CODEX ALIMENTARIUS COMMISSION





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Agenda Item 4.2

CX/MAS 21/41/5 March 2021

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

41st Session Virtual 17 – 21 and 25 May 2021

REVIEW OF METHODS OF ANALYSIS IN CXS234: 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 *the Standard for Methods of Analysis and Sampling* (CXS 234-1999). The Committee agreed to continue the review of all methods related to fats and oils through an EWG chaired by the Netherlands.
- 2. Due to the COVID-19 pandemic, CCMAS41 was postponed from May 2020 to May 2021, and in order to use the additional time at the disposal of the Committee, an interim report was provided by the EWG (CX/MAS 20/41/5) on which comments were requested through <u>CL 2020/29-MAS</u> in order for the EWG to consider the comments and continue their discussion. The comments were compiled in CX/MAS 20/41/5 Add.1.
- 3. The paper is an update of CX/MAS 20/41/5.

EWG PROCESS AND DISCUSSION

- 4. See CX/MAS 20/41/5 for information on the process followed by the EWG . The list of participants is in Appendix III.
- 5. The EWG considered the comments in response to CL 2020/29-MAS from Canada, Egypt, Mexico, Norway, Peru, Uruguay, USP, FOSFA International and GOED (CX/MAS 20/41/5 Add.1).
 - General considerations and decisions
- 6. In response to the comments commodity, provision, methods, principle and typing have been changed in the revised list of methods listed in CXS 234 for consideration by CCMAS and its Endorsement Working Group (Appendix I).
- 7. General comments, i.e. on historical usage and relationships between various global standards, will require additional discussion and are indicated in <u>Appendix I</u> where applicable. It is suggested that CCMAS provide additional information on those methods for further consideration by the Endorsement Working Group.
- 8. The addition of new methods or provisions to CXS 234 was not considered by the EWG, except for method updates. New methods for endorsement should follow the standard procedure of CCMAS.
 - Selection of Type II methods from multiple Type III methods
- 9. The provision 'Fatty acid composition' of Fish Oils, comprised a large number of Type III methods. No Type II method was provided in CXS 234. Therefore the EWG invited AOCS, Australia, Canada and USA to apply the method described in CX/MAS 20/41/10 (Discussion paper on rules to select Type II methods from multiple Type III methods)¹ with slight modifications to provide additional insights on the applicability of the rules for selection of Type II methods from multiple Type III methods. Their insights are available here. The

¹ The updated paper will be issued as CX/MAS 21/41/10

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recommended Type II methods for this provision is provided in Appendix I. The EWG would like to thank the respondents for their efforts.

Methods of analysis for provisions in the Standard for olive oils and olive pomace oils (CXS 33–1981)

- 10.CCFO26 (2019) was informed of review of methods: fats and oils package in CCMAS and its EWG. CCFO is currently revising the CXS33-1981. The EWG chair, Mr Juan Ramón Izquierdo, has provided section 8 of the revised standard for information and a consolidated reconciliation report of comments of CCFO members which is available here. The EWG Chair also elaborates on the choice of certain methods for provisions. Where applicable, changes were suggested to CXS 234 in Appendix I.
- 11.As many changes for CXS 33-1981 consider new methods not endorsed in CXS 234, it is suggested that CCMAS awaits the request of CCFO to endorse the new methods proposed. After endorsement, changes can be applied in CXS 234.

Other matters

12. Appendix II contains all methods which have not been reviewed by the EWG.

RECOMMENDATIONS

- 13. The Committee is invited to:
 - Consider Appendix I and endorse the proposed changes to CXS 234.
 - Provide methods where historical usage and relations between various global standards are of importance for this workable package to the Endorsement Working Group.
 - Agree that CCMAS consider any new methods in CXS 33 once these have been finalized by CCFO and submitted to CCMAS for endorsement..
 - Consider Appendix II and determine whether the review and updating of these methods are warranted.

APPENDIX I

PART A - METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

Comments CX/MAS 20/41/5 Add.1 applicable to multiple lines:

- Egypt: Remove the word (drying) from the principle description
- Norway: Insoluble impurities, principle could be shortened to "Gravimetry, drying at 103 °C.

