are calculated using robust statistical methods and used as assigned values, since no reference value is available. The standard deviation from the dataset is taken as standard deviation for proficiency assessment to calculate the z-scores, provided that the standard deviation is below a value which was agreed between the participants as being fit for purpose. In cases where the calculated value is higher, this limit is used to calculate the z-scores. Outlook and future plans: The first rounds showed that there is a need for those PT Schemes, and there has been a gradual increase in number of participants. There are plans to increase on the frequency, expand the scope, and extend participation to other regions. The quality of the participants is varying and the laboratories benefit from the comparison with other labs. Evaluation workshops that follow each PT round are being used for networking between laboratories.

BENEFITS OF PARTICIPATING IN THE 2007 NPL ENVIRONMENTAL RADIOACTIVITY PROFICIENCY TEST EXERCISE

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Analytical Sciences performed well in the 2007 National Physical Laboratory (NPL) Environmental Radioactivity Proficiency Test Exercise. Excellent agreement was achieved for all alpha-emitting nuclides measured by radiochemistry and alpha spectrometry. Tritium was successfully separated from interfering beta-emitters by distillation and measured by liquid scintillation counting. All gamma-emitting nuclides were identified in both samples. The samples were also used to conduct an internal inter-comparison between instruments and with separate groups performing similar measurements within AWE. Participation in the exercise was invaluable in supporting continued UKAS accreditation for these processes.

The exercise also served to highlight areas for improvement and limitations to the current methods, including coincidence summing correction and density correction to improve the accuracy of measurements made using gamma spectrometry. A neutron-activated concrete sample was also supplied; lower than expected results were obtained for total and leachable tritium. This was due to the lower efficiency of our methods for extracting tritium produced by neutron activation within the concrete matrix than for surface contamination with tritiated water HTO(L) or tritium gas HT(g).

PROFICIENCY TESTING: WASTEWATER SAMPLING UNCERTAINTY

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This contribution deals with the results of the study Sampling uncertainty undertaken by CSlab company which was supported under contract with Czech Office for Standards, Metrology and Testing as part of the Metrology Programme 2007 Task No VIII/7/07. The study was aimed at evaluation of sampling uncertainty and total uncertainty of measurement for selected analytical wastewater characteristics such as Chemical Oxygen Demand (dichromate value), total inorganic nitrogen, total phosphorus, total suspended solids and dissolved inorganic salts. The monitoring of these parameters is of great interest due to the fact that monitoring is required by legislative regulations (Directive No 293/2002 of Ministry of the Environment of the Czech Republic) that define a charge for wastewater disposal into surface water related to violation limits for the selected chemical characteristics in effluent samples.

The value of measurement uncertainty was estimated both on the basis of the results of Proficiency Testing study of wastewater sampling performed at wastewater treatment plant with 50 participating sampling teams and on the basis of experiment performed with 8 selected participants. The participants collected two samples, divided them into two subsamples and performed duplicate analysis of each sample in the laboratory using recommended methods of analysis. In sum 32 values for each characteristics were obtained in this way and the data were subjected to ANOVA statistical analysis. To verify metrological traceability of results, participating laboratories were supplied with a control sample with certified values of the analytical parameters monitored in this study.

The reproducibility coefficients of variation for the parameters under study were greater than those estimated in this study for measurement uncertainty for analysis and sampling and than the expanded uncertainties supplied by individual laboratories. This is due to the fact that the PT study included a greater number of laboratories, producing robust set of data and that the selected parameters were investigated for a longer time interval. Sampling uncertainty was very low, lower than expected (inlet for activation was homogeneous).

Metrological traceability of analytical results of participating laboratories was verified with use of a reference material. The data obtained indicates that the majority of participating laboratories produce reliable and accurate results.

Experimental determination of sampling uncertainty contribution is very cost intensive, especially for single laboratory experiment. The most convenient and robust way to estimate sampling uncertainty is sampling Proficiency Testing. This conclusion is in agreement with recommendations of Nordtest Report TR 604.

INTERLABORATORY COMPARISON SCHEME FOR FUEL SECTOR, LABKAR IN TURKEY

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Petroleum and natural gas sector is one of the powerful and economically important industries. The new petroleum law which has the main aim of regulating the fuel market had an important effect on the quality of measurements in assessing the conformance to ISO/EN/TS standards. Several fuel testing laboratories have emerged which required accreditation thus a scheme to evaluate their proficiency in measurements had become an important requirement.

The interlaboratory comparison scheme LABKAR had evolved from this need. It is run twice every year to cover the seasonal requirements on fuel properties.

The samples are gasoline, diesel oil, LPG, lubricating oil and biodiesel. Presently there are 49 members of LABKAR and 108 samples as a whole are distributed in each round. The samples are provided from the producers.

The data are collected through a web based environment. Statistical analysis is done according to ISO 13528 "Statistical methods for use in Proficiency Testing by interlaboratory comparisons". Fist of all the homogeneity of the samples is tested by randomly selecting 10 samples from the ones that will be used in the scheme. All analysis in the scheme requires the determination of the standard deviation for the scheme. For this, the reproducibility standard deviations obtained from the relevant ISO/EN/TS standards are used since single replication of measurements is made by each participant meaning that repeatability is not concerned. Next, the consensus value from the participants is calculated for each analysis as the robust mean of the data which is described in ISO 13528 and used as the assigned value. Distribution of the data is observed to check if the robust mean calculation is valid or not. But the results are not reported to the labs. Actually up to now, no situation is met where it is not valid. Then z-scores are calculated if the standard uncertainty of the assigned value is negligible. Else it is mentioned in the report that the uncertainty of the assigned value is not negligible therefore z-scores are not reported for labs not to receive wrong action and warning signals. In the calculation of z-scores, the standard deviation of the scheme is used rather than the standard deviation estimate from the participants. Besides graphs for the z-scores of the lab obtained in each round for the same analysis is given in the report so that they can visualize their performance over the rounds.

The quality of the scheme will be evaluated with selected indicators.

RESULTS AND UTILITY OF PROFICIENCY TEST IN THE DAIRY FIELD

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Since 1994, Laboratorio Standard Latte (LSL), an organization of the Italian Breeders' Association (AIA), organises Proficiency Tests (PT) on cow, sheep and buffalo milk, according to ILAC G13. Results are elaborated according to ISO 5725-2. For each PT, samples are prepared, subject to homogeneity testing, sent to laboratories who analyse them and send the results to LSL.

