CODEX ALIMENTARIUS COMMISSION

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Agenda Item 4.2

CX/MAS 20/41/5
March 2020

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

41st Session

Budapest, Hungary, 11 - 15 May 2020

REVIEW OF METHODS OF ANALYSIS IN CXS 234 FATS AND OILS WORKABLE PACKAGE

(Prepared by the Electronic Working Group chaired by the Netherlands)

INTRODUCTION

1. At its 40th session CCMAS agreed to continue efforts on the workable packages for the review and update of CODEX STAN 234-1999 (CXS 234-1999). The Committee agreed to continue the review of all methods related to fats and oils which was initiated by AOCS in document CX/MAS 19/40/3 Add.3 as described in REP 19/MAS, para.27.

2. CCMAS noted that AOCS had begun the review of the workable package on fats and oils and that the table of methods presented in CX/MAS 19/40/3 Add.3 would serve as basis for further consideration.

3. CCMAS further agreed to establish an EWG chaired by the Netherlands working in English, to continue the review of the fats and oils workable package, using CX/MAS 19/40/3 Add.3 as the basis for the review, and to work in close coordination with the relevant SDOs.

EWG PROCESS

4. The original terms of reference of the EWG stated in REP 18/MAS, para47 were to update the methods in the workable package of fats and oils. All methods concerned in CXS 234 were therefore considered and the methods were grouped by provision. This is in order to (i) have a complete and transparent overview of the provision and its endorsed analysis methods and their respective commodities and (ii) to avoid duplicate review work.

5. The electronic working group was initiated and operated through coordination of the EWG chair via e-mail. The EWG chair managed all communication within the EWG and kept track of all documents and comments. The list of participants is attached in Appendix IV.

6. All Codex participants were welcome and members joining the EWG were asked to be willing to review a number of methods and provide feedback on the methods. To this end, EWG Participants were asked to supply the list of methods (e.g. ISO, AOCS, AOAC, COI, EN, NMKL) to which they had access. Based on the responses each participant was invited to review certain methods for a certain provision group (e.g. arsenic, fatty acid composition, etc.). As a means of control, some EWG members were asked to review the same provision but for different commodity group(s).

7. To assist in the method review, the chair created a method review sheet to be used by the reviewers during the review. The review sheet was supplemented with content from the information document “Comprehensive guidance for the process of submission, consideration and endorsement of methods for inclusion in CXS 234”. Method assignments, method review sheets and additional instructions for completing the review, saving and naming review files were distributed via e-mail.

8. Based on the comments, recommendations and conclusions from the review sheets, three tables (appendices I-III) were prepared to help explain and track changes to CXS 234. For ease of review and comparison, all the tables utilize the information (Commodity, Provision, Method, Principle, Type) as is done in CXS 234. Additional information (Codex Standard, Committee) will need to be added when updating to the new CXS 234 format.

9. Appendix I contains all methods listed in CXS 234 and their proposed changes to CXS 234. Almost all methods in this workable package required textual formatting and uniformization of terminology used. The current CXS 234 methods were formatted as strikethrough when needed, and the proposed reviewed method is listed below the strikethrough method. Changes to method definitions (identical and complementary) and method typing were also proposed here.

10. Appendix II contains the list of methods sorted by provision, the reviewer comments and the suggested changes to CXS 234 which were implemented in Appendix I. Furthermore, Appendix II contains proposed edits or actions that require further decisions, such as retyping or removal of a method. All comments and rationale are presented below the specific commodity-provision combination or below the provision. Where necessary, the EWG chair added explanatory comments.

11. Appendix III contains all methods which have not been reviewed by any member of the EWG.

RECOMMENDATIONS

12. The Committee is invited to:

- Consider Appendix I and endorse the proposed changes to CXS 234.
- Consider Appendix II and provide questions and comments on the information listed to guide further work, and make recommendations on the removal of methods, proposed retyping or additional information on the status of the methods listed.
- Consider Appendix III and determine whether review and updating of these methods are warranted.

APPENDIX I

PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

Commodity	Provision	Method	Principle	Type
~~**Fat spreads and blended spreads**~~	~~Fat content~~	~~ISO 17189 | IDF 194~~	~~Gravimetry~~	~~I~~
Fat spreads and blended spreads	Total fat	ISO 17189 | IDF 194	Gravimetry. Direct determination of fat using solvent extraction.	I
~~**Fats and oils**~~	~~Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, & propyl gallate~~	~~AOAC 983.15; or AOCS Ce 6-86~~	~~Liquid chromatography~~	~~II~~
Fats and oils	Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, and propyl gallate	AOAC 983.15	Liquid chromatography	II
Fats and oils	Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, and propyl gallate	AOCS Ce 6-86	Liquid chromatography	III
~~**Fats and Oils (all)**~~	~~Arsenic~~	~~AOAC 942.17~~	~~Colorimetry (molybdenum blue)~~	~~III~~
Fats and Oils (all)	Arsenic	AOAC 963.21 and AOAC 942.17	Kjeldahl flask digestion and colorimetry (molybdenum blue)	III
~~**Fats and Oils (all)**~~	~~Arsenic~~	~~AOAC 952.13~~	~~Colorimetry (diethyldithiocarbamate)~~	~~II~~
Fats and Oils (all)	Arsenic	AOAC 963.21 and AOAC 952.13	Kjeldahl flask digestion and colorimetry (diethyldithiocarmabate)	II
Fats and Oils (all)	Arsenic	AOAC 986.15	Atomic absorption spectrophotometry	III
~~**Fats and Oils (all)**~~	~~Insoluble impurities~~	~~ISO 663~~	~~Gravimetry~~	~~I~~
Fats and Oils (all)	Insoluble impurities	ISO 663	Calculation from total insoluble content in *n*-hexane or light petroleum. Gravimetry, drying at 103 ^{o}C	I
~~**Fats and Oils (all)**~~	~~Lead~~	~~AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91~~	~~Atomic absorption spectrophotometry (direct graphite furnace)~~	~~II~~
Fats and Oils (all)	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
~~**Fats and Oils (all)**~~	~~Matter volatile at 105°C~~	~~ISO 662~~	~~Gravimetry (open-drying)~~	~~I~~
Fats and Oils (all)	Moisture and volatile matter	ISO 662	Gravimetry, drying at 105 ^{o}C	I
~~**Fats and Oils (all)**~~	~~Soap content~~	~~BS EN ISO 10539 or AOCS Cc 17-95~~	~~Gravimetry~~	~~I~~
Fats and Oils (all)	Soap content	ISO 10539 / AOCS Cc 17-95	Titrimetry (Colorimetric)	I

~~**Fats and Oils not covered by individual standards**~~	~~Acid value~~	~~ISO 660; or AOCS Cd 3d-63~~	~~Titrimetry~~	~~I~~
Fats and Oils not covered by individual standards	Acidity: acid value	ISO 660 / AOCS Cd 3d-63	Titrimetry	I
~~**Fats and Oils not covered by individual standards**~~	~~Copper and Iron~~	~~AOAC 990.05; or ISO 8294; or AOCS Ca 18b-91~~	~~Atomic absorption spectrophotometry (direct graphite furnace)~~	~~II~~
Fats and Oils not covered by individual standards	Copper and Iron	AOAC 990.05 / ISO 8294 / AOCS Ca 18b-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
~~**Fats and Oils not covered by individual standards**~~	~~Peroxide value~~	~~AOCS Cd 8b-90 ISO 3960~~	~~Titrimetry using iso-octane~~	~~I~~
Fats and Oils not covered by individual standards	Peroxide value	AOCS Cd 8b-90 / ISO 3960	Titrimetry (Colorimetric)	I
~~**Fish oils**~~	~~Acid value~~	~~AOCS Ca 5a-40 AOCS Cd 3d-63 ISO 3960 NMKL 38~~	~~Titration~~	~~I~~
Fish oils	Acidity: acid value	AOCS Ca 5a-40 / AOCS Cd 3d-63 / ISO 660 / NMKL 38	Titrimetry	I
~~**Fish oils**~~	~~Fatty acid composition~~	~~AOCS Ce 1a-13~~	~~Capillary GLC~~	~~III~~
~~**Fish oils**~~	~~Fatty acid composition~~	~~AOCS Ce 2-66~~	~~Preparation of methyl esters by fatty acids~~	~~III~~
Fish oils	Fatty acid composition	AOCS Ce 2-66 and AOCS Ce 1a-13	Gas Chromatography of methyl esters	IV
~~**Fish oils**~~	~~Fatty acid composition~~	~~AOCS Ce 1b-89~~	~~GLC~~	~~III~~
Fish oils	Fatty acid composition	AOCS Ce 1b 89	Gas Chromatography of methyl esters	III
~~**Fish oils**~~	~~Fatty acid composition~~	~~AOCS Ce 2b-11~~	~~Alkali hydrolysis~~	~~III~~
Fish oils	Fatty acid composition	AOCS Ce 2b-11 and AOCS Ce 1i-07 or AOCS Ce 1j-07	Gas Chromatography of methyl esters	III
~~**Fish oils**~~	~~Fatty acid composition~~	~~AOCS Ce 1-07~~	~~Capillary GLC~~	~~III~~
~~**Fish oils**~~	~~Fatty acid composition~~	~~ISO 12966-2~~	~~Gas chromatography~~	~~III~~
~~**Fish oils**~~	~~Fatty acid composition~~	~~ISO 5508~~	~~Gas chromatography~~	~~III~~

Fish oils	Fatty acid composition	ISO 12966-2 and ISO 12966-4 / AOCS Ce 2-66 and AOCS Ce 1i-07	Gas Chromatography of methyl esters	II
Fish oils	p-anisidine	European Pharmacopoeia 2.5.36 / AOCS Cd 18-90 / ISO 6885	Spectrophotometry	I
~~**Fish oils**~~	~~Peroxide value~~	~~AOCS Cd 8b-90 ISO 3960 NMKL 158~~	~~Titration~~	~~I~~
~~**Fish oils**~~	~~Peroxide value~~	~~European Pharmacopoeia 2.5.5 (Part B Iso-octane as solvent)~~	~~Titration~~	~~I~~
Fish oils	Peroxide value	AOCS Cd 8b-90 / ISO 3960 / NMKL 158 / European Pharmacopoeia 2.5.5	Titrimetry (Colorimetric)	I
~~**Fish oils**~~	~~Phospholipids~~	~~USP-FCC 10 2S (Krill oil): Phospholipids Nuclear Magnetic Resonance, Appendix IIC~~	~~NMR Spectroscopy~~	~~I~~
Fish oils	Phospholipids	USP-FCC 11 1S	NMR Spectroscopy	IV
~~**Fish oils**~~	~~Triglycerides~~	~~AOCS Cd 11d-96~~	~~HPLC-ELSD~~	~~III~~
Fish oils	Triglycerides	AOCS Cd 11d-96	Liquid chromatography (ELSD)	II
~~**Fish oils**~~	~~Triglycerides~~	~~European Pharmacopoeia 1352 (Omega-3 acid triglycerides): Oligomers and partial glycerides~~	~~HPLC-RI~~	~~III~~
Fish oils	Triglycerides	European Pharmacopoeia 1352	Liquid chromatography (RI)	III
~~**Fish oils**~~	~~Triglycerides~~	~~USP 40-NF35 (Omega-3 Acid Triglycerides):Content of oligomers and partial glyceride~~	~~HPLC-RI~~	III
Fish oils	Triglycerides	USP 40-NF35	Liquid chromatography (RI)	III
~~**Fish oils**~~	~~Vitamin A~~	~~EN 12823-1 (Determination of vitamin A by high performance liquid chromatograph — Part 1: Measurement of all-E-retinol and 13-Z-retinol~~	~~LC~~	~~III~~
~~**Fish oils**~~	~~Vitamin A~~	~~European Pharmacopoeia Monograph on Cod Liver Oil (Type A), monograph 01/2005:1192, with LC end point 2.2.29~~	~~LC~~	~~III~~
Fish oils	Vitamin A (all-E-retinol and 13-Z-retinol)	EN 12823-1	Liquid chromatography	II
Fish oils	Vitamin A (all-E-retinol)	European Pharmacopoeia 2398	Liquid chromatography	III

