CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda item 4 **CX/MAS 20/41/5 Add.1**

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ORIGINAL LANGUAGE ONLY

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

REVISION OF THE RECOMMENDED METHODS OF ANALYSIS AND SAMPLING (CXS 234 – 1999) FATS AND OILS PACKAGE

Comments in reply to CL 2020/29/OCS-MAS

Comments of Canada, Egypt, Mexico, Norway, Peru, Uruguay, FOSFA International, GOED

NOTE: CCMAS41 has been postponed to 17 – 21 May 2021. In order to ensure work continuity comments were requested on the two workable packages. See background information in CL 2020/29/OCS-MAS. The comments compiled in this document will be made available to the EWG on the fats and oils package, for discussion and preparation of a revised proposals for consideration by CCMAS41.

Background

1. This document compiles comments received through the Codex Online Commenting System (OCS) in response to CL 2020/29/OCS-MAS issued in March 2020. Under the OCS, comments are compiled in the following order: general comments are listed first, followed by comments on specific sections.

Explanatory notes on the appendix

2. The comments submitted through the OCS are hereby attached as **Annex I** and are presented in table format.

ANNEX I

<table>
<tr><th colspan="2">GENERAL COMMENTS</th></tr>
<tr><td colspan="2">URUGUAY

COMMENT PAGE 10
For olive oil and olive pomace, for acidity / acid number add method COI / T.20 / Doc. No. 34.

COMMENT PAGE 11
For olive oil and olive-pomace oil, for peroxide index add method COI / T.20 / Doc Nº 35.

COMMENT pages 15 and 17
METHODS FOR As, Fe and Cu in fats and oils
Regarding the methods for As, Cu and Fe, it is considered appropriate to consider the inclusion as a method for As in fats and oils, Pb, Cu and Fe in unrefined fats and oils of the standard proposed by Reviewer II:

• EN 15763: 2009 (Foodstuffs - Determination of trace elements - Determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion)
Which is equivalent to:
• US FDA 4.7 version 1.1, 2015 (Inductively Coupled Plasma-Mass Spectrometric Determination of Arsenic, Cadmium, Chromium, Lead, Mercury, and Other Elements in Food Using Microwave Assisted Digestion)
• AOAC Method 2013.06 (Arsenic, Cadmium, Mercury, and Lead in Foods (Pressure Digestion and Inductively Coupled Plasma-Mass Spectrometry))

In general, Uruguay understands that when numerical criteria are applicable to the provision considered, this is the best alternative for CCMAS.</td></tr>
<tr><td colspan="2">Peru
2. Opiniones generales sobre las propuestas de cambios a los métodos relacionados con las grasas y aceites de la Norma CXS 234 y otras observaciones específicas sobre esos métodos.

El Perú agradece al Grupo de trabajo por medios electrónicos (GTE) presidido por los Países Bajos por el trabajo realizado en la revisión de EXAMEN DE LOS MÉTODOS DE ANÁLISIS EN LA NORMA CXS 234 CONJUNTO MANEJABLE DE GRASAS Y ACEITES y el informe que se presenta en el documento CX/MAS 20/41/5, que nos permite realizar el siguiente comentario.

El Perú mediante la Comisión Técnica Nacional de Aceites y Grasas ha participado en el citado GTE; y evidencia que el CX/MAS 20/41/5 recoge todas las sugerencias que se presentó, las cuales ratifica.</td></tr>
<tr><th colspan="2">Specific comments</th></tr>
<tr><td>Based on the changes proposed in Appendix I and the comments provided in Appendix II to underpin these changes:</td><td>USP
Comments on Fats and Oils Workable Package APPENDIX II from USP:

Please consider the historical relationships that exist between various global standards development organizations (SDOs) in regards to harmonization of the technical aspects</td></tr>
</table>

	of methods for use in trade. These relationships are, in many cases, longstanding and may exist in either formal or informal agreements - as such they have formed a solid basis for use by industry to support global trade. The existence of different styles of complementary methods with harmonized technical content should ideally not be confused with the methods themselves - that is, the methods can be technically the same without the same style and format. Additionally, we should be cognizant of the historical usage of specific methods as it affects global trade. In trade there may be reasons for multiple Type I methods to be used to meet the needs of a specific trade agreement or contract. In these cases the specific method can be (and are) documented in the trade agreement / supplier contract / certificates of analysis. When deciding how to reassess existing methods and determine the method Type, particularly the sole Type I method, this usage in trade should be a paramount concern. Many of these methods are quite old, historical versions from IUPAC are no longer available (as such they have been absorbed by other groups) and they have been used heavily since long before modern method validation guidance was available. Please recognize the work of trade with SDOs and the needs of the respective trade groups potentially impacted by the decisions made here. The decision to accept one method to the exclusion of another one potentially could be perceived as being a trade barrier and I believe we would want to avoid this perception.
	USP Comments on Fats and Oils Workable Package from USP: 1. BHA, BHT, TBHQ and PG in Fats and Oils - We believe the AOCS method would be the method preferred by industry in the case of this matrix. If the AOCS method has been updated appropriately to allow modern columns and quantitation and is the method used in trade, it seems inappropriate to NOT apply Type II status to the AOCS (industry) method. Suggest AOCS method is Type II; AOAC method is Type III. 2. Fatty Acid Composition of Fish Oils (by GC analysis of FAME) - We suggest AOCS C3 2-66 and Ce1i-07 be Type II with ISO 12966-2 and ISO 12966-4/AOCS Ce 2-66 and AOCS Ce 1j-07 be Type III (method for unknown fats/oils or mixtures). 3. Fatty Acid Composition of Named Animal Fats (by GC of FAME) - We suggest replacing AOCS Ce 1f-96 with AOCS Ce 1j-07, which is a better GC method for animal fats. 4. Titre of Named Animal Fats - Please Note AOCS Cc 12-59 appears to have first been published in 1959. As such it seems this method has been used by industry for a very

	long period of time already, regardless of the requirement multiple types of equipment based on temperature. If this is the historically used method by industry for trade, it is the appropriate Type I method. 5. Apparent Density of Named Vegetable Oils - Please delete the reference to the use of an "appropriate conversion factor" with the ISO 6883 method. 6. Relative Density of Named Vegetable Oils - Please delete the reference to the use of "an appropriate conversion factor" with the ISO 6883 method. 8. Unsaponifiable Matter in Named Vegetable Oils - We suggest removing ISO 18609 because it utilizes a different solvent (hexanes). 9. ALL METHODS applying to Olive Oils and Olive Pomace Oils - There is a great deal of simultaneous review of all relevant methods that has been occurring in CCFO as part of the revision of the olive oili and olive pomace oil standard. Based on this ongoing activity, we suggest it would be be in the best interest of CCMAS to wait for the results of the work of the CCFO WG to make changes to these methods. This will help Codex avoid duplication of efforts and allows appropriate input from the Commodity Committee for consideration when CCMAS takes up this item in the future. *Category : SUBSTANTIVE*
	Egypt Egypt agrees the proposed provision name as described in Appendix I & II. Egypt agrees the proposed principle description with some editorial comments on Appendix I, Appendix II as following: • Remove the word (drying) from the principle description. • Correct (NMR) at principle description to be (Nuclear Magnetic Resonance). • Correct (Sensory analysis by trained panel) to be (Sensory analysis by a panel) Egypt agrees the proposed methods as described in Appendix I & II.
Do you agree with the proposed provision name? If no provide comments.	**Canada** Fats and oils: AOCS Ce 6-86. Provision: Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, & propyl gallate Suggest Provision should be: Synthetic antioxidants Fats and oils: AOAC 983.15. Provision: Butylhydroxyanisole, butylhydroxytoluene, tert-

