

**REPORT OF THE 26th MEETING OF INTERNATIONAL ORGANISATIONS WORKING  
IN THE FIELD OF METHODS OF ANALYSIS AND SAMPLING  
(INTER-AGENCY MEETING; IAM-26)**

09.30 – 13.00h, Saturday, 1st March 2014

**Present**

AACC International	Anne Bridges
AACC International	Paul Wehling
AOAC International	Stan Bacler
AOAC International	Erik Konings
AOAC International	Darryl Sullivan
AOAC International	John Szpylka
AOCS	Ray Shillito
AOCS	Richard Cantrill (Secretary)
BIPM	Ralf Josephs
CEN	Duncan Arthur
Codex Alimentarius	Verna Carolissen
Codex Alimentarius	Hidetaka Kobayashi
Codex Alimentarius	Patrick Sekitoleko
EURACHEM	Steve Ellison
ICUMSA	Roger Wood (Chair)
ISO/IDF	Jaap Evers
ISO/IDF	Roger Kissling
ISO/TC 34	Sandrine Espeillac
ISO/TC 34/SC 5	Marcel de Vreeze (repl. Sophie More)
IUPAC	Christoph von Holst
IUPAC	Zoltan Mester
NMKL	Hilde Skår Norli
Invited	Arpad Ambrus
Invited	Greg Noonan

**Apologies**

Apologies were received from Roland Poms (ICC).

The attendees were welcomed by Dr. Roger Wood (Chair) who thanked Dr Ambrus for kindly offering to host the meeting at the Hungarian Food Safety Office. The Chair stressed the need to complete the discussions in a timely manner. There were no additions to the agenda.

**1. Report of the Previous Meeting IAM-25, 2013**

The representatives of ISO/TC 34/SC 5 and IDF corrected the roles of ISO and IDF in the preparation of targeted guidelines for the quantitative determination of melamine and cyanuric acid by LC-MS/MS in milk and milk products (ISO/TS 15496 | IDF/RM 230, published in 2010) and the CEN in developing a separate method with a broader scope of application (also to be validated for e.g. infant formula soy, candy bars, milked-based sweets).

## 2. **Matters arising from the Previous Meeting not otherwise on the Agenda**

### **Validation of in-line methods of analysis**

The Chair indicated that he hoped some results would be forthcoming from ICUMSA in time for next's year meeting.

## 3. **Method validation issues**

### **AOAC Expert Review Panel Methods Progress – use of proficiency test data**

AOAC representatives explained the process they are developing for use in the generation of method performance data from rounds of proficiency testing data. Guidance documents will be available shortly. In essence, proficiency testing samples will be tested in progressive rounds prescribing the use of a particular method rather than various methods at the same time. To obtain repeatability values, results would be required in duplicate. A number of concerns were raised about data confidentiality, and the use of a method before it can be validated. Attention was drawn to the availability of a Codex document from CCPR which can be regarded as a precedent in Codex for the use of PT data to estimate precision parameters. The Chair recalled the existence of documents that were presented to Codex in 2002 but were not developed further (previously circulated to IAM members). Though studies had yet to be performed this way, a similar approach had been developed by NMKL for method comparison. It was noted that in order to meet current Codex requirements, studies on infant formula in the AOAC SPIFAN program were being conducted using the conventional approach.

*Outcome: The IAM members noted positive progress in this field and looked forward to further information on the development of the AOAC procedures.*

### **Use of Standard Method Performance Requirements in ISO/IDF (SMPR)**

A discussion paper from ISO/IDF prepared for the ISO/TC 34 plenary meeting was discussed. A stakeholder panel is used to develop the criteria a relevant method of analysis must meet. A number of participants questioned the author in regards to the global composition and balance of the stakeholder panel, the comparison of methods with those from other pharmacopoeial sources, the difference between this proposal and criteria or processes already in use. The author explained that great care is taken to ensure balanced representation on the stakeholder panel.

*Outcome: the proposal will be discussed at upcoming meetings of ISO/TC 34 and ISO/IDF.*

### **International guidelines for the validation of qualitative methods – update**

AOCS reported on a meeting held the previously day that included members of the specific ISO Working Group (ISO/TC 34/SC 16/WG 5), liaisons from IUPAC, MoniQA Association, IAM, AACCI and Eurachem. The main discussion revolves around the determination of confidence limits when the detection level falls in the 85-99% probability of detection where a large number of replicates may be required. Four model approaches have been proposed and these models will be tested using simulation data sets. A document is in preparation for consideration by the working group and submission to ISO as a CD for vote.

*Outcome: A draft will be produced and data from simulation sets generated using agreed parameters will be used to check the different approaches which we await further experimental data from GMOVAl and other sources.*

#### **Requirements regarding modifications of proprietary methods**

A number of organizations consider the level of editorial/scientific work required to modify the method and determine the level of revalidation that might be required.

If the provider of a proprietary method does not supply sufficient information, then the method should be considered Type IV.

*Outcome: IAM members recommend that if the provider of a proprietary method does not supply sufficient information, then the method should be considered Type IV.*

#### **4. Revision of ISO 5725**

The project has been returned to Preliminary Work Item status (PWI) as the project had failed to provide a CD or Draft Standard within the appropriate timescale and, following a change in responsibility of the existing project leader, there is currently no project leader available to carry on this ambitious project. The participants considered the current standard and agreed that while it might be useful to add more recent statistical methods as permitted options, the standard largely met their present needs, so immediate revision was not felt to be a priority.

