



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

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**LISTING OF TYPE IV METHODS IN CXS 234 WHEN A TYPE I METHOD IS LISTED FOR THE SAME
COMMODITY AND PROVISION**

(Prepared by the EWG led by Uruguay and Brazil)

BACKGROUND

1. During the 42th Session of CCMAS (CCMAS42), the Committee recalled that at that session, an approach was taken to have both a Type I and Type IV method when there was a justifiable and motivating reason. Using such an approach would require changes to the information document: *Comprehensive guidance for the process of submission, consideration, and endorsement of methods for inclusion in CXS 234*, to describe this situation. CCMAS agreed to establish an electronic working group (EWG) to develop a discussion paper to address this issue.
2. The electronic working group held discussions from October 2023 to January 2024. Participants were: Argentina, Australia, Brazil, Canada, China, Costa Rica, Ghana, Hungary, India, Indonesia, New Zealand, Nigeria, Korea, Panama, Saudi Arabia, Singapore, Switzerland, Thailand, Uganda, United Kingdom, Uruguay AOAC, IDF/FIL, International Fruit and Vegetable Juice Association (IFU).
3. This discussion paper summarizes the work carried out by the EWG and aims to give recommendations and questions to be used as a basis for discussion by CCMAS43 (Appendix I).

RECOMMENDATIONS

4. CCMAS is invited to consider the discussion paper (Appendix 1) and in particular questions, conclusions and recommendations provided in paras 40 -45 of the discussion paper.

APPENDIX I

DISCUSSION PAPER ON THE LISTING OF TYPE IV METHODS IN CXS234 WHEN A TYPE I METHOD IS LISTED FOR THE SAME COMMODITY AND PROVISION**1. INTRODUCTION****1.1 *Type I and type IV Method definitions***

1. The Codex Procedural Manual defines a Type I method as “a method which determines a value that can only be arrived at in terms of the method per se and serves by definition as the only method (Codex Procedure Manual).
2. The Procedural Manual defines a Type IV method as “A method which has been used traditionally or else has been recently introduced but for which the criteria required for acceptance by the Committee on Methods of Analysis and Sampling have not yet been determined”.
3. During CCMAS42 it was noted that there was no hard rule to not endorse a Type IV method when there was a Type I and that the performance data had been submitted for review and indicated that the method was fit for purpose ([REP23/MAS, para. 42](#)).

1.2 *Type I methods relevant discussions*

4. In relation to Type I methods there has been numerous relevant CCMAS discussions throughout the years:

Type I equivalency

5. During CCMAS35, the Committee agreed not to develop numerical criteria for Type I methods, however procedure for establishing equivalence to Type I should be considered. The Committee agreed to pursue the work further through the establishment of an electronic working group to prepare a discussion paper which would consider different approaches for different classes of Type I methods ([REP14/MAS](#)).
6. During CCMAS36, CCMAS agreed to continue consideration of the development of procedures/guidelines for determining equivalency to Type I methods, but with caution, since such criteria could have many unintended implications. It was noted that it would be important to clearly define the concept of equivalent methods and whether equivalency would apply between Type I methods, or other methods with Type I methods and that the development of criteria should not disrupt the current concept of Type I methods. Related comments (CX/MAS 15/36/5) explain that there is little guidance from regulatory agencies or scientific associations such as AOAC or ISO on the exact procedures for establishing the equivalence of analytical methods; although in recent years, the pharmaceutical industry has been required to establish procedures for evaluating method equivalence based on regulations for the bioequivalence of orally administered drug products. The discussion paper on development of procedures/guidelines for determining equivalency to Type I methods ([CX/MAS 15/36/5](#)) contains statistical approaches and raises several questions for discussions. CCMAS took into account that although this paper described a number of statistical approaches for establishing equivalency between methods it clearly did not address specific details in implementing any of these methods. And if a general approach could be recommended, there were still outstanding questions as to the application of that approach to specific methods. For instance:
 - i. Based on the discussion and the range of methods is it practical to establish one set of equivalence criteria for all Codex Methods?
 - ii. If such criteria or even general procedures for evaluating equivalence were established where would they reside in Codex, as part of the Procedural Manual or in a Guidance document?
7. Three options were identified for the purpose of determining method equivalency:
 - i. the two-sample t-test;

