

codex alimentarius commission



FOOD AND AGRICULTURE
ORGANIZATION
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Agenda Item 8

CX/MAS 04/9

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Twenty-fifth Session

Budapest, Hungary, 8 – 12 March 2004

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS

This document contains the Methods of analysis proposed by the following Committees in Draft Standards and Proposed Draft Standards under elaboration or as a revision of the methods included in adopted standards.

Part I Methods of Analysis

- A. Codex Committee on Fats and Oils
- B. Codex Committee on Fish and Fishery Products
- C. *Ad hoc* Intergovernmental Task Force on Fruit and Vegetable Juices

Part II Sampling

Codex Committee on Fats and Oils

Note: Document CX/MAS 04/9-Add.1 on the updating of the methods for additives and contaminants will be presented separately.

A. CODEX COMMITTEE ON FATS AND OILS¹

1. Draft Standard for Fat Spreads and Blended Spreads (at Step 6)²

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note
Fat Spreads and Blended Spreads	Milk fat content (butyric acid)	AOAC 985.16	AAS	see below CCFO discussion
		IUPAC 2.310, AOAC 990.27 or AOCS Ca 5c-87 (97).	Gravimetry	

The Committee considered the question from the Codex Committee on Methods of Analysis and Sampling concerning the determination of milk fat. As proposed by the Working Group on Methods of Analysis and Sampling (CRD 10), the Committee noted that butyric acid was an indicator but that the concentration was subject to variation. It therefore recommended, in the absence of a single agreed factor, to convert the butyric acid concentration into milk fat concentration, and to report the range in which the milk fat concentration of a sample would lie. It was also agreed that the method would read Milk Fat Content (butyric acid) for clarification purposes. The Committee agreed to retain the methods of analysis for salt content and for vitamins at this stage, as these ingredients were mentioned in the standard. However, it noted that there were no specific levels for these substances and that this question might require further consideration (ALINORM 03/17, para. 60).

2. Standard for Olive Oils and Olive Pomace Oils³

COMMODITY	PROVISION	METHOD	PRINCIPLE	Note
Olive Oils and Olive Pomace Oils	Organoleptic characteristics	COI/T.20/Doc. no. 15.		
	Free acidity	ISO 660 or AOCS Cd 3d-63(99).	Titrimetry	
	Fatty acid composition	COI/T.20/Doc. no. 24 and ISO 5508 or AOCS Ch 2-91(02) or AOCS Ce 1f-96 (02)	Gas chromatography of methyl esters	
	<i>Trans</i> fatty acids content	COI/T.20/Doc no. 17 or ISO 15304:2001 or AOCS Ce 1f-96 (02)	Gas chromatography	
	Wax content	COI/T.20/Doc. no. 18 or AOCS Ch 8-02 (02)	Titrimetry	
	Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 or AOCS Ce 5b-89 (97).	Calculation	

¹ ALINORM 03/17, Appendices II, III and IV

² The other methods will be presented to the next session of the CCMAS (April 2005) following the next session of the CCFO (February 2005)

³ The Standard was adopted by the 26th Session of the Commission. The methods had not been endorsed due to the timing of the sessions of CCFO and CCMAS

Olive Oils and Olive Pomace Oils	Sterol composition and content	COI/T.20/Doc. no. 10 or ISO 12228:1999 or AOCS Ch 6-91 (97).	Gas chromatography	
	Erythrodiol content	IUPAC 2.431.	Gas chromatography	
	Stigmastadienes	COI/T.20/Doc. no. 11 or ISO 15788-1:1999 or AOCS Cd 26-96 (02).	Gas chromatography	
	Peroxide value	ISO 3960:2001 or AOCS Cd 8b-90 (02).	Titrimetry	
	Absorbency in ultra-violet	COI/T.20/Doc. no. 19 or ISO 3656:2001 or AOCS Ch 5-91 (01).	Absorption in ultra violet	
	Alpha-tocopherol	ISO 9936:1997	HPLC	
	Arsenic	AOAC 952.13	Colorimetry (diethyldithiocarbamate)	Type II method for fats and oils
	Arsenic	AOAC 942.17	Colorimetry (Molybdenum blue)	Type III methods for fats and oils
	Arsenic	AOAC 985.16	Colorimetry	
	Lead	AOAC 994.02 or ISO 12193:1994 or AOCS Ca 18c-91(97)	AAS	Type II method for fats and oils
	Traces of halogenated solvents	COI/T.20/Doc. no. 8.		
	Moisture and volatile matter	ISO 662:1998.	Gravimetry	
	Insoluble impurities in light petroleum	ISO 663:2000.	Gravimetry	
	Iron and copper	ISO 8294:1994 or AOAC 990.05.	Atomic absorption (direct graphite furnace)	Type II method for fats and oils
	Saponification value	ISO 3657:2002 or AOCS Cd 3-25 (02)	Titrimetry	
	Unsaponifiable matter	ISO 3596:2000 or ISO 18609:2000 or AOCS Ca 6b-53 (01)	Titrimetry	Type I method for fats and oils
	Fatty acids in the 2-position of the triglycerides	ISO 6800:1997 or AOCS Ch 3-91 (97).	Gas chromatography	
	Relative density	IUPAC 2.101, with the appropriate conversion factor.	Pyknometry	
	Refractive index	ISO 6320:2000 or AOCS Cc 7-25 (02)	Refractometry	Type II method for fats and oils
	Iodine value	ISO 3961:1996 or AOAC 993.20 or AOCS Cd 1d-92 (97)	Wijs-Titrimetry	Type I method for fats and oils