^{**}Excerpt comment from Appendix III considering olive oils and olive-pomace oils.

| Commodity | Provision | Method | Principle | |
|---------------------------------|--|-----------------------------|---|-----|
| | | | | |
| Fat spreads and blended spreads | Fat content | ISO 17189 IDF 194 | Gravimetry | Ŧ |
| Fat spreads and blended spreads | Total fat | ISO 17189 IDF 194 | Gravimetry. Direct determination of fat using solvent extraction. | I |
| Fats and oils | tert-butylhydroquinone, & propyl gallate | | Liquid chromatography | #- |
| Fats and oils | Butylhydroxyanisole, butylhydroxytoluene, AOAC 983.15 Liquid chromatography tert-butylhydroquinone, and propyl gallate Synthetic antioxidants | | II | |
| Fats and oils | Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, and propyl gallate. Synthetic antioxidants *USP: AOAC Type III; AOCS Type II, check method updates *Canada: ISO/TC 34/SC 11 is coordinating a collaborative study on AOCS Ce 6-86. Check for results and retype if desired by trade. | AOCS Ce 6-86 | Liquid chromatography | III |
| Fats and Oils (all) | Arsenic- | AOAC 942.17 | Colorimetry (molybdenum blue) | # |
| 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) | # |
| 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 | Ш |

^{*}Excerpt comment from CX/MAS 20/41/5 Add.1, please refer to this document for full comment.

| | *Norway: AOAC 942.17 and 952.13 surplus | | | |
|---|---|---|---|----|
| | in 1993. Suggest AOAC 986.15 as Type II, Kjeldahl methods as Type III. *Mexico: AOAC 986.15 Type II, AOAC 963.21 and AOAC 952.13 Type III. | | | |
| Fats and Oils (all) | Insoluble impurities | ISO 663- | Gravimetry- | ⊢ |
| Fats and Oils (all) | Insoluble impurities | ISO 663 | Calculation from total insoluble content in <i>n</i> -hexane or light petroleum. Gravimetry, drying at 103 °C | I |
| Fats and Oils (all) | Lead- | AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91- | Atomic absorption spectrophotometry (direct graphite furnace) | # |
| 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) | + |
| Fats and Oils (all) | Moisture and volatile matter | ISO 662 | Gravimetry, drying at 105 °C | ı |
| Fats and Oils (all) | Soap content | BS EN ISO 10539 or AOCS Cc 17-95 | Gravimetry | ţ |
| 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 | 1 |
| 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) | # |
| 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 | 1 |
| 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 | + |

| 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 | # |
| Fish oils | Fatty acid composition | AOCS Ce 2-66 | Preparation of methyl esters by fatty- acids | # |
| Fish oils | Fatty acid composition | AOCS Ce 2-66 and AOCS Ce 1a-13 | Gas Chromatography of methyl esters | III/IV/Re moval |
| | Type II from Type III selection suggests ei- | | | |
| | ther (i) remain Type III, (ii) Type IV or (iii) re- moval from STAN 234. | | | |
| | *FOSFA: Suggested combination for marine | | | |
| | oils does not make sense. Should be combi- | | | |
| | nation of AOCS Ce 2-66 and AOCS Ce1i- 07. | | | |
| Fish oils | Fatty acid composition | AOCS Ce 1b-89 | GLC | ##- |
| Fish oils | Fatty acid composition | AOCS Ce 1b 89 | Gas Chromatography of methyl esters | Ш |
| Fish oils | Fatty acid composition | AOCS Ce 2b-11 | Alkali hydrolysis | # |
| 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 |
| | Type II from Type III selection comes unany- mously to conclusion: Type III | | | |
| Fish oils | Fatty acid composition | AOCS Ce 1-07 | Capillary GLC | ##- |
| Fish oils | Fatty acid composition | ISO 12966-2 | Gas chromatography | ##- |
| Fish oils | Fatty acid composition | ISO 5508 | Gas chromatography | # |
| Fish oils | Fatty acid composition | ISO 12966-2 and ISO 12966-4 | Gas Chromatography of methyl esters | III |
| | Type II from Type III selection comes unany- mously to conclusion: Type III | | | |
| Fish oils | Fatty acid composition | AOCS Ce 2-66 and AOCS Ce 1i- 07 | Gas Chromatography of methyl esters | II |
| | Type II from Type III selection comes unany- mously to conclusion: Type II | | | |
| Fish oils | p-anisidine | European Pharmacopoeia 2.5.36 / AOCS Cd 18-90 / ISO 6885 | Spectrophotometry | I |
| | *Canada: Cd 18-90 chosen as Type I method by fish oil industry | | | |
| Fish oils | Peroxide value | AOCS Cd 8b-90 ISO 3960 NMKL 158 | Titration | 1 |