~~**Fish oils**~~	~~Vitamin D~~	~~EN 12821 (Determination of vitamin D by high performance liquid chromatography – Measurement of cholecalciferol (D3) or ergocalciferol (D2))~~	~~LC~~	~~III~~
~~**Fish oils**~~	~~Vitamin D~~	~~NMKL 167 (Cholecalciferol (vitamin D3) and Ergocalciferol (vitamin D2)). Determination by HPLC in foodstuffs~~	~~LC~~	~~III~~
Fish oils	Vitamin D (Vitamin D2 and D3)	EN 12821 / NMKL 167	Calculation from vitamin D2 or D3 concentration, preparative column chromatography and liquid chromatography	II
~~**Named Animal Fats**~~	~~Acidity~~	~~ISO 660; or AOCS Cd 3d-63~~	~~Titrimetry~~	~~I~~
Named Animal Fats	Acidity: acid value	ISO 660 / AOCS Cd 3d-63	Titrimetry	I
~~**Named Animal Fats**~~	~~Copper and Iron~~	~~AOAC 990.05; or ISO 8294; or AOCS Ca 18b-91~~	~~Atomic absorption Spectrophotometry (direct graphite furnace)~~	~~II~~
Named Animal Fats	Copper and Iron	AOAC 990.05 / ISO 8294 / AOCS Ca 18b-91	Atomic absorption Spectrophotometry (direct graphite furnace)	II
~~**Named Animal Fats**~~	~~GLC ranges of fatty acid composition~~	~~ISO 5508 and ISO 12966-2; or AOCS Ce 2-66 and Ce 1e-91 or Ce 1f-96~~	~~Gas chromatography of methyl esters~~	~~II~~
Named Animal Fats	Fatty acid composition	ISO 12966-2 and ISO 12966-4 / AOCS Ce 2-66 and Ce 1f-96	Gas Chromatography of methyl esters	II
~~**Named Animal Fats**~~	~~Iodine value (IV)~~	~~ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92~~	~~Wijs-Titrimetry~~	~~I~~
Named Animal Fats	Iodine value	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Titrimetry (Wijs)	I
~~**Named Animal Fats**~~	~~Peroxide value~~	~~AOCS Cd 8b-90; or ISO 3960~~	~~Titrimetry using iso-octane~~	~~I~~
Named Animal Fats	Peroxide value	AOCS Cd 8b-90 / ISO 3960	Titrimetry (Colorimetric)	I
~~**Named Animal Fats**~~	~~Refractive index~~	~~ISO 6320; or AOCS Cc 7-25~~	~~Refractometry~~	~~II~~
Named Animal Fats	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
~~**Named Animal Fats**~~	~~Relative density~~	~~ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95~~	~~Pycnometry~~	~~I~~
Named Animal Fats	Relative density	ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95	Pycnometry	I

~~**Named Animal Fats**~~	~~Saponification value~~	~~ISO 3657; or AOCS Cd 3-25~~	~~Titrimetry~~	~~I~~
Named Animal Fats	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry (Colorimetric)	I
~~**Named Animal Fats**~~	~~Titre~~	~~ISO 935; or AOCS Cc 12-59~~	~~Thermometry~~	~~I~~
Named Animal Fats	Titre	ISO 935	Thermometry	I
Named Animal Fats	Titre	AOCS Cc 12-59	Thermometry	IV
~~**Named Animal Fats**~~	~~Unsaponifiable matter~~	~~ISO 3596; or ISO 18609; or AOCS Ca 6b-53~~	~~Titrimetry after extraction with diethyl ether~~	~~I~~
Named Animal Fats	Unsaponifiable matter	ISO 3596 / ISO 18609 / AOCS Ca 6b-53	Gravimetry, drying at 103 °C and titrimetry (colorimetry)	I
~~**Named Vegetable Oils**~~	~~Acidity~~	~~ISO 660; or AOCS Cd 3d-63~~	~~Titrimetry~~	~~I~~
Named Vegetable Oils	Acidity: acid value	ISO 660 / AOCS Cd 3d-63 / AOCS Ca 5a-40	Titrimetry	I
~~**Named Vegetable Oils**~~	~~Apparent density~~	~~ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95~~	~~Pycnometry~~	~~I~~
Named Vegetable Oils	Apparent density	ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95	Pycnometry	I
Named Vegetable Oils	Baudouin test (modified Villavecchia or sesame seed oil test)	AOCS Cb 2-40	Colour reaction	I
~~**Named Vegetable Oils**~~	~~Carotenoids, total~~	~~BS 684 Section 2.20~~	~~Spectrophotometry~~	~~II~~
Named Vegetable Oils	Carotenoids, total	BS684-2.20	Spectrophotometry	II
~~**Named Vegetable Oils**~~	~~Copper and Iron~~	~~ISO 8294; or AOAC 990.05; or AOCS Ca 18b-91~~	~~AAS~~	~~II~~
Named Animal Fats	Copper and Iron	AOAC 990.05 / ISO 8294 / AOCS Ca 18b-91	Atomic absorption Spectrophotometry (direct graphite furnace)	II
~~**Named Vegetable Oils**~~	~~Crismer value~~	~~AOCS Cb 4-35 and AOCS Ca 5a-40~~	~~Turbidity~~	~~I~~
Named Vegetable Oils	Crismer value	AOCS Cb 4-35 and AOCS Ca 5a-40	Calculation from individual fatty acid composition (gas chromatography of methyl esters) and turbidity	I
~~**Named Vegetable Oils**~~	~~GLC ranges of fatty acid composition~~	~~ISO 5508 and ISO 12966-2; or AOCS Ce 2-66 and Ce 1--62 or Ce 1h-05~~	~~Gas chromatography of methyl esters~~	~~II~~
Named Vegetable Oils	Fatty acid composition	ISO 12966-2 and ISO 12966-4 / AOCS Ce 2-66 and AOCS Ce 1h-05	Gas Chromatography of methyl esters	II

Named Vegetable Oils	Free fatty acids	ISO 660 / AOCS Cd 3d-63 / AOCS Ca 5a-40	Titrimetry	I
Named Vegetable Oils	Halphen test	AOCS Cb 1-25	Colorimetry	I
~~**Named Vegetable Oils**~~	~~Insoluble impurities~~	~~ISO 663~~	~~Gravimetry~~	~~I~~
Named Vegetable Oils	Insoluble impurities	ISO 663	Calculation from total insoluble content in *n*-hexane or light petroleum. Gravimetry, drying at 103 °C	I
~~**Named Vegetable Oils**~~	~~Iodine value (IV)~~	~~ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92; or NMKL 39~~	~~Wijs-Titrimetry~~	~~I~~
Named Vegetable Oils	Iodine value	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Titrimetry (Wijs)	I
~~**Named Vegetable Oils**~~	~~Lead~~	~~AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91~~	~~Atomic Absorption~~	~~II~~
Named Vegetable Oils	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
~~**Named Vegetable Oils**~~	~~Moisture & volatile matter at 105°C~~	~~ISO 662~~	~~Gravimetry~~	~~I~~
Named Vegetable Oils	Moisture and volatile matter	ISO 662	Gravimetry, drying at 105 °C	I
~~**Named Vegetable Oils**~~	~~Peroxide value (PV)~~	~~AOCS Cd 8b-90; or ISO 3960~~	~~Titrimetry~~	~~I~~
Named Vegetable Oils	Peroxide value	AOCS Cd 8b-90 / ISO 3960	Titrimetry (Colorimetric)	I
~~**Named Vegetable Oils**~~	~~Refractive index~~	~~ISO 6320; or AOCS Cc 7-25~~	~~Refractometry~~	~~II~~
Named Vegetable Oils	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
~~**Named Vegetable Oils**~~	~~Reichert value and Polenske value~~	~~AOCS Cd 5-40~~	~~Titrimetry~~	~~I~~
Named Vegetable Oils	Reichert-Meissl value and Polenske value	AOCS Cd 5-40	Calculation from soluble and insoluble volatile fatty acids. Titrimetry (Colorimetric).	I
~~**Named Vegetable Oils**~~	~~Relative density~~	~~ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95~~	~~Pycnometry~~	~~I~~
Named Vegetable Oils	Relative density	ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95	Pycnometry	I
~~**Named Vegetable Oils**~~	~~Saponification value (SV)~~	~~ISO 3657; or AOCS Cd 3-25~~	~~Titrimetry~~	~~I~~
Named Vegetable Oils	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry (Colorimetric)	I
~~**Named Vegetable Oils**~~	~~Slip point~~	~~ISO 6321 for all oils; AOCS Cc 3b-92 for all oils except palm oils; AOCS Cc 3-25 for palm oils only~~	~~Open ended capillary tube~~	~~I~~

