14.00 – 18.00h, Friday, 7th March 2008

Present

AOCS     Richard Cantrill (Secretariat)
AOCS     Ray Shillito
BIPM      Robert Wielgosz
BIPM     Ralf Josephs
CEN      Duncan Arthur (CEN/TC 275)
Codex     Selma Doyran
EURACHEM    Steve Ellison
Hungarian Food Safety Office  Arpad Ambrus (Observer)
ICC      Roland Poms
IDF      Aurélie Dubois
IDF     Rinus van Schaik (ISO/TC 34/SC 5)
IDF     Jaap Evers
IDF     Harrie van den Bijgaart
ISO     Jean-Baptiste Finidori (ISO/TC 34)
IUPAC     Christoph von Holst
IUPAC     Roger Wood (Chair)
NMKL     Hilde Skaar Norli
Observer    Michael Sussman

Apologies

Apologies were received from A. Fajgelj (IAEA), M-A. Godshall (USDA), D. Mittanck (AOCS), A. Pohland (AOAC), M-N. Bourquin (ISO), J-C. Ruf (OIV), and M. Blondel (OIV).

The attendees were welcomed by Dr. Wood (Chair) who thanked Dr Arpad Ambrus, Deputy Director of the Hungarian Food Safety Office, for again kindly hosting the meeting.

1. **Report of the Previous Meeting IAM-19, 2007**

   This was accepted without modification.

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

   There were none.

3. **Criteria Approach to the Adoption of Methods**

   The participants discussed the CCMAS paper on the Criteria Approach. NMKL indicated that sampling uncertainty was not part of the process. The participants commented on the effect the criteria approach will have on the activities of Standards Development Organisations (SDOs). It was noted that the criteria approach does not replace the need for official methods of analysis. However, it was agreed that the laboratory data requirements, though partly available from
routine method validation, are both time-consuming and expensive to generate. Some members felt strongly that the use of the criteria approach and heavier reliance on single laboratory validation while acceptable to regulatory bodies, was not the most practical outcome for analytical laboratories. It was postulated that the use of the criteria approach could lead to an increase in the number of disputes. However, the EC has implemented the cascade approach which gives the order of priority of methods analysis: EC method > fully validated collaboratively tested method produced by a recognised SDO > other fully validated collaboratively tested methods > single laboratory validated method. IAM members recognise the need to collect the results of method validation and make them available to the users of the methods.

The participants then considered the document in detail. It was agreed that methods should be selected which fulfil the criteria laid down in Codex provisions and CCMAS should inform commodity committees of the need to ensure that methods to be endorsed by Codex are “fit-for-purpose”. In discussion of Section 3 of the Criteria Approach document, IAM members agreed that methods which do not have adequate performance characteristics for their intended Codex purpose should be retyped or delisted. Some members of the group were of the opinion that it was better to have no method within the Codex system than a method that was not able to meet the required purpose (i.e. was not “fit-for-purpose”). The representative from ISO/TC 34 was concerned that the criteria approach should not replace listing of specific methods as analysts need information regarding the possible sources of suitable methods since the Procedural Manual indicates that the criteria approach will replace the need for specific methods of analysis. It was also pointed out that some of the older ISO references may have already been revised and may now have better precision data. Finally the IAM asks if there is a need for guidance on the use of the criteria approach by Commodity and other committees?

IDF noted that it had concerns regarding the validity of some of the criteria outlined in section I of CX/MAS08/29/7 and that IDF’s comments have been formally submitted to the Working Group through the normal CCMAS procedures.

With regard to the criteria approach, IAM offers to develop position papers for CCMAS on the following issues:

a. Implementation of a hierarchical method selection process;

b. How SDOs view and use the criteria


The most recent version of the Codex discussion document reflects the latest version of the VIM, to be released shortly. Most of the definitions are taken from the VIM, though some are from ISO 3534. These definitions are used in both Appendix 1 and 2. IAM participants considered the definitions in Appendix 2 and agreed to the inclusion of many of the other terms suggested. Previous problems with the Issue of LOD/LOQ have been solved by the inclusion of the ISO 11843 definitions used for critical value and detection limit. Although some new concepts not currently in the Procedural Manual have been proposed as a result of the definitions given in
Appendix 2 the IAM participants agreed only to the exclusion of alpha and beta. It was agreed that the remainder of the terms should be added to Appendix 1. IAM agrees that such terms and definitions should be removed from the Procedural Manual and replaced with a reference to this document (when finalised). Although some terms may have different definitions it was considered that these should be the preferred definitions for Codex purposes.

IAM members are to be encouraged to use the revised document once completed.

