



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

42nd Session
Budapest, Hungary
13 – 16 June 2023 with report adoption on 20 June 2023 (virtual)

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

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Background

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 the 40th Session of the Committee on Methods of Analysis and Sampling (CCMAS40) (2019), Switzerland 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. The initial discussion paper was published in February 2020 (CX/MAS 20/41/10), in which Switzerland proposed several rules to facilitate the selection of a Type II method when multiple Type III methods exist.
4. Due to the Covid-19 pandemic, and making use of the additional time at the disposal of the Committee, comments were requested through a CL (CL 2020/31/OCS-MAS) to aid the further development of the discussion paper.
5. At CCMAS41, which was held virtually on 17 – 21 and 25 May 2021, the revised rules considering comments in response to CL 2020/31/OCS-MAS were discussed ([REP21/MAS, paragraph 119](#)). It was agreed to circulate the proposed rules for selection of Type II methods from multiple Type III methods again for comments, further revision by Switzerland and consideration at CCMAS42.
6. In March 2022 comments were requested through a CL (CL 2022/23/OCS-MAS). Comments were received from Brazil, Canada, Colombia, Egypt, European Union, Kenya, Mexico, Peru, Philippines, Singapore and Thailand and available [here](#).
7. The revised rules (Appendix I) consider these comments.
8. **Summary of main changes** Prerequisite ii, additional consideration iii of the prerequisites and decision rules i and ii: reworded or wording added for clarification.

Validation of the rules

9. To test the proposed selection rules, specific commodity-provision combinations with multiple Type III methods included in CXS 234 were used (see Appendix II).
10. The rules were also tested by the EWG on the review of fats and oils package and their analysis is presented in [CX/MAS 21/41/5](#) and in Appendix III to this document.

Conclusion

11. From the examples of Sodium and Potassium in infant formula and Copper in milkfat products (Appendix II) and the analysis of the EWG on the review of fats and oils package (Appendix III), the proposed rules 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.

Recommendation

12. The Committee is invited to agree on the revised rules 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.](#)

Rules to select Type II methods from multiple Type III methods

(changes are in **bold/underlined** or ~~striketrough~~ mode)

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 (as per the Procedural Manual) is available for the classification of analytical methods as Type II or III. For this reason, we propose to apply the following rules¹:

Prerequisites for inclusion in Codex standards for Type III chemical or physical Methods

- i. The method is validated according to an internationally recognized protocol and the validation data published
- ii. The method should fulfil **the General Criteria for the Selection of Methods of Analysis of the Procedural Manual** ~~the general method performance criteria in the Procedural Manual.~~
- iii. The method is easily accessible, e.g. from SDO websites
- iv. Codex (commodity) committees, country delegations or NGO's submitting methods of analysis to CCMAS for consideration need to provide technical information using the template MAS/40 CRD 28 (cf. CCMAS40 CRD05)
- v. The validation covers the analytical range for the provision (e.g. MRL).

Additional considerations in cases where results from several Type III methods for the same commodity-provision combination are compared and the Criteria Approach is not an option:

- i. All methods should measure the same analyte (specific chemical entity to be determined), especially if the methods contain differing analysis steps or sample preparation (e.g. Vitamin B6 with or without enzymatic digestion). If available, the assumption can be confirmed by an equivalence study.
- ii. The methods are preferably validated on the same matrices. In absence of methods covering the commodity of the provision, a potential suitable method validated on matrices of similar composition (in terms of fat, protein and carbohydrate content) can be considered.
- iii. Check availability of results of **round robins** or proficiency tests² in order to detect systematic differences between methods.

¹ In some situations, CCMAS may decide not to apply these selection rules, e.g. for ethical, economic or safety reasons. This decision must be duly justified.