| Fish oils | Peroxide value | European Pharmacopoeia 2.5.5 (Part B Iso-octane as solvent) | Titration | 1 |
|-----------|--|--|--|-----|
| Fish oils | Peroxide value *Canada: EP 2.5.5 chosen as the method by CCMAS *GOED: Different chemical nomenclature is | AOCS Cd 8b-90 / ISO 3960 / NMKL 158 / European Pharma- copoeia 2.5.5 | Titrimetry (Colorimetric) | I |
| | used for same solvent in different methods | | | |
| Fish oils | Phospholipids | USP-FCC 10 2S (Krill oil): Phos- pholipids Nuclear Magnetic Res- onance, Appendix IIC | NMR Spectroscopy | ł |
| Fish oils | Phospholipids *Canada: USP does not publish validation data. refer to JAOCS article | USP-FCC 11 1S | Nuclear Magnetic Resonance Spectroscopy | IV |
| Fish oils | Triglycerides | AOCS Cd 11d-96 | HPLC-ELSD | |
| Fish oils | Triglycerides | AOCS Cd 11d-96 | Liquid chromatography with evapora- tive light scattering detection | II |
| Fish oils | Triglycerides | European Pharmacopoeia 1352- (Omega-3 acid triglycerides): Oli- gomers and partial glycerides | HPLC-RI | ##- |
| Fish oils | Triglycerides | European Pharmacopoeia 1352 | Liquid chromatography with refractive index detection | III |
| Fish oils | Triglycerides | USP 40-NF35 (Omega-3 Acid- Triglycerides):Content of oligo- mers and partial glyceride | HPLC-RI | III |
| Fish oils | Triglycerides | USP 40-NF35 USP 40 NF37 | Liquid chromatography with refractive index detection | 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- | ##- |
| Fish oils | Vitamin A | European Pharmacopoeia Mono- graph on Cod Liver Oil (Type A), monograph 01/2005:1192, with LC end-point 2.2.29 | LC- | ₩ |
| 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 |
| | · · · · · · · · · · · · · · · · · · · | 1 | , <u>, , , , , , , , , , , , , , , , , , </u> | |

