# REPORT OF THE 15TH MEETING OF INTERNATIONAL ORGANISATIONS WORKING IN THE FIELD OF METHODS OF ANALYSIS AND SAMPLING (INTERAGENCY MEETING)

#### **Budapest, Hungary 15<sup>th</sup> November 2002**

#### **Present**

AOAC INTERNATIONAL Al Pohland (Secretary)

Markus Lipp

Roger Wood (Chairman)

AOCS Richard Cantrill

Kim Magin

AAFCO Alan Hanks

CEN TC 275 Braxton Reynolds

CIPAC Alan Hanks
EOQ Pal Molnar
EURACHEM Marie Walsh
ICC Radomir Lasztity
ICUMSA Roger Wood
IDF Edward Hopkin
ISO Martha Petro Turza

Nadine Normand

NMKL Hilde Skar Norli

Harriet Wallin

OIV Alain Blaise

Jean-Claude Ruf

Mary Kelly

#### **Apologies**

Apologies were received from Scott Coates (AOAC RI), H. Glattes (ICC), Arpad Ambrus (IAEA), Ales Fajgelj (IUPAC), Selma Doyran (Codex) and Jeronimas Maskeliunas (Codex), Marianna Toth (EOQ) and Pauline Jones (ISO).

The attendees were welcomed by the Mrs Martha Petro-Turza of the Hungarian Standards Institution, which was, as previously, kindly hosting the meeting.

#### 1. Report of the previous meeting IAM-14, 2001

This was accepted without modification, except that in Section 10 it was clarified that the EURACHEM publication referred to gave a glossary of terms and their translation into various languages of interest to analytical chemists.

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#### 2. Matters arising from the previous meeting not otherwise on the agenda

There were no matters arising from previous meeting not otherwise on the agenda. However, it was noted that a number of the actions agreed at the previous meeting had not been completed by the participants at that meeting.

#### 3. Review of IAM membership

The membership of the IAM was discussed. It was agreed that an invitation to participate in the activities of the IAM would be made to those organisations which had been listed in the "Directory of Organisations" which had, until recently, been prepared for CCMAS delegates and which are recognised "International Organisations" in the sense accepted by the current IAM membership.

These organisations, which includes the current IAM members, are given below

AACC American Association of Cereal Chemists

AAFCO American Association of American Feed Control Officials

AOAC\* AOAC INTERNATIONAL
AOAC RI\* AOAC Research Institute
AOCS\* American Oil Chemists' Society

CEN\* European Committee for Standardization

CIPAC Collaborative International Pesticides Analytical Council

CODEX\* Codex Alimentarius Commission of the FAO/UN

EAL European Co-operation for Accreditation of Laboratories

EOQ European Organisation on Quality

**EURACHEM\*** 

FECS Federation of European Chemical Societies IAEA\* International Atomic Energy Agency

ICC\* International Association for Cereal Science and Technology ICUMSA\* International Commission for Uniform Methods of Sugar Analysis

IDF\* International Dairy Federation

IFG International Federation of Glucose Manufacturers

IFU International Fruit Juice Union

ILAC International Laboratory Accreditation Conference

IOCCC International Organisation for Cocoa, Chocolate and Confectionery

IOOC International Olive Oil Council

ISO\* International Organization for Standardization IUPAC\* International Union of Pure and Applied Chemistry

NFPA\* National Food Processors' Association NMKL\* Nordic Committee on Food Analysis

OIV\* International Organisation for Wine and Vine

WHO\* World Health Organisation

It was also agreed that the organisations would be asked to prepare a simple "News Report" which could be regularly up-dated as necessary and which could be circulated to the membership, ideally on a six-monthly basis.

<sup>\*:</sup> current IAM member.

In addition other organisations which were listed in the "Directory" would be informed of the activities of the IAM, as are participants to the Codex Committee on Methods of Analysis and Sampling through the report back to each Plenary Session of CCMAS.