- ii. the limit of agreement method; and
 - iii. the Two one-sided t-test or TOST method.
8. It was noted that it would be important to clearly define the concept of equivalent methods and whether equivalency would apply between Type I methods, or other methods with Type I methods and that the development of criteria should not disrupt the current concept of Type I methods. Problems could arise in international trade in a dispute situation, especially if methods were found to be equivalent to Type I methods, which would require a decision to be made as to which method was the defining method. In this Session, views were expressed that the approach could provide an opportunity to replace old and outdated, and difficult to replace Type I methods; that it should not change the current typing system or current levels in commodity standards; that while equivalent methods might help the analytical community in the light of technological advances, it had to be clear that the equivalent methods would be used only for routine control purposes and that in dispute situations, the Type I method should be preferred.
9. Opinions were also expressed that currently once a method had been classified Type I, it was difficult to be replaced. By showing equivalence or superiority, this would allow a Type I method to be replaced. This was especially important in the light of technological advances. The Committee noted that there were several other statistical approaches that could be considered, such as the NMKL NordVal Protocol No.2, as well as other national protocols.
10. During CCMAS37, the Committee continued developing the equivalence procedure for Type I methods. Comments pointed out that while the procedure was intended for determining a statistical approach for establishing equivalence to existing Type I methods, the recommended procedures could be applicable to establishing equivalence between any two methods, regardless of type (Type I – IV), but that before the procedures could be further developed the Committee should provide guidance to the questions raised in CX/MAS 16/37/4 which included the following:
 - i. For methods measuring a composition or characteristic (e.g. moisture content) it would be required that the two methods be equivalent across the entire range of the method. However, for provisions where a maximum limit is established would it be acceptable to establish equivalency around that limit, but not worry about equivalency at some value well above the limit?
 - ii. This paper has focused primarily on quantitative methods, but procedures for qualitative may also be useful. Such procedures would have a very different format/approach, so would they be included in a single document or would separate documents be developed for quantitative and qualitative methods?
11. During the review of the workable package the Committee had raised the need to ‘Clarify rules for determining when a defining method should be Type I or Type IV method’ (MAS/39 CRD/2).
12. There were general agreements in some topics as follow:
 - i. that precision figures for Type I methods are an important aspect of assessing the performance of the methods. Moving forward any newly developed/proposed Type I should present precision figures as part of the data reviewed during the endorsement process. However, there was also agreement that while having such data for long standing methods would be beneficial, lack of such data would not cause a change in the method type or revocation of a method.
 - ii. not to have a general rule to extend the typing of the method if a defining method has been subjected to an international collaborative study involving commodities A, B and C, and the method is generally known to work on commodity D, but this commodity was not included in the study.
13. The Committee could not reach consensus on the use and scope of the equivalency approach and agreed to reconsider this matter in the future when more information became available.

14. During CCMAS39 the Committee agreed the definition / interpretation of “identical methods” and thus there was no need to define “technically equivalent methods” in the Standard CXS234.

Recent work

15. There were recent instances where co-existing Type I and IV have been endorsed by CCMAS and this has mainly been in relation to recent Workable package reviews, where co-existing Type I and Type IV has been used or proposed to resolve specific situations of multiple Type I methods for the same commodity/provision combination. Examples are explained below and includes review of methods of:

Review of Methods for Herbs and Spice

- Review of Methods for Dried Oregano
- Review of Methods for milk and milk products
- Review of Methods for Named Animal Fats

16. During CCMAS42 (REP23/MAS), the Committee agreed to establish an EWG to develop a discussion paper to:

- identify in CXS 234 all commodities and provisions where there are both Type I and Type IV methods listed;
- assess the reasons for having set both Type I and Type IV methods identified.
- discuss criteria and approaches for when Type I and Type IV methods can co-exist; and
- if necessary, make recommendations for changes to the information document and CXS 234.

