CODEX ALIMENTARIUS COMMISSION



Food and Agriculture Organization of the United Nations



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Agenda Item 5

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CODEX COMMITTEE ON FATS AND OILS

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PROPOSED DRAFT REVISION TO THE STANDARD FOR OLIVE OILS AND OLIVE POMACE OILS (CXS 33-1981): REVISION OF SECTIONS 3, 8 AND APPENDIX

(Comments of Canada¹)

In response to <u>CL 2023/61/OCS-FO</u>: Request for Comments (at Step 3) on proposed draft revision to the *Standard for Olive Oils & Olive Pomace Oils* (CXS 33-1981): Revision of Sections 3, 8 and Appendix, and CX/FO 24/28/8, Canada would like to make the following comments and suggestions.

Canada thanks the EWG Chair (Spain), co-chair (Argentina) and participants for their work in preparing the methods table and taking into account CRD 24 presented at CCFO27. Canada notes the request from the Codex Alimentarius Commission and Codex secretariat that, when refining the methods of analysis in Codex Standards, committees are reminded that the only listing of methods in Codex Standards will be in CXS 234-1999. A single paragraph will be inserted into each standard to reflect that the reader should refer to CXS 234-1999 for the most updated guidance in method selection.

A. To create the attached tables a stepwise approach was used to fulfill the above requirements and highlight some issues for resolution. The steps in this approach starting from the current published version of CXS 33-1981 (2021) were:

- 1. Identify additional or changed provisions between CXS 33 (2021) and proposed revised version.
- 2. Identify additional or changed methods between CXS 33 (2021) and proposed revised version.
- 3. Identify additional or changed method principle between CXS 33 (2021) and proposed revised version.
- 4. Identify original method Typing.
- 5. Provide commentary for proposed additional changes.
- 6. Highlight cells with changes.

Results are shown in Table A. Comparison of CXS 33-1981 (2021) with CXS 33-1981 (Proposed revision 2024)

B. On the assumption that the methods comments provided in Table A would be substantially accepted, Table B was constructed to reflect the current published version of CXS 234-1999 (2021), the proposed changes for CXS 33-1981 (Proposed Revision 2024), and a revised version of olive oil provisions and methods for CXS 234-1999 reflecting those changes.

- 1. Insertions, deletions and method typing were identified, according to the CCMAS informational document: **Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234.**
- 2. Note: there were interim steps in this process

Results are show in Table B. Comparison of CXS 234-1999 (2021) with Proposed changes to both CXS 33-1981 (2021) and CXS 234-1999 (Proposed Revision 2024)

C. On completion of the modifications for CXS 234 shown in Table B, the columns entitled Provision, Method, Principle and Type were formatted according to *Comprehensive guidance for the process of submission*,

¹ Canada would like to acknowledge the work done by our subject matter expert Dr. Richard Cantrill and the MoniQA Association in developing this document for CCFO28.

consideration and endorsement of methods for inclusion in CXS 234 for future presentation to CCMAS for endorsement and final adoption by CAC.

Results are show in Table C. Proposed corrections for insertion into a revised CXS 234-1999 (2021) Clean version.

Proposal

- 1. Items identified in the commentary column below will be discussed and agreed-upon by olive oil experts prior to approval by CCFO28.
- 2. Any changes arising from the discussion of Table A shall be transferred to Tables B and C prior to final approval by CCFO28 and submission to CCMAS.
- 3. This work can be completed during the CCFO28 meeting either by an *ad hoc* small group of experts, during a physical working group meeting or in plenary, as time allows.

