



Food and Agriculture
Organization of the
United Nations



World Health
Organization

Viale delle Terme di Caracalla, 00153 Rome, Italy - Tel: (+39) 06 57051 - E-mail: codex@fao.org - www.codexalimentarius.org

Agenda Item 8

CX/MAS 20/41/10
February 2020

JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

41st Session
Budapest, Hungary, 11 - 15 May 2020

DISCUSSION PAPER ON CRITERIA TO SELECT TYPE II METHODS FROM MULTIPLE TYPE III METHODS

Prepared by Switzerland

Executive Summary

1. With regard to the inclusion of Codex analytical methods in CXS 234-1999, clarification of the criteria for selecting the appropriate Type II (reference method) from several Type III methods (alternative approved methods) is necessary.
2. At CCMAS40, Switzerland has agreed to prepare a discussion paper on the criteria for the selection of Type II methods from several Type III methods ([REP19/MAS, paragraph 38](#)) for the next meeting of the Committee.
3. In this discussion paper, Switzerland proposes several criteria to facilitate the selection of a Type II method when multiple Type III methods exist.

Recommendation

4. The Committee is invited to consider the proposed criteria presented in Appendix I for inclusion in the information document: [Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234](#).

Appendix I**Discussion paper on criteria to select Type II methods from multiple Type III methods****Introduction**

It is not uncommon that several analytical methods are proposed for a single commodity – provision combination. However, only one of these can be designated as the reference method (Type II method). The following paragraphs give guidance on the selection of a Type II method from multiple Type III methods.

Codex Methods of Analysis

According to the Procedural Manual, the Codex analytical methods are primarily intended as international methods for the verification of provisions in Codex standards. They should be used for reference, in calibration of methods in use or introduced for routine testing and control purposes.

Purpose of Reference Methods (Type II)

Definition as per the Procedural Manual: A Type II method is the one designated Reference Method where Type I methods do not apply. It should be selected from Type III methods (as defined below). It should be recommended for use in cases of dispute and for calibration purposes.

Purpose of Alternative Approved Methods (Type III)

As per description in the Procedural Manual, a Type III method is one which meets the criteria required by the Committee on Methods of Analysis and Sampling for methods and may be used for control, inspection or regulatory purposes.

In the event of multiple Type III methods for the same provision-commodity combination, it is expected that these methods, although they might use different approaches, should result in equivalent decisions (compliant vs. non-compliant).

Current situation

Currently only general guidance (Procedural manual) is available for the classification of analytical methods as Type II or III. For this reason, we propose to apply the following additional criteria:

Inclusion criteria for Type III chemical or physical Methods

- i. A potential Type III method should fulfil the following criteria, in addition to the general criteria for the selection of methods of analysis (cf. Procedural Manual, p. 76):
 - The method is easily accessible, e.g. from SDO websites
 - The method is validated according to an internationally recognised protocol and the validation data published
- ii. All methods should measure the same analyte (chemical entity).
- iii. The validation covers the analytical range for the provision (e.g. MRL).
- iv. The methods are preferably validated on the same matrices.
- v. If the methods contain differing analysis steps (e.g. Vitamin B6 with or without enzymatic digestion), verify that these methods still measure the same provision.
- vi. Check results of proficiency testing in order to detect systematic differences between methods (e.g. NIST <https://nvlpubs.nist.gov/nistpubs/ir/2019/NIST.IR.8266.pdf>).

Decision criteria for choosing the best method (=Type II) among multiple Type III methods

- i. The method explicitly validated for the commodity stated should be preferred: e.g. if a method for copper in infant formula is required, a method specifically validated for this commodity should be preferred to a method validated for milk powder.
- ii. The method validated for the larger panel of matrices should be preferred.
- iii. The method where a certified reference material, preferably from a matrix similar to that used in the scope of the method, was included in the validation should be preferred.
- iv. The method with the better specificity should be preferred.
- v. The method with the better precision data (if this precision difference is relevant to the question asked) should be preferred.

Additional considerations for selection Type II when several Type III methods fulfil all above criteria:

- Methods with less safety concerns (i.e. not using toxic solvents or reagents) should be preferred.
- Methods with lowest ethical concerns should be preferred (i.e. which do not use animal testing).

Methods with lowest economic costs should be preferred.

To test the proposed selection criteria, the following commodity-provision combinations with multiple Type III methods included in CXS-234 were used:

- Copper in milkfat products (1 Type II, 2 Type III and 1 Type IV methods)
- Sodium and Potassium in infant formula (1 Type II and 3 Type III methods)

1 **Table 1: Selection criteria for Type III / II methods**

				Type III										Type II				
											additional considerations for selection Type II among multiple Type III methods							
				method easily accessible	validated according to international recognized protocol	measure same analytes	validation covers analytical range provision	validated on same matrices	different analytical steps	Proficiency test results available	safety concerns	lowest ethical concerns	lowest economic costs	validated for commodity	validated for larger panel matrices	certified reference material included pref. Similar matrix scope	better selectivity /specificity	better precision data
sodium/potassium infant formula	AOAC 2015.06	ICPMS	Type II	x	x	x	x	yes		?				x	x	x		x
	AOAC 2011.14	ICPOES	Type III	x	x	x	x	yes		?				x	x	x		
	ISO 8070 IDF 119	FAAS	Type III Was Type II	x	x	x	x	no	option dry ashing	?				no, milk products only	x	yes, milk powder		
	AOAC 986.24	ICPOES	Was Type III	x	x	x	?	no	no internal standardization	?	x					?		
Copper in milkfat products	AOAC 2015.06	ICPMS	Type II	x	x	x	x	yes		?				yes, butter	x	yes, infant formula	x	
	ISO 5738 IDF 76	photometry	Type III	x	x	x	x	no		?				yes, butter, butterfat	x	no		x
	AOAC 2011.14	ICPOES	candidate Type III	x	x	x	no	yes	no	?				yes, butter	x	yes, infant formula		
	AOAC 960.40	Photometry	Type IV	x	?	?	NA	NA						? scope not specified for butter or milk fat		?		

Considerations selection Type II method Sodium/Potassium in infant formula:

- AOAC 986.24 cannot be considered as Type II because of difference in analytical steps as compared to other Type III methods, which may have implications on the results. In addition, this method has 'Safety concerns' (Perchloric acid destruction). Method is rightfully revoked by the SDO and CXS-234.
- ISO 8070 | IDF 119, has an option to use dry ashing as a sample preparation, which is not appropriate for the determination of sodium. In addition, the method is not validated for Infant Formula. In conclusion, this method has several drawbacks as compared to the other 2 candidate Type II methods: AOAC 2011.14 and AOAC 2015.06.
- Comparing AOAC 2015.06 and AOAC 2011.14, which are both validated on the same samples, AOAC 2015.06 has better precision data and therefore should be preferred as Type II method.

Considerations selection Type II method Copper in milkfat products:

- The validation of AOAC 2011.14 does not cover the range of the provision and consequently cannot be considered as Type III method.
- Although ISO 5738 | IDF 76 based on photometry seems to have better precision data, AOAC 2015.06 based on ICP-MS has a better selectivity/specificity and therefore should be preferred as Type II.

Conclusion

The proposed criteria are suitable for the selection of the appropriate Type II method when multiple Type III methods exist, and may therefore support CCMAS in the process of consideration and endorsement of methods for inclusion in CXS234