This study aims to underline two different aspects from these PTs: i) to focus the attention on the organisation of a PT by comparing the performances of 25 associated laboratories (AA-Lab, which are weekly controlled) with 50 not associated laboratories (NO-AA-Lab: Universities, private labs, dairy industry); ii) to compare a reference method with a screening method for aflatoxin M1.

Every two months AIA-LSL organises a PT on 10 cow milk samples at different concentration of fat, protein and lactose which are analyzed with Infrared instruments. The mean reproducibility values, for the routine PT, are 0.084% and 0.121% for fat, 0.068% and 0.083% for protein, 0.063% and 0.106% for lactose, for AA-Labs and No-AA-Labs respectively. It is clear that in a controlled system, such as that of the AA-Labs, agreement among laboratories is better than among non controlled labs.

Since 2004, LSL organises a PT on Aflatoxin M1 in cow milk twice per year. LSL sends milk samples, contaminated with ca. 25 ppt and 50 ppt of aflatoxin M1, to 75 labs. Analyses are performed by ELISA (screening test) and/or by HPLC(reference method).

From 2004 to 2008, repeatability values, for each method, decreased from 9 ppt (nanograms/kg) to 4 ppt at 25 ppt and from 14 ppt to 7 ppt at 50 ppt. The reproducibility values, instead, remained constant for each level and method (ca. 6 ppt at 25 ppt and ca. 11 ppt at 50 ppt).

At regulation limit (50 ppt), since 2004, the screening test has improved, obtaining results closer to the reference method. Although ELISA seems to overestimate in comparison with the reference method, the results are in the range of reproducibility standard deviation (SR=12 ppt).

QUALITY ASSURANCE OF THE RESULTS IN THE DIAGNOSTIC LABORATORY: THE ROLE OF PROFICIENCY TESTING

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The EN ISO/IEC Standard 17025:2005 "General requirements for the competence of testing and calibration laboratories" requires the application of assurance quality criteria in the microbiological methods and underlines the importance of the inter-laboratory circuit as basic instrument to verify the assurance of the quality results. However, some microbiological labs have difficulty in applying these rules. In fact concepts such "quality assurance of results", "quality continuous improvement", "surveying and evaluation of lab trend" have been already acquired in Food microbiological laboratories, while in Diagnostic microbiological ones these concepts are not completely applied because of the complexity of diagnostic activity it is difficult to identify parameters useful for quality assurance. Moreover there are very expensive and few available circuits. However is important for the labs to take part in Proficiency Tests to evaluate their performances. At the same time the inter-laboratory comparison methods allow them to ensure results reliability, as required by ISO guidance, and to stimulate technicians into discussion, exchange of technical details, with the aim to point out the main differences in diagnostic microbiological diagnosis and non conformity management.

This paper describes methods used to set up a qualitative test about isolation and identification of *Taylorella equigenitalis,* intended for diagnostic laboratories and organized by Clinical Diagnostic Laboratory of *Istituto Zooprofilattico Sperimentale delle Venezie*. The agreement between expected results and the results of Proficiency Testing is evaluated by the Cohen's Kappa statistic. During the preparation of inter-laboratory circuit, the main problem was to produce test-samples the most similar to real ones. So it was necessary to consider different aspects of bacterial behaviour (biochemical characteristics and interactions between them), preservation of the test-samples (freeze-drying), and media recipe.

EXTERNAL QUALITY ASSESSMENT FOR THE DETECTION OF HCV RNA, HIV RNA AND HBV DNA IN PLASMA BY NUCLEIC ACID AMPLIFICATION TECHNOLOGY: A NOVEL APPROACH

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Background and objectives. To increase the viral safety of blood and blood products, Nucleic acid Amplification Technology (NAT) for detection of HCV, HIV and HBV has been introduced as part of the screening of single donations by most blood centres and blood product manufacturers either on a voluntary basis or as required by national laws.

Participation in EQA programmes is important for a NAT laboratory in order to ensure that its system works efficiently, not only with respect to the analytical phase but to the whole process, namely from receipt/storage of the samples to data transcription on the reporting sheet. Obviously, a failure at any of these stages would affect the performance of the laboratory.

The present study was aimed at assessing the performance of the participating laboratories in determining the possible contamination of plasma with HCV, HIV and HBV using a novel approach. In fact, panels were prepared taking into account viral concentrations of about three times the 95%DL of the methods most commonly used by laboratories. Moreover, in order to verify the consistency of the results obtained in separate runs on different days, possibly by different operators, three samples with the same viral concentration were included in each panel.

Materials and methods. A panel of 12 samples, three negative and three positive for each virus, was distributed to the EQA participants. The positive samples were prepared, using the respective WHO standards, in order to obtain a viral concentration of about three times 95%DL of the methods.

Results and discussion. A total of 65 panels were distributed to 59 laboratories that agreed to participate in the study. Six laboratories tested an additional panel by using a second NAT assay.

Overall, the participants' performance was satisfactory. In particular, 49 of the 59 participants (83%) were able to correctly identify all samples. Regarding the remaining 10 laboratories, in three cases a deviation from the laboratory's procedure that could be attributed to an operator's mistake, was observed, in two cases a possible cross-contamination occurred while in the remaining five cases the failure to detect the positive samples couldn't be ascribed to any relevant deviation in the laboratory's procedure. In conclusion, this study points out that despite the high level of automation reached by NAT assays, human errors can still occur. Therefore, participation in these studies is still a valid tool for laboratories involved in NAT blood screening to assess their proficiency.

PROFICIENCY TEST ON INCURRED AND SPIKED PESTICIDE RESIDUES IN CEREALS

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In 2006 the European Commission appointed seventeen new Community Reference Laboratories, CRLs, covering areas of food and feed safety and animal health. The National Food Institute was designated to be CRL for Pesticide Residues in Cereals and Feedingstuff, CRL-CF. One of the main tasks for the CRLs is to organise Proficiency Tests for all the National Reference Laboratories from the EU Member States. However, Official Laboratories, OFLs, which are involved in the control pesticides in food, were also invited to participate in the PTs organised by the four CRLs covering pesticide residues.

Two PTs on incurred and spiked pesticide in cereals have been organised by the CRL-CF, with 63 and 72 participants, respectively. The presentation will focus on: i) the production of homogenised test material with incurred and spiked pesticide; ii) the website for results submission; iii) the calculation of results; iv) experience gained from the results submitted by the laboratories. The test material for both PTs was wheat. The Faculty of Agricultural Sciences, University of Aarhus, performed the field spraying. The test materials were further spiked in the laboratory with additional pesticides. After mixing, milling and homogenisation, the test materials were packed in containers and homogeneity and stability test were performed. The test material contained 10-15 different pesticides and the laboratories were asked to identify and quantify the pesticide residue in the test material using there own standard procedures.