	butylhydroquinone, & propyl gallate Suggest Provision should be: Synthetic antioxidants
	Norway We agree with the proposed provision names.
¿Está de acuerdo con el nombre de la disposición propuesto? En caso negativo, formule sus observaciones.	**Perù** Se aprueba los cambios propuestos por el GTE para el CXS 234 (incluido el nombre de la disposición, la descripción del principio y los métodos).
Do you agree with the proposed principle description? If no provide comments.	**Norway** • General, Insoluble impurities: The principle could be shortened to “Gravimetry, drying at 103 ºC”. • Fish oils, Triglycerides: Under the principle the name of the detectors should be written out, or else it appears as an abbreviation for liquid chromatography
¿Está de acuerdo con la descripción del principio propuesto? En caso negativo, formule sus observaciones.	**Perù** Se aprueba los cambios propuestos por el GTE para el CXS 234 (incluido el nombre de la disposición, la descripción del principio y los métodos).
Do you agree on the proposed methods (and updates thereof if applicable) in conjunction with the method typing? If no provide comments.	**GOED** For Commidity “Fish Oils” and Provision “Fatty acid composition”, GOED recommends the inclusion of European Pharmacopoeia method, assay 2.4.29 (Gas Chromatography of methyl esters). This method only expresses the results as mg/g (wt%) instead of area%. If only methods giving fatty acid content in area% are given as recommended methods in the CODEX standard than this will not be harmonized with some United States Pharmacopeia, European Pharmacopoeia and industry monographs. The expression of the quantity of fatty acids is recommended to be as wt% in mg/g, and not in area% (detector response). For Commodity “Fish oils” and Provision “Acidity: acid value”, GOED recommends the inclusion of European Pharmacopoeia method, assay 2.5.1. This method uses 1:1 ethanol : petroleum ether solvent system, but most laboratories use 1:1 ethanol : diethyl ether.
	FOSFA International - Fish oils: Fatty acid composition: The trade often uses European Pharmacopoeia 5.0 Method 2.4.29. - The various AOCS fatty acid composition methods for Fish oils rich in omega-3s do not quite make sense. Should be combination Ce2-66 and Ce1i-07 specifically for marine oils - Olive Oils and Olive Pomace Oils: Trans fatty acids content: ISO 15304. Method

	obsolete - been replaced by ISO 12966-2 and 12966-4 (although it does not include validation data for olive oil) - Phosphorus determination should be included in Named Vegetable oils - it is an important trade method to determine degumming efficiency (FOSFA contractual method) ISO 10540-1 = colorimetric; ISO 10540-2 = Atomic Absorption; ISO 10540-3 = ICP - Determination trace elements (lead, copper and iron) should include an ICP method ie ISO 21033 with ICP-OES

	Canada NOTES: 1) For Copper and Iron provisions as well as Lead in various fats and oils commodities: Suggest method AOCS Ca 17a-18 as Type II; with atomic absorption methods as Type III. Typing suggestion follows method harmonization in this group of commodities which has been an ongoing project for more than 30 years and many methods published by fats and oils SDOs are also based on original IUPAC methods, which were discontinued about 20 years ago, and now of limited availability. 2) The methods for olive oil and olive pomace oils are under review in a WG at CCFO. This review will be carried out as part of the revision of the olive oil and olive pomace oil standard. CCMAS should wait until the results of the review are submitted to avoid duplication of effort. Some of the current International Olive Council (IOC) methods may not be fully harmonized with ISO and AOCS methods at this time. It is important to note that there is a very strong and enduring relationship between the various standards development organizations (SDOs). Under both formal and informal arrangements, methods are harmonized for use in trade, hence there are many complementary methods in both ISO and AOCS formats where the technical content is the same, but the method is written in the style is that of the respective organization. There is also a long-standing arrangement that AOCS will republish IOC methods in AOCS style. In addition, method harmonization in this group of commodities has been an ongoing project for more than 30 years and many methods published by fats and oils SDOs are also based on original IUPAC methods, which were discontinued about 20 years ago, and now of limited availability. It is important to consider the active usage of a method when deciding whether a method is Type II or Type III and also deciding which method from from the list should be selected as Type I. In some cases multiple Type I methods are routinely used in trade and the particular method is stated in the terms of a contract so that no misunderstanding occurs. Similarly, certificates of analysis should name the method used and the version number to avoid unnecessary confusion. Existing methods that were adopted by SDOs prior to the development of guidance for method validation and the generation of performance data were adopted without the validation information and remain in use because of decades or even a century of regular use. These older methods may be monitored through proficiency testing, but SDOs have few resources to requalify such methods and users are equally reluctant to participate in these types of studies. Some methods have been developed within a particular region and, as such, give rise to groups of Type I methods that are sufficiently different (i.e., use of different solvents, titration agents, indicator solutions,