5. Recovery Correction in Collaborative Trials

Christoph von Holst indicated there had been little progress since the last meeting of IAM and indicated that the previous results would be made available though the IAM website (www.aocs.org/meetings/iam). The discussion covered different instances of measurement uncertainty with and without recovery correction. It was considered to be a trade-off between bias and precision. No progress had been made on the suggestion that Christoph von Holst and Steve Ellison produce a discussion paper for future IAM consideration - including recovery in the determination of analyte concentrations, role of recovery correction in method validation and recovery and laboratory bias.

6. Exchange of Reports and Information/Concerns of Members

It was agreed that information on current work programmes of the IAM Members should be made freely available. It was therefore agreed that IAM members would continue to supply the IAM Secretariat with the following information for posting on the IAM Website:

• All member work programmes through down-loadable files or as links to member websites.
• Links to publicly available newsletters and news items of individual IAM members
• Lists of published standards and links to list of newly published standards

6.1 An update of the European Framework 6 Project “MoniQA” was given by ICC. In summary (from www.moniqa.org):

“Aims & Goals of the MoniQA project MoniQA (full title: Towards harmonisation of analytical methods for monitoring food quality and safety in the food supply chain) seeks to establish long-lasting cooperation amongst leading research institutions, industrial partners, and the small- and medium-sized businesses that dominate European food manufacture and retail in order to ensure food quality and safety for consumers.

By implementing joint research programmes and promoting exchanges of researchers, the project partners hope to develop solutions that will be well accepted by consumers, manufacturers, and regulatory bodies as well as other groups involved in the food chain. Significantly, researchers will also investigate the food quality and safety implications of new processing technologies, to identify future research needs.
MoniQA will allow participants to work together in areas other than research, including training. It will also mean that knowledge as well as equipment and personnel can be shared globally among the partners.

In the long term, the consortium hopes that the project will form the basis of a global network of food safety and quality experts, which will continue to exist long after the initial project has come to an end."

6.2 ISO/IWA Bulk Commodity Grain Sampling

IAM delegates heard about a new ISO initiative taking place in 2008 to determine the baseline practices used in the sampling of bulk commodities to aid in the revision of ISO standards:

The project outline states that “The purpose of the IWA is to provide the basic foundations for all sampling documents used for commodity grain sampling. Since there are regional, governmental, voluntary consensus and intergovernmental standards (Codex Alimentarius), the IWA proposes to bring together experts from all of these constituencies. Additionally, surveyors and official sampling organizations will be invited to present a real-world perspective on the schemes which are currently used and their rationale. Since it is unlikely that a commodity grain shipment can (or will) be sampled according to several different protocols, the IWA will aim to identify the basis of the different sampling schemes and provide the statistical background to enable the development of an acceptable single scheme. This should provide a platform for assessing the possible precision of methods of analysis used for determining quality parameters.”

7. Incorporation of change of methods/method corrections in the Codex Alimentarius Commission

The IAM noted with thanks that the most recent list of methods of analysis in Codex Standards had been made available and would be posted on the IAM website. IAM members are encouraged to review the list and bring changes to the attention of CCMAS through the Methods Endorsement Working Group. Special attention should be paid to the methods in specifications in both active and stood-down committees.

8. International guidelines for the validation of qualitative methods through collaborative trials

The development of these guidelines has become a joint IUPAC/MoniQA cooperation using professional statisticians. It will address the sensitivity of test kits giving yes/no answers. Models will be developed to address the portion of correct answers, and estimate impact of results at a range of concentrations of analyte. Attention was drawn to the fact that many major kit manufacturers and groups working on statistical approaches were not currently involved.
9. **Uncertainty of sampling Update (Nordtest and Eurachem Guides)**

The Eurachem and Nordtest Guides on the estimation of sampling uncertainty have been published within the last year. Workshops have been/will be held to aid in interpretation and implementation of the Guides.

10. **Measurement Uncertainty and the Horwitz Equation**

A recent publication was presented and thoroughly reviewed. A number of the assumptions and omissions were discussed and some technical reservations were expressed regarding the use of older publicly available data which may be affected by previous lab QA practices. Some animated discussion ensued regarding this statistical approach and the applicability of the Horwitz equation, however there was acknowledgement that the Horwitz equation may not always be an accurate predictor of the performance of a method.

11. **CEN TC 275 WG0 Report**

The output of this Working group was presented with special emphasis on measurement uncertainty and the modular approach to method validation. There were a number of comments on the work program and a feeling that such important work should not to conflict with the ISO/CEN relationship and the activities of many SDOs.

12. **Review of Secretariat and Chairmanship**

The members recognized that AOCS was again hosting a formal session of IAM. AOCS agreed to continue this role for the next year. The committee also recognized the fine work of the Chair Roger Wood and encouraged him to remain in office for a further year.