#### These organisations are:

AGAL Australian Government Analytical Laboratories

AMC Analytical Methods Committee of the Royal Society of Chemistry (UK)

APA Association of Public Analysts (UK)

BgVV Federal Institute for Health Protection of Consumers and Veterinary Medicine

(Germany)

CFIA Canadian Food Inspection Agency

EU European Commission (DG AGRI, DG SANCO and DG RESEARCH)

FDA Food and Drug Administration (US)

FIS Food Inspection Service (The Netherlands)

FSA Food Standards Agency (UK)
USDA US Department of Agriculture

# 4. Review of actions taken by Codex in response to the results of the 14<sup>th</sup> IAM meeting and other actions by Codex affecting IAM

#### 4.1 Criteria Approach

It was noted that it was likely that the criteria approach would be further developed by CCMAS at its 24<sup>th</sup> Session. It was noted that as a result of the adoption of this approach it was probable that the users of analytical methods would require more information than was currently included in "Standard Methods".

It was noted that the criteria required in the Codex discussion documents indicated that assessments for:

- accuracy
- applicability (matrix, concentration range and preference given to 'general' methods)
- detection limit
- determination limit
- precision; repeatability intra-laboratory (within laboratory), reproducibility interlaboratory (within laboratory and between laboratories), but generated from collaborative trial data rather than measurement uncertainty considerations
- recovery
- selectivity
- sensitivity
- linearity

may be required to be assessed. At the present time few, if any "Standard Methods" included all such information. This would be discouraging to the users of methods and is an issue that will have to be addressed if/when the criteria approach is implemented in the food sector.

#### 4.2 Single-Laboratory Validation

It was noted that this would be extensively discussed at the 24<sup>th</sup> Session of CCMAS. It was also noted that a number of the papers to be discussed were of a general nature and not a simple "how-to" guide for the bench analyst. It was also noted that a number of such papers had recently, or are about to be, published.

The NMKL representatives agreed to up-date the Chapter "A Critique on Available In-House Method Validation Documentation" in "Principles and Practices of Method Validation" edited by A Fajgelj and A Ambrus, UK RSC 2000 in the light, particularly, of the recent papers prepared by the EU, IAEA, IUPAC and the NMKL. In addition AOAC is preparing a "how-to" guide for use in its new Single Laboratory Validation Programme.

#### 5. Electronic Compendium of Analytical Methods (e-CAM)

The AOAC project on developing an electronic compendium of analytical methods (e-CAM) was introduced and explained by Al Pohland. The rationale behind the project was explained and its progress to date.

It was noted that the inclusion of methods developed by organisations other than the AOAC was necessary to ensure a worldwide appeal of the system. To this end Memoranda of Agreements were being discussed between some International Organisations and the AOAC. It was noted that e-CAM would serve as a reference point to aid the analyst obtain summary information about the performance characteristics of available methods of analysis submitted to the system applicable to any particular determination. In particular it may be expected that *summary* information on the following attributes of a method would be included in e-CAM:

- Method Title, Scope (Abstract)
- References (publications and regulations)
- Principle and Summary of Method
- Definitions
- Matrix (Sample Information)
- Method performance
- Interferences
- Physical and Environmental Conditions
- Apparatus and Equipment
- Reagents and Supplies
- Standards,
- Sampling Procedures
- Test Sample Preparation
- Analytical Procedure
- QA/QC Procedures
- Calculation and Interpretation
- Appendices: Tables; Diagrams; Flowcharts; Supplemental Validation Data
- Bibliography

It was stressed that any copyright would remain with the organisation submitting the method to e-CAM, and that the analyst would have to obtain the text of the method from the submitting organisation in the usual way. However, it was anticipated that simple Website links to the submitting organisation would be set up to aid the analyst retrieve the texts.

Participants considered that the system would be of benefit to both the organisations and, more importantly, to the users of the methods of analysis. It was reported that it was hoped that the "almost final" electronic version of the system would be ready for  $\beta$ - testing in June 2003.

It was noted that the system would not directly affect existing Codex Standards, but would aid users of Codex Standards when the criteria approach was adopted, as it would readily demonstrate whether methods were available which met the performance standards then being demanded by Codex.

It was also noted that the documentation regarding e-CAM would be re-drafted by the AOAC to be applicable to the members of the IAM, and would be sent to all organisations invited to be associated with the IAM (see Section 3 of this Report).

## 6. Discussion of IAM role and involvement in issues relating to Quality Assurance in Food analysis

It was noted that there were several recent publications/activities in the quality assurance for analytical laboratory area which are of interested to the IAM members, notably:

- The NMKL procedure No. 9 for evaluation of results, derived from the analysis of certified reference materials.
- ISO/CEN work in the control of media for microbiological determinations.
- ISO activities in the development of recommendations for estimating measurement uncertainty in microbiological determinations.