Before the deadline for result submission, the laboratories had to report their results via the website. The labs were also ask to inform on which of the 40-50 possible pesticides they have analysed for, and give information on the methods they have used.

When the results were received from the laboratory, the calculations were done in accordance with "The International harmonized protocol for the Proficiency Testing of analytical Chemistry laboratories". Firstly, all false negatives and false positives were identified. Secondly, z-scores where calculated for all pesticides. To evaluate each laboratory's overall performance, and including all the pesticides analysed, a Weighted Sum of z-scores (WSZ) was calculated. This function was only applied to laboratories with sufficient scope.

The two PTs showed big differences in the results, which depended on whether the laboratories added water to the samples prior to extraction or not. The differences were up to 300%. This information is now included in the EU guideline "Method validation and quality control procedures for pesticide residues analysis".

REPRODUCIBILITY IN PROFICIENCY TESTS IN PROGRAMA INTER*2000*-Q

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Programa INTER*2000* (www.inter2000.cat) is a Proficiency Testing (PT) Scheme for the agro-food sector laboratories operated by the Laboratori Agroalimentari of the Department of Agriculture, Food and Rural Action, of Generalitat (Catalonia Government).

INTER*2000*-Q is the PT Scheme for chemical analysis. In 2007, 197 parameters were tested in samples of meat products, dairy products, fish products, oil, beverages, cereals, soil, water, etc. Last year, a total of 1,105 samples were distributed to participants and 8,728 results were statistically analyzed.

The INTER*2000* PT Scheme is managed in accordance with the requirements of IUPAC Technical Report "The international harmonized protocol for the Proficiency Testing of analytical chemistry laboratories", ISO13528:2005 standard "Statistical methods for use in Proficiency Testing by interlaboratory comparisons", and the ILAC-G13:2000 "Guidelines for the Requirements for the Competence of Providers of Proficiency Testing Schemes".

Concerning statistics in INTER*2000*-Q, the assigned value (μ) (mu) is calculated after removing extreme outliers as the robust average of all results reported by participating laboratories according to ISO13528:2005 "Statistical methods for use in Proficiency Testing by interlaboratory comparisons" Section 5.6. The test variability σ (sigma) is set as the robust standard deviation of reported results according to ISO13528:2005 Section 6.6. Following INTER*2000*-Q protocol, this assigned value (μ) (mu) and this variability σ (sigma) are used to calculate z-score.

In this communication we summarize the reproducibility achieved in different test of INTER*2000*-Q and compare this reproducibility with the precision models described by Horwitz in 1982 and further developed by Thompson in 2000. Analytical results in liquid samples usually show a better reproducibility than in solid samples, and the %RSD values are not always in accordance with Horwitz or Thompson precision models.

According INTER*2000*-Q protocol, the data set in which is applied the ISO13528:2005 statistics is also checked for normality using Lillieforts (Kolmogorov-Smirnoff) test. In this communication we also discuss the correlation between the percentage of participant laboratories with satisfactory z-score and the normality test results.

CHALLENGES IN MICROBIOLOGICAL PROFICIENCY TEST IN PROGRAMA INTER*2000*-M. *CLOSTRIDIUM PERFRINGENS*, *ESCHERICHIA COLI*, *LISTERIA MONOCYTOGENES*, *SALMONELLA SPP*., *ENTEROBACTER SAKAZAKII* AND *CAMPYLOBACTER SPP*. PERFORMANCES

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Programa INTER*2000* (www.inter2000.cat) is a Proficiency Testing Scheme for the agro-food sector laboratories operated by the Laboratori Agroalimentari of the Department of Agriculture, Food and Rural Action, of Generalitat (Catalonia Government).

INTER*2000* scheme is managed in accordance with the requirements of IUPAC Technical Report "The international harmonized protocol for the Proficiency Testing of analytical chemistry laboratories", ISO13528:2005 "Statistical methods for use in Proficiency Testing by interlaboratory comparisons", and ILAC-G13:2000 "Guidelines for the Requirements for the Competence of Providers of Proficiency Testing Schemes".

Programa INTER*2000* consists of two schemes: INTER*2000*-Q for physical-chemical tests and INTER*2000*-M for microbiological tests. In INTER*2000*-M scheme, 7 Proficiency Tests were carried out in 2007. In these tests, 32 microorganisms were analyzed in different types of samples such as meat products, dairy products, prepared meals and water. 1,187 samples were distributed to participating laboratories and 4,537 microbiological results where statistically treated.

All test materials of INTER*2000*-M scheme consists of a real food sample or matrix, and a vial containing freeze-dried microorganisms. Matrixes are irradiated with gamma rays (Cobalt-60) in order to guarantee their complete sterility, and are free of preservatives that could inhibit microbiological growing. Strains from CECT (Spanish Type Culture Collection) are used to obtain the freeze-dried microorganisms. The use of sterilized products and type strains, together with a management of the tests in accordance with the international standards, allow us to achieve reliable data on the behaviour of microorganisms in reconstitution procedures and on laboratories improvement of performance in our Proficiency Tests. In this communication, we outline the results for the analysis of *Clostridium perfringens*, *Escherichia coli*, *Listeria monocytogenes*, *Salmonella spp*., *Enterobacter sakazakii* and *Campylobacter spp*. in different food samples of INTER*2000*-M scheme. We also discuss the difficulties for the analysis of *Clostridium perfringens* and the successful analysis of important microorganisms for food safety such as *Enterobacter sakazakii* in milk powder or *Campilobacter spp*. in minced meat.

PROFICIENCY TESTING IN A DEVELOPING COUNTRY: THE BOTSWANA SITUATION

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This paper represents the current state of affairs in Botswana regarding participation and performance in Proficiency Testing (PT) Schemes by Botswana laboratories and is subject to change as developments occur, and therefore should be viewed as a "living document".

Botswana is an approximately 600,000 sq. km semi-desert, landlocked, developing country in Southern Africa, with a population estimated at 1.8 million.

The country has an import based economy and a small industrial base. Consequently, scientific research and testing is limited, resulting in the country having approximately 115 laboratories comprising of Medical, Mining, Academic, Research and Industrial testing laboratories.

A majority (95%) of these labs are non-commercial and only 2% are accredited. Of all these laboratories, 60% participate in some form of PT Scheme offered by international, regional and/or local Proficiency Testing providers. A small percentage of these laboratories perform well in these schemes, while the rest perform unsatisfactorily.