17. This document prepared by the EWG aims to respond to these concerns.

2. ELECTRONIC WORKING GROUP FOR THE DISCUSSION PAPER ON LISTING OF TYPE IV METHODS IN CXS 234 WHEN A TYPE I METHOD IS LISTED FOR THE SAME COMMODITY AND PROVISION

18. During the work of the EWG, the identification of commodities for which Type IV methods were identified when a Type I method is listed in CXS 234 for the same commodity and provision was carried out. Considering this criterion, the following methods were identified:

Commodity	Provision	Method	Principle	Type
Laver products	Moisture content	AOAC 925.45	Gravimetry, drying at atmospheric pressure	I
Laver products	Moisture content	AOAC 925.45B	Gravimetry, drying at atmospheric pressure	IV

19. Reviewing the background of the endorsement of these methods, it was found that during CCMAS38 the method was originally endorsed as Type IV (REP17/MAS) and retyped as Type I during the CCMAS41 taking in account moisture content validation data (REP21/MAS).

20. AOAC 925.45 method has part A (vacuum drying), part B (drying at atmospheric pressure), part C (drying on pumice stone), and part D (drying on Quartz sand). Considering that in CXS234 in both cases the principle is described as atmospheric pressure, it is found that AOAC 925.45 and AOAC 925.45B is the same method and this could be a typographical error in the Codex Standard. Currently the same method is found as Type I and IV in CXS234 simultaneously, which should be reviewed.

21. In order to address the task, the EWG Members were invited to provide comments and suggestions in the following issues:

- i. Identify situations where Type I and Type IV methods for the same provision and commodity can co-exist, and their restrictions;

- ii. Identify reasons why the co-existence of Type I and Type IV methods for the same commodity and provision would be necessary;
 - iii. Identify selection criteria for Type IV methods;
 - iv. Identify situations in which it would be possible to include additional Type IV methods those Type I methods already included in the CXS234.
22. The comments of the participants of this electronic working group are summarized below.

2.1 Situations where Type I and Type IV methods for the same provision and commodity can co-exist, and their restrictions

23. There were multiple situations identified where they both type I and type IV methods have been proposed for the same provision and commodity for endorsement/adoption in CCMAS/CAC. Examples are:

Review of Herbs and Spice Methods

24. CCMAS40 did not endorse the methods of analysis submitted by the Committee on Spices and Culinary Herbs (CCSCH) and noted that several methods were submitted for endorsement as Type I methods, even though the methods were not identical ([REP19/MAS](#)).

Review of Methods for Dried Oregano

25. CCMAS41 considered methods for Dried Oregano and endorsed a method for 'Foreign Matter' using 'ISO 927' assigned a 'Type I' with the subsets of 'Whole dead insect' using the same 'ISO 927' assigned as 'Type IV' to allow the other method 'MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General method for spices herbs and botanicals (V 32)' was also endorsed ([REP21/MAS](#)).

Review of Methods for milk and milk products

26. CCMAS42 reviewed of methods of milk and milk products in relation to methods for the determination of moisture and endorsed ISO 5537 | IDF 26 as Type I method and a method described in Annex D as Type IV on an exceptional basis only with a footnote explaining that the method described in CXS 234 is listed as Type IV "due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26" ([REP23/MAS](#)).

Review of Method for Named Animal Fats – Titre and Named Vegetable Oils – Unsaponifiable matter

CCMAS42 endorsed additional methods as Type I and Type IV for the same commodity and provision ([MAS42/CRD02](#)).

27. During endorsement of methods for 'Named Animal Fats' for provision 'Titre' where two methods had historically been named with a "or", the Committee included a footnote "a AOCS Cc 12-59 is the preferred method in certain regions. Due to differences in practical application of AOCS Cc 12-59 compared to ISO 935, it is listed as a Type IV method."

28. CCMAS endorsed ISO 3596 / AOCS Ca 6b-53 as Type I and ISO 18609 as Type IV for Named Vegetable Oils – Unsaponifiable matter .

Additional comments to other committees

Questions send back to CCSCCH

29. CCMAS42 sent back the following questions:
- Draft Standard for spices derived from dried fruits and berries (Part A – allspice, juniper berry and star anise) - There are Type I and Type IV methods listed for the provisions "whole dead insects" and "insect fragments". While listing both a Type I and Type IV is allowed, there should be a compelling reason for the listing. Would it be possible to explain the reasoning for this request?