Olive oils and olive pomace oils; Provisions	CXS 33 (2021)	CXS 33 Proposed	Principle	Preliminary Typing	Commentary
Absorbency in ultra-violet	COI/T.20/Doc. no. 19 or ISO 3656:2002 or AOCS Ch 5-91 (01)	COI/T.20/Doc. No. 19 ISO 3656 AOCS Ch 5-91	Absorption in <u>ultra-violet</u>	Ш	Uses 2 alternative solvents with different conditions and results – suggest change to Type I
Acidity, free (acid value)	ISO 660 or AOCS Cd-3d 63	ISO 660 AOCS Cd 3d-63 <u>COI/T.20/Doc. No 34</u>	Titrimetry	1	ISO/AOCS – Codex general method for fats and oils; IOC - Same principle – with solvent restrictions – suggest change to Type IV
Alpha-tocopherol	ISO 9936:1997.	ISO 9936 AOCS Ce 8-89	HPLC (UV or fluorescence)	 	
Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 or AOCS Ce 5b-89	COI/T.20/Doc. no. 20	Analysis of triglycerides by HPLC and calculation	1	TG by HPLC and calculation from FAC using COI/T.20/Doc. No 33 - Suggest inclusion of method
Erythrodiol + uvaol	COI/T.20/doc. No 30- 2011	COI/T.20/Doc. No 26	Separation and gas chromatography (FID)	11	See sterols – Depending on CCMAS, include separation methods in CXS 234
Fatty acid composition	COI/T.20/Doc. no. 24 and ISO 5508:1990 and AOCS Ch 2- 91(02) or AOCS Ce 1f-96 (02). For Sample preparation ISO 5509:2000 or AOCS Cc 2-66(97)	COI/T.20/Doc. No 33 AOCS Ch 2-91 ISO 12966-2 ISO 12966-4	Gas chromatography (FID) of methyl esters	 	COI/T.20/Doc. No 33 Include methyl ester prep - Suggest inclusion of ME prep for AOCS and ISO AOCS Ce 2-66 and AOCS Ch 2-91 / Ce 1h-05 ISO 12966-2 and ISO 12966-4
Fatty acids in 2-position of triglycerides	ISO 6800:1997 or AOCS Ch 3-91 (97).				Provision replacement / alignment Suggest remove entry from CXS 234
2-glyceryl monopalmitate percentage		COI/T.20/Doc. No 23	Gas chromatography (FID)	<u>II</u>	New method/provision

Olive oils and olive pomace oils; Provisions	CXS 33 (2021)	CXS 33 Proposed	Principle	Preliminary Typing	Commentary
<u>Fatty acid ethyl ester</u> <u>content</u>		<u>COI/T.20/Doc. No 28</u>	Gas chromatography (FID)	II	New method/provision
Halogenated solvents, traces	COI/T.20/Doc. no. 8	ISO 16035	Gas chromatography (FID)	II	Replacement method
Insoluble impurities in light petroleum	ISO 663:2000	ISO 663	Gravimetry	I	
lodine value	ISO 3961:1996 or AOAC 993.20 or AOCS Cd 1d-92 (97) or NMKL 39(2003)	ISO 3961 AOAC 9930.20 AOCS Cd 1d-92 NMKL 39	Wijs-Titrimetry	1	
Iron and copper	ISO 8294:1994 or AOAC 990.05	ISO 8294 AOAC 990.05	AAS	II	
Lead		*Performance criteria examples? ISO 12193 AOAC 994.02 AOCS Ca 18c-91	AAS	11	Lead using criteria approach; examples: ISO 12193 AOAC 994.02 AOCS Ca 18c-91
Moisture and volatile matter	ISO 662:1998	ISO 662	Gravimetry	I	
Organoleptic characteristics	COI/T.20/Doc. no. 15	COI/T.20/Doc. no. 15	Panel test	I	
Peroxide value	ISO 3960:2001 or AOCS Cd 8b-90 (03)	ISO 3960 AOCS Cd 8b-90 <u>COI/T.20/Doc. No 35</u>	Titrimetry	I I IV	COI/T.20/Doc. No 35 uses chloroform not isopropanol. Therefore, these methods are not identical – Suggest include as Type IV?
Pomace oils		NMKL 39			No provision in standard - Wrong method (IV) – Suggest delete