13. **Other Business**

At the last meeting of CCMAS it was thought appropriate to put together a workshop to help CCMAS delegates appreciate the application of method performance and analytical uncertainty. The IAM through its sponsoring organisations AOCS, BIPM, ICC (MoniQA) and NMKL, organised and hosted this meeting on Sunday 9 March, 2008 at the Hotel Helia. It was attended by more than 75 delegates from 29 countries. The many presentations from experts in the field provided for a highly informative workshop with ample audience participation. Presentations will be available through both the MoniQA and IAM websites.

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

Friday 14.00h before CCMAS in Budapest
Pros and cons of correction for recovery: Summary on the results from interlaboratory studies

Christoph von Holst

1. Introduction

European legislation (e.g. Commission Directive 2003/78/EC in the field of mycotoxin analysis) specifies that in some fields of food and feed analysis the analytical results should be corrected by recovery. A Commission paper from 2005 gives more details on this topic (see reference list).

Quite different approaches currently exist in order to assess the recovery rate of analytical measurements. Depending on the respective analyte/matrix combinations the following procedures are frequently applied:

- The "true" recovery rate of an individual analytical measurement is determined by adding isotope labelled standards or surrogate standards to the sample. The concentration of the target analyte in the unknown samples is then corrected by the "true" recovery rate (method 1). It is considered to provide the best estimate of the recovery rate.

- The recovery rate is determined when validating the method and the obtained value for the recovery rate is taken as such in routine analysis for correcting the results of analysis (method 2).

- In each batch of samples with unknown content of the target analyte a matrix matched sample with known concentration of the target analyte is included. The batch of samples is analysed under repeatability conditions, i.e. on the same day, by the same technician utilising identical equipment. The concentration of the target analyte in the unknown samples is then corrected by the recovery rate calculated from the analysis of the fortified sample (method 3).

Why correcting for recovery? Main factors in this context that adversely influence the accuracy of the analytical result are related to the bias of the method and of the laboratory. Moreover it is assumed that the use of recovery information may reduce the impact of both types of bias. It is therefore expected that correcting for recovery improves the agreement between the result of analysis and the target (or true?) concentration.

This document summarises the results from statistical calculations conducted on the results from various interlaboratory studies. In particular, the calculated average
concentrations and the precision under reproducibility conditions were compared, depending on whether the participating laboratories have corrected for recovery rate or not. The results are partly based on a former publication dealing with the same topic (see reference list).

2. Approach for the comparison

Since method 1 is well established, it has not been further considered in this document. Its applicability only depends on the availability of appropriate isotope labelled or surrogate standards.

The following studies are included in this investigation.

**JRC validation study**
- Target analyte: Aflatoxin B1 in six different matrices
- The participants were asked to analyse unknown samples along with blank samples fortified in the participants’ laboratory using the standard compound delivered by the coordinator

**CSL validation study**
- Target analyte: Zearalenone and Deoxynivalenol
- The participants were asked to analyse unknown samples along with blank samples fortified in the participants’ laboratory. However, the origin of standards have not been specified

**European Commission’s Pesticide proficiency study PT 4**
- Target analytes: 14 out of a list of 41 pesticides
- Laboratories reported the results along with information on recovery
- No information about how and when the recovery rates were determined. It is assumed that same of the laboratory used the recovery information obtained in the validation exercise of the method.

3. Results

*Impact on mean value:* In all three studies the fast majority of the analyte/matrix combinations yielded higher values for the mean concentrations when the laboratories applied correction for recovery.

*Impact on precision:* Contrary to the impact on the mean value, the influence of the recovery rate was different between the interlaboratory studies

- JRC validation study: In almost all cases the reproducibility precision was lower when the results was corrected for recovery
- CSL validation study: Only about half of the studies (5/11) showed lower precision values, when the results were corrected for recovery. In the other cases, correction for recovery led to an increase of the variation of the analytical results.

- European Commission’s Pesticide proficiency study PT 4: In all cases correcting for recovery reduced the variation of the results, but the effect in terms of absolute reduction of the observed standard deviation was minor.

Looking more carefully at the results from the JRC validation study with the highest positive influence of the recovery correction on the precision of the results revealed that the correlation between the analytical results of the unknown and the spiked samples plays an important role. This aspect is supported by applying the law of error propagation.

4. Conclusions

The accuracy of analytical results can be improved when using recovery information. However, this depends strongly on the respective experimental design, how the recovery has been determined.

The results of the study clearly shows that the discussion on the use of recovery information needs always to take into account the circumstances under which the recovery data have been obtained. Future studies should therefore focus on these aspects. For example, the above mentioned correlation requires further evaluation, as well as other reasons that might positively influence the correction of recovery on the accuracy of the analytical results.

5. Reference