Co-existence of Type I and Type IV methods for the same commodity and provision already included in CXS 234

- there are two methods for Mammalian excreta in Black, white and green pepper as in CXS 234:1999 (2001),
 - one is a Type IV (USFDA technical bulletin V.39 B) (for pepper whole)
 - one is Type I AOAC 993.27 (for ground pepper).

30. While both methods identify the determination of mammalian excreta in the same commodity, one is used for 'whole' pepper and the other is for 'ground'. Doubts were expressed about whether the two methods are for the same commodity and provision, but for different forms of the pepper, and suggested that this is an example of when the retention of both methods as they are currently written (i.e. a Type I and a Type IV method) should be supported. It appears that this was the logic used in CCMAS38 (May 2017) when both methods were endorsed for adoption.

31. The question is if pepper whole and ground pepper is considered as the same provision.

2.2 Reasons why the co-existence of Type I and Type IV methods for the same commodity and provision would be necessary

32. Electronic group participants identified some reasons for the co-existence of Type I and Type IV methods:

- The difficulty to replace Type I methods especially important in the light of technological advances (REP15/MAS para. 93).
- Restrictions regarding its practicability and applicability for routine use and the preference of methods in certain regions.
- Restrictions regarding the accessibility to equipment and calibration of the method
- Selection criteria for the co-existence of type I and type IV methods

33. EWG participants considered three key points regarding the development of selection criteria for co-existence of Type I and Type IV methods:

Co-existence of Type I and IV methods as an exception

34. The first scenario may be to consider the co-existence of Type I and IV methods as a 'exception' and not a 'rule' to utilize when no other alternative is available and the rationale for a Type I and IV co-existence will be justified in CXS 234 by a footnote, so that as soon as the justification and motivation is no longer applicable, the method(s) can be re-typed and changed accordingly. Such an option requires minimal adjustment to the Procedural Manual and changes could be made to the CCMAS information document, *comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS234*, to describe this situation. This approach was addressed during CCMAS42 where Type IV and Type I methods for the same commodity and provision were endorsed when there is a 'justifiable and motivating reason' and/or if the implementation of the Type I method is challenging in some members due the legislation/climate/economic reasons.

Criteria for the Co-existence of Type I and IV methods

35. Another scenario may be to develop Criteria for Type I and Type IV co-existence, so that they become a mechanism which can be utilised as often as the criteria is satisfied.

36. Some criteria were identified by the EWG to the selection of Type IV methods when a Type I exists, which are as follows:

- Compliance with the general criteria for the selection of methods of analysis.
- Selection based on method performance characteristics.
- When applicable, selection based on results of participation in proficiency tests and reference materials.
- Selection on the basis of their practicability and applicability for routine use (example, frequent application of the method in different countries)

- Preferably methods with which the specification has been established in the product standard.
 - Preferably select methods that avoid or minimize use of toxic reagents.
 - Preferably select methods that avoid or minimize generation of hazardous waste.
 - Preferably select methods that have historically been used and are fit for purpose.
37. Some general questions were identified to be addressed by CCMAS before criteria are developed, that includes:
- The necessity to segregate into 'Type IV Rational methods' and 'Type IV Defining methods' to apply selection criteria for methods and numeric criteria. The Codex Procedural Manual includes a definition unique for both methods (independent if they are rational or defining methods) as follows: A Type IV method is a method which has been used traditionally or else has been recently introduced but for which the criteria required for acceptance by CCMAS have not yet been determined. Example: chlorine by X ray fluorescence, estimation of synthetic colours in foods.
 - A potential statistical approach and the restriction of the statistical procedure and methods scope.

Decision to do not have Co-existence of Type I and IV methods

38. The last proposed scenario may be to not have Type I and Type IV methods for the same commodity and provision. In this case, changes to the Procedural Manual and CCMAS information document should be made, and any nomination for multiple 'defining methods' will have to be denied and commodity committees informed of this restriction. Such a situation could give rise to 'work-arounds' including using a different 'provision' name to allow the two methods to co-exist which could mean both 'provisions' need to be assessed and be conforming for a commodity; or one method for a specific range or application and another method required for another range or application within that commodity, in which case a dispute could arise in situations where the results are close to the extremes of that range or application.