Olive oils and olive pomace oils; Provisions	CXS 33 (2021)	CXS 33 Proposed	Principle	Preliminary Typing	Commentary
Relative density	IUPAC 2.101, with the appropriate conversion factor	ISO 6883 AOCS Cc 10c-95	Pycnometry	I	Changed to match other provisions in fats and oils standards
Refractive index	ISO 6320:2000 or AOCS Cc 7-25 (02)		Refractometry	П	No method stated: suggest retain previous entry
Sampling	ISO 661:1989 and ISO 5555:2001				No method stated: suggest retain previous entry
Saponification value	ISO 3657:2002 or AOCS Cd 3-25 (03)	ISO 3657 AOCS Cd 3-25	Titrimetry	1	Year of publication remove per CCMAS protocol
Sterol composition and total sterols <u>4α-desmethylsterol and</u> total sterol content	COI/T.20/Doc. no. 10 or ISO 12228:1999 or AOCS Ch 6-91 (97)	COI/T.20/Doc. No 26 ISO 12228-2 AOCS Ch 6-91	Gas chromatography (FID)		4α -desmethyl- not in original provision nor in title of methods quoted. COI method - Revised version with two IS incorporating E+U and aliphatic alcohols – Have reservations about ISO and AOCS being suitable for the combined analysis without further work or confirmation Need to include method of separation?
Stigmastadienes	COI/T.20/Doc. no. 11 or ISO 15788-1:1999 or AOCS Cd 26-96 (03) or ISO 15788- 2:2003	COI/T.20/Doc. no. 11 ISO 15788-1 AOCS Cd 26-96	Gas chromatography (FID)	 	
Stigmastadienes		ISO 15788-2	HPLC	111	Separated based on principle
trans fatty acids content	COI/T.20/Doc no. 17 or ISO 15304:2002 or AOCS Ce 1f-96 (02).	COI/T.20/Doc no. 33 ISO 12966-4 AOCS Ce 1h-05	Gas chromatography (FID) of methyl esters		COI/T.20/Doc no. 33 Include methyl ester prep: ISO 12966-2 and ISO 12966-4 AOCS Ce 2-66 and AOCS Ce 1h-05 Comment as per FAC

Olive oils and olive pomace oils; Provisions	CXS 33 (2021)	CXS 33 Proposed	Principle	Preliminary Typing	Commentary
Unsaponifiable matter	ISO 3596:2000 or ISO 18609:2000 or AOCS Ca 6b-53 (01)	ISO 3596 AOCS Ca 6b-53	Gravimetry	I	ISO 18609 removed as gives systematic lower values; not identical
Wax content	COI/T.20/Doc. no. 18 or AOCS Ch 8-02	COI/T.20/Doc. no. 18 AOCS Ch 8-02	Gas chromatography (FID)	 	
1,2 Diglycerides		ISO 29822	Gas chromatography (FID)	П	
Pyropheophytin "a"		ISO 29841	HPLC with UV/VIS or fluorescence detection	II	

Olive oils and olive pomace oils; Provisions	CXS 234 (2021)	CXS 33 Proposed (from Table A)	CXS 234 Proposed	Principle	Туре
Absorbency in ultra-violet	COI/T.20/Doc. No. 19 or ISO 3656 or AOCS Ch 5-91	COI/T.20/Doc. No. 19 ISO 3656 AOCS Ch 5-91	<u>COI/T.20/Doc. No. 19 / ISO</u> <u>3656 / AOCS Ch 5-91</u>	Absorption in <u>ultra-violet</u>	1
Acidity, free (acid value)	ISO 660 or AOCS Cd 3d-63	ISO 660 AOCS Cd 3d-63 <u>COI/T.20/Doc. No 34</u>	<u>ISO 660 / AOCS Cd 3d-63</u> <u>COI/T.20/Doc. No 34</u>	Titrimetry	۱ <u>IV</u>
Alpha-tocopherol	ISO 9936	ISO 9936 AOCS Ce 8-89	ISO 9936 AOCS Ce 8-89	HPLC (UV or fluorescence)	
Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 or AOCS Ce 5b-89	COI/T.20/Doc. no. 20	<u>COI/T.20/Doc. no. 20 and</u> <u>COI/T.20/Doc. No 33</u>	Analysis of triglycerides by HPLC and GC followed by calculation	I
Erythrodiol + uvaol	COI/T.20/Doc.no. 30	COI/T.20/Doc. No 26	<u>COI/T.20/Doc. No 26</u>	Separation and gas chromatography (FID)	11
Fatty acid composition		COI/T.20/Doc. No 33 AOCS Ch 2-91 ISO 12966-2 ISO 12966-4	<u>COI/T.20/Doc. No 33</u> <u>AOCS Ce 2-66 and AOCS</u> <u>Ch 2-91 / Ce 1h-05</u> <u>ISO 12966-2 and</u> <u>ISO 12966-4</u>	Gas chromatography <u>(FID)</u> of methyl esters	
Fatty acids in 2-position of triglycerides					
2-glyceryl monopalmitate percentage		COI/T.20/Doc. No 23	COI/T.20/Doc. No 23	Gas chromatography (FID)	Ш
Fatty acid ethyl ester content		COI/T.20/Doc. No 28	<u>COI/T.20/Doc. No 28</u>	Gas chromatography (FID)	11
Halogenated solvents, traces	COI/T.20/Doc. no. 8	ISO 16035	ISO 16035	Gas chromatography <u>(FID)</u>	11