Lack of legislation and the socio-economic status of the country have resulted in a significant lack of appreciation and implementation of Quality Assurance Programmes.

Low literacy and consumer education levels have resulted in a population that does not demand for quality in products and services rendered. Consequently, testing laboratories are not committed to ensuring the quality of their services, more so that the majority of these labs are non-profit oriented, this has resulted in lax laboratory practices.

Due to over-reliance and domination of the local manufacturing industry by South Africa, local non-accredited laboratories are often overlooked in preference to South African and other international laboratories. This lack of credibility has resulted in a lack of drive for excellence and growth of the local laboratories and consequently, failure to appreciate the need to participate in Proficiency Testing Schemes.

The scope of PT Schemes in Botswana is very limited; the country has only two PT providers, which are:

- the Botswana Bureau of Standards and on ad-hoc basis;
- the Quality Assurance office under the Ministry of Health.

Both the schemes provided by these organizations are non-accredited. The Ministry of Health offers PT in areas of Medical Microbiology, Chemistry, Hematology, TB-ZN staining and CD4 cell-count using routine patient samples as reference material.

Similarly, the Botswana Bureau of Standards offers a scheme in Civil Engineering studies, Geochemistry, Food and Water Chemistry and Microbiology using purchased Certified Reference Materials.

THE ITALIAN EXTERNAL QUALITY ASSESSMENT SCHEME IN MOLECULAR GENETIC TESTING COORDINATED BY THE NATIONAL CENTRE FOR RARE DISEASES

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Genetic testing services in the European Union have increased their activity in the past few years. Several External Quality Assessment (EQA) Schemes have been funded by international groups, national governments and private subscription.

In 1999 the Istituto Superiore di Sanità (ISS) underlined the importance of EQA within the official document "Guidelines for genetic testing". In 2004 the "Guidelines for medical genetics services", issued by the Italian Government, stressed the need for Medical Genetics Services to participate to internal and external quality assessment programs at regional, national and international level.

The Italian EQA Scheme on genetic testing started in 2001. The scheme, financially supported by the National Health System and coordinated by the National Centre for Rare Diseases (ISS), covers both molecular genetic (cystic fibrosis-CF, β-thalassemia-BT, fragile-X syndrome-XF, adenomatous polyposis coli-APC) and cytogenetics (prenatal, postnatal and oncological diagnosis). Public laboratories are enrolled on a voluntary basis and participation is free of charge. Six DNA validated samples are sent, once per year, to laboratories for each pathology; clinical and technical information are included. Laboratories send back to ISS: raw data, interpretation of results and a written report. A panel of National experts evaluate all data.

Five trials have been performed and concluded and the sixth one is actually ongoing. Eighty two laboratories have been enrolled up to now. Results from the first five years showed an improvement both in the use and in the interpretation of genetic tests. The average genotyping error rate observed over the five years was 0.6%, 0.3%, 5% and 4.7% in the CF, BT, XF and APC scheme respectively. The percentage of complete reports increased over the five years. However, lack of information or inadequacy in reporting were frequently observed. In order to harmonize our scheme with the existing European ones, a web-based system was developed and introduced in 2008 trial, thus simplifying accessibility and participation of laboratories.

In conclusion the overall genotyping, technical and report results obtained during these five year survey show an increasing improvement of quality in the majority of the participating diagnostic genetic laboratories in Italy.

IDENTIFICATION AND TYPING OF VEROCYTOTOXIN-PRODUCING *E. COLI* (VTEC): INTER-LABORATORY STUDY AMONG EUROPEAN NATIONAL COMMUNITY LABORATORIES

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Verocytotoxin-producing *E. coli* (VTEC) are so-called because of their ability to produce Verotoxin (VT) responsible of illness in humans. The main virulence factors are represented by the genes *vtx1*, *vtx2* and *eae,* coding VT1, VT2 and intimin, respectively. Duties of the EU-Community Reference Laboratory for VTEC in food and feed (CRL-VTEC) (Reg. 882/2004 EC,) include the organisation of Proficiency Tests among the National Reference Laboratories (NRLs) for *E. coli*, for the assessment of the capability of identification and typing of VTEC strains.

The first NRL VTEC inter-laboratory study has been conducted in 2007 and involved 21 NRLs from 17 EU Member States. Five *E. coli* strains were distributed in blind to the participating NRLs. They were requested to identify serogroup (O antigen), capability to produce VT, presence of *vtx1*, *vtx2* and *eae* genes, using the methods currently applied by the laboratory. The strains belonged to serogroups O157, O111, O26, O145, O103. Three of them produced VT and carried the *vtx1* gene while all carried the *eae* gene. Presence of *vtx2* gene characterised three strains but in one of them it was undetectable by PCR, if certain primers were used.

NRLs performance was evaluated in term of agreement (*Cohen's kappa*), sensitivity and specificity. As a whole, 403 single tests were performed. Five NRLs performed all the requested tests. All the NRLs had excellent agreement with the gold standard values ($K>0.75$), and 15 NRLs had a perfect agreement. Overall sensitivity was 0.97 (95% CI: 0.93-0.99) and specificity was 0.99 (95% CI: 0.97-1.00).

Methods for identification of the O serogroup included slide agglutination or PCR amplification of serogroup-associated genes and were applied by 17 NRLs. Serogroups O157, O26 and O111 were correctly identified while 2 NRLs failed to identify O103, and one O145. VT production was analysed by 7 NRLs, by Vero-cell assay, immuno-enzymatic or latex commercial kits. All the VT-positive strains were correctly identified while one NRL provided a wrong result for one VT-negative strains. PCR detection of virulence genes was performed by 20 NRLs. Detection of *vtx* genes was correctly performed by 19 NRLs; *eae* gene was correctly identified by 16 of the 18 NRLs who did the test.

In conclusion, the first interlaboratory study was successful, as the NRLs performance was in general satisfactory with a good accuracy of results. However increasing the number of participating NRLs and improving the capabilities to perform serotyping even for O157, is necessary.

RESULTS OF THE FIRST NATIONAL INTERLABORATORY STUDY FOR THE ASSESSMENT OF THE OVINE PRP GENOTYPE

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Scrapie is a contagious disease responsible for serious damages to sheep populations. Since no *in vivo* diagnostic tests nor vaccines are available, traditional strategies for the control of infectious diseases are ineffective against scrapie. Host susceptibility to scrapie is influenced by the genotype at the prion protein (PrP) gene. Polymorphisms at codon 136, 154 and 171 have been identified as the main determinants. Codon 136 encodes alanine (A) or valine (V), codon 154 arginine (R) or histidine (H), and codon 171, glutamine (Q), arginine or histidine. These polymorphisms are combined in five main alleles: $A_{136}R_{154}Q_{171}$ (ARQ), VRQ, AHQ, ARH and ARR.