Named Vegetable Oils	Slip point	ISO 6321 / AOCS Cc 3b-92 for all oils except palm oils or AOCS Cc 3-25 for palm oils only	Open ended capillary tube	I
~~**Named Vegetable Oils**~~	~~Soap content~~	~~BS 684 Section 2.5 withdrawn for BS EN ISO 10539 or AOCS Cc 17-95~~	~~Gravimetry~~	~~I~~
~~**Named Vegetable Oils**~~	~~Sterol content~~	~~ISO 12228; or AOCS Ch 6-91~~	~~Gas chromatography~~	~~II~~
Named Vegetable Oils	Sterol composition and total sterols	ISO 12228-1 / AOCS Ch 6-91	Thin-layer chromatography and gas chromatography	II
~~**Named Vegetable Oils**~~	~~Tocopherol content~~	~~ISO 9936; or AOCS Ce 8-89~~	~~HPLC~~	~~II~~
Named Vegetable Oils	Tocopherol content	ISO 9936 / AOCS Ce 8-89	Liquid chromatography	II
~~**Named Vegetable Oils**~~	~~Unsaponifiable matter~~	~~ISO 3596; or ISO 18609; or AOCS Ca 6b-53~~	~~Gravimetry~~	~~I~~
Named Vegetable Oils	Unsaponifiable matter	ISO 3596 / ISO 18609 / AOCS Ca 6b-53	Gravimetry, drying at 103 °C and ti-trimetry (colorimetry)	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Absorbency in ultra-violet~~	~~COI/T.20/Doc. No. 19; or ISO 3656; or AOCS Ch 5-91~~	~~Absorption in ultra violet~~	~~II~~
Olive Oils and Olive Pomace Oils	Absorbency in ultra-violet	COI/T.20/Doc. No. 19 / ISO 3656 / AOCS Ch 5-91	Spectrophotometry	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Acidity, free (acid value)~~	~~ISO 660; or AOCS Cd 3d-63~~	~~Titrimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Acidity: acid value	ISO 660 / AOCS Cd 3d-63	Titrimetry	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Alpha-tocopherol~~	~~ISO 9936~~	~~HPLC~~	~~II~~
Olive Oils and Olive Pomace Oils	Alpha-tocopherol	ISO 9936	Liquid chromatography	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Iron and copper~~	~~ISO 8294; or AOAC 990.05~~	~~AAS~~	~~II~~
Olive Oils and Olive Pomace Oils	Copper and Iron	AOAC 990.05 / ISO 8294	Atomic absorption Spectrophotometry (direct graphite furnace)	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Difference between the actual and theoreti-cal ECN 42 triglyceride content~~	~~COI/T.20/Doc. no. 20; or AOCS Ce 5b-89~~	~~Analysis of triglycerides of HPLC and calculation~~	~~I~~
Olive Oils and Olive Pomace Oils	Difference between the actual and theoreti-cal ECN 42 triglyceride content	COI/T.20/Doc. no. 20	Calculation from experimental values of triacylglycerols with equivalent car-bon number 42 (liquid chromatog-raphy) and theoretical value of triacyl-glycerols with an equivalent carbon number of 42 (calculated from the fatty acid composition obtained with gas	I

			chromatography). Calculation from individual fatty acids composition (gas chromatography of methyl esters), triacylglycerols (liquid chromatography) and theoretical composition of triacylglycerols.	
~~**Olive Oils and Olive Pomace Oils**~~	~~Erythrodiol + uvaol~~	~~COI/T.20/Doc.no. 30~~	~~Gas chromatography~~	~~II~~
Olive Oils and Olive Pomace Oils	Erythrodiol and uvaol	COI/T.20/Doc.no. 26	Calculation of relative percentage of the sum of erythrodiol and uvaol with respect to the sum of all sterols, erithrodiol, and uvaol. Thin-layer chromatography and gas chromatography (trimethylsilyl esthers)	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Halogenated solvents, traces~~	~~COI/T.20/Doc. no. 8~~	~~Gas chromatography~~	~~II~~
Olive Oils and Olive Pomace Oils	Tetrachloroethylene, traces	COI/T.20/Doc. no. 8	Gas chromatography	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Insoluble impurities in light petroleum~~	~~ISO 663~~	~~Gravimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Insoluble impurities	ISO 663	Calculation from total insoluble content in *n*-hexane or light petroleum. Gravimetry, drying at 103 ^{o}C	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Iodine value~~	~~ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92; or NMKL 39~~	~~Wijs-Titrimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Iodine value	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Titrimetry (Wijs)	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Lead~~	~~AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91~~	~~AAS~~	~~II~~
Olive Oils and Olive Pomace Oils	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Moisture and volatile matter~~	~~ISO 662~~	~~Gravimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Moisture and volatile matter	ISO 662	Gravimetry, drying at 105 ^{o}C	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Organoleptic characteristics~~	~~COI/T.20/Doc. no. 15~~	~~Panel test~~	~~I~~
Olive Oils and Olive Pomace Oils	Organoleptic characteristics	COI/T.20/Doc. no. 15	Sensory analysis by trained panel	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Peroxide value~~	~~ISO 3960; or AOCS Cd 8b-90~~	~~Titrimetry~~	~~I~~

Olive Oils and Olive Pomace Oils	Peroxide value	AOCS Cd 8b-90 / ISO 3960	Titrimetry (Colorimetric)	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Refractive index~~	~~ISO 6320; or AOCS Cc 7-25~~	~~Refractometry~~	~~II~~
Olive Oils and Olive Pomace Oils	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Relative density~~	~~ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95~~	~~Pycnometry~~	~~I~~
Olive Oils and Olive Pomace Oils	Relative density	ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95	Pycnometry	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Saponification value~~	~~ISO 3657; or AOCS Cd 3-25~~	~~Titrimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry (Colorimetric)	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Sterol composition and total sterols~~	~~COI/T.20/Doc. no. 30; or ISO 12228-2; or AOCS Ch 6-91~~	~~Gas chromatography~~	~~II~~
Olive Oils and Olive Pomace Oils	Sterol composition and total sterols	COI/T.20/Doc. no. 26 / ISO 12228-2 / AOCS Ch 6-91	Thin-layer chromatography and gas chromatography	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Stigmastadienes~~	~~COI/T.20/Doc. no. 11; or ISO 15788-1; or AOCS Cd 26-96~~	~~Gas chromatography~~	~~II~~
Olive Oils and Olive Pomace Oils	Stigmastadienes	COI/T.20/Doc. no. 11 / ISO 15788-1 / AOCS Cd 26-96	Preparative column chromatography and gas chromatography	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Stigmastadienes~~	~~ISO 15788-2~~	~~HPLC~~	~~III~~
Olive Oils and Olive Pomace Oils	Stigmastadienes	ISO 15788-2	Preparative column chromatography and gas chromatography	III
~~**Olive Oils and Olive Pomace Oils**~~	~~Trans fatty acids content~~	~~COI/T.20/Doc no. 17; or ISO 15304; or AOCS Ch 2a-94~~	~~Gas chromatography of methyl esters~~	~~II~~
Olive Oils and Olive Pomace Oils	Trans fatty acids content	COI/T.20/Doc no. 33	Gas chromatography of methyl esters	II
Olive Oils and Olive Pomace Oils	Trans fatty acids content	ISO 15304	Gas chromatography of methyl esters	III
Olive Oils and Olive Pomace Oils	Trans fatty acids content	AOCS Ch 2a-94	Gas chromatography of methyl esters	III
~~**Olive Oils and Olive Pomace Oils**~~	~~Unsaponifiable matter~~	~~ISO 3596; or ISO 18609; or AOCS Ca 6b-53~~	~~Gravimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Unsaponifiable matter	ISO 3596 / ISO 18609 / AOCS Ca 6b-53	Gravimetry, drying at 103 °C and titrimetry (colorimetry)	I

~~**Olive Oils and Olive Pomace Oils**~~	~~Wax content~~	~~COI/T.20/Doc. no. 18; or AOCS Ch 8-02~~	~~Gas chromatography~~	~~II~~
Olive Oils and Olive Pomace Oils	Wax content	COI/T.20/Doc. no. 18 / AOCS Ch 8-02	Gas chromatography	II

APPENDIX II

PART A – METHODS OF ANALYSIS BY PROVISION AND NAMES

*Comments with an asterisk (*) were taken and edited for relevance from CX/MAS 19/40/3 Add.3.

Commodity	Provision	Method	Principle	Type
~~**Olive Oils and Olive Pomace Oils**~~	~~Absorbency in ultra-violet~~	~~COI/T.20/Doc. No. 19; or ISO 3656; or AOCS Ch 5-91~~	~~Absorption in ultra violet~~	~~II~~
Olive Oils and Olive Pomace Oils	Absorbency in ultra-violet	COI/T.20/Doc. No. 19 / ISO 3656 / AOCS Ch 5-91	Spectrophotometry	II
The provision is not listed or identified somewhere else. The three methods are not correctly validated and do not meet all the criteria for validation of methods defined by CCMAS. These methods are Type II methods, though very similar, there are slight differences between the listed methods.				
~~**Fish oils**~~	~~Acid value~~	~~AOCS Ca 5a-40 AOCS Cd 3d-63 ISO 3960 NMKL 38~~	~~Titration~~	~~I~~
Fish oils	Acidity: acid value	AOCS Ca 5a-40 / AOCS Cd 3d-63 / ISO 660 / NMKL 38	Titrimetry	I
~~**Fats and Oils not covered by individual standards**~~	~~Acid value~~	~~ISO 660; or AOCS Cd 3d-63~~	~~Titrimetry~~	~~I~~
Fats and Oils not covered by individual standards	Acidity: acid value	ISO 660 / AOCS Cd 3d-63	Titrimetry	I
~~**Named Animal Fats**~~	~~Acidity~~	~~ISO 660; or AOCS Cd 3d-63~~	~~Titrimetry~~	~~I~~
Named Animal Fats	Acidity: acid value	ISO 660 / AOCS Cd 3d-63	Titrimetry	I
~~**Named Vegetable Oils**~~	~~Acidity~~	~~ISO 660; or AOCS Cd 3d-63~~	~~Titrimetry~~	~~I~~
Named Vegetable Oils	Acidity: acid value	ISO 660 / AOCS Cd 3d-63 / AOCS Ca 5a-40	Titrimetry	I
Named Vegetable Oils	Free fatty acids	ISO 660 / AOCS Cd 3d-63 / AOCS Ca 5a-40	Titrimetry	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Acidity, free (acid value)~~	~~ISO 660; or AOCS Cd 3d-63~~	~~Titrimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Acidity: acid value	ISO 660 / AOCS Cd 3d-63	Titrimetry	I
Reviewer I: ISO 660 is an omnibus method containing all possibilities covered in the two AOCS methods and the NMKL method. Please consider the considerable discussion on this method listing in CCMAS 40 (REP19/MAS)). AOCS recommends this method for proficiency testing in its fish oil series.				
REP19/MAS - Committee on Fats and Oils (CCFO). Methods of analysis for acid value and free fatty acids for virgin palm oil and crude palm kernel oil. CCMAS noted the explanation provided by the Observer from AOCS that the three methods ISO 660 / AOCS Cd 3d-63 / AOCS Ca 5a-40 could be used				