#### **5. CCMAS papers**

##### **CX/MAS 14/35/5 - Discussion paper on considering procedures for establishing criteria**

This item elicited considerable discussion. It was agreed that there were two distinct issues under discussion. 1) Type I methods might be considered to be of two subcategories: the first involving technique and instrument dependency such as moisture and the second involving the measurement of an analyte such as the KOH equivalence for free fatty acid content where the actual composition is not known but may be estimated by use of a conversion factor. 2) The analysis of multiple components of a similar class of compounds (such as individual aflatoxins) but the specification requires the determination of the total content by summing individual components.

It was concluded that not all criteria can be applied to Type I methods since this requires a decision to be made since “trueness” cannot be defined. “HorRat type” criteria only apply when there is a relationship with mass fraction. However some type of guidelines could be developed. Should a conversion factor be employed, it would be the purview of the relevant committee to agree on this value, not the method developer.

In the case of multi-analyte determinations the participants indicated it would be impractical to define each component and set criteria for each analyte and thus the “HorRat value” could not be applied to the sum of analytes.

*Opinion: There is no need to establish criteria for Type I methods, However, there is a need to consider the statistical treatment for the sum of analytes identified in a single determination.*

### **CX/MAS 14/35/6 - Discussion paper on elaboration of procedures for regular updating of methods**

The participants discussed the paper for the elaboration of procedures for regular updating of methods in the Codex system. The participants agreed that there is confusion regarding the process to update methods of analysis listed in Codex Standard 234. Although it was assumed that all methods of analysis from the Codex commodity committees are listed in the document, there was concern that some methods may still be only found in the respective Standards. The participants discussed the role IAM members should play in the revision of the methods of analysis and sampling Standards listed in Standard 234 and also the issue of dormant committees. While the process for revision of standards in documents from active committees is clear, the process for the revision of the other group of standards is less clear. It was agreed that Standard 234 would be more useful if it were made available as a searchable database, though the Codex Secretariat would be responsible for the creation and maintenance of the database and IAM members would be responsible for keeping their method entries current.

The participants also discussed the required information for Table 1. It was felt that much of the information required could already be found within the method or its supporting documents, where available. It was also possible that some of the requirements for precision data could not be fulfilled as the method were developed and in use from a time before the introduction of the concept of collaborative trials. It was felt that it is unlikely that new trials will be conducted and grandfathered methods should be excluded from this requirement. It was also pointed out that coefficients of variation would have to be quoted for every level of analyte and matrix, thus making the table unwieldy. It was felt that the current requirements of Standard 234 were sufficient to point the analyst in the right direction, though the addition of a column denoting responsibility for updating would be of benefit. The relationship between Table 1 and Table 2 was also discussed though no clear conclusion of any linkage between the two was made.

*Opinion: The IAM members feel that 1) the requirements for Table 1 should be restricted to those already in Standard 234 possibly supplemented with an entry listing which body is responsible for updating the method and the date the method was last reviewed. 2) It is the role of the relevant SDOs to maintain their own listings. 3) There should be a defined roadmap for the updating of methods for specifications developed or maintained by active committees. 4) There should be a defined roadmap for the updating of methods from dormant committees. 5) There should be a CCMAS agenda item requesting "notification of changes to methods" where SDOs can bring forward suggested changes or other items for discussion. 6) The Codex Secretariat should send out the current version of Standard 234 three months in advance of the Codex meeting and SDOs should send their changes to the secretariat prior to the two-month deadline for document submission. This could be achieved with a Circular Letter.*

### **CX/MAS 14/35/7- Discussion paper on Sampling in Codex standards**

The participants discussed the purpose and contents of the discussion paper. It was felt that there was a need to explain the rationale between different forms of sampling and sampling uncertainty. It was felt that the development of this document would be enhanced by the inclusion of examples of sampling plans and the rationale behind them. This document would then augment the sampling information contained in GL 50. The Commodity Committees would benefit from having access to this information

and would be able to ask CCMAS and the IAM authors for help. The disposition of the paper in the Codex system was also discussed, though no resolution was agreed upon.

*IAM will monitor with interest whether a member body will be willing to lead an e-Working Group with the assignment to revise the document and add examples so that it may be published as an informational text within the Codex system or as an annex to GL50.*

6. **IAM/MoniQA Workshop**

This year's workshop attracted more than 60 pre-registrants and a number of on-site registrations from CCMAS delegates. Under the banner of MoniQA Association and Inter-Agency Meeting, the meeting was sponsored by ICC, NMKL and AOCS together with donations collected at the door. An afternoon of discussions on sampling kept the room full till the end. Comments and suggestions for future workshops are welcomed.

7. **Exchange of Reports and Information/Concerns of Members**

In view of lengthy discussions on the Codex papers, no urgent issues were identified.

8. **IAM Management**

AOCS agreed to continue to hold the secretariat for the next year together with the Chair, Roger Wood.

9. **Any Other Business.**

No items were raised under other business.

10. **Provisional Date and Place of Next Meeting**

The next meeting of IAM will be held prior to the next meeting of CCMAS but the exact details depend on the structure of the next CCMAS meeting, especially whether a physical working group on endorsement of methods will be held prior to CCMAS.