The assessment of PrP genotype of the host is crucial for eradication of outbreaks and for implementing preventive strategies based on breeding programs for the selection of genetically-resistant populations, which are currently applied in EU. Neverthless no standard methods are available for this purpose. Thus laboratories involved in scrapie surveillance have developed different analytical methods.

The first national interlaboratory study was organised in Italy in 2005 with the aim to assess the capabilities of 12 peripheral laboratories to identify the PrP genotype at codons 136, 154 and 171. Laboratories were requested to determine the PrP genotype on a set of 20 samples of ovine DNA. Samples were prepared by extracting DNA from blood of 15 sheep whose PrP genotype was previously assessed. The samples were randomly allocated in every set which included the following genotypes: ARQ/ARQ, ARQ/AHQ, ARQ/ARH, ARQ/VRQ, ARQ/ARR, AHQ/AHQ, ARR/AHQ, ARR/ARH, ARR/ARR, ARR/VRQ.

Participants were requested to perfom genotyping using their analytical methods currently applied. Performance of each laboratory was evaluated by calculating the level of agreement (*Cohen's kappa*) of the results with the true genotypes. Furthermore sensitivity and specificity for the different methods were calculated by grouping results obtained with the same method. These included Real Time-PCR, Sequencing, Hybridization, Pirosequencing and Primer Extension.

Nine laboratories reported a perfect agreement (K=1) while two and one laboratories failed to correctly assess the genotype of one (K=0.94) and three samples (K=0.83), respectively. In particular errors were reported for codons 154 and 171.

A 100% sensitivity and specificity was reported for RT-PCR, Pirosequencing and Primer Extension resulted (100%) while Hybridization and Sequencing had lower accuracy.

In conclusion, even if no standard method was available and several analytical methods were used by the participants, the performance of laboratories resulted satisfactory and all the samples carrying the PrP genotypes most frequently reported in ovine population, were correctly identified.

APPLICATION OF INDUCTIVELY COUPLED PLASMA-SECTOR FIELD MASS SPECTROMETRY FOR VERIFICATION OF GRAVIMETRICALLY PREPARED INTERLABORATORY COMPARISON WATER SAMPLES

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The Center for Analytical Chemistry at the IFA-Tulln organises a Proficiency Testing (PT) Scheme for external quality assurance in water analysis. The periodical interlaboratory comparisons are part of the national monitoring programme on ground and surface water quality in Austria that is supervised by the Austrian Federal Ministry of Agriculture, Forestry, Environment and Water Management. Assessment of laboratories performance is based on reference concentrations, which are calculated from the weights of the substances and standards used to produce the samples. These values have to be confirmed by analytical methods providing smallest possible measurement uncertainties in order to check integrity, homogeneity and stability of the interlaboratory comparison samples.

In 2006, a high resolution inductively coupled plasma - sector field mass spectrometry (ICP-SFMS) method was developed and validated for this purpose. Since June 2006 this method has been applied for the analysis of PT water samples prepared within the course of the interlaboratory comparisons. The performance of the new method in the run of the PT-Scheme is discussed. The characteristics of the ICP-SFMS method are presented in comparison to the atomic absorption spectrometry (AAS) methods employed so far.

In general, ICP-SFMS is faster and more sensitive than AAS. For most trace elements the limits of quantification reached by ICP-SFMS are well below those obtained by AAS. Recoveries, repeatability and intermediate precision were at comparable levels. Moreover, total dissolved phosphorus and boron, which have been previously determined by spectrophotometric methods, can be determined by ICP-SFMS. For the major ions Ca, Mg, Na and K the ICP-SFMS method did not reach the precision of Flame-AAS. In this case the existing AAS method appeared to be most suitable. The time required for analysis could be reduced from one week to one day per interlaboratory comparison round.

PETROLEUM PRODUCT QUALITY CONTROL LABORATORY. ANALYSIS AND INTERPRETATION OF RESULTS OF INTERLABORATORY COMPARISONS

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Petroleum Product Quality Control Laboratory is in possession of accreditation certificate and participates in the round-robin interlaboratory testing programme organised by the American Society for Testing and Materials - ASTM.

Within the scope of this programme, since the year 1993 the laboratory has been performed testing of automotive petrol, jet fuel, diesel fuel, since 2006 also biodiesel fuel, and since 2008 the re-formulated petrol, according to the standard methods. Majority of these methods pertains to the laboratory accreditation area.

In this work we have presented the laboratory successfulness in the year 2007 for all the methods our laboratory compares with the world laboratories.

Considering that the laboratory has a large number of available results from previous years of participation in such a programme, we have taken the example of density testing performed to automotive petrol, jet fuel, and diesel fuel for representing chronologically z-values in the period from 1994 to 2007.

Many years of experience in participating in interlaboratory comparisons represent a significant reference for the laboratory, but also the evidence of a proper functioning of the laboratory quality control system.

COMPARISON OF DIFFERENT APPROACHES TO THE STATISTICAL EVALUATION OF PROFICIENCY TESTS

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The objective of this study was to compare different ways of z-score calculation for the results from Proficiency Testing programmes. The comparison was carried out on the data from Proficiency Tests for four different matrices (soil, plant, feedstuff and sludge) and for all parameters included in the Proficiency Testing programmes organised by our institute from the year 2005 to 2007. It represented 99 samples (three periods per year and eleven samples distributed in each period); the number of determined parameters was 115 in each period. The total number of investigated data sets was 953.

Three approaches to z-score calculation were compared by using:

- the arithmetical average without outliers and the standard deviation calculated after elimination of the outliers;
- robust values of the average and the standard deviation calculated according to the Algorithm A from the standard ISO 13528;
- the robust average calculated according to the Algorithm A from the standard ISO 13528 and the standard deviation calculated by the Horwitz equation.

The procedure according to Algorithm A was found to be the most suitable for the studied data sets. The evaluation described in point 1 showed to be very strict due to usually very small values of the standard deviations. The method using the standard deviation calculated by the Horwitz equation detected very few laboratories as outliers in case of low analyte concentrations. On the other hand this approach was very strict for high concentrations of analytes.

Statistical evaluation of our Proficiency Testing results is based on the median and the median of absolute deviations, calculation of z-scores together with a numerical and graphical presentation and Horn pivot test for limited number of participants.

On the basis of the achieved results it was decided to change the statistical method for the z-score calculation adopting the robust average and robust standard deviation calculated according to the Algorithm A from the standard ISO 13528.