² e.g. NIST <https://nvlpubs.nist.gov/nistpubs/ir/2019/NIST.IR.8266.pdf>

Decision rules for choosing the best method (=Type II) among multiple Type III methods³

- i. **As the scope of methods of analysis are aligned with various matrices from many groups of commodities (Codex Procedural Manual, General Criteria for the Selection of Methods of Analysis, CXS 234-1999 Recommended Methods of Analysis and Sampling), the method explicitly validated for the commodity stated in the Codex provision 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 **more than one matrix from a specific commodity the larger panel**⁴ of ~~matrices~~ should be preferred. E. g. a method validated for milk-based and soy protein-based infant formulae should be preferred to a method validated only for milk-based infant formula.
- iii. The method with the best selectivity should be preferred.
- iv. The method with the best precision data (if this precision difference is relevant to the question asked) should be preferred.
- v. 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.

³ The decision rules should be considered in the order presented.

⁴ ~~Larger panel means~~ **Different types matrices of one matrix belonging to one commodity**. E.g. infant formula includes milk-based, soy-based, hydrolyzed protein based.

Appendix II**Validation of the proposed decision rules**

To test the proposed selection rules, 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 rules for Type III / II methods**

				Type III						Type II					
				method easily accessible	validated according to international recognized protocol	measures same analytes as every other method being compared	validation covers analytical range provision	validated on same matrices	indicate methods with similar analytical steps	Proficiency test results available	validated for commodity	validated for larger panel matrices	best selectivity	best precision data	certified reference material included pref. Similar matrix scope
sodium/potassium infant formula	AOAC 2015.06	ICPMS	Type II	yes	yes	yes	yes	yes		?	yes	yes		yes	yes
	AOAC 2011.14	ICPOES	Type III	yes	yes	yes	yes	yes		?	yes	yes			yes
	ISO 8070 IDF 119	FAAS	Type III Was Type II	yes	yes	yes	yes	no	option dry ashing	?	no, milk products only	yes			yes, milk powder
	AOAC 986.24	ICPOES	Was Type III	yes	yes	yes	yes	no	no internal standardization	?					?
Copper in milkfat products	AOAC 2015.06	ICPMS	Type II	yes	yes	yes	yes	yes		?	yes, butter	yes	x		yes, infant formula
	ISO 5738 IDF 76	photometry	Type III	yes	yes	yes	yes	no		?	yes, butter, butterfat	yes		yes	no
	AOAC 2011.14	ICPOES	candidate Type III	yes	yes	yes	no	yes		?	yes, butter	yes			yes, infant formula
	AOAC 960.40	Photometry	Type IV	yes	?	?	no	no		?	? 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. (MAS40/CRD05 for precision data)

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. (MAS40/CRD06 for precision data)
- 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 and therefore should be preferred as Type II.

Conclusions

From the examples of Sodium and Potassium in infant formula and Copper in milkfat products, the proposed rules 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.

Appendix III**Considerations by the fats and oils EWG in applying the proposed rules in the selection of a Type II method for fatty acid composition of fish oils**

The fats and oils EWG applied the rules on 6 (combinations of) methods for determining fatty acid composition in fish oil. As summarized in Table 2, the members came to the same conclusion for 5 (combinations of) methods, whereas no consensus was found for AOCS Ce 2-66 and AOCS Ce 1a-13 (the insights by the reviewers is available [here](#)). However, from the comments by the EWG it was understood that AOCS Ce 2-66 and AOCS Ce 1a-13 are not a method but a guidance with no validation data and should be removed from CXS 234 (as suggested by 2 reviewers).

Table 2: fats and oils EWG method typing Method	Type
AOCS Ce 2-66 and AOCS Ce 1a-13	No consensus (III/IV/Removal)
AOCS Ce 1b 89	III
AOCS Ce 2b-11 and AOCS Ce 1i-07	III
AOCS Ce 2b-11 and AOCS Ce 1j-07	III
SO 12966-2 and ISO 12966-4	III
AOCS Ce 2-66 and AOCS Ce 1i-07	II