Members agreed to provide information on other recent initiatives, publication of standards, in the area.

It was also noted that with the publication of ISO 17025 there would be an enhanced emphasis on traceability to be made for the accredited analytical determination. Here CITAC/EURACHEM has produced 2 QA guides for chemistry in the 17025 context and for microbiology.

To aid the laboratory it was noted that a EURACHEM/CITAC Guide on "Traceability in Chemical Measurement" was currently undergoing development; it was initially discussed at an International Workshop held in Lucerne, June 2002.

It was also commented at a list of readily available *quality control* materials (as well as reference materials) was being collated in the UK developed to aid the analyst and participants were invited to contribute to that list.

#### 7. IUPAC/ISO/AOAC INTERNATIONAL Harmonisation Programme

It was reported that the current IUPAC/ISO/AOAC Protocol for Proficiency Testing: The International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories, already adopted by Codex by reference, was being revised by the IUPAC Working Party for Harmonization of Quality Assurance Schemes for Analytical Laboratories in the light of experience over the past 9 years since its publication. [The editors of the review are Michael Thompson, Steve Ellison and Roger Wood].

It was anticipated that the review would consider, in particular:

- the choice of target standard deviations
- the measurement uncertainty of the assigned value
- the problems with "satisfactory" not detected values
- the possibility of using proficiency testing results for method validation

The draft revision would be discussed at an International Workshop, to be held in Vienna in 2003, organised by IAEA. Members of the IAM would be notified of the Workshop.

#### 8. Proficiency Testing

See Section 7 above, and

## 8.1 The Continuous Assessment of Standard Methods of Analysis - Effects of Proficiency Testing

At the present time IAM members prepare and publish standard methods of analysis. There is little follow-up or dialogue between the membership and the users of methods of analysis. Because participation in Proficiency Testing Schemes is now so widespread, it would seem that there is now an opportunity for the continuous assessment of adopted methods of analysis if sufficient participants state that they use a defined method exactly as written.

IAM members, while discussing this issue, were very interested but stated that it should be made clear by PT users under what circumstances methods were applied. Any variation in method would preclude the use of data to re-evaluate precision characteristics, most notably the  $RSD_R$  values.

The participants also noted that it may be possible to give information on whether specific methods of analysis may be validated through proficiency testing scheme results if sufficient participants "happened" to use the same method. IUPAC would consider this possibility when its Working Party on the Harmonisation of Quality Assurance Schemes revised the International Harmonised Proficiency Testing Protocol.

#### 8.2 Blind Proficiency Testing

It was noted that the use of blind proficiency testing (i.e. when analysts were not aware that the sample they were analysing was a proficiency testing sample but would assume that it was a routine sample for analysis) was likely to increase, and that the results from such exercises should be handled with caution. In particular it was essential that the Analytical Laboratory Community was not unfairly discredited by such exercises.

### 9. Incorporation of Change of Methods/Method Corrections in the Codex Alimentarius Commission.

Concern was expressed by participants that methods of analysis were frequently incorrectly referenced in Codex Alimentarius publications, and in particular Volume XIII.

It was also noted that where methods were up-dated by an organisation, this was also not reflected in Codex publications.

The participants therefore requested the Codex Secretariat to ensure the IAM participants which had methods of analysis/sampling/other publications referenced in Volume XIII were consulted in time to make corrections before future revisions of the Volume were published.

#### 10. Harmonisation of Analytical Terminology in Accordance with International Standards

A paper listing current analytical terminology was discussed. It was agreed that participants would be asked to update the current paper and for it to be then published on the Web. It was also noted that the VIM was currently being up-dated and note of this would have to be taken when revising the current paper.

#### 11. Policies and Practices of IAM Members Regarding Proprietary Laboratory Techniques

The above was briefly discussed and participants agreed to send in their policies in this area, particularly with respect to the adoption of so-called "black-box" techniques.

### 12. Discussion of the Utility, Objectives, and Future Direction of the IAM; Including Revision of the Terms of Reference

Participants considered the meeting to be of value, and would continue to be if the actions agreed were undertaken by participants in the IAM.

The Terms of Reference of the IAM were briefly discussed. These have been revised by the Secretariat in the light of discussions and current draft is attached as Annex I.