TEN YEARS INTERLABORATORY STUDIES IN THE FIELD OF ANALYSIS OF NOT PERMITTED AND AUTHORIZED VETERINARY DRUGS

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One of the tasks of the Community Reference Laboratories (CRL), in accordance with article 32 (1b) and 33 (1c) of Council Regulation 882/2004/EC, is to organise interlaboratory studies for the benefit of the National Reference Laboratories (NRL) and the Routine Field Laboratories (RFL).

The aim of the interlaboratory studies of the CRL Berlin is to promote the residue analysis of ß-agonists, coccidiostats, nitroimidazoles, anthelmintics and NSAIDs in different matrices.

Since more than 10 years, in the CRL, inhouse RM has been produced and different kind of incurred material from animal studies were used for interlaboratory comparisons.

One of the main applications of programmes for Proficiency Tests is the assessment of the ability of laboratories to perform the required tests for a whole substance group (especially with multi methods) competently in order to reach some day a EU-wide comparability of results.

The poster provides a detailed picture of how to evaluate (robust statistics for median and standard deviation, z-sores, combination scores and point score system) interlaboratory tests for the determination of authorized (MRL substances) and not permitted veterinary drugs.

In view of false negative and false positive results, it was regarded as not sufficient to include only the z-scores and combination scores for the assessment of the laboratories' proficiency. Therefore a point score system was established that accounts for the overall evaluation of the examination results as well as for the false negative and false positive examination results.

The point score system is different between authorized and not permitted substances and offers the possibility to compare qualitative and semiquantitative screening methods without considering the quantification of the measurement results.

Examples of different kinds of evaluation will be presented.

Some times, the correct quantification (especially of MRL substances) needs to be improved. These results underline the relevance of validating according to the criteria of Commission Decision 2002/657/EC.

UNCERTAINTY ASSESSMENT BY PT SCHEMES

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The uncertainty of result has a fundamental role in compliance assessment and/or in comparison with limits. Laboratories should know if the estimation of uncertainty correspond to the target uncertainty.

Real results from interlaboratory comparison organised by Metrological and Testing Laboratory of ICT Prague, designated laboratory according to CIMP MRA, were assessed with zeta-score and with the aid of "naji plot". Two real samples were prepared, simulated calibration solutions of cations and/or anions in surface water matrix. Target uncertainty was estimated with the use of GUM procedure. There were two ways for assessment of the laboratory results: z-score and zeta score. "Naji plot" was used for combined assessment of the results.

Invitation for further comparison is presented. Four samples will be sent to possible participants. One sample is the calibration solution consist of three metal ions: Pb, Cu and Cd, second consist of As, third consist of Se. Fourth sample is olive oil with important pesticides added into matrix.

RECENT DEVELOPMENTS IN EXTERNAL QUALITY ASSESSMENT FOR ENVIRONMENTAL AND OCCUPATIONAL LABORATORY MEDICINE

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Organisers of External Quality Assessment Schemes (EQAS) for Occupational and Environmental Laboratory Medicine (OELM) in Europe, North America and Korea are collaborating (www.occupational-environmental-laboratory.com) to improve the effectiveness of their activities, with regards to:

- stimulating improvements in analytical results;
- establishing conformity of assessment among Schemes;
- enhancing the practice of EQA, including whenever possible, to warrant traceability of EQAS to primary standards.

In addition to previously reported collaborative work, other on-going projects have addressed:

- implementation of the EU Directive on Chemical Agents at Work into national legislation, with reference to differences in binding biological limits for lead in blood and their consequences on setting harmonised target of performance for EQA;
- the definition of achievable harmonised quality specifications for the total allowable error of assays of the concentration of Cu, Se and Zn in serum;
- differences among values assigned to the same EQA samples by different methods according to ISO 13528 and their effect on the evaluation of performance (details presented in a separate paper).

New developments include:

- investigation of the causes of poor reproducibility and lack of recovery of spiked amounts observed in EQAS for mercury in urine (details presented in a separate paper). Further progress should address the reliability of the assigned values and the analytical methods applied by the participants;

- use of EQAS data to calculate the measurement uncertainty of scheme participants. Preliminary work has been undertaken to determine how schemes should be organised and managed to provide the appropriate information;
- use of robust statistics compared with the calculations more usually applied to scheme data.

Membership of the Network is open to interested EQAS organisers. Meetings are held annually to review progress of current projects, discuss arising problems and consider ideas for any new work. The next meeting is 8th-10th October 2008 in Rome at the Istituto Superiore di Sanità.

AN ASSESSMENT OF VALUES ASSIGNED BY DIFFERENT METHODS IN EQAS FOR TRACE ELEMENTS IN HUMAN SERUM AND THEIR IMPACT ON THE EVALUATION OF LABORATORY PERFORMANCE

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External Quality Assessment Schemes provide laboratories with the means to assess the quality of their measurement results in comparison with others using a range of control materials covering the expected range of matrices and concentrations. Several methods are used to determine the assigned values in EQAS, including formulation, determination by reference method, consensus of expert laboratories and overall consensus of the results reported by participants. ISO 13528 gives guidance on how to estimate the uncertainty of assigned values according to the method used to obtain them, but, unless a traceability chain to a stated reference can be documented, the reliability of assigned values may be questioned. However, it is also acknowledged that in fields where sufficient experience exists, supported by access to appropriate standards and good understanding of the measurement principles, consensus values should be close to traceable values and therefore provide a reliable estimate of the measurand value. Essential trace elements, particularly copper, selenium and zinc, are measured in serum to assess nutritional status, diagnose specific diseases and as part of epidemiological studies on the role of these micronutrients in the development of degenerative diseases. Measurements are performed mostly by atomic spectrometry techniques although colorimetric procedures are also used for copper and zinc. Since reference materials for these tests are very limited and generally not well characterised, participation in EQAS plays a major role in assessing the laboratory performance and support the quality of analytical work in this field. Following a recent trial of the International Measurement Evaluation Programme (IMEP-17, www.imep.ws) devoted to minor and trace constituents of human serum, including copper, selenium and zinc, a limited number of vials of two test materials became available to which values traceable to SI units had been assigned using methods of higher metrological order. The Network of EQAS Organisers in Occupational and Environmental Laboratory Medicine

exploited the opportunity to use them as a common reference traceable to the SI in order to transfer values to a batch of three secondary reference materials, covering the range of concentrations of interest, which were distributed to laboratories participating in seven different EQAS in Europe and North America. The differences between values (and their uncertainty) assigned by each organisers using his own established procedure and those assigned by comparison with those carried by the certified test materials were assessed as well as the possible impact on the evaluation of laboratory performances.

