



JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
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**DISCUSSION PAPER ON THE UPDATE REFERENCES OF METHODS OF ANALYSIS
AND RELATED TEXTS**

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BACKGROUND

In the 26th CCMAS Session, in 2005, the reference to the year of publication in methods of analysis was discussed, considering the ISO/IEC 17025, referred under CAC/GL 27, which requires that the most updated versions of analysis methods be used. However, the year of publication mentioned in methods endorsed by the CCMAS was often outdated and reference was made to methods that no longer exist. The Committee noted that the update of the year of publication reflected only editorial changes, and that when the method was substantially amended the reference number was revised. The proposal to delete the year of publication was not approved, but the Committee agreed to include a note for clarification purposes in the list of methods (CODEX STAN 234-1999) to the effect that the most updated version of the method should be used. The Observer from AOCS expressed the view that the inclusion of a note would only be a temporary measure and that only the deletion of the year of publication would entirely address the problem.

In 2009, in its 30th session the Committee considered a request to regularly update the references in Codex methods of analysis, as the methods developed by international organizations were updated and this should be reflected in Codex methods. The Delegation of AOCS, on behalf of IAM, offered that the members of IAM would provide their updated references to the relevant Codex Committees. It was noted that active Codex commodity committees regularly considered the update of the methods in the commodities under their responsibility, but that references might become outdated, when committees were adjourned or standards were not reviewed for a long time. Once more it was proposed the reference to the year in the method be deleted in order to solve this problem, but the Committee recalled that the matter had been discussed in the past and that this had not been agreed upon in view of the need to retain the reference year for regulatory purposes. The Committee recalled that under ISO/IEC 17025, referred under CAC/GL 27, analysts were required to use the most updated version of methods of analysis and that it had been agreed upon to insert a note to CODEX STAN 234 to this effect. The Committee concluded that the IAM member organizations could provide information for update as regards methods of analysis and that for the update of other texts such as the Guidelines, the need for update and revision needed to be raised by a member.

During the thirty third session of the Codex Committee on Methods of Analysis and Sampling it was noted that e.g. the IUPAC methods or other methods requiring updating were still listed in standards developed by adjourned committees. The Delegation of Brazil indicated that several adopted methods were no longer in use or otherwise required an update and that the reference to documents developed by other organizations in some Guidelines should also be revised, and proposed this issue be considered at the next session. The Secretariat recalled that while updates of methods were the responsibility of the relevant committee when active, CCMAS could review the methods when the committees were adjourned. It was also noted that the standard setting organizations provided their updates to the Committee for consideration under the item on endorsement. The Committee agreed that Brazil would prepare a discussion paper on the update of references to methods of analysis and other texts for consideration at the next session.

In many occasions it was observed that there is a concern about keeping references updated. This paper aims to describe some issues that may be of help in the Committee debate. The delegation of Switzerland assisted in preparing this document.

REFERENCES EVALUATION

Tables I to VII show several examples of methods that need to be revised or amended.

Table I – Methods of Analyses not found or withdrawn

| COMMODITY | PROVISION | METHOD | PRINCIPLE | TYPE |
|--|---|--|--|------|
| Peanuts (Cereals, shell-fruits and derived products (including peanuts)) | Sum of aflatoxins B1, B2, G1 and G2 | EN 12955 : 1999-07 | HPLC with post column derivatization and immuno affinity column clean up | III |
| Soy protein products | Fat | - CAC/RM 55-1976 - Method 1 | Gravimetry (extraction) | I |
| Named Vegetable Oils | Relative density | IUPAC 2.101 with the appropriate conversion factor | Pycnometry | I |
| Margarine | Vitamin E | - IUPAC 2.411 | TLC followed by spectrophotometry or GLC | II |
| Margarine | Water | - CAC/RM 17-1969 (described in the Standard) | Gravimetry | I |
| Milk powders and cream powders | Acidity, titratable | ISO 6091:1980 | Titrimetry, titration to pH 8.4 | I |
| Milkfat products | Milkfat | IDF Standard 24:1964 | Gravimetry (calculation from solids-not-fat and water content) | IV |
| Milkfat products | Vegetable fat(sterols) | IDF Standard 54:1979 ISO 3594:1976 | Gas liquid chromatography | II |
| Fish Sauce (CODEX STAN 302 – 2011) | Determination of amino acid nitrogen by determining formaldehyde nitrogen | (AOAC 2.066) and subtracting by ammoniacal nitrogen (AOAC 2.065) | | |
| Fish Sauce (CODEX STAN 302 – 2011)SAUCE | Determination of sodium chloride | FAO 1981 | | |
| Honey | Sugars added (for sugar profile) | AOAC 998.18 | Carbon isotope ratio mass spectrometry | I |