| Fish oils | Vitamin D | EN 12821 (Determination of vitamin D by high performance liquid | LC | ₩ |
|-------------------|--|---|--|-----|
| | | chromatography Measurement | | |
| | | of cholecalciferol (D3) or ergocal- | | |
| | | ciferol (D2)) | | |
| Fish oils | Vitamin D | NMKL 167 (Cholecalciferol (vita- | LC | Ш |
| | | min D3) and Ergocalciferol (vita- | | |
| | | min D2)). Determination by | | |
| | | HPLC in foodstuffs | | |
| Fish oils | Vitamin D (Vitamin D2 and D3) | EN 12821 / NMKL 167 | Calculation from vitamin D2 or D3 con- | Ш |
| | | | centration, preparative column chro- | |
| | | | matography and liquid chromatog- | |
| | | | raphy | |
| Named Animal Fats | Acidity- | ISO 660; or AOCS Cd 3d-63 | Titrimetry- | + |
| 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 | Atomic absorption Spectrophotometry | # |
| | | AOCS Ca 18b-91 | (direct graphite furnace) | |
| Named Animal Fats | Copper and Iron | AOAC 990.05 / ISO 8294 / AOCS | Atomic absorption Spectrophotometry | Ш |
| | | Ca 18b-91 | (direct graphite furnace) | |
| Named Animal Fats | GLC ranges of fatty acid composition- | ISO 5508 and ISO 12966-2; or | Gas chromatography of methyl esters | # |
| | | AOCS Ce 2-66 and Ce 1e-91 or | | |
| | | Ce 1f-96 | | |
| 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 |
| | *Canada: Replace AOCS Ce 1f-96 with Ce | | | |
| | 1j-07. Retype to Type III, including the ISO | | | |
| | methods. Suggest AOCS Ce 2-66 and Ce1j- | | | |
| | 07 as Type II. | | | |
| Named Animal Fats | lodine value (IV) | ISO 3961; or AOAC 993.20; or | Wijs-Titrimetry | ⊢ |
| | | AOCS Cd 1d-92 | | |
| Named Animal Fats | lodine value | ISO 3961 / AOAC 993.20 / AOCS | Titrimetry (Wijs) | - 1 |
| | | Cd 1d-92 / NMKL 39 | | |
| Named Animal Fats | Peroxide value | AOCS Cd 8b-90; or ISO 3960 | Titrimetry using iso-octane | + |
| Named Animal Fats | Peroxide value | AOCS Cd 8b-90 / ISO 3960 | Titrimetry (Colorimetric) | 1 |
| Named Animal Fats | Refractive index- | ISO 6320; or AOCS Cc 7-25 | Refractometry | # |
| Named Animal Fats | Refractive index | ISO 6320 / AOCS Cc 7-25 | Refractometry | Ш |
| Named Animal Fats | Relative density | ISO 6883, with the appropriate | Pycnometry - | ļ. |
| | | conversion factor; or AOCS Cc | | |
| | | 10c-95 | | |

| 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- | + |
| 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- | ⊢ |
| Named Animal Fats | Titre | ISO 935 | Thermometry | I |
| Named Animal Fats | Titre *USP: Historically important method, Type I *Canada: Make oldest method Type I (AOCS) | 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 diethylether- | ⊢ |
| Named Animal Fats | Unsaponifiable matter | ISO 3596 / ISO 18609 / AOCS Ca 6b-53 | Gravimetry, drying at 103 °C and titimetry (colorimetry) | I |
| Named Vegetable Oils | Acidity- | ISO 660; or AOCS Cd 3d-63 | Titrimetry- | |
| 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- | F |
| 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- | # |
| 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 | # |
| 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 | + |
| 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 | # |
|----------------------|--|---|---|--------------|
| 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 5 ^a -40 | Titrimetry | I |
| Named Vegetable Oils | Halphen test | AOCS Cb 1-25 | Colorimetry | ı |
| Named Vegetable Oils | Insoluble impurities | ISO 663 | Gravimetry- | Ļ |
| Named Vegetable Oils | Insoluble impurities | ISO 663 | Calculation from total insoluble content in <i>n</i> -hexane or light petroleum. Gravimetry, drying at 103 °C | I |
| Named Vegetable Oils | lodine value (IV) | ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92; or NMKL 39 | Wijs-Titrimetry | ⊢ |
| Named Vegetable Oils | lodine value | ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39 | S Titrimetry (Wijs) | |
| Named Vegetable Oils | Lead- | AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91 | r- Atomic Absorption | |
| Named Vegetable Oils | Lead | AOAC 994.02 / ISO 12193 / AOCS Ca 18c-91 | Atomic absorption spectrophotometry (direct graphite furnace) | |
| Named Vegetable Oils | Moisture & volatile matter at 105°C- | ISO 662 | Gravimetry | F |
| Named Vegetable Oils | Moisture and volatile matter | ISO 662 | Gravimetry, drying at 105 °C | |
| Named Vegetable Oils | Peroxide value (PV) | AOCS Cd 8b-90; or ISO 3960- | Titrimetry | Ļ |
| Named Vegetable Oils | Peroxide value | AOCS Cd 8b-90 / ISO 3960 | Titrimetry (Colorimetric) | |
| Named Vegetable Oils | Refractive index | ISO 6320; or AOCS Cc 7-25 | Refractometry- | # |
| 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 | |
| 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- | + |
| 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- | Ļ. |
|--------------------------------------|---|---|---|----|
| Named Vegetable Oils | Saponification value | ISO 3657 / AOCS Cd 3-25 | Titrimetry (Colorimetric) | ı |
| 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 | ⊢ |
| 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- | ⊢ |
| Named Vegetable Oils | Sterol content | ISO 12228; or AOCS Ch 6-91 | Gas chromatography | # |
| 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 | # |
| Named Vegetable Oils | Tocopherol content | ISO 9936 / AOCS Ce 8-89 | Liquid chromatography | Ш |
| Named Vegetable Oils | Unsaponifiable matter | ISO 3596; or ISO 18609; or AOCS Ca 6b-53 | Gravimetry- | Ļ |
| Named Vegetable Oils | Unsaponifiable matter *Canada: solvents differ between methods | ISO 3596 / ISO 18609 / AOCS Ca 6b-53 | Gravimetry, drying at 103 °C and titrimetry (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 | # |
| Olive Oils and Olive Pomace Oils | Absorbency Absorbance 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- | ⊢ |
| 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- | #- |
| Olive Oils and Olive Pomace Oils | Alpha-tocopherol | ISO 9936 | Liquid chromatography | II |
| | **add AOCS Ce 8-89 | | | |
| Olive Oils and Olive- Pomace Oils | Iron and copper- | ISO 8294; or AOAC 990.05- | AAS | # |
| Olive Oils and Olive Pomace Oils | Copper and Iron | AOAC 990.05 / ISO 8294 | Atomic absorption Spectrophotometry (direct graphite furnace) | Ш |

| | **AOAC 990.05 removed, addition of not endorsed methods ISO 21033 and AOCS Ca 18b-91 | | | |
|-------------------------------------|--|---|--|----|
| 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 | H |
| 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. | Ī |
| Olive Oils and Olive Pomace Oils | Erythrodiol + uvaol | COI/T.20/Doc.no. 30- | Gas chromatography | # |
| 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, eri- throdiol, and uvaol. Thin-layer chroma- tography and gas chromatography (tri- methylsilyl esthers) | II |
| Olive Oils and Olive Pomace Oils | Halogenated solvents, traces- | COI/T.20/Doc. No. 8 | Gas chromatography | # |
| Olive Oils and Olive Pomace Oils | Tetrachloroethylene, traces **Replace COI with ISO 16035 (not endorsed) | COI/T.20/Doc. No. 8 | Gas chromatography | II |
| Olive Oils and Olive Pomace Oils | Insoluble impurities in light petroleum- | ISO 663 | Gravimetry- | ⊢ |
| Olive Oils and Olive Pomace Oils | Insoluble impurities | ISO 663 | Calculation from total insoluble content in <i>n</i> -hexane or light petroleum. Gravimetry, drying at 103 °C | I |
| Olive Oils and Olive Pomace Oils | lodine value | ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92; or NMKL 39 | Wijs-Titrimetry | + |
| Olive Oils and Olive Pomace Oils | lodine 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- | #- |
|---------------------------------------|---|---|---|----------|
| 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- | H |
| Olive Oils and Olive Pomace Oils | Moisture and volatile matter | ISO 662 | Gravimetry, drying at 105 °C | I |
| Olive Oils and Olive- Pomace Oils- | Organoleptic characteristics | COI/T.20/Doc. No. 15- | Panel test | + |
| Olive Oils and Olive Pomace Oils | Organoleptic characteristics | COI/T.20/Doc. No. 15 | Sensory analysis by a trained panel | I |
| Olive Oils and Olive Pomace Oils | Peroxide value | ISO 3960; or AOCS Cd 8b-90 | Titrimetry | H |
| Olive Oils and Olive Pomace Oils | Peroxide value | AOCS Cd 8b-90 / ISO 3960 | Titrimetry (Colorimetric) | I |
| | **USA/Australia: prefer usage of method not using chloroform. No COI defined. | | | |
| Olive Oils and Olive Pomace Oils | Refractive index- | ISO 6320; or AOCS Cc 7-25 | Refractometry- | #- |
| 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 Pycnometry Pycnometry Pycnometry | + |
| 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 | ⊢ |
| 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 | #- |
| Olive Oils and Olive Pomace Oils | Sterol composition and total sterols **Australia/USA/USP: concerns about | 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 | COI/T.20/Doc. No. 26 Stigmastadienes | Col/T.20/Doc. No. 11; or ISO- 15788-1; or AOCS Cd 26-96 | Gas chromatography | # |

| Olive Oils and Olive | Stigmastadienes | Col/T.20/Doc. No. 11 / ISO | Preparative column chromatography | П |
|----------------------|---------------------------|--|--------------------------------------|-----|
| Pomace Oils | | 15788-1 / AOCS Cd 26-96 | and gas chromatography | |
| Olive Oils and Olive | Stigmastadienes | ISO 15788-2 | HPLC- | ##- |
| Pomace Oils | | | | |
| Olive Oils and Olive | Stigmastadienes | ISO 15788-2 | Preparative column chromatography | Ш |
| Pomace Oils | - | | and gas chromatography | |
| Olive Oils and Olive | Trans fatty acids content | COI/T.20/Doc no. 17; or ISO | Gas chromatography of methyl esters | # |
| Pomace Oils | • | 15304; or AOCS Ch 2a-94 | 5 . , , , | |
| Olive Oils and Olive | Trans fatty acids content | COI/T.20/Doc no. 33 | Gas chromatography of methyl esters | Ш |
| Pomace Oils | • | | 0 1 , , , | |
| Olive Oils and Olive | Trans fatty acids content | ISO 15304 | Gas chromatography of methyl esters | III |
| Pomace Oils | • | ISO 12966-2 and 12966-4 | 0 1 , | |
| Olive Oils and Olive | Trans fatty acids content | AOCS Ch 2a-94 | Gas chromatography of methyl esters | III |
| Pomace Oils | • | | 0 1 , , , | |
| Olive Oils and Olive | Unsaponifiable matter | ISO 3596; or ISO 18609; or | Gravimetry- | + |
| Pomace Oils | • | AOCS Ca 6b-53 | • | |
| Olive Oils and Olive | Unsaponifiable matter | ISO 3596 / ISO 18609 / AOCS | Gravimetry, drying at 103 °C and ti- | ı |
| Pomace Oils | • | Ca 6b-53 | trimetry (colorimetry) | |
| Olive Oils and Olive | Wax content | COI/T.20/Doc. no. 18; or AOCS | Gas chromatography | # |
| Pomace Oils | | Ch 8-02 | 0 . , | |
| Olive Oils and Olive | Wax content | COI/T.20/Doc. no. 18- 28 / AOCS | Gas chromatography | II |
| Pomace Oils | | Ch 8-02 | 5 1 7 | |

PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

APPENDIX II

| Commodity | Provision | Method | Principle | Туре |
|-----------------------------|--------------------|--|-------------------|------|
| Named Vegetable Oils | Apparent density | ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95 | Pycnometry- | + |
| 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- | Ł |
| Named Vegetable Oils | Relative density | ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95 | Pycnometry | I |
| Olive Oils and Olive Pomace | Relative density | ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95- | Pycnometry- | Ł |
| Oils | | | | |
| Olive Oils and Olive Pomace | Relative density | ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95 | Pycnometry | I |
| Oils | | | | |
| Named Animal Fats | Relative density | ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95 | Pycnometry- | ļ |
| 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 | # |
| Named Vegetable Oils | Carotenoids, total | BS684-2.20 | Spectrophotometry | II |

APPENDIX III

LIST OF PARTICIPANTS

CHAIR

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Thailand

Chanchai Jaengsawang Rungrassamee Mahakhaphong

United Kingdom

Bhavna Parmar

United States of America

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