INSTABILITY OF MERCURY IN SPECIMENS OF HUMAN URINE FOR EXTERNAL QUALITY ASSESSMENT

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Organisers of External Quality Assessment Schemes (EQAS) for trace elements observe that with urine samples spiked with inorganic mercury there is failure to recover all the added mercury and the range of results reported by participants is much wider than is typically seen with other assays. Loss of mercury by volatilization is a possible explanation for these observations. However, EQAS samples are prepared to contain oxidizing preservatives *e.g.* nitric or sulfamic acids. Furthermore, the under-recoveries are inconsistent suggesting there may be features associated with the urine matrix that could make the mercury unavailable for measurement. To investigate possible factors that may be relevant to the stability of mercury in urine the following experiments were set up:

- a sample of Hg in water, with various "stabilizers" added was sent to UK EQAS participants. - To assess only laboratory analytical performance, without the additional factor of the urine matrix and to compare the effectiveness of stabilizers;
- urine was collected from volunteers who also completed a three-day food diary. Hg, Ca, Mg, Se, uric acid, phosphate, creatinine, reducing substances and protein were measured. Inorganic mercury was spiked into the urine, various stabilizers were added and the mercury determined following storage at different temperatures. - To evaluate results with reference to the urine analysis and the food diaries;
- to compare the stability of physiological and spiked mercury, urine was collected from individuals with occupational exposure to mercury. The mercury was determined following storage at different temperatures.

The results showed that: mercury will be lost from spiked solutions if stabilizing agents are not added; even in stabilized samples the urine matrix produces an under-recovery of added mercury; the technique used is not relevant but aspects of the sample preparation may influence results; there is no simple relationship between recovery of mercury and urinary components. Further experiments should be carried out.

THE HUMIC ACID PITFALL

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Natural waters often contain humic substances, which originate from plant degradation. They are known to adsorb organic compounds, *e.g.* contaminants in the water. This complicates the reliable measurement of their levels, as analytical recovery can be incomplete.

The fraction that cannot be recovered on extraction with some common organic solvents has recently been quantified by Bercaru, as part of her doctoral thesis, for several Polyaromatic Hydrocarbons (PAHs). It ranges between 10% and 60%, depending on the hydrophobicity of the congener.

A compensation for adsorption losses is often made through determination of the recovery, or measurement against an internal (isotope or other) standard. In either case, a period of time is needed for the surrogate material to reach the adsorption equilibrium. Laboratories that do not respect this period take the risk to underestimate the measurand.

This was observed during a recent IMEP Interlaboratory Comparison (ILC) that was organised in support of the EU Water Framework Directive (WFD). The ILC test sample contained a mixture of eight PAHs and humic acid in a water matrix. Certification measurements were carried out by two different reference laboratories. Fifty-nine routine laboratories mainly from Europe participated in the ILC.

Both routine and experienced reference laboratories appeared to run into the *humic acid pitfall* of measurand underestimation due to adsorption.

KDLL PT-SCHEME USEFUL TO PREDICT PRECISION OF (TRACE) ELEMENTS OVER A CONCENTRATION RANGE OF SEVEN DECADES

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The Agricultural Laboratories Quality Service (KDLL) as a service of Product Board Animal Feed (PDV) has offered, since 1988 a broad support in providing Proficiency Testing to ensure the quality of the analytical work in the agricultural sector. This Proficiency Testing Scheme is ILAC G13 accredited and the PDV has contracted out the KDLL activities to DUCARES B.V. The KDLL Proficiency Testing Scheme is focused on chemical, microbiological and microscopic parameters in animal feed and raw materials.

Examples of the KDLL scheme are: PCBs, OCs, mycotoxins, minerals, trace elements, vitamins, fatty acids, mineral oils and veterinary drugs.

A wide range of data sets has been gathered in the past four years for minerals and trace elements. The following elements are measured in the several feed and premixes: calcium, magnesium, sodium, potassium, iron, copper, zinc, manganese, cadmium, cobalt, lead, selenium, and in the last year: mercury, arsenic and chromium. The used general techniques are AAS, AES, ICP-OES, ICP-MS and graphite furnace method.

The results of this Proficiency Testing can be useful to predict the statistical performance of the minerals and trace elements over a wide concentration range. When the matrix contained added minerals and (trace) elements (for example in premix), the relative error is significantly higher compared to normal processed feed.

The median ratio of reproducibility/repeatability is 3.3 which implicates that the systematic error between laboratories isn't important. Although individual elements show an increasing relative error at their detection limits, when combining all elements there is a linear relation between the concentration and the relative error (repeatability as well as reproducibility) over a concentration range of seven decades.

EVALUATION OF PROFICIENCY TESTING METHODS FOR AFLATOXINS IN FEED

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Reliable analytical chemical methods together with a proper implementation are of crucial importance for the monitoring of the quality of foods, feed and raw materials. To improve the analytical quality of the measurements in the Netherlands, the Product Board Animal Feed (PDV) started the Agricultural Laboratories Quality Service (KDLL) program with tailor-made Proficiency Testing and harmonization of methods.

This program is carried out by DUCARES and is ILAC G13:2007 accredited. Currently, DUCARES organizes approximately thirty Proficiency Tests pro annum for analytical-chemical-, physicochemical-, microscopic- and microbiological research methods in the areas of contaminant analysis, macro- and micronutrient analysis and microbiology.

The PDV method (OSP-1), based on HPLC with Br_2/I_2 derivatisation, is recommended for aflatoxin B_1, but other methods such as immunoaffinity are accepted. In general the results of aflatoxins B_2, G_1, and G_2 are also reported by the participants.

Method OSP-1 describes the boundary values for the repeatability r and reproducibility R as a linear relationship with the average concentration. From the aflatoxine-B_1 data it appears that r is only slightly dependent on the concentration. This is true for the OSP-1 method, for immunoaffinity based methods and for these and all other methods evaluated as one group combined. The R values however always show a linear dependency on the concentration: both OSP and immunoaffinity data have linear trendlines parallel with the OSP-1 equations, but with a larger constant. All methods combined show an even steeper trendline between R and the concentration, thus a combination of methods increases the reproducibility. A same phenomenon is observed for all other aflatoxins. The slope of the trendlines are steeper than for aflatoxin-B_1. The main conclusion is that the methods for aflatoxin-B_1 can also be used for the other aflatoxins. The OSP-1 relationship between r (R) and the concentration should not be applied.