Table II – Methods of Analyses mentioned incorrectly

| COMMODITY | PROVISION | METHOD | PRINCIPLE | TYPE | CORRECT METHOD |
|--|-----------------|---------------------------------|--------------------------------------|------|----------------|
| Processed meat and poultry products | Tin | AOAC 985 (Codex general method) | Atomic absorption spectrophotometry) | II | AOAC 985.16 |
| Bouillons and Consommés (soups and broths) | Nitrogen, total | AOAC 928:08 | Kjeldahl | II | AOAC 928.08 |

Table III– Methods of Analyses for other analyte or with wrong description or type

| COMMODITY | PROVISION | METHOD | PRINCIPLE | TYPE | REMARKS |
|---|--|---|---|------|---|
| Fats and Oils (all) | Arsenic | - AOAC 985.16 (Codex general method) | Atomic absorption spectrophotometry | III | Method for “Elemental Analysis/Tin |
| Cocoa Products and Chocolate | Cocoa butter | AOAC 963.15 IOCCC 14-1972 | Gravimetry (Soxhlet extraction) | I | The description of the analyte should be just fat |
| Fruit juices and Nectars | Glucose and fructose – Determination of glucose, fructose and saccharose | EN 12630 IFU Method No. 67 (1996) NMKL 148 (1993) | High performance liquid chromatography (HPLC) | II | CODEX STAN 247-2005 is as Type II and in CODEX STAN 234 as Type III |
| Sugar (dextrose anhydrous and dextrose monohydrate) | D-Glucose | ISO 5377:1981 | Titrimetry | I | The method measures ‘reducing power’ and Dextrose Equivalent (DE) |
| Follow-up formula | Dietary fibre, total | AOAC 991.43 | Gravimetry (enzimatic digestion) | | A lot of follow-up-formula may contain GOS/FOS which (in some countries) will be defined as fibre and will not be measured using AOAC 991.43. |

Table IV – Methods of Analyses outdated

| COMMODITY | PROVISION | METHOD | PRINCIPLE | TYPE | UPDATE METHOD VERSION |
|--|---|---|--|------|--|
| Degermed maize (corn); meal and maize (corn) grits | Moisture | - ISO 712:1998 | Gravimetry | I | ISO 712:2009 |
| Fruit Juices and Nectars | Benzoic acid and its salts; sorbic acid and its salts | IFU Method No. 63 (1995) NMKL 124 (1997) | High performance liquid chromatography (HPLC) | II | IFU No 63 (Revised in 2005) NMKL (2007) |
| Cocoa butter | Unsaponifiable matter | AOCS Ca 6b-53 (01) | Titrimetry after extraction with diethyl ether | I | AOCS Ca 6b-53 de 2011 |
| Fats and oils | Vitamin D | AOAC 936.14 | Bioassay | II | This method is based on a rat bioassay originating from 1936. More appropriate methods are currently available and validated for margarine, e.g. EN 12821 (European Standard): Determination of vitamin D by HPLC. |

Table V – Methods of Analyses Replaced

| COMMODITY | PROVISION | METHOD | PRINCIPLE | TYPE | REMARKS |
|----------------------|--------------|--------------------------|------------|------|--|
| Named Vegetable Oils | Soap content | - BS 684 Section 2.5; or | Gravimetry | I | Replaced by BS EN ISO 10539:2002 |
| Fermented milks | Milk fat | AOAC 905.02 | Gravimetry | I | Replaced by AOAC 989.05 |

Table VI – Description of Methods of Analyses are not unique

| COMMODITY | PROVISION | METHOD | PRINCIPLE | TYPE | REMARKS |
|------------------------|-------------------|----------------------|----------------------|------|--|
| Edible casein products | Casein in protein | IDF Standard 29:1964 | Titrimetry, Kjeldahl | I | ISO 17997-2 IDF 029-2:2004 - Milk - Determination of the casein-nitrogen content - Part 2: Direct method ISO 17997-1 IDF 029-1:2004 - Milk - Determination of the casein-nitrogen content - Part 1: Indirect method (Reference method) IDF 092:1979 is to determine protein content. IDF 29 (1964) was not found |

Table VII –Methods of Analyses are mentioned only in Commodities Standards

| COMMODITY | PROVISION | METHOD | PRINCIPLE | TYPE | REMARKS |
|--------------------------|---|--|---|------|--|
| Honey | Determination of Sugars added to honey (Authenticity) | AOAC 977.20 for sugar profile, AOAC 991.41 internal standard for SCIRA (stable carbon isotope ratio analysis). | Liquid Chromatography Carbon isotope ratio mass spectrometry | I | CODEX STAN 234 mentioned AOAC 998.18 (doesn't exist) |
| Honey | Determination of Diastase Activity | AOAC 958.09 | Spectrophotometry | | This method is mentioned only in a CODEX STAN 12-1981 |
| Honey | Determination of hydroxymethylfurfural (HMF) content | AOAC 980.23 | Spectrophotometry | I | This method is mentioned only in a CODEX STAN 12-1981 |
| Fruit juices and Nectars | Isocitric acid-D | EN 1139 (1999) IFU Method No. 54 (1984) | Enzymatic determination | II | This method is mentioned only in a CODEX STAN 247-2005 |

Additionally, there is a decision to update the reference documents mentioned in CAC/GL 28-1995 in the report of the 28th Session of CCMAS (paragraphs 16 and 17), but the Guidelines still mention the former references.