#### 13. Any Other Business

#### 13.1 Acknowledgement of Other Organisations' Methods

It was noted that there have been recent instances of methods being adopted by an organisation where the method has been developed and usually collaboratively studied under the auspices of another and that this has not been acknowledged.

This practice was not appreciated as it was recognised:

- as being unhelpful to the user of the method not to know its true pedigree (its traceability),
   and
- discourteous to the developing organisation.

It was stressed that this was not a copyright issue as such.

#### 13.2 Rounding of Results

It was noted that the procedure for the Reporting of Analytical Results prepared by Professor Peter Wilrich for the previous IAM was being up-dated and would shortly be available for comment.

#### 14. Publication of IAM Report

It was agreed that the Report of the  $15^{th}$  IAM would be published on the AOAC Website (www.aoac.org) once it was finalised.

#### 15. Provisional Date of the Next Meeting

The next Inter-Agency meeting will take place prior to the 25th Session of CCMAS, at Budapest, Hungary

#### ANNEX I: LIST OF ACTIONS AGREED AT THE 15TH INTER-AGENCY MEETING

The following actions arose from the 15th Inter-Agency Meeting:

- 1. Invitation to participate in the activities of IAM to be extended (Secretariat).
- 2. Members to prepare regular contributions to a News Report (all members). This to be provided on the 1<sup>st</sup> January and 1<sup>st</sup> July each year, commencing 1<sup>st</sup> July, 2003 (all members).
- 3. NMKL representatives to up-date the Chapter "A Critique on Available In-House Method Validation Documentation" in "Principles and Practices of Method Validation" edited by A Fajgelj and A Ambrose, UK RSC 2000 in the light of recent papers in the area (NMKL).
- 4. Documentation regarding the Electronic Compendium of Analytical Methods (e-CAM) to be re-drafted for IAM purposes (Secretariat).
- 5. Information to be provided on other recent initiatives, publication of standards, in the quality assurance area (all members).
- 6. Contributions to a list of readily available quality control materials to be made (all participants).
- 7. The Codex Secretariat is to be requested that all IAM participants, who have methods of analysis/sampling/other publications, referenced in Volume XIII are consulted in time to make changes regarding any amendments needed before publication of documents.
- 8. Participants would update the current the current definitions paper (all participants).
- 9. Participants to provide secretariat any information on their approach to the adoption of propriety laboratory techniques (all members).
- Terms of Reference to be revised and circulated to members (the Secretariat).
- IAM members recommended to insert the full "pedigree" of methods in their adopted Standards (all members).

#### **Terms of Reference of the Inter-Agency Meeting**

#### The Inter-Agency Meeting (IAM)

The meeting of the international organisations working in the field of analysis and sampling of food products and associated quality assurance measures (hereafter referred to as the "Inter-Agency Meeting") comprises representatives of interested international (governmental and non-governmental organisations active in this field.

#### Terms of Reference

The aim of the Inter-Agency Meeting is to promote co-operation between international organisations and to support the needs of the Codex Alimentarius Commission. In this respect, the Inter-Agency Meeting:

- 1. Develops and/or recommends common rules for the validation of methods of analysis and sampling and associated quality assurance measures;
- 2. Assists the Codex Alimentarius Commission in its need for methods of analysis and sampling and associated quality assurance measures;
- 3. Promotes Co-ordination between the organisations concerned;
- 4. Addresses problems which require international collaboration; and
- 5. Promotes uniformity of terminology relative to its field of activity.

#### Sessions

The Inter-Agency Meeting is normally held in association with the sessions of the Codex Committee on Methods of Analysis and Sampling (CCMAS).

#### Secretariat

A participating organisation to the IAM assumes the Secretariat function for 3 sessions, and may be renewable for a further 3 sessions.

The Secretariat function is currently assumed by the AOAC INTERNATIONAL; this will cease, or be renewed, at the end of the 16<sup>th</sup> Session of the IAM.

#### Chairman

The Chairman of the Inter-Agency Meeting is appointed for the duration of one meeting. This appointment starts at the end of the Session of CCMAS to which the IAM session is attached, and will cease at the end of the subsequent Session of CCMAS.

It is expected, but not a requirement, that the current Chairman and Secretariat will be provided by the same IAM member organisation.

#### Amendments

Adopted circa 1977.

Amended March 1997 (IAM -12).

Amended November 2002 (IAM-15).