Table B. Comparison of CXS 234-1999 (2021) with Proposed changes to both CXS 33-1981 (2021) and CXS 234-1999 (Revised 2024)

Olive oils and olive pomace oils; Provisions	CXS 234 (2021)	CXS 33 Proposed (from Table A)	CXS 234 Proposed	Principle	Туре
Insoluble impurities in light petroleum	ISO 663	ISO 663	ISO 663	Gravimetry	I
lodine value	ISO 3961 or AOAC 993.20 or AOCS Cd 1d92 or NMKL 39	ISO 3961 AOAC 9930.20 AOCS Cd 1d-92 NMKL 39	ISO 3961 / AOAC 9930.20 / AOCS Cd 1d-92 / NMKL 39	Wijs-Titrimetry	1
Iron and copper	ISO 8294 or AOAC 990.05	ISO 8294 AOAC 990.05	ISO 8294 / AOAC 990.05	AAS	II
Lead	AOAC 994.02 or ISO 12193 or AOCS Ca 18c-91	*Performance criteria examples? ISO 12193 AOAC 994.02 AOCS Ca 18c-91	<u>Use performance criteria</u>	AAS	11
Moisture and volatile matter	ISO 662	ISO 662	ISO 662	Gravimetry	1
Organoleptic characteristics	COI/T.20/Doc. no. 15	COI/T.20/Doc. no. 15	COI/T.20/Doc. no. 15	Panel test	1
Peroxide value	ISO 3960 or AOCS Cd 8b-90	ISO 3960 AOCS Cd 8b-90 <u>COI/T.20/Doc. No 35</u>	ISO 3960 / AOCS Cd 8b-90 COI/T.20/Doc. No 35	Titrimetry	I IV
Pomace oils		NMKL 39	Delete provision in CXS 234		
Relative density	IUPAC 2.101, with the appropriate conversion factor. See comment above	ISO 6883 AOCS Cc 10c-95	ISO 6883 / AOCS Cc 10c- 95	Pycnometry	I
Refractive index	ISO 6320 or AOCS Cc 7-25		ISO 6320 / AOCS Cc 7-25	Refractometry	11
Sampling	ISO 661:1989 and ISO 5555:2001		ISO 5555 and ISO 661		

Olive oils and olive pomace oils; Provisions	CXS 234 (2021)	CXS 33 Proposed (from Table A)	CXS 234 Proposed	Principle	Туре
Saponification value	ISO 3657 or AOCS Cd 3-25	ISO 3657 AOCS Cd 3-25	ISO 3657 / AOCS Cd 3-25	Titrimetry	I
Sterol composition and total sterols $\frac{4\alpha}{1000}$ desmethylsterol and $\frac{4\alpha}{10000}$	COI/T.20/Doc. no. 30 ISO 12228-2 or AOCS Ch 6-91	COI/T.20/Doc. No 26 ISO 12228-2 AOCS Ch 6-91	<u>COI/T.20/Doc. No 26</u>	Gas chromatography (FID)	11
Stigmastadienes	COI/T.20/Doc. no. 11 or ISO 15788-1 or AOCS Cd 26-96	COI/T.20/Doc. no. 11 ISO 15788-1 AOCS Cd 26-96	COI/T.20/Doc. no. 11 ISO 15788-1 AOCS Cd 26-96	Gas chromatography (FID)	
Stigmastadienes	ISO 15788-2	ISO 15788-2	<u>ISO 15788-2</u>	HPLC	Ш
<i>trans</i> fatty acids content	COI/T.20/Doc no. 17 or ISO 15304 or AOCS Ch 2a-94	COI/T.20/Doc no. 33 ISO 12966-4 AOCS Ce 1h-05	<u>COI/T.20/Doc no. 33</u> <u>ISO 12966-2 and ISO</u> <u>12966-4</u> <u>AOCS Ce 2-66 and AOCS</u> Ce 1b-05	Gas chromatography (FID) of methyl esters	
Unsaponifiable matter	ISO 3596 or ISO 18609 or AOCS Ca 6b-53	ISO 3596 AOCS Ca 6b-53	<u>ISO 3596 / AOCS Ca 6b-</u> 53	Gravimetry	1
Wax content	COI/T.20/Doc. no. 18 or AOCS Ch 8-02	COI/T.20/Doc. no. 18 AOCS Ch 8-02	COI/T.20/Doc. no. 18 AOCS Ch 8-02	Gas chromatography (FID)	
1,2 Diglycerides		ISO 29822	ISO 29822	Gas chromatography (FID)	Ш
Pyropheophytin "a"		ISO 29841	ISO 29841	HPLC with UV/VIS or fluorescence detection	Ш