to calculate both acid value and free fatty acids. Each of the methods used an alkali titrant and indicators that change colour as the pH changes. He reported that AOCS 5a-40 used an indicator that comes closest to neutral pH and the same applied to ISO 660. The Observer further noted that while the methods might not fully meet the definition for "identical", from the chemical point of view, they could be considered identical and would provide the same result. Based on this explanation, CCMAS agreed to endorse the three methods for both acid value and free fatty acids.				
Reviewer II suggests the addition of COI/T.20/Doc. No 34 (cold method) for this provision as proposed in COI/T.15/NC No 3/Rev. 14 (trade standards applying to olive oils and olive pomace oils). This method is not endorsed by CCMAS.				
***Method ISO 3960 tests for peroxide value not acid value. ISO 660 tests for acid value and is reviewed here. The methods do not share the same validation data.**				
~~**Olive Oils and Olive Pomace Oils**~~	~~Alpha-tocopherol~~	~~ISO 9936~~	~~HPLC~~	~~II~~
Olive Oils and Olive Pomace Oils	Alpha-tocopherol	ISO 9936	Liquid chromatography	II
~~**Named Vegetable Oils**~~	~~Tocopherol content~~	~~ISO 9936; or AOCS Ce 8-89~~	~~HPLC~~	~~II~~
Named Vegetable Oils	Tocopherol content	ISO 9936 / AOCS Ce 8-89	Liquid chromatography	II
Reviewer I: According to CXS 210-1999 (Table 4) range for total content (tocopherols + tocotrienols) varies between 20 mg/kg for almond oil to 3 720 mg/kg in corn oil. ISO method uses mg/kg, AOCS method uses µg/g as unit. No mentions were found in relation to using a different test portion size if needed because of a higher or lower amount of tocopherol content is found in the sample. The ISO standard method includes information about interlaboratory test, repeatability limit, reproducibility limit. No information about LOD and LOQ, but it is assumed that ISO has performed such determinations. The same applies for the AOCS method, it includes information about interlaboratory test, repeatability limit, reproducibility limit, adopted from an IUPAC study. But no information about LOD and LOQ, but it is assumed that AOCS has performed such determinations. There are differences in the HPLC mobile phase. AOCS uses mixture of isopropanol in hexane (0,5:99,5 v/v). ISO uses tetrahydrofuran in n-heptane (3,85 % by volume). But ISO other possible mobile phases used for the interlaboratory test in 2003. Also, a difference in the stationary phase: ISO gives two alternatives: microparticulate diol or microparticulate silica; AOCS only mentions the silica. The division factors used in both standards are equal.				
Reviewer II: EN 12822:2014, Tocopherol content, Liquid chromatograpy. EWG Chair: no further information and comparison was provided with the ISO and AOCS methods by reviewer II.				
~~**Fats and Oils (all)**~~	~~Arsenic~~	~~AOAC 942.17~~	~~Colorimetry (molybdenum blue)~~	~~III~~
Fats and Oils (all)	Arsenic	AOAC 963.21 and AOAC 942.17	Kjeldahl flask digestion and colorimetry (molybdenum blue)	III
No validation data found for the commodity/provision pairing. Validation data is only for "apple digests" (JAOAC 46, 246(1963)). Although there is a lack of validation data, the committee has previously said. Lack of such (validation) data would not cause a change in the method type or revocation of a method." Additional notes; Note 1: AOAC lists 942.17 as "surplus" and this method may no longer be relevant. If AOAC no longer considers this method as part of the Official Methods of Analysis (OMA) then it should no longer be included in CXS 234. Note 2: AOAC 942.17 requires a sample digestion step and uses the complementary method AOAC 963.21 (Kjeldahl Flask Digestion). 963.21 should be included in CXS 234 as a complementary method. Note 3: Suggest setting performance criteria for this provision/commodity pair.				
~~**Fats and Oils (all)**~~	~~Arsenic~~	~~AOAC 952.13~~	~~Colorimetry (diethyldithiocarbamate)~~	~~II~~

Fats and Oils (all)	Arsenic	AOAC 963.21 and AOAC 952.13	Kjeldahl flask digestion and colorimetry (diethyldithiocarmabate)	II
No validation data found for the commodity/provision pairing. Validation data is only for "apple digests" (JAOAC 46, 246(1963). Although there is a lack of validation data, the committee has previously said " Lack of such (validation) data would not cause a change in the method type or revocation of a method." Additional notes: Note 1: AOAC lists 952.13 as "surplus" and this method may no longer be relevant. If AOAC no longer considers this method as part of the Official Methods of Analysis (OMA) then it should no longer be included in CXS 234. Note 2: AOAC 952.13 requires a sample digestion step and uses the complementary method AOAC 963.21 (Kjeldahl Flask Digestion). 963.21 should be included in CXS 234 as a complementary method. Note 3: suggest to set performance criteria for this provision/commodity pair.				
Fats and Oils (all)	Arsenic	AOAC 986.15	Atomic absorption spectrophotometry	III
No validation data found for the commodity/provision pairing. Although there is a lack of validation data, the committee has previously said " Lack of such (validation) data would not cause a change in the method type or revocation of a method." Suggest setting performance criteria for this provision/commodity pair.				
General comment of second reviewer: EN 15763:2009 (ICP-MS); Elemental Analysis Manual for Food and Related Products, U.S. Food and Drug Administration (Version 1.1, 2015) (ICP-MS).				
Named Vegetable Oils	Baudouin test (modified Villavecchia or sesame seed oil test)	AOCS Cb 2-40	Colour reaction	I
Global industry standard from 1940, validated by use grandfathered. No published (validation) data.				
~~**Fats and oils**~~	~~Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, & propyl gallate~~	~~AOAC 983.15; or AOCS Ce 6-86~~	~~Liquid chromatography~~	~~II~~
Fats and oils	Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, and propyl gallate	AOAC 983.15	Liquid chromatography	II
Fats and oils	Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, and propyl gallate	AOCS Ce 6-86	Liquid chromatography	III
Main conclusion of reviewer is displayed. Collaborative trial data for three matrices provided (in AOAC method); Collaborative trial data for Oils & Lard. While verified appropriately for Final Action in 1994. The collaborative data provided does not provide all the 'generalized analytical Characteristics' mentioned in PM (although they may be obtainable form the original data). For example, an applicable concentration range and not a LOD/LOQ is provided. The methods are not identical or complementary – very similar but suspect that while the AOAC method was revised in accordance with J. AOAC Int. 76, 765(1993), to include scope broadened with 2 additional antioxidants plus the Butter oils matrix; the mobile phase in AOAC is a variation allow in AOCS method, injection volume different, and chromatography example is different. We suspect the AOCS method is adopted from the previous version of the AOAC and the previous IUPAC Method 2.642 Determination of antioxidants by high performance liquid chromatography (1987).				

The methods are not 'identical' so both cannot be endorsed as Type II, but if one was to be endorsed, the more recently revised and more comprehensive AOAC 983.15 would be chosen. As commented Blumhorst et al.[1] the AOCS Official Method Ce 6-86 "Antioxidants, Liquid Chromatographic Method" was originally developed to confirm the correct antioxidant was added at the specified concentration to refined oils. But BHT recoveries and estimated LOQ do not meet the Codex Criteria (requiring LOQ ≤ 15 mg/kg and % Rec. within 90 – 107%). This Blumhorst et al. work, showed quantification of tert-butylhydroquinone (TBHQ) in crude canola/rapeseed oil using liquid chromatography (LC) with ultraviolet (UV) detection was compromised by an interfering peak. While Collison (2019)[2] suggested AOCS Method Ce 6-86 was written at a time when HPLC was not a well-developed technique. It is based on a method from the late 1970's, requires the use of very low performance columns and poor choice of mobile phases. The method has been updated to allow use of modern columns and a section has been added to describe how to use the method to determine the absence of antioxidants as well as their quantitation. Using the AOCS Ce 6-86 method to briefly discuss the need for AOCS methods updates. This would suggest the method lacks sufficient specificity, and that in accordance with 'Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results', for an LC-UV/VIS (single wavelength) method at least two different chromatographic systems or second, independent detection method is used. If a suitable type II reference method could be identified, the Method AOAC 983.15 and AOCS Ce 6-86 could be retained as Type III.				
***AOCS Ce 6-86 Reference # 2 should be Page, B.D., J. Assoc. Off. Anal. Chem. 66:729 (1983), not From Page, B.D., J. Assoc. Off. Anal. Chem. 66:729 (1983) and Reference # 3 should be Horwitz, W., J. Assoc. Off. Anal. Chem. 67:432 (1984), not Horwitz, W., Ibid. 67:432 (1984).**				
~~**Fats and Oils not covered by individual standards**~~	~~Copper and Iron~~	~~AOAC 990.05; or ISO 8294; or AOCS Ca 18b-91~~	~~Atomic absorption spectrophotometry (direct graphite furnace)~~	~~II~~
Fats and Oils not covered by individual standards	Copper and Iron	AOAC 990.05 / ISO 8294 / AOCS Ca 18b-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
~~**Named Animal Fats**~~	~~Copper and Iron~~	~~AOAC 990.05; or ISO 8294; or AOCS Ca 18b-91~~	~~Atomic absorption Spectrophotometry (direct graphite furnace)~~	~~II~~
Named Animal Fats	Copper and Iron	AOAC 990.05 / ISO 8294 / AOCS Ca 18b-91	Atomic absorption Spectrophotometry (direct graphite furnace)	II
~~**Named Vegetable Oils**~~	~~Copper and Iron~~	~~ISO 8294; or AOAC 990.05; or AOCS Ca 18b-91~~	~~AAS~~	~~II~~
Named Vegetable Fats	Copper and Iron	AOAC 990.05 / ISO 8294 / AOCS Ca 18b-91	Atomic absorption Spectrophotometry (direct graphite furnace)	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Iron and copper~~	~~ISO 8294; or AOAC 990.05~~	~~AAS~~	~~II~~
Olive Oils and Olive Pomace Oils	Copper and Iron	AOAC 990.05 / ISO 8294	Atomic absorption Spectrophotometry (direct graphite furnace)	II
Methods validated with 1 fat (cocoa butter) and 2 oils (soya bean and groundnut). No animal fat, fish oil or olive oils were used in the collaborative validation study. All three methods (AOAC, ISO, AOCS) share validation data (Pure & Appl. Chem., Vol. 60, No. 6, pp. 893-900, 1988.). Suggest setting performance criteria for these methods.				
***The methods are Identical as they all derivate from same initial method (IUPAC2.631) and they are based on the same set of validation data.**				

[1] Blumhorst et al. Identification of a TBHQ-Interfering Peak in Crude Canola Oil Using AOCS Official Method Ce 6-86 and its Chromatographic Resolution J Am Oil Chem Soc (2017) 94:1323–1328

[2] Mark W. Collison* (2019), Presentation at 2019 AOCS ANNUAL MEETING & EXPO Update of AOCS Ce 6-86 Antioxidants Method and an overview of the need for methods updates.