Below, other examples of references that are outdated in the Codex Documents:

- CAC/GL 27-1997 (Guidelines for the assessment of the competence of testing laboratories involved in the import and export control of foods) makes reference to ISO Guide 58 instead of ISO/IEC 17011, the updated one.
- In some paragraphs of CAC/GL 54-2004 (Guidelines on Measurement Uncertainty) there are references to 17025:1999 and in others to 17025:2005. The same document makes reference to the outdated norms VIM 1993, ISO GUM 1993, EURACHEM/CITAC Guide Quantifying Uncertainty In Analytical Measurement (Second Edition) 2000 instead 2008, 2008, 2012 editions, respectively, that are the current ones.
- The CAC GL 72 2009 (Guideline on Analytical Terminology) has as reference VIM 2007 and ISO Guide 30 1992 instead 2012 and 2008 editions respectively.
- Procedure Manual also mentions the outdated version of 17025:1999 and ISO 8402 that was withdraw.

Another very important issue to consider is the availability of the methods for all interested parties in a clear way. There are reference to CAC/Vol IX- Ed.1- Part III, CAC Volume 13, and CAC/RM documents in many CODEX STANDARDS , for example, that cannot be found on line.

METHODS OF ANALYSES IN CODEX STANDARDS

It was observed that depending on the Codex Committee the way of make a reference of methods of analyses is different as showed below:

- a) **Complete description** as the determination of drained weight in the Codex Standard for Canned Finfish (Codex Stan 119-1981)
- b) **Mention the Method of Reference** as the Determination of Arsenic in the Codex Stan 210-1999 (Standard for named vegetable oils) that is according to AOAC 952.13; AOAC 942.17; or AOAC 986.15.
- c) **Table with the provision, method, principle and type** as in the Codex Stan 241- 2003 (Canned Bamboo Shoots)
- d) **Advice to see relevant Codex texts on methods of analysis and sampling** as the Codex Stan 240-2003 (Coconut Milk and Coconut Cream)
- e) **Advice to see Codex Stan 234-1999** as the Codex Stan 280-1973 (milkfat products)

The information provided in the Codex documents should be presented uniformly, as these differences may lead to some confusion. For example, the footnote in Codex Stan 234-1999 that states the most updated version of the method should be used, in application of ISO/IEC 17025 merely for commodities mentioned in this standard. How to apply the CAC/GL 27, which requires the use of the most updated versions of methods of analysis for commodities not covered by the Codex Stan 234-1999?

Additionally, the latest version of the method of analysis recommended must be used for accreditation by the 17025, unless there is a justification for not using it. Generally, new versions bring improvements, while the mention of an old version in a Codex Standard requires a laboratory to use an outdated norm.

UPDATING METHODS OF ANALYSES AND THEIR TYPES

According to the Procedure Manual, the *Codex Alimentarius Commission* and its subsidiary bodies are committed to revision as necessary of Codex standards and related texts to ensure that they are consistent with and reflect current scientific knowledge and other relevant information. When required, a standard or related text shall be revised or removed in accordance with the Procedures for the Elaboration of Codex Standards and Related Texts. Each member of the *Codex Alimentarius Commission* is responsible for identifying and presenting to the appropriate committee any new scientific and other relevant information which may warrant revision of any existing Codex standards or related texts.

The Commission decides that a standard should be elaborated and also which subsidiary body or other body should undertake the work. Decisions to elaborate standards may also be taken by subsidiary bodies of the Commission and subsequently be approved by the Commission

CCMAS is responsible for carrying out the steps of the elaboration procedures of the methods of analysis and sampling of general application to foods.