3. Standard for Named Vegetable Oils⁴

Amendments proposed to the methods in the current standard for Named Vegetable Oils

COMMODITY	PROVISION	METHOD PRESENTLY GIVEN IN STANDARD	SUGGESTED REPLACEMENT	PRINCIPLE	NOTE Current Type
Named Vegetable Oils	Acidity	IUPAC 2.201; or ISO 660: 1996; or AOCS Cd 3d-63	ISO 660: 1996; or AOCS Cd 3d-63 (99)	Titrimetry	I
	Apparent density	ISO 6883: 2000, with the appropriate conversion factor; or AOCS Cc 10c-95	ISO 6883: 2000, with the appropriate conversion factor; or AOCS Cc 10c-95 (02)	Pyknometry	I
	Arsenic	AOAC 952.13 or IUPAC 3.136	AOAC 952.13	Colorimetry (diethyldithiocarbamate)	II
	Arsenic	AOAC 942.17	AOAC 942.17 or AOAC 985.16	Colorimetry (molybdenum blue)	III
	Copper and iron	ISO 8294: 1994; IUPAC 2.631; AOAC 990.05; or AOCS Ca 18b-91	ISO 8294: 1994; or AOAC 990.05; or AOCS Ca 18b-91 (97)	AAS (direct graphite furnace)	II
	Crismer value	AOCS Cb 4-35 (97)	AOCS Cb 4-35 (97) and AOCS Ca 5a-40 (97)	Turbidity	I
	GLC ranges of Fatty acid composition	IUPAC 2.301, 2.302 and 2.304; or ISO 5508: 1990 and ISO 5509: 2000; or AOCS Ce 2-66, Ce 1e-91 or Ce 1f-96	ISO 5508: 1990 and ISO 5509: 2000; or AOCS Ce 2-66 (97); or Ce 1e-91 (01); or Ce 1f-96 (02)	Gas chromatography of methyl esters	II
	Insoluble impurities	IUPAC 2.604; or ISO 663: 2000	ISO 663: 1998	Gravimetry	I

⁴ A Draft Amendment to the Standard was adopted by the 26th Session of the Commission. The amendments to the methods were not endorsed due to the timing of the sessions of CCFO and CCMAS