2.3 Situation in which it would be possible to include additional Type IV methods to those Type I methods already included in CXS 234

39. No specific methods were presented to consider as an example of the co-existence of type I and IV methods.

3. QUESTIONS FOR DISCUSSION

40. During the analysis of co-existence of Type IV methods in CXS 234 when a Type I method is listed for the same commodity and provision, multiple questions are raised to for consideration by the Committee:

- i. Analyse whether it is more appropriate, the co-existence of Type I and IV methods as an exception, the development of criteria for the co-existence of Type I and IV methods or decide to not have co-existence of Type I and IV methods.
- ii. Clarify the relationship between the concepts of co-existence and equivalence of Type I and Type IV methods.
- iii. Clarify the concept of equivalent methods and whether equivalency would apply between Type I methods, or other methods.
- iv. If is it more practical to establish one equivalence criteria for all Codex Methods.
- v. If such criteria or even general procedures for evaluating co-existence or equivalence of Type I and Type IV methods were established, where would they reside in Codex, as part of the Procedural Manual or in a Guidance document?
- vi. Discuss what extent the 'Guidelines for establishing numeric values for the criteria' could be applied.

4. CONCLUSIONS

41. During the analysis of criteria to include Type IV methods in CXS 234 when a Type I method is listed for the same commodity and provision there was no consensus in relation to the development of selection criteria for co-existence of Type I and Type IV methods.

42. Some participants suggested selection criteria for the selection of Type IV methods when Type I exist in CXS234.

43. Other participants consider that the number of instances of co-existing Type I and IV can be minimised and only considered on a case-by-case basis when a 'justifiable and motivating reason' is provided.

44. In practice, the co-existence of Type I and Type IV methods currently exists in the international field, for example,

- Empirical normalized methods (Type I methods) used by laboratories in different regions that establish a reference method related with a routine or alternative methods such as the examples included in the Annex.
- Many Reference Materials are available in the international market characterized through proficiency testing results as a consensus of participants that use different methodologies corresponding with provisions of Type I methods.
- Many Proficiency Testing exercises are available in the international market where assigned values are set as a consensus of participants that use different methodologies corresponding with provisions of Type I methods.

5. RECOMMENDATIONS

45. It is recommended that CCMAS consider to:

- continue with the selection of Type IV methods on a case-by-case basis when a 'justifiable and motivating reason' is provided until appropriate selection criteria are developed.
- Re-establish the EWG to develop co-existence or equivalence criteria for Type I and Type IV methods.

Annex: List of Reference and Routine/Alternative Methods

Commodity/Provision	Reference Method	Routine/Alternative Methods
Cheese/Fat	ISO 23319/IDF 250:2022. Cheese and processed cheese products, caseins and caseinates- Determination of fat content- Gravimetric method.	ISO 3433/IDF 222:2008. Cheese- Determination of fat content-Van Gulik method.
		ISO 21543/IDF 201:2020. Milk products-Guidelines for the application of near infrared spectrometry.
Milk powder/Titrable Acidity	ISO 6091/IDF 86:2010. Dried milk- Determination of titrable acidity (Reference method).	ISO 6092:1980. Dried milk Determination of titratable acidity (Routine method)
Milk and milk Products/Lactose	ISO 22662:2007 IDF 198:2007. Milk and milk products- Determination of lactose content by high-performance liquid chromatography (Reference method)	ISO 26462:2010 IDF 214:2010. Milk- Determination of lactose content Enzymatic method using difference in pH
		ISO 5765-1:2002 IDF 79-1:2002. Dried milk, dried ice-mixes and processed cheese. Determination of lactose content. Part 1: Enzymatic method utilizing the glucose moiety of the lactose
		ISO 5765-2:2002 IDF 79-2:2002. Dried milk, dried ice-mixes and processed cheese- Determination of lactose content. Part 2: Enzymatic method utilizing the galactose moiety of the lactose
Butter/Salt content	ISO 15648:2004 IDF 179:2004. Butter. Determination of salt content. Potentiometric method	ISO 1738:2004 IDF 12:2004. Butter. Determination of salt content