AOCS general comment on Copper and Iron provision: AOCS recently adopted Official Method Ca 17a-18, "Determination of Trace Elements in Oil by Inductively Coupled Plasma Optical Emission Spectroscopy (22-Element Method),".				
General comment of second reviewer: EN 15763:2009 (ICP-MS); Elemental Analysis Manual for Food and Related Products, U.S. Food and Drug Administration (Version 1.1, 2015) (ICP-MS).				
~~**Named Vegetable Oils**~~	~~Crismer value~~	~~AOCS Cb 4-35 and AOCS Ca 5a-40~~	~~Turbidity~~	~~I~~
Named Vegetable Oils	Crismer value	AOCS Cb 4-35 and AOCS Ca 5a-40	Calculation from individual fatty acid composition (gas chromatography of methyl esters) and turbidity	I
Global industry standard from 1935, validated by use – grandfathered. No published (validation) data.				
~~**Olive Oils and Olive Pomace Oils**~~	~~Difference between the actual and theoretical ECN 42 triglyceride content~~	~~COI/T.20/Doc. no. 20; or AOCS Ce 5b-89~~	~~Analysis of triglycerides of HPLC and calculation~~	~~I~~
Olive Oils and Olive Pomace Oils	Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20	Calculation from experimental values of triacylglycerols with equivalent carbon number 42 (liquid chromatography) and theoretical value of triacylglycerols with an equivalent carbon number of 42 (calculated from the fatty acid composition obtained with gas chromatography). Calculation from individual fatty acids composition (gas chromatography of methyl esters), triacylglycerols (liquid chromatography) and theoretical composition of triacylglycerols	I
Reviewer I and EWG Chair: In AOCS Ce 5b-89 the calculation for the provision is not present.				
Reviewer II: COI method Scope: "Determination of the absolute difference between the experimental values of triacylglycerols (TAGs) with equivalent carbon number 42 (ECN42HPLC) obtained by determination in the oil by high performance liquid chromatography and the theoretical value of TAGs with an equivalent carbon number of 42 (ECN 42 theoretical) calculated from the fatty acid composition."				
~~**Olive Oils and Olive Pomace Oils**~~	~~Erythrodiol + uvaol~~	~~COI/T.20/Doc.no. 30~~	~~Gas chromatography~~	~~II~~
Olive Oils and Olive Pomace Oils	Erythrodiol and uvaol	COI/T.20/Doc.no. 26	Calculation of relative percentage of the sum of erythrodiol and uvaol	II

			with respect to the sum of all sterols, erithrodiol, and uvaol. Thin-layer chromatography and gas chromatography (trimethylsilyl esthers)	
Reviewer: According to COI/T.15/NC No 3/Rev. 14 this method has been updated to COI/T.20/Doc. no. 26 (Rev.4 2018 DETERMINATION OF THE STEROL COMPOSITION AND CONTENT AND ALCOHOLIC COMPOUNDS BY CAPILLARY GAS CHROMATOGRAPHY).				
EWG Chair: Validation data present for all relevant analytes				
***Reference ISO 5725-5 is listed as 1994 but has since been updated to 1998.**				
~~**Fat spreads and blended spreads**~~	~~Fat content~~	~~ISO 17189 | IDF 194~~	~~Gravimetry~~	~~I~~
Fat spreads and blended spreads	Total fat	ISO 17189 | IDF 194	Gravimetry. Direct determination of fat using solvent extraction.	I
EWG Chair: Validation data for Butter, Dairy spread and vegetable oil spread present. Used terminology of EWG dairy.				
~~**Fish oils**~~	~~Fatty acid composition~~	~~AOCS Ce 1a-13~~	~~Capillary GLC~~	~~III~~
~~**Fish oils**~~	~~Fatty acid composition~~	~~AOCS Ce 2-66~~	~~Preparation of methyl esters by fatty acids~~	~~III~~
Fish oils	Fatty acid composition	AOCS Ce 2-66 and AOCS Ce 1a-13	Gas Chromatography of methyl esters	IV
Main conclusions of reviewer I, part I. Complementary methods - AOCS Ce 1a-13 (Determination of Fatty Acids in edible oils and fats by capillary GLC) and Ce 2-66 (Preparation of Methyl Esters of Fatty Acids). A number of Commodity – Fish Oils and Provision - Fatty Acid composition methods are provided in CXS 234:2019 but all currently endorsed as Type III. Thus, a type II method requires endorsement, but in the absence of Method Performance or SLV/MLT data we cannot recommend the complementary 'AOCS Ce 1a-13 and AOCS Ce 2-66'. Further with the endorsement of a suitable Type II method(s), as 'AOCS Ce 1a-13 and AOCS Ce 2-66' has been used traditionally, this method(s) could be considered a Type IV.				
Main conclusions of reviewer I, part II. Not properly validated. Method Performance or SLV/MLT data provided for this method, but states in Note 8 "representative values may be found in the tables contained in AOCS Ce 1h-05, Ce 1i-07 and Ce Ij-07". However, the methods specified for representative values are not identical to 'AOCS Ce 1a-13 and AOCS Ce 2-66' having very different internal standards etc. The AOCS Ce 1i-07 is an alternative in CXS 234 and supported by "Statistical Analysis of the Collaborative Study in Support of the Official Method AOCS Ce 1i-07: Determination of Saturated, cis-Monounsaturated and cis-Polyunsaturated Fatty Acids in Marine and Other Oils Containing Long Chain Polyunsaturated Fatty Acids by Capillary GLC				
Reviewer II: Ce 1a-13 provides relatively generic guidance related to GC analysis and is identified for analysis of vegetable or non-ruminant oils and fats, so it may not be a good method to be quoted unless paired with a method developed to address the matrix (i.e., Ce 1i-7 for fish oils). Perhaps the combination of Ce 2-66 and Ce 1a-13 could be removed from consideration for fish oils given that we would be required to include 2 analytical methods (Ce 1a-13 and Ce 1i-07) to get the result for fish oils.				
~~**Fish oils**~~	~~Fatty acid composition~~	~~AOCS Ce 1b-89~~	~~GLC~~	~~III~~

Fish oils	Fatty acid composition	AOCS Ce 1b 89	Gas Chromatography of methyl esters	III
Main conclusion of reviewer I: The CXS 329-2017 Table 1 requires coverage of C14:0 to C22:6 (n-3) fatty acids and range of concentration from ND (defined as <0.05% which presumably is the required LOD concentration) up to 42.5% expressed as percentage of total fatty acid. However, AOCS Ce 1a-13 and AOCS Ce 2-66 provides no Method Performance SLV/MLT data, simply stating "representative values may be found in the tables contained in AOCS Ce 1h-05, Ce 1i-07 and Ce lj-07", which is not identical to 'AOCS Ce 1a-13 and AOCS Ce 2-66' having very different internal standards etc. A number of Commodity – Fish Oils and Provision - Fatty Acid composition methods are provided in CXS 234:2019 but currently all endorsed as Type III. Thus a Type II method needs to be selected and endorsement, but as the AOCS Ce 1b-89 Method Performance /MLT data doesn't cover the Recovery range, provide certified reference material accuracy data and specifically demonstrate compliance with the Detection limit specified in CXS 329-2017 Table 1 – it is recommended to be retained as a Type III.				
Reviewer II: Ce 1b-89 suggest *replacing* this method with Ce 1i-07 given the relative summary statistics, etc.				
The method is properly validated, better than the 'AOCS Ce 1a-13 and AOCS Ce 2-66' alternative.				
~~**Fish oils**~~	~~Fatty acid composition~~	~~AOCS Ce 2b-11~~	~~Alkali hydrolysis~~	~~III~~
Fish oils	Fatty acid composition	AOCS Ce 2b-11 and AOCS Ce 1i-07 or AOCS Ce 1j-07	Gas Chromatography of methyl esters	III
Reviewer I: Requires a 'Complementary' instrumental method, which for marine oils would be AOCS Ce 1i-07, but for the MLT data provided is for Ce 1j-07. The AOCS Ce 2b-11 and Ce 1j-07 Method performance/MLT data available provided with AOCS Ce 2b-11 is not convincing, particularly when AOCS Ce 2b-11 suggests a 'Complementary' instrumental method for marine oils to be AOCS Ce 1i-07.				
Reviewer II: Ce 2b-11 and Ce 1i-07 be considered Type III as Ce 2b-11 is a method for the preparation of methyl esters in finished foods so it may not be as good a choice as the above noted combination and Ce 1i-07 is used for the analysis, there are collaborative study data. Ce 2b-11 and Ce 1j-07: Note that Ce 1j-07 is identified for analysis of unknown, dairy or of ruminant origin rather than fish oils so perhaps it should be considered as a Type III not a Type II. There are collaborative study data for DHA/EPA.				
~~**Fish oils**~~	~~Fatty acid composition~~	~~AOCS Ce 1-07~~	~~Capillary GLC~~	~~III~~
~~**Fish oils**~~	~~Fatty acid composition~~	~~ISO 12966-2~~	~~Gas chromatography~~	~~III~~
~~**Fish oils**~~	~~Fatty acid composition~~	~~ISO 5508~~	~~Gas chromatography~~	~~III~~
Fish oils	Fatty acid composition	ISO 12966-2 and ISO 12966-4 / AOCS Ce 2-66 and AOCS Ce 1i-07	Gas Chromatography of methyl esters	II
Reviewer II: Ce 2-66 and Ce 1i-07 be considered Type II as Ce 2-66 is a method for the preparation of methyl esters in all oils (with the exception of milk fats), combined with Ce 1i-07 where there are extensive collaborative study data relevant specifically to fish oils.				
EWG Chair: Up for discussion, endorsing type II methods concerning Fish oils. Consider comment on AOCS Ce 2b-11 and AOCS Ce2c-11 for FAME preparation. ISO 12966-3 is a fast alternative for ISO 12966-2. Ce 1i-07 uses supelcowax, ISO 12966-4 uses SP-2560 or CP-Sil 88. ISO 5508 was replaced by ISO 12966-4. ISO 12966-4 does contain validation data on fish oils tested on the SP-2560 column. Ce 1i-07 contains explicit validation data for fish oil, EE, Tuna Oil, Ara, DHA. ISO 12966-4 contains validation data per kind of fatty acid (SAFA, MUFA, PUFA) not specifying fish oils.				
Reviewer I additional comments. Actually Masson et al. (2015)[3] highlighted that Fish oil FAs are complex to separate and to identify, considering their different polarity related to chain length, double bonds from 2 to 6 and positional and geometric isomers with the same carbon number. So undertook work aimed to improve the AOCS Ce 1j-07 Method for the FA composition of a mixture of soybean and sunflower oil, fish oil, and butterfat, using a				