	temperatures) but are based on regional preferences. The decision to accept one method to the exclusion of a second one may be perceived as a barrier to trade. Fish oils: Provision: Phospholipids: US Pharmacopoeia does not publish validation data, refer to JAOCS article Fish oils: Provision: Peroxide value: European Pharmacopoeia 2.5.5 was chosen at CCMAS on physical examination of the method to check the use of iso-octane. Fish oils: Provision: p-Anisidine: AOCS Cd 18-90 was chosen as Type I by fish oil industry and tested in AOCS proficiency program (marine oil; GOED) Named Vegetable oils: Provision Halphen Test: AOCS Cb 1-25: Suggest referring to CCFO to establish continued relevance of test Named Vegetable Oils: Provision: Crismer Value: AOCS Cb 4-35 and AOCS Ca 5a-40: Suggest confirming with CCFO whether this test is still relevant Named Vegetable Oils: Provision: Unsaponifiable matter: Both ISO 3596 and AOCS 6b-53 methods use diethyl ether, ISO 18609 uses hexanes, AOCS Ca 6a-40 uses petroleum ether. Named Vegetable Oils: Provision: Apparent Density: ISO 6883, suggest Removal of "with the appropriate conversion factor" Named Vegetable Oils: Provision: Relative Density: ISO 6883, suggest Removal of "with the appropriate conversion factor" Named Animal Fats: Provision Titre: ISO 935 and AOCS Cc 12-59 are not the same method and these methods will give different results, given that the AOCS method was published in 1959, which should be identified as Type I? Named Animal Fats: Provision Fatty Acid composition: Among ISO 12966-2 and ISO 12966-4 / AOCS Ce 2-66 and AOCS Ce 1f-96, suggest replacing Ce 1f-96 with Ce 1j-07 and suggest AOCS Ce 2-66 and Ce 1j-07 as Type II because the AOCS method has a better GC method for analysis. Ce 1j-07 is newer than Ce 1f-96 which has been deemed surplus.Fish oils: Provision Fatty Acid composition: IS0 12966-2 and ISO 12966-4 methods are for unknown fats and oils or mixtures, rather than fish oils, similar to AOCS Ce 2-66 + AOCS 1j-07, so they should be Type III

	Fish oils: Provision Fatty acid composition: Suggest that AOCS Ce 2-66 + Ce 1i-07 should be Type II because there are collaborative study data provided in the method for fish oils. AOCS 2b-11 was validated in various food matrices and Ce 1j-07 is for use with unknown fats or fats from dairy or ruminant origins Fats and oils: Current Provision: Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, & propyl gallate – AOAC 983.15: given the note below, suggest that this method may become Type III

	Norway • Fats and oils, Arsenic: We recognise that both the Arsenic methods, AOAC 942.17 and AOAC 952.13, were surplus in 1993. Therefore, it could be considered to select AOAC 986.15 using AAS as type II and the Kjeldahl methods as type III methods. • General: We welcome method performance criteria for provisions such as arsenic, lead, copper and iron. • Fish oils, Phospholipids: The USP-FCC 10 2S (type I) is suggested to be deleted in this document, however, this method is not listed in CXS 234. For this parameter only a type IV method is listed. • Fish oils, Fatty acid composition: It is important that both type II and type III methods are validated on fish oils. We agree that one of the type III methods should be selected as the type II method. However, it seems that two type III methods are turned into type II. If there are technical differences (such as different extractions) between ISO 12966-2 + ISO 12966-4 and AOCS Ce 2-66 + AOCS Ce1i-07, only one of them should be a type II method, the other should remain as type III.
¿Está de acuerdo con los métodos propuestos (y actualizaciones, si procede) junto con la clasificación de los métodos? En caso negativo, formule sus observaciones.	**Perù** Se aprueba los cambios propuestos por el GTE para el CXS 234 (incluido el nombre de la disposición, la descripción del principio y los métodos).
European Pharmacopoeia 2.5.5 Part B utilizes trimethylpentane instead of iso-octane, still this method is considered identical. Only identical type I methods should be endorsed	**GOED** On page 24, regarding “European Pharmacopoeia 2.5.5 Part B utilizes trimethylpentane instead of iso-octane, still this method is considered identical. Only identical type I methods should be endorsed,“ it is not surprising that the methods are considered identical since iso-octane and trimethylpentane are the same solvent. A different chemical nomenclature is used for the same solvent in different methods. For all commodities associated with Provision “Relative density” and “Apparent density”, GOED recommends adding method ASTM D7777-13(2018)e1 “Standard test method for density, relative density, or API gravity of liquid petroleum by portable digital density meter” as a more modern method than the old “pycnometer” methods. Here is a link https://www.astm.org/Standards/D7777.htm with additional information.
	Canada Fats and oils: Current Provision: Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, & propyl gallate – Suggestion re: Typing of AOCS Ce 6-86: a collaborative study of the AOCS Ce 6-86 method is being coordinated with ISO/TC 34/SC 11 (fats and oils). In the near future, perhaps this will be the choice for Type II because it will have more recent collaborative study data, unless trade prefers the AOAC method.