Methods of analysis to specific commodities were originally assigned by commodity committees or ad hoc Intergovernmental Task Forces. However, six of these committees/task forces are still active while eleven were adjourned, abolished or dissolved. See Table below:

| Commodity Committee/ad hoc Task Force | Status |
|--|-----------|
| Cereals, Pulses and Legumes and derived products | Adjourned |
| Cocoa products and chocolate | Adjourned |
| Fats and oils and related products | Active |
| Fish and fishery products | Active |
| Foods for special dietary uses | Active |
| Fresh Fruit and vegetables | Active |
| Edible Ices | Abolished |
| Meat | Abolished |
| Fruit and vegetable juices | Dissolved |
| Milk and milk products | Adjourned |
| Natural mineral waters | Adjourned |
| Processed fruit and vegetable | Active |
| Processed meat and poultry products | Abolished |
| Quick frozen foods | Abolished |
| Soups and broths | Abolished |
| Sugars | Active |
| Vegetable Proteins | Adjourned |

Source: *Codex Alimentarius* website

According to the Procedure Manual, updating of references consequential to the adoption, amendment or revision of Codex standards and other texts of general applicability (including the provisions in the Procedural Manual), updating of methods of analysis and sampling as well as alignment of provisions, for consistency, to those in similar standards or related texts adopted by the Commission may be handled by the Commission in the same manner as amendments of an editorial nature.

Proposals for the amendment or revision of Codex standards and related texts should be submitted to the Commission by the subsidiary body concerned, by the Secretariat, or by a member of the Commission where the subsidiary body concerned is not in existence or has been adjourned *sine die*. If the amendment proposed is of an editorial nature, the preparation of a project document is not required.

The Commission decides whether the amendment or revision of a standard is necessary. In the case of an amendment of an editorial nature, it will be open to the Commission to adopt the amendment at Step 8. In the case of an amendment proposed and agreed upon by a subsidiary body, it will also be open to the Commission to adopt the amendment at Step 5 of the Uniform Procedure. In other cases, the Commission will approve the proposal as new work and the approved new work will be referred for consideration to the appropriate subsidiary body, if such body is still in existence. If such body is not in existence, the Commission will determine how best to deal with the new work.

In the case of methods of analysis, when the commodities committees are not active it is justified that the CCMAS be responsible for the task, although in the Procedural Manual it says that where Codex subsidiary bodies have been abolished or dissolved, or Codex committees have been adjourned *sine die*, the Secretariat keeps under review all Codex standards and related texts elaborated by these bodies and determines the need for any amendments, in particular those arising from decisions of the Commission. If the need for amendments of an editorial nature is identified then the Secretariat should prepare proposed amendments for consideration and adoption by the Commission. If the need for amendments of a substantive nature is identified, the Secretariat, in cooperation with the national secretariat of the adjourned Committee if applicable, should prepare a working paper containing the reasons for proposing amendments and the wording of such amendments as appropriate, and request comments from members of the Commission.

There is a concern in other organizations to keep their standards updated. For example, all ISO standards are reviewed at least every 5 years. . A majority of the Participating-members of the ISO Technical Committees (TC) or Subcommittee (SC) decides whether an International Standard should be confirmed, revised or withdrawn (www.iso.org).

CONCLUSIONS

The CAC/GL 27 recommends the application of ISO/IEC 17025 that require the use of the most updated version of methods of analysis.

The footnote in Codex Stan 234-1999 that states the most updated version of the method should be used, in application of ISO/IEC 17025 it does not solve the problem of the use of the outdated version for those methods out of this standard.

The year of publication reflects only editorial changes and when the method is substantially amended the reference number is changed. For this reason, it is not necessary mention the year of publication of methods of analysis, unless the use of a specific revision be justified.

The current way to refer to methods of analyses in many documents and different forms makes it difficult to keep references updated.

The mechanisms for updating methods of analyses and other CCMAS documents have not been sufficient to keep them in adequate status and the recommend methods may be increasingly disconnected from the reality of analytics. This situation may affect the credibility of Codex's recommendations.

RECOMENDATIONS

1. Consider the possibility to amend Codex Standards, Procedural Manual and other relevant documents of CCMAS to mention the number of the norm alone.
2. When possible, it is advisable to use the criteria approach, rather than referring to specific methods.
3. Harmonize the way of mentioning methods of analysis in Codex Standards.
4. The Commodities Codex Standards could just make reference to a general document with all the methods of analyses. This approach allows permanent and dynamic revision.
5. As a last recommendation, the suggestion is that before each CCMAS, the Codex secretariat would issue a list of all methods from standard 234 whose endorsement date is older than 5 years for consideration by the method endorsement session¹. For each method in this list, CCMAS has to decide between confirmation or revision. Revision apply to method that CCMAS considers as no longer adequate, because e.g. insufficiently sensitive to protect consumer's health adequately.
 - 5.1. Cases where a Commodity Committee is still active: the CCMAs will either propose to the Commodity Committee an appropriate new method or motivate the commodity Committee to make a proposal
 - 5.2. Cases where the Commodity Committee was adjourned: CCMAS will perform the update under his own responsibility.

¹ This would require a minor editorial change in the Codex Stan 234 in the form of one column "endorsement date"