Named Vegetable Oils	Iodine value (IV)	Wijs - IUPAC 2.205/1; ISO 3961: 1996; AOAC 993.20; or AOCS Cd 1d-1992 (97); or by calculation - AOCS Cd 1b-87 (97)	Wijs - ISO 3961: 1996; or AOAC 993.20; or AOCS Cd 1d-1992 (97); or by calculation – AOCS Cd 1b-87 (97) -The method to be used for specific named vegetable oils is stipulated in the Standard	Wijs-Titrimetry	I
	Lead	IUPAC 2.632; AOAC 994.02 ; ISO 12193: 1994; or AOCS Ca 18c-91	AOAC 994.02 ; or ISO 12193: 1994; or AOCS Ca 18c-91 (97)	Atomic Absorption (direct graphite furnace)	II
	Moisture & volatile matter at 105°C	IUPAC 2.601; or ISO 662: 1998	ISO 662: 1998	Gravimetry (open drying)	I
	Peroxide value (PV)	IUPAC 2.501 (as amended); or AOCS Cd 8b – 90 (97); or ISO 3961: 1998	AOCS Cd 8b-90 (02); or ISO 3961: 2001	Titrimetry using iso-octane	I
	Refractive index	IUPAC 2.102; ISO 6320: 2000; or AOCS Cc 7-25	ISO 6320: 2000; or AOCS Cc 7-25 (02)	Refractometry	II
	Reichert value and Polenske value	IUPAC 2.204	AOCS Cd 5-40 (97)	Titrimetry	I
	Saponification value (SV)	IUPAC 2.202 or ISO 3657: 1988	ISO 3657: 2002; or AOCS Cd 3-25 (02)	Titrimetry	I
	Slip point	ISO 6321: 1991 and Amendment 1: 1998 for all oils; or AOCS Cc 3b-92; or AOCS Cc 3-25 (97) for Palm Oils only.	ISO 6321:2002 for all oils; or AOCS Cc 3b-92 (02); or AOCS Cc 3-25 (97) for Palm Oils only	Open ended capillary tube	I
	Soap content	BS 684 Section 2.5	BS 684 Section 2.5; or AOCS Cc 17-95 (97)	Gravimetry	I
	Sterol content	ISO 12228:1999; or IUPAC 2.403	ISO 12228: 1999; or AOCS Ch 6-91 (97)	Gas chromatography	II
	Tocopherol content	IUPAC 2.432; or ISO 9936: 1997; or AOCS Ce 8-89	ISO 9936: 1997; or AOCS Ce 8-89 (97)		
	Unsaponifiable matter	IUPAC 2.401 (part 1-5); or ISO 3596:2000; or ISO 18609: 2000	ISO 3596: 2000; or ISO 18609: 2000; or AOCS Ca 6b-53 (01)	Titrimetry	I

B. CODEX COMMITTEE ON FISH AND FISHERY PRODUCTS⁵

Draft Standard for Salted Atlantic Herring and Salted Sprats (at Step 8)

Draft Amendment to the Standard for Quick Frozen Fish Sticks (Fish Fingers), Fish Portions and Fish Fillets – Breaded or in Batter (at Step 7)

COMMODITY	PROVISION	METHOD	PRINCIPLE
Salted Atlantic Herring and Salted Sprat	Water content	AOAC 950.46B	air drying
Quick Frozen Fish Sticks	Fish content (declaration)	AOAC 966.15 and calculation (see below)	see below

Section 7. Sampling, Examination and Analysis

7.4 Estimation of Fish Content

According to AOAC Method 966.15. In cases where there is some remaining doubts over the composition of the fish core then the method of analysis as outlined below could be used, i.e. as a reference method.

Determination of Fish Content

The fish content of a fish finger (fish stick) is calculated by using the following equation

$$\% \text{Fish Content} = \frac{\text{Weight of ingoing fish}}{\text{Weight of final product}} \times 100$$

For most products therefore, the fish ingredient weight is that of the raw ingredient. Any figure placed or declared on a product label would be a typical quantity reflecting the producer's normal manufacturing variations, in accordance with good manufacturing practice.

Checking of fish content by chemical analysis

The percentage fish content, corrected for the non-fish flesh nitrogen contributed by the carbohydrate coating, is calculated as follows.

$$\% \text{Fish} = \frac{(\% \text{ total nitrogen} - \% \text{ non - fish flesh nitrogen})}{\text{N factor} *} \times 100$$

* appropriate N (nitrogen) factor

The non-fish flesh nitrogen is calculated as follows: % non-fish flesh nitrogen = % carbohydrate x 0.02

⁵ ALINORM 04/27/18, Appendix II and Appendix VII (Draft Amendment to the Standard for Quick Frozen Fish Sticks (Fish Fingers), Fish Portions and Fish Fillets – Breaded or in Batter : declaration of fish content)

Where the carbohydrate is calculated by difference: % carbohydrate = 100 – (% water + % fat + % protein + % ash)

References

Determination of nitrogen: ISO 937:1978

Determination of moisture: ISO 1442:1997

Determination of total fat: ISO 1443:1973

Determination of ash: ISO 936: 1978

Table 2: Interim Nitrogen factors to be used for white fish as an ingredient (i.e. after GMP)

SPECIES	Nitrogen %
<u>White fish:</u>	
Cod	2.66
Minced Cod	2.61
Coley/Saithe	2.69
European Hake	2.64
Haddock	2.72
Ling	2.78
Plaice	2.46
Alaskan Pollack	2.59
Whiting	2.68
White fish mean	2.65