Considerar el Apéndice II y proporcionar preguntas y comentarios sobre la información enumerada para orientar más y formular recomendaciones sobre la eliminación de los métodos, la propuesta de volver a escribir o información adicional sobre la situación de los métodos enumerados.	**Perù** Se aprueba los cambios propuestos por los revisores, trabajo realizado durante la vigencia del GTE
3.-Examinar el Apéndice III y determinar si se justifica la revisión y actualización de esos métodos. -¿Es necesario llevar a cabo un examen de los métodos? -En caso afirmativo, ¿estaría dispuesto a examinar dichos métodos?	**Perù** Estamos conforme con la revisión del Apéndice III y se considera que si se justifica el examen y la actualización de los métodos citados
Olive Oils and Olive Pomace Oils Absorbency in ultra-violet COI/T.20/Doc. No. 19 / ISO 3656 / AOCS Ch 5-91 Spectrophotometry	**Mexico** Específico (Pág. 18 y 23) Se sugiere modificar la palabra “absorbency” por “absorbance”, el cual es un término más aceptado y relevante en el ámbito de la química analítica. Cambio general con fin de hacer más clara y técnica la guía.
	Mexico General Appendix II Part A Se sugiere establecer criterios numéricos para la determinación de cobre y hierro en grasas y aceites en general Se acepta que el método AOAC 983.15 se mantenga como tipo II y el AOCS Ce 6-86 como tipo III para la determinación de antioxidantes en aceites y grasas Se acepta la sugerencia de establecer criterios numéricos para la determinación de arsénico en grasas y aceites Se está de acuerdo en que el método ISO 3960 aplica para índice de peróxido y no para índice de acidez Se esta de acuerdo en que permanezca la referencia a la ISO 660

	Mexico General Appendix I Part A Se requiere detallar la referencia de la Farmacopea Europea 1352 (método general o monografía) para la determinación de triglicéridos en aceites de pescado por Cromatografía de Líquidos (IR) Se requiere actualizar la referencia de la USP a USP 42 NF 37 La farmacopea cuenta con metodologías por Cromatografía de Gases y puede haber confusión Se sugiere que el método AOAC 986.15 para la determinación de arsénico en grasas y aceites sea considerado como tipo II y los AOAC 963.21 and AOAC 952.13 como tipo III Los métodos de absorción atómica tienen mejores límites de cuantificación que los métodos colorimétricos Se está de acuerdo con el reemplazo del concepto “agua” por “humedad” Se está de acuerdo en establecer criterios numéricos para la determinación de plomo en mantequilla, caseína comestible y suero en polvo
	Mexico Específico Appendix II El método ADPI para la determinación de partículas quemadas en leche y crema en polvo debe permanecer como tipo IV El método ha sido usado tradicionalmente y no se tienen criterios para satisfacer su aceptación ya que es un método semicuantitativo que se basa en una inspección visual Para la determinación de calcio se sugiere considerar criterios numéricos en lugar de tres métodos Las metodologías de ICP-espectrometría de masas, ICP-espectrometría de emisión y Absorción Atómica por flama son apropiadas para cuantificar los niveles requeridos de calcio

	Se sugiere que el método ISO/DIS 23970|IDF 252 para la determinación de melanina en leche y productos lácteos sea considerado como tipo II La metodología de Cromatografía de Líquidos acoplada a detección de masas es ampliamente selectiva
	Mexico Tabla G2.3: Lead methods for further review Se sugiere que el método general AOAC 972.25 para la determinación de plomo en mantequilla sea considerado como tipo II La metodología de Absorción Atómica es ampliamente utilizada en el análisis de metales contaminantes en alimentos debido a su practicabilidad y su seguridad ha sido establecida a través de la validación de la misma