The analytical method requires an extensive clean-up procedure. Along with the feed samples a reference solution with aflatoxin-B_1 is sent, which does not need any clean-up. From the data it appears that the behavior is similar to the aflatoxin-B_1 (all methods) data. Apparently the clean-up procedure has only limited impact on the results. Furthermore, it is concluded that the intralab error is small, but that big differences are found between the labs. Calibration and instrumentation may be the most important reasons.

In the poster results of the Proficiency Testing from the past years will be discussed.

THE ORGANIZATION OF PROFICIENCY TESTS FOR RABIES SEROLOGY

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For many years, quarantine confinement has proven to be an efficient method against rabies introduction in rabies free territories, however it was unsatisfactory for animal welfare and for the owner. Between 1993 and 2000, many rabies free countries have alleviated their quarantine measures and adopted a scheme requiring a rabies vaccination followed by a serological control. This alternative measure allows to guarantee the safety of free movements of pets and preserves the rabies free status of the countries. For laboratories willing to carry out the rabies serological controls, the European Commission decided to establish a system of community approval of such laboratories in order to guarantee an effective control system. The European Commission has designated a Specific Institute to coordinate the approval of the laboratories. The main task of this institute is to organize rabies Proficiency Tests for laboratories already agreed or willing to be agreed to perform rabies serological controls. These Proficiency Tests allow to determine the performance of individual laboratories for specific tests and to evaluate the laboratories' continuing performance. To evaluate the laboratories' performance, 4 criteria are specifically analysed. They are the classical criteria that are considered when validating a serological tests: the specificity, the intra-laboratory repeatability and consistency and the inter-laboratory consistency.

After 13 rabies Proficiency Tests, 95.9% of laboratories obtained satisfactory results and 98.74% of participating laboratories succeeded for their first participation in rabies Proficiency Tests whatever the OIE prescribed method used (RFFIT or FAVN test). The organization of these Proficiency Tests and the statistical analyses performed on the results given by the participating laboratories will be presented on the poster as well as some results obtained from the ring tests.

ANALYTICAL PERFORMANCE IS IMPROVED BY REGULAR PARTICIPATION IN PROFICIENCY TESTING: AN ANALYSIS OF DATA FROM THE AQUACHECK PROFICIENCY TESTING SCHEME

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Introduction. The Aquacheck Proficiency Testing (PT) Scheme is open to participants from any industry and is designed to promote quality and comparability in the measurement of a wide range of analytes in clean waters, wastewater, soils and sludges. A vital aspect of PT Scheme design is the monitoring and improvement of the quality of a laboratory's measurements over an extended period of time. Analysis of historical and current data, obtained from PT Schemes can assist in the evaluation of methods and instrumentation, educate laboratory staff and demonstrate the quality of results to third parties. In this poster results are presented from the analysis of historic data from the Aquacheck scheme and used to reach conclusions about historical laboratory performance, considering issues of frequency of participation, scope of participation and performance improvement over time

Methods. Changes in analytical performance, based on historical data from the Aquacheck scheme, have been assessed using two measures. The spread of results returned by all participants, defined by the percentage relative standard deviation (%RSD) and the performance (z) scores awarded to the individual participants. Several groups of analytes, inorganic determinands, heavy metals and organic compounds, have been assessed and where possible "more difficult" analytes have been included to ensure quality improvement in the data returned is not confined to routinely analysed components.

Results. Over the period analysed up to 700 laboratories a year participated in the various rounds of the Aquacheck PT Scheme. Improvements in laboratory performance are seen for groups containing trace organics; a reduction in the %RSD of 10% is observed for each analyte, but this is not consistently replicated in groups which contain heavy metals and general water quality parameters. Improvements of the same order of magnitude are observed when the percentage of acceptable z-scores, those between +2 and -2, is assessed for the trace organic analytes; again the same scale of improvement is not observed when the heavy metal and general water quality parameter groups are assessed. Analysis of the frequency of participation showed that those laboratories who participated in all of the distributions for an analyte group reported 10-15% more acceptable (z-scores between +2 and -2) results than those labs who took part with a greatly reduced frequency. The increase in acceptable results was observed in analyte groups which contained trace organic compounds and those which contained heavy metals.

Conclusions. The Aquacheck PT Scheme has been established for over 20 years and as such the data produced by the participants is a reasonably well ordered system, as can be seen by the %RSD and the overall percentages of satisfactory z-scores. There is evidence to show that analytical performance has improved for several analytes, important in the determination of water quality, but this is not replicated in all of the analyte groups

assessed. For a range of analytes those laboratories which participate in the Aquacheck scheme frequently have a more consistent and accurate performance than those laboratories whose participation is less frequent.

EUROPEAN INTERLABORATORY STUDY FOR TESTING EMISSIONS OF VOLATILE ORGANIC COMPOUNDS FROM CONSTRUCTION PRODUCTS USING EMISSION TEST CHAMBERS

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An interlaboratory study with 29 institutes located in Europe was organized by BAM to get an overview of the variation of the emission test chamber method, which is used to evaluate emissions from materials into indoor air. The test method is based on the international standards ISO 16000-9 (emission test chamber) and ISO 16000-6 (analysis).

The interlaboratory study was divided into three steps. In the first step liquid samples had to be analysed using Tenax thermal desorption. For the second step sampling was done at an emission test chamber at BAM and for the third step emission chamber tests were performed by the participants at their own chambers.

For every step there was a questionnaire to get detailed information about the analytical method (thermal desorption coupled with gas chromatography/mass spectrometry) and the test chamber.

The problem is that there is no reference material for emission testing because the material alteration has a big influence on emission behaviour.

For step 3 a sealant, which has been tested for homogeneity was chosen as test material.

The mean values and standard deviations of the results from fourfold analysis were calculated. Results with high intralaboratory deviations were excluded following the Cochran test with a level of confidence of 95%. Additionally the Grubbs test was used to identify outliers.

The standard deviations did not vary much from step to step although step 3 was the most complex part. The standard deviation for step 3 ranged from 20% up to 38% depending on the chemical compound. This is much better than for former round robin tests and is surprising because the questionnaire showed a large variation for the analytical equipment and for the test chambers.

The small standard deviation might be due to the very good homogeneity of the test material and the easy preparation of it for the chamber test.

Only three laboratories were not successful in achieving a z-score of less or equal 2 for 80% of all compounds analysed in step 2 and 3.

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Stampato da Tipografia Facciotti srl
Vicolo Pian Due Torri 74, 00146 Roma

Roma, luglio-settembre 2008 (n. 3) 9° Suppl.