[3] Masson et al. (2015), Fatty acid composition of soybean/sunflower mix oil, fish oil and butterfat applying the AOCS Ce 1j-07 method with a modified temperature program, Grasas y Aceites, Vol 66, No 1 (2015)

modified temperature program, included Reference Standard GLC 463 (contains 52 FAMEs with a wide spectrum of FAs from 4:0 to 24:1) and achieved good results for BCR-163 certified reference material. While Santercole el al. (2012)[4], concluded that use of poly(ethylene glycol) (PEG) columns do not resolve most unsaturated FA geometric isomers and better results were obtained using the SP-2560 capillary column than the Supercowax -10, 30 m indicated in the Official AOCS Method Ce 1i-07 (AOCS, 2007a) for marine oils. While the Li et al. (2019)[5] study compared American Oil Chemists' Society (AOCS) Official Methods Ce 2b-11 and Ce 2c-11 for determining EPA and DHA in foods and dietary supplements and found that AOCS Ce 2c-11 produces significantly higher analyzed values, which could be attributed to a more comprehensive breakdown of the sample matrix and derivatization of fatty acids. Their subsequent food matrix extension validation of AOCS Ce 2c-11 demonstrated that the method produces true, accurate, sensitive, and precise determinations of EPA, DHA, and total omega-3 PUFA in foods and dietary supplements containing added marine oil, including those formulated with emulsified and microencapsulated oils.				
***AOCS Ce 1-07 should be listed as Ce 1i-07; AOCS Ce 2b-11 Reference #5 should be listed J. Chromatogr. Sci. not J. Chromat. Sci.; AOCS Ce 1a-13 Reference 7 is actually 2014: ISO 12966-1:2014 and not 2013, ISO/DIS 12966-1.**				
~~**Named Vegetable Oils**~~	~~GLC ranges of fatty acid composition~~	~~ISO 5508 and ISO 12966-2; or AOCS Ce 2-66 and Ce 1--62 or Ce 1h-05~~	~~Gas chromatography of methyl esters~~	~~II~~
Named Vegetable Oils	Fatty acid composition	ISO 12966-2 and ISO 12966-4 / AOCS Ce 2-66 and AOCS Ce 1h-05	Gas Chromatography of methyl esters	II
Ce 1-62 does not contain validation data, whilst Ce 1h-05 contains elaborated validation data on vegetable oils and non-ruminant animal oils and fats. It is recommended to remove the general Ce 1-62 from CXS 234 or retain it as at type IV method (in combination with AOCS Ce 2-66).				
~~**Named Animal Fats**~~	~~GLC ranges of fatty acid composition~~	~~ISO 5508 and ISO 12966-2; or AOCS Ce 2-66 and Ce 1e-91 or Ce 1f-96~~	~~Gas chromatography of methyl esters~~	~~II~~
Named Animal Fats	Fatty acid composition	ISO 12966-2 and ISO 12966-4 / AOCS Ce 2-66 and Ce 1f-96	Gas Chromatography of methyl esters	II
AOCS Ce 1e-91 scope is applicable to FAMEs obtained from vegetable oils and fats. Animal fats are not mentioned in the method scope, therefore this method should be Type III (validation data is available). AOCS Ce 1f-96 "is specially designed to evaluate, by a single capillary GLC procedure, the level of trans isomers as formed during (high-temperature) refining or during hydrogenation of vegetable oils or fats (see Notes, 1 and 2). The method may also be used to report all other fatty acids, for example to obtain saturated fatty acid (SAFA), monounsaturated fatty acid (MUFA), and polyunsaturated fatty acid (PUFA) levels from the same sample and same analysis."				
~~**Olive Oils and Olive Pomace Oils**~~	~~Halogenated solvents, traces~~	~~COI/T.20/Doc. no. 8~~	~~Gas chromatography~~	~~II~~
Olive Oils and Olive Pomace Oils	Tetrachloroethylene, traces	COI/T.20/Doc. no. 8	Gas chromatography	II
Reviewer: The method only partially tests for the provision, the method is only partially validated and is probably validated for olive oil for tetrachloroethylene only. Section "5.3 Halogenated solvents" of CXS_033e_2015 reports that these commodities shall have "maximum content of each halogenated solvent of 0.1 mg/kg, maximum content of the sum of all halogenated solvents 0.2 mg/kg". The method under evaluation measures the content in				

[4] Santercole et al. (2012), Comparison of separations of fatty acids from fish products using a 30-m Supelcowax-10 and a 100-m SP-2560 column. Lipids 47, 329–344. http://dx.doi.org/10.1007/s11745-011-3645-y.

[5] Li etal. (2019), Matrix Extension Validation of AOCS Ce 2c-11 for Omega-3 Polyunsaturated Fatty Acids in Conventional Foods and Dietary Supplements Containing Added Marine Oil, Journal of the American Oil Chemists' Society 96(5) February 2019.

only one halogenated solvent, tetrachloroethylene. Note 8.1 indicates "the determination of carbon tetrachloride, 1,1,1 trichloroethane, dibromochloromethane and bromoform has also been found satisfactory". No directions are given on how to add these analytes to the method. Also the term "satisfactory" is qualitative and no validation/performance data are provided. More halogenated solvents may be present in these commodities other than tetrachloroethylene and the other halogenated solvents reported in Note 8.1, which cannot be measured applying this method unless it is extended. Therefore, the method may not provide the accurate measurement of "sum of all halogenated solvents".				
EWG Chair: The provision named in COI/T.20/Doc. No. 8 is (partially) validated for tetrachloroethylene. As this is the only method in CXS 234, it is Type II. This leaves the provision "Halogenated solvents, traces" not covered by any method in CXS 234. It is suggested that CCFO provides CCMAS with a method which covers this provision. In COI/T.15/NC No.3/Rev. 14 Trade standard applying to olive oils and olive pomace oils section 11.19: "Detection of traces of halogenated solvents: According to COI/T.20/Doc. No 8, "Determination of tetrachloroethylene in olive oils by gas-liquid chromatography".				
Named vegetable oils	Halphen test	AOCS Cb 1-25	Colorimetry	I
No validation data are provided. This is an old legacy method.				
~~**Fats and Oils (all)**~~	~~Insoluble impurities~~	~~ISO 663~~	~~Gravimetry~~	~~I~~
Fats and Oils (all)	Insoluble impurities	ISO 663	Calculation from total insoluble content in *n*-hexane or light petroleum. Gravimetry, drying at 103 °C	I
~~**Named Vegetable Oils**~~	~~Insoluble impurities~~	~~ISO 663~~	~~Gravimetry~~	~~I~~
Named Vegetable Oils	Insoluble impurities	ISO 663	Calculation from total insoluble content in *n*-hexane or light petroleum. Gravimetry, drying at 103 °C	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Insoluble impurities in light petroleum~~	~~ISO 663~~	~~Gravimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Insoluble impurities	ISO 663	Calculation from total insoluble content in *n*-hexane or light petroleum. Gravimetry, drying at 103 °C	I
***Specific usage of light petroleum for olive oils and olive pomace oils is not mentioned in the method.**				
~~**Olive Oils and Olive Pomace Oils**~~	~~Iodine value~~	~~ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92; or NMKL 39~~	~~Wijs-Titrimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Iodine value	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Titrimetry (Wijs)	I
~~**Named Animal Fats**~~	~~Iodine value (IV)~~	~~ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92~~	~~Wijs-Titrimetry~~	~~I~~
Named Animal Fats	Iodine value	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Titrimetry (Wijs)	I

STAN 211-1999 does not specify NMKL 39, suggestion to add this method.				
~~**Named Vegetable Oils**~~	~~Iodine value (IV)~~	~~ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92; or NMKL 39~~	~~Wijs-Titrimetry~~	~~I~~
Named Vegetable Oils	Iodine value	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Titrimetry (Wijs)	I
EWG Chair: The commodity "Fats and Oils (all)" is not specified for this provision.				
~~**Fats and Oils (all)**~~	~~Lead~~	~~AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91~~	~~Atomic absorption spectrophotometry (direct graphite furnace)~~	~~II~~
Fats and Oils (all)	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
~~**Named Vegetable Oils**~~	~~Lead~~	~~AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91~~	~~Atomic Absorption~~	~~II~~
Named Vegetable Oils	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Lead~~	~~AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91~~	~~AAS~~	~~II~~
Olive Oils and Olive Pomace Oils	Lead	AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Identical methods which have the same principle, the same chemicals in the same concentrations, in the same procedure/sequence and the same measuring equipment but are published by different SDOs and written in differing styles (even though very some minor difference which I believe have been individually included with various revisions). All appear to have been derived re-published from 'Standard Methods for the Analysis of Oils, Fats and Derivatives, International Union of Pure and Applied Chemistry, 7th edn., Blackwell Scientific Publications, 1987, IUPAC Method 2.632. The methods are based on the same validation data of the IUPAC method.				
Method validated with 1 fat (cocoa butter) and 1 oil (soy bean oil). No animal fat or olive oils were used in the collaborative validation study. All three methods (AOAC, ISO, AOCS) share validation data (Pure & Appl. Chem., Vol. 63, No. 8, pp. 1183-1190, 1991.). Suggest to CCMAS to set performance criteria.				
General comment of second reviewer: EN 15763:2009 (ICP-MS); Elemental Analysis Manual for Food and Related Products, U.S. Food and Drug Administration (Version 1.1, 2015) (ICP-MS).				
~~**Fats and Oils (all)**~~	~~Matter volatile at 105°C~~	~~ISO 662~~	~~Gravimetry (open-drying)~~	~~I~~
Fats and Oils (all)	Moisture and volatile matter	ISO 662	Gravimetry, drying at 105 °C	I
~~**Named Vegetable Oils**~~	~~Moisture & volatile matter at 105°C~~	~~ISO 662~~	~~Gravimetry~~	~~I~~
Named Vegetable Oils	Moisture and volatile matter	ISO 662	Gravimetry, drying at 105 °C	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Moisture and volatile matter~~	~~ISO 662~~	~~Gravimetry~~	~~I~~

Olive Oils and Olive Pomace Oils	Moisture and volatile matter	ISO 662	Gravimetry, drying at 105 ºC	I
***Reference ISO 5725:1986 has since been updated to ISO 5725:1994/1998.**				
~~**Olive Oils and Olive Pomace Oils**~~	~~Organoleptic characteristics~~	~~COI/T.20/Doc. no. 15~~	~~Panel test~~	~~I~~
Olive Oils and Olive Pomace Oils	Organoleptic characteristics	COI/T.20/Doc. no. 15	Sensory analysis by trained panel	I
The standard does not present validation information				
***Method is only for VIRGIN olive oils. Reference COI/T.28/Doc. No. 1 September 2007 has since been revised to COI/T.28/Doc. No.1 Rev. 3 2018. Reference COI/T.20/Doc. No.14 Rev.3 November 2011 has since been revised to COI/T.20/Doc. No. 14 Rev. 5 June 2018. Reference ISO/IEC 17025:05 has since been revised to ISO/IEC 17025:2017.**				
Fish oils	p-anisidine	European Pharmacopoeia 2.5.36 / AOCS Cd 18-90 / ISO 6885	Spectrophotometry	I
ISO 6885 contains validation data for fish oils, AOCS Cd 18-90 contains validation data for crude rapeseed and refined palm oil, European Pharmacopoeia 2.5.36 does not contain any validation data.				
***Methods do not share the same validation data. AOCS Cd 18-90 reference #3 should be listed as 51:I7, not 51:17.**				
~~**Fats and Oils not covered by individual standards**~~	~~Peroxide value~~	~~AOCS Cd 8b-90 ISO 3960~~	~~Titrimetry using iso-octane~~	~~I~~
Fats and Oils not covered by individual standards	Peroxide value	AOCS Cd 8b-90 / ISO 3960	Titrimetry (Colorimetric)	I
~~**Named Animal Fats**~~	~~Peroxide value~~	~~AOCS Cd 8b-90; or ISO 3960~~	~~Titrimetry using iso-octane~~	~~I~~
Named Animal Fats	Peroxide value	AOCS Cd 8b-90 / ISO 3960	Titrimetry (Colorimetric)	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Peroxide value~~	~~ISO 3960; or AOCS Cd 8b-90~~	~~Titrimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Peroxide value	AOCS Cd 8b-90 / ISO 3960	Titrimetry (Colorimetric)	I
Reviewer II: proposes COI/T.20/DOC. No. 35. This method has not been endorsed by CCMAS for this provision and commodity (EWG Chair).				
Reviewer I: COI/T.20/Doc. No 35/Rev.1 uses chloroform for the analysis of olive and olive pomace oils and is not considered equivalent or identical to the AOCS and ISO method.				
~~**Named Vegetable Oils**~~	~~Peroxide value (PV)~~	~~AOCS Cd 8b-90; or ISO 3960~~	~~Titrimetry~~	~~I~~
Named Vegetable Oils	Peroxide value	AOCS Cd 8b-90 / ISO 3960	Titrimetry (Colorimetric)	I
Global industry standard; methods use different versions of collaborative study data.				