Fats and oils and related products	Provision	Method(s)	Principle	Туре
Olive oils and olive pomace oils	Absorbency in ultra- violet	COI/T.20/Doc. No. 19 / ISO 3656 / AOCS Ch 5-91	Absorption in ultra-violet	I
Olive oils and olive pomace oils	Acidity, free (acid	ISO 660 / AOCS Cd 3d-63	Titicanta	I
	value)	COI/T.20/Doc. No 34	litrimetry	IV
Olive oils and olive pomace oils		ISO 9936		II
	Alpha-tocopherol	AOCS Ce 8-89	HPLC (UV or fluorescence)	III
Olive oils and olive pomace oils	Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 and COI/T.20/Doc. No 33	Analysis of triglycerides by HPLC and GC followed by calculation	I
Olive oils and olive pomace oils	1,2 Diglycerides	ISO 29822	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Erythrodiol + uvaol	COI/T.20/Doc. No 26	Separation and gas chromatography (FID)	II
Olive oils and olive pomace oils		COI/T.20/Doc. No 33		II
	Fatty acid composition	AOCS Ce 2-66 and AOCS Ch 2-91 / Ce 1h-05	Gas chromatography (FID) of methyl esters	III
		ISO 12966-2 and ISO 12966-4		III
Olive oils and olive pomace oils	2-glyceryl monopalmitate percentage	COI/T.20/Doc. No 23	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Fatty acid ethyl ester content	COI/T.20/Doc. No 28	Gas chromatography (FID)	11
Olive oils and olive pomace oils	Halogenated solvents, traces	ISO 16035	Gas chromatography (FID)	II

Table C. Proposed corrections for insertion into CXS 234-1999 (2021) [clean version]

Olive oils and olive pomace oils	Insoluble impurities in light petroleum	ISO 663	Gravimetry	I
Olive oils and olive pomace oils	lodine value	ISO 3961 / AOAC 9930.20 / AOCS Cd 1d-92 / NMKL 39	Wijs-Titrimetry	I
Olive oils and olive pomace oils	Iron and copper	ISO 8294 / AOAC 990.05	AAS	II
Olive oils and olive pomace oils	Lead	Use performance criteria*		
Olive oils and olive pomace oils	Moisture and volatile matter	ISO 662	Gravimetry	I
Olive oils and olive pomace oils	Organoleptic characteristics	COI/T.20/Doc. no. 15	Panel test	I
Olive oils and olive pomace oils	Peroxide value	ISO 3960 / AOCS Cd 8b-90		I
		COI/T.20/Doc. No 35	litrimetry	IV
Olive oils and olive pomace oils	Pyropheophytin "a"	ISO 29841	HPLC with UV/VIS or fluorescence detection	II
Olive oils and olive pomace oils	Relative density	ISO 6883 / AOCS Cc 10c-95	Pycnometry	I
Olive oils and olive pomace oils	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
Olive oils and olive pomace oils	Sampling	ISO 5555 and ISO 661		
Olive oils and olive pomace oils	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry	I
Olive oils and olive pomace oils	Sterol composition and total sterols	COI/T.20/Doc. No 26	Gas chromatography (FID)	II
Olive oils and olive pomace oils		COI/T.20/Doc. no. 11		II
	Stigmastadienes	ISO 15788-1	Gas chromatography (FID)	
		AOCS Cd 26-96		
		ISO 15788-2	HPLC	

Olive oils and olive pomace oils		COI/T.20/Doc no. 33		II
	<i>trans</i> Fatty acids content	ISO 12966-2 and ISO 12966-4	Gas chromatography (FID) of methyl esters	
		AOCS Ce 2-66 and AOCS Ce 1h-05		111
Olive oils and olive pomace oils	Unsaponifiable matter	ISO 3596 / AOCS Ca 6b-53	Gravimetry	I
Olive oils and olive pomace oils	Wax contant	COI/T.20/Doc. no. 18	Cas obromotography (EID)	II
		AOCS Ch 8-02	Gas chromatography (FID)	111

*Example of suitable methods: ISO 12193, AOAC 994.02, AOCS Ca 18c-91