Additional species important for international trade proposed by Canada, South Africa and the USA but for which there are no nitrogen factors at present:

- Pacific Salmon, Atlantic Salmon, Halibut, Sole, Pacific Cod, Pacific Tomcod, Pacific Whiting, Yellowfin Sole and American Catfish
- South Africa Hake (*Merluccius capensis* and *Merluccius paradoxus*)

C. *AD HOC* INTERGOVERNMENTAL TASK FORCE ON FRUIT AND VEGETABLE JUICES⁶

⁶ ALINORM 03/39A, Appendices II and III

Proposed Draft General Standard for Fruit Juices and Nectars ⁷

The methods listed below were forwarded to the CCMAS by the Task Force on Fruit and Vegetable Juices. However, several methods do not correspond to provisions in the standard. The list below is presented in conformity with the usual format for endorsement of methods of analysis and identifies those provisions that are included in the standard, mainly for permitted ingredients and additives and those that are not specified. The report of the Task Force is available on the Codex website at the following address <ftp://ftp.fao.org/codex/alinorm03/al0339Ae.pdf> and will be available at the CCMAS session for information.

COMMODITY	PROVISION	METHOD	PRINCIPLE
Fruit Juices and Nectars	Not specified	Determination of Acetic acid EN 12632 or IFU Method No 66 (1996)	Enzymatic determination
	Not specified	Determination of Alcohol (ethanol) IFU Method No 52 (1983/1996)	Enzymatic determination
	Not specified	Determination of anthocyanins IFU Method No 71 (1998)	HPLC
	Ascorbic acid-L (additives)	IFU Method No 17a (1995)	HPLC
Fruit Juices and Nectars	Ascorbic-L (additives)	AOAC 967.21 IFU Method No 17	Indophenol method

⁷ *Methods of analysis for the proposed draft Codex General Standard for Fruit Juices and Nectars (extract from the report of the TFFJ)*

The CCMAS reminded the Task Force that the methods proposed by the Codex commodity committee for endorsement should correspond to provisions in Codex standards. In order to overcome this problem and recognizing that a single method cannot be used to determine the overall authenticity, composition, and quality and that a combination of methods may be used, the Task Force added the following wording to Section 3 - Essential Composition and Quality Factors.

Section 3.3 - Authenticity

Authenticity is the maintenance of the product's essential physical, chemical, organoleptical, and nutritional characteristics of the fruit(s) from which it comes.

Section 3.4 - Verification of Composition, Quality, and Authenticity

Fruit juices and nectars should be subject to testing for authenticity, composition, and quality where applicable and where required. The analytical methods used should be those found in Section 8, Methods of Analysis and Sampling.

The Task Force noted that the question of establishing specific values for fruit juices corresponding to each method of analysis was complicated and required a new approach. The Task Force was of the view that previous fruit juice standards applied to a very small number of juices from specific fruits and that the current General Standard being developed covered a much wider range of juices, their mixtures and other fruit juice products and that a number of different methods were used internationally, therefore it was not possible to agree on the specific values at this stage. The Task Force concluded that the approach used by this Task Force should be to develop and only then agree upon specific values for all the varieties of fruit juices and other products covered by the General Standard on Fruit Juices and Nectars using the proposed methodology (ALINORM 03/39A, paras. 69-71 and 76).