~~**Fish oils**~~	~~Peroxide value~~	~~AOCS Cd 8b-90 ISO 3960 NMKL 158~~	~~Titration~~	~~I~~
~~**Fish oils**~~	~~Peroxide value~~	~~European Pharmacopoeia 2.5.5 (Part B Iso-octane as solvent)~~	~~Titration~~	~~I~~
Fish oils	Peroxide value	AOCS Cd 8b-90 / ISO 3960 / NMKL 158 / European Pharmacopoeia 2.5.5	Titrimetry (Colorimetric)	I
European Pharmacopoeia 2.5.5 Part B utilizes trimethylpentane instead of iso-octace, still this method is considered identical. Only identical type I methods should be endorsed.				
***For AOCS Cd 8b-90, define "normal fats and oils" in scope of the method. The methods do not share the same validation data.**				

~~**Fish oils**~~	~~Phospholipids~~	~~USP-FCC 10 2S (Krill oil): Phospholipids Nuclear Magnetic Resonance, Appendix IIC~~	~~NMR Spectroscopy~~	~~I~~
Fish oils	Phospholipids	USP-FCC 11 1S	NMR Spectroscopy	IV
Method requires suitable equipment and analysis. Global experience may be limited. Validation data published method in FCC and some study data in an AOCS publication. USP-FCC data are considered intellectual property and not generally accessible. NMR is considered to be a primary technology ,so the Method Type is not Type I - Method could be Type II (as there is no other) or Type IV (limited capacity and still under development).				
*** Method as received by USP is listed as USP-FCC 11 1S not USP-FCC 10 2S. Method does not contain validation or precision data.**				

~~**Named Animal Fats**~~	~~Refractive index~~	~~ISO 6320; or AOCS Cc 7-25~~	~~Refractometry~~	~~II~~
Named Animal Fats	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
~~**Named Vegetable Oils**~~	~~Refractive index~~	~~ISO 6320; or AOCS Cc 7-25~~	~~Refractometry~~	~~II~~
Named Vegetable Oils	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
~~**Olive Oils and Olive Pomace Oils**~~	~~Refractive index~~	~~ISO 6320; or AOCS Cc 7-25~~	~~Refractometry~~	~~II~~
Olive Oils and Olive Pomace Oils	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
In CXS 234 for other uses of refractometry it is being used to measure “soluble solids” which is not a well-defined compound. Therefore, the result is not really quantitating a known analyte(s) but simply using a physical measuring and applying it to that group (soluble solids). That is similar to ash or moisture. In the use for AOCS Cc 7-25 the refractive index is being measured and reference is used to assist in that. There are other methods that could measure the refractive index where the answer isn’t dependent on the technique, therefore it is Type II.				

~~**Named Vegetable Oils**~~	~~Reichert value and Polenske value~~	~~AOCS Cd 5-40~~	~~Titrimetry~~	~~I~~
Named Vegetable Oils	Reichert-Meissl value and Polenske value	AOCS Cd 5-40	Calculation from soluble and insoluble volatile fatty acids. Titrimetry (Colorimetric).	I

Comment of reviewer: Why is the Kirschner value not added here (i.e. butyric acid), next to the Reichert-Meissl (soluble volatile fatty acids) and Polenske value (insoluble volatile fatty acids)?				
~~**Named Animal Fats**~~	~~Saponification value~~	~~ISO 3657; or AOCS Cd 3-25~~	~~Titrimetry~~	~~I~~
Named Animal Fats	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry (Colorimetric)	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Saponification value~~	~~ISO 3657; or AOCS Cd 3-25~~	~~Titrimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry (Colorimetric)	I
~~**Named Vegetable Oils**~~	~~Saponification value (SV)~~	~~ISO 3657; or AOCS Cd 3-25~~	~~Titrimetry~~	~~I~~
Named Vegetable Oils	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry (Colorimetric)	I
The two methods should be considered identical. They are based on the same principle and use same chemicals. There are minor negligible differences in the two procedures, the concentration of the KOH solution in ethanol is slightly different but it does not affect results. The two methodologies are published by different SDOs and written in different styles. Validation of both methods is based on a collaborative study conducted by DIN in 2000. The results of that study were included in both methods, implying there is not difference between the two procedures.				
~~**Named Vegetable Oils**~~	~~Slip point~~	~~ISO 6321 for all oils; AOCS Cc 3b-92 for all oils except palm oils; AOCS Cc 3-25 for palm oils only~~	~~Open ended capillary tube~~	~~I~~
Named Vegetable Oils	Slip point	ISO 6321 / AOCS Cc 3b-92 for all oils except palm oils or AOCS Cc 3-25 for palm oils only	Open ended capillary tube	I
~~**Fats and Oils (all)**~~	~~Soap content~~	~~BS EN ISO 10539 or AOCS Cc 17-95~~	~~Gravimetry~~	~~I~~
~~**Named Vegetable Oils**~~	~~Soap content~~	~~BS 684 Section 2.5 withdrawn for BS EN ISO 10539 or AOCS Cc 17-95~~	~~Gravimetry~~	~~I~~
Fats and Oils (all)	Soap content	ISO 10539 / AOCS Cc 17-95	Titrimetry (Colorimetric)	I
ISO 10539 has validation data, AOCS Cc refers to "A study among seven industrial organizations indicated that this method is suitable only for refined oils. Yukagaku (Japan) 39:1056 (1990)". As both methods have been around for a long time, methods are considered identical, therefore Type I. It is unclear why a separate line has been created for "Named vegetable oils".				
***AOCS method Cc 17-95 is only applicable to refined oils and not crude oils. AOCS method Cc 17-95 does not have validation data but does reference Codex Alimentarius method CAC/RM 13-1969 which could not be located.**				
~~**Olive Oils and Olive Pomace Oils**~~	~~Sterol composition and total sterols~~	~~COI/T.20/Doc. no. 30; or ISO 12228-2; or AOCS Ch 6-91~~	~~Gas chromatography~~	~~II~~
Olive Oils and Olive Pomace Oils	Sterol composition and total sterols	COI/T.20/Doc. no. 26 / ISO 12228-2 / AOCS Ch 6-91	Thin-layer chromatography and gas chromatography	II
***CXS 33-1981 lists COI/T.20/Doc.no. 10 as method for sterols, but COI/T.20/Doc. No. 30 is listed above. IOC members have doc.No.10 and doc. No. 30 are no longer used and replaced by COI/T.20/Doc.no. 26. Doc.no.26 was reviewed.**				

~~**Named Vegetable Oils**~~	~~Sterol content~~	~~ISO 12228; or AOCS Ch 6-91~~	~~Gas chromatography~~	~~II~~
Named Vegetable Oils	Sterol composition and total sterols	ISO 12228-1 / AOCS Ch 6-91	Thin-layer chromatography and gas chromatography	II
Reviewer I: ISO 12228 Now withdrawn. ISO 12228-1: specifies a procedure for the gas chromatographic determination of the content and composition of sterols in animal and vegetable fats and oils. However, the determination of the contents and composition of sterols in olive and olive pomace oils is to be carried out using ISO 12228-2. ISO 12228-2 specifies a procedure for the gas chromatographic determination of the contents and composition of sterols and triterpene dialcohols in olive and olive pomace oils. For the determination of the contents and composition of sterols in all other animal and vegetable fats and oils, ISO 12228-1 is to be used.				
Reviewer II: COI/T.20/Doc. no. 30 and ISO 12228-2 (Olive oils and olive pomace oils) methods are identical. The COI method may also be used for testing other oils and fats. AOCS Ch 6-91 method has the same principle, uses almost the same chemicals with some different solvents for TLC. The GLC conditions such as capillary column have some differences. COI/T.20/Doc. no. 30 and ISO 12228-2 share validation data. AOCS Ch 6-91 lacks reference data.				

~~**Olive Oils and Olive Pomace Oils**~~	~~Stigmastadienes~~	~~COI/T.20/Doc. no. 11; or ISO 15788-1; or AOCS Cd 26-96~~	~~Gas chromatography~~	~~II~~
Olive Oils and Olive Pomace Oils	Stigmastadienes	COI/T.20/Doc. no. 11 / ISO 15788-1 / AOCS Cd 26-96	Preparative column chromatography and gas chromatography	II
Addition of EWG chair to reviewer I comments: Methods share validation data, principles are identical.				
Reviewer II suggests addition of COI/T.20/Doc. no. 16 (rev. 2 determination of sterenes in refined vegetable oils). Method COI/T.20/Doc. no. 11 has range of 0.01-4.0 mg/kg whilst method COI/T.20/Doc. no. 16 has an operating range of >4.0 mg/kg.				
***AOCS Cd 26-96 reference to COI/T.20/Doc. No. 11 rev.2 2001 has since been updated to rev.3 2017. COI/T.20/Doc. No.11 reference to ISO 5725-5 is listed as 1994 but has since been updated to 1998.**				

~~**Olive Oils and Olive Pomace Oils**~~	~~Stigmastadienes~~	~~ISO 15788-2~~	~~HPLC~~	~~III~~
Olive Oils and Olive Pomace Oils	Stigmastadienes	ISO 15788-2	Preparative column chromatography and gas chromatography	III
~~**Named Animal Fats**~~	~~Titre~~	~~ISO 935; or AOCS Cc 12-59~~	~~Thermometry~~	~~I~~
Named Animal Fats	Titre	ISO 935	Thermometry	I
Named Animal Fats	Titre	AOCS Cc 12-59	Thermometry	IV
None of the two methods include validation data. However, both are old, widely accepted legacy methods. The two methods are not identical, nor complementary. Both methods share the same principle and are fairly similar, but they have critical differences. Critically, ISO 935:1988 describes a single apparatus setting that allows measuring titre values above 30°C. AOCS Cc 12-59 requires the assembling of two different apparatuses: one for titre values below 35°C, and one above for titre values above 35°C. There are also other differences in the sample preparation. As example, ISO 935:1988 (section 9.1) requires neutralizing the free fatty acids with sulfuric acid solution (generally 50 mL) until reaching neutral pH, verified with methyl orange, then wash 3 times with 150 mL of hot sodium chloride solution. Differently, Method AOCS Cc 12-59 requires addition of 50 mL of sulfuric acid solution without checking final pH (presumably turning acid, 1(c)), then wash the free fatty acids by adding an unspecified amount of water and boiling (1(d)), and repeat last step until the washing water is neutral (1(e)). Also, in ISO 935:1988 purified free fatty acids are dried by mixing with 5				

grams of anhydrous sodium sulfate (last step of 9.1). In AOCS Cc 12-59 the free fatty acids are dried by heating at 130°C for an unspecified length of time (1(g)). Method ISO 935:1988 should remain type I, while AOCS Cc 12-59 should become type IV. As described above, the two methods are not identical, and none includes validation data. Method ISO 935:1988 (applicable for titre values above 30°C) describes a single procedure for determining the titre of all commodities reported in CXS 211-1999 (32-49°C); AOCS Cc 12-59 instead requires using one apparatus for titre values above 35°C, and another one for titre values below 35°C. In some instances, AOCS Cc 12-59 does not indicate precise amounts of reagent/solvents (example, water for washing free fatty acids) or timings (example, how long the free fatty acids are dried at 130°C). As result, ISO 935:1988 should be preferred over AOCS Cc 12-59.				
~~**Olive Oils and Olive Pomace Oils**~~	~~Trans fatty acids content~~	~~COI/T.20/Doc no. 17; or ISO 15304; or AOCS Ch 2a-94~~	~~Gas chromatography of methyl esters~~	~~II~~
Olive Oils and Olive Pomace Oils	Trans fatty acids content	COI/T.20/Doc no. 33	Gas chromatography of methyl esters	II
Olive Oils and Olive Pomace Oils	Trans fatty acids content	ISO 15304	Gas chromatography of methyl esters	III
Olive Oils and Olive Pomace Oils	Trans fatty acids content	AOCS Ch 2a-94	Gas chromatography of methyl esters	III
Reviewer I: ISO 15304 and AOCS Ch 2a-94 are not dedicated to olive oil measurements. Both methods contain validation data on olive oils. AOCS 2a-94 contains one validation sample consisting of a mixture of refined olive oil, refined olive-pomace oil, refined soybean oil and lampante virgin olive oil. ISO 15304 contains no validation data for olive pomace oils. Both methods should therefore be typed as Type III, as te COI method is dedicated to olive oils and olive pomace oils and has validation data for these oils.				
Reviewer II: Provision should be named "Fatty acids and *trans*-fatty acids composition and content".				
***CXS 33-1981 lists AOCS Ce 1f-96 as a method for trans fatty acids, but it is not listed here. IOC members state that COI/T.20/Doc. No. 17 is no longer used and replaced by COI/T.20/Doc. No. 33.; Doc. No. 33 was reviewed. The methods are Technically Equivalent. It is recommended that ISO 15304 be changed to Type III instead of Type II, due to the validation study does not include olive pomace oils and that method COI/T.20/Doc. No. 33 is a better fit for trans fatty acid measurement. It is also recommended that AOCS Ch 2a-94 be changed to Type III instead of Type II, due to the method being "recommended practice" and not an official method and that the validation data does not contain virgin or olive pomace oils. It is also recommended that AOCS 2a-94 be replaced with AOCS 1h-05 as a Type III method for fatty acid analysis.**				
EWG Chair: AOCS Ce 1h-05 does not contain validation data for olive oils and olive pomace oils, whilst AOCS Ch2a-94 does. AOCS Ce 1f-96 as mentioned by CXS 33-1981 does contain validation data on olive oil. It is recommended to CCMAS to evaluate the endorsement of the two AOCS methods for this commodity provision as Type III methods.				
~~**Fish oils**~~	~~Triglycerides~~	~~AOCS Cd 11d-96~~	~~HPLC-ELSD~~	~~III~~
Fish oils	Triglycerides	AOCS Cd 11d-96	Liquid chromatography (ELSD)	II
~~**Fish oils**~~	~~Triglycerides~~	~~European Pharmacopoeia 1352 (Omega-3 acid triglycerides): Oligomers and partial glycerides~~	~~HPLC-RI~~	~~III~~
Fish oils	Triglycerides	European Pharmacopoeia 1352	Liquid chromatography (RI)	III

~~**Fish oils**~~	~~Triglycerides~~	~~USP 40-NF35 (Omega-3 Acid Triglycerides):Content of oligomers and partial glyceride~~	~~HPLC-RI~~	III
Fish oils	Triglycerides	USP 40-NF35	Liquid chromatography (RI)	III
Reviewer I: Methods use size exclusion chromatography and different HPLC detectors. The methods use different detectors, however, with the use of reference standards the methods can be calibrated. EP and FCC performance data are not available. AOCS method has extensive validation data in the method. One method should be chosen as Type II by the relevant stakeholders, in this case, as validation data is available, the AOCS method is proposed as Type II.				
Reviewer II: Cd 11d-96 suggest Type II given the clear separation of the triglycerides from the others.				
***AOCS Ca 6b-53 tests for mono and diglycerides and not triglycerides and does not specifically mention that the test is for fish oils.**				

~~**Named Animal Fats**~~	~~Unsaponifiable matter~~	~~ISO 3596; or ISO 18609; or AOCS Ca 6b-53~~	~~Titrimetry after extraction with diethyl ether~~	~~I~~
Named Animal Fats	Unsaponifiable matter	ISO 3596 / ISO 18609 / AOCS Ca 6b-53	Gravimetry, drying at 103 °C and titrimetry (colorimetry)	I
~~**Named Vegetable Oils**~~	~~Unsaponifiable matter~~	~~ISO 3596; or ISO 18609; or AOCS Ca 6b-53~~	~~Gravimetry~~	~~I~~
Named Vegetable Oils	Unsaponifiable matter	ISO 3596 / ISO 18609 / AOCS Ca 6b-53	Gravimetry, drying at 103 °C and titrimetry (colorimetry)	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Unsaponifiable matter~~	~~ISO 3596; or ISO 18609; or AOCS Ca 6b-53~~	~~Gravimetry~~	~~I~~
Olive Oils and Olive Pomace Oils	Unsaponifiable matter	ISO 3596 / ISO 18609 / AOCS Ca 6b-53	Gravimetry, drying at 103 °C and titrimetry (colorimetry)	I
ISO 3596 and AOCS Ca 6b-53 are Identical; ISO 18609 use another solvent. The three methods are not correctly validated and don't meet all the criteria for validation of methods required by CCMAS.				
***ISO 3596 and AOCS Ca 6b-53 have differences in sample weight and extraction volumes.**				

~~**Fish oils**~~	~~Vitamin A~~	~~EN 12823-1 (Determination of vitamin A by high performance liquid chromatograph — Part 1: Measurement of all-E-retinol and 13-Z-retinol~~	~~LC~~	~~III~~
~~**Fish oils**~~	~~Vitamin A~~	~~European Pharmacopoeia Monograph on Cod Liver Oil (Type A), monograph 01/2005:1192, with LC end-point 2.2.29~~	~~LC~~	~~III~~
Fish oils	Vitamin A (all-E-retinol and 13-Z-retinol)	EN 12823-1	Liquid chromatography	II
Fish oils	Vitamin A (all-E-retinol)	European Pharmacopoeia 2398	Liquid chromatography	III
The EN 12823 method has been validated in an interlaboratory study with samples of margarine and milk powder with all-E-retinol levels ranging from 653 µg/100 g to 729 µg/100 g and with 13-Z-retinol levels ranging from 30 µg/100 g to 39 µg/100 g, no fish oil validation data is available. The EP 2398				

method is applied to cod liver oil and has its application range for vitamin A of 1500 µg/100 g to 15000 µg/100 g considering only all-trans-retinol. No validation data is presented. The EN method should therefore by Type II and the EP method Type III.				
~~**Fish oils**~~	~~Vitamin D~~	~~EN 12821 (Determination of vitamin D by high performance liquid chromatography – Measurement of cholecalciferol (D3) or ergocalciferol (D2))~~	~~LC~~	~~III~~
~~**Fish oils**~~	~~Vitamin D~~	~~NMKL 167 (Cholecalciferol (vitamin D3) and Ergocalciferol (vitamin D2)). Determination by HPLC in foodstuffs~~	~~LC~~	~~III~~
Fish oils	Vitamin D (Vitamin D2 and D3)	EN 12821 / NMKL 167	Calculation from vitamin D2 or D3 concentration, preparative column chromatography and liquid chromatography	II
Both methods are similar and share the same validation data concerning fish oils and can therefore be seen as identical. The EN12821 method has been validated in interlaboratory tests on fortified and non-fortified samples such as margarine, milk, milk powder, liquid infant formula, cooking oil and fish oil at levels from 0,4 ug/100 g to 14 ug/100 g (i.e. 0.004mg/kg to 0.14 mg/kg). EN12821 therefore contains additional validation data, but no additional data on the commodity concerned here. As there is no Type II method defined and both methods are identical, it is advised to endorse both methods as Type II.				
~~**Olive Oils and Olive Pomace Oils**~~	~~Wax content~~	~~COI/T.20/Doc. no. 18; or AOCS Ch 8-02~~	~~Gas chromatography~~	~~II~~
Olive Oils and Olive Pomace Oils	Wax content	COI/T.20/Doc. no. 28 / AOCS Ch 8-02	Gas chromatography	II
Reviewer: According to COI/T.15/NC No.3/Rev.14 Trade standard applying to olive oils and olive pomace oils section 11.20: "Determination of the content of waxes and alkyl esters, According to COI/T.20/Doc. No 28/Rev.2, "Determination of the content of waxes, fatty acid methyl esters and fatty acid ethyl esters by capillary gas chromatography".				
***COI/T.20/Doc. No.18 reference ISO 5725-5 is listed as 1994 but has since been updated to 1998.**				

APPENDIX III

PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

Commodity	Provision	Method	Principle	Type
~~**Named Vegetable Oils**~~	~~Apparent density~~	~~ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95~~	~~Pycnometry~~	~~I~~
Named Vegetable Oils	Apparent density	ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95	Pycnometry	I
~~**Named Vegetable Oils**~~	~~Relative density~~	~~ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95~~	~~Pycnometry~~	~~I~~
Named Vegetable Oils	Relative density	ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95	Pycnometry	I
~~**Olive Oils and Olive Pomace Oils**~~	~~Relative density~~	~~ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95~~	~~Pycnometry~~	~~I~~
Olive Oils and Olive Pomace Oils	Relative density	ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95	Pycnometry	I
~~**Named Animal Fats**~~	~~Relative density~~	~~ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95~~	~~Pycnometry~~	~~I~~
Named Animal Fats	Relative density	ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95	Pycnometry	I
~~**Named Vegetable Oils**~~	~~Carotenoids, total~~	~~BS 684 Section 2.20~~	~~Spectrophotometry~~	~~II~~
Named Vegetable Oils	Carotenoids, total	BS684-2.20	Spectrophotometry	II

APPENDIX IV

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