	Not specified	Determination of ash in fruit products AOAC 940.26 (JAOAC 23, 314 (1940)) EN 1135 (1994) - IFU Method No 9 (1989)	Gravimetry
	Not specified	Determination of Beet sugar in fruit juices AOAC 995.17 - JAOAC 79, 917 (1996)	Deuterium NMR
		Determination of Benzoic acid as a marker in orange juice AOAC 994.11 - JAOAC 78, 80 (1995)	HPLC
	Carbon dioxide (additives and processing aids)	IFU Method No 42 (1976)	Titrimetry (back-titration after precipitation)
	Not specified	Determination of C ¹³ /C ¹² ratio of ethanol derived from fruit juices Method Submitted to AOAC	Stable isotope mass spectrometry
	Not specified	Determination of Carbon stable isotope ratio of apple juice AOAC 981.09 - JAOAC 64, 85 (1981)	Stable isotope mass spectrometry
	Not specified	Determination of Carbon stable isotope ratio of orange juice AOAC 982.21 - JAOAC 65, 608 (1982) J.Agric.Food Chem, 29, 803-804 (1981)	Stable isotope mass spectrometry
	Not specified	Determination of Carotenoid, Total/individual groups EN 12136 (1997) - IFU Method No59 (1991)	Precipitation/fractionation
	Not specified	Determination of Centrifugable pulp EN 12134 - IFU Method No 60 (1991/1998)	Centrifugation/% value
	Not specified	Determination of Chloride (expressed as sodium chloride) EN12133 IFU Method No 37 (1968)	Electrochemical titrimetry
	Not specified	Determination of Chloride in vegetable juice AOAC 971.27 (Codex general method)	Titration
Fruit Juices and Nectars	Citric acid (additives)	AOAC 986.13 JAOAC 69, 594 (1986) JAOAC 77, 411 (1994)	HPLC
	Citric acid, enzyme (additives)	EN 1137 IFU Method No 22 (1985)	Enzymatic determination
	Not specified	Determination of Essential oils AOAC 968.20 - IFU 45b	(Scott) distillation, titration
	Not specified	Determination of Fermentability IFU Method No 18 (1974)	Microbiological method

	Not specified	Determination of Formol number EN 1133 (1994) IFU Method No 30 (1984)	Potentiometric titration
	Not specified	Determination of Free amino acids EN 12742 IFU Method No 57 (1989)	Chromatography
	Not specified	Determination of Fumaric acid IFU Method No 72 (1998)	HPLC
	Glucose and fructose (permitted ingredients)	EN 12630 IFU Method No 67 (1996)	HPLC
	Not specified	Determination of sorbitol EN 12630 IFU Method No 67 (1996)	
	Glucose-D and fructose-D (permitted ingredients)	EN 1140 IFU Method No 55 (1985)	Enzymatic determination
	Not specified	Determination of Gluconic acid IFU Method No 76 (2001)	
	Not specified	Determination of Glycerol IFU Method No 77 (2001)	
	Not specified	Determination of hesperidin and naringin EN 12148 (1996) - IFU Method No 58 (1991)	HPLC
	HFCS & HIS in apple juice (permitted ingredients)	JAOAC 84, 486 (2001)	CAP GC Method
	Not specified	Determination of Hydroxymethylfurfural IFU Method No 69 (1996)	HPLC
	Not specified	Determination of Isocitric acid-D EN 1139 - IFU Method No 54 (1984)	Enzymatic determination
Fruit Juices and Nectars	Not specified	Determination of Lactic acid- D and L EN 12631 (1999) IFU Method No 53 (1983/1996)	Enzymatic determination
	Malic acid (additives)	AOAC 993.05, JAOAC 69, 594 (1986); JAOAC 77, 411 (1994)	Enzymatic determination and HPLC
	Not specified	Determination of (L-malic/total malic acid ratio in apple juice) AOAC 993.05, JAOAC 69, 594 (1986) JAOAC 77, 411 (1994)	
	Malic acid-D	EN 12138 IFU Method No 64 (1995)	Enzymatic determination

	Malic acid-D in apple juice	AOAC 995.06	HPLC
	Malic acid-L	EN 1138 (1994) IFU Method No 21 (1985)	Enzymatic
	Not specified	Determination of Naringin and neohesperidin in orange juice AOAC 999.05 JAOAC, Vol. 83, No.5 (2000), pp1155-1165	HPLC
	Pectin (additives)	IFU Method No 26 (1964/1996)	Precipitation/photometry
	Not specified	Determination of pH-value EN 1132 (1994) IFU Method No 11 (1968/1989)	Potentiometry
	Not specified	Determination of Phosphorus/Phosphate EN 1136 (1994) IFU Method No 50 (1983)	Photometric determination
	Preservatives in fruit juices ⁸	IFU Method No 63 (1995)	HPLC
	Not specified	Determination of Proline EN 1141 (1994) IFU Method No 49 (1983)	Photometry
	Quinic, malic & citric acid in cranberry juice cocktail and apple juice (permitted ingredients and additives)	AOAC 986.13 JAOAC 69, 594 (1986)	HPLC
	Not specified	Determination of Recoverable oil AOAC 968.20 - IFU Method No 45b	Distillation and titration Scott method
	Not specified	Determination of Relative density EN 1131 (1993) IFU Method No 1 (1989) & IFU Method No General sheet (1971)	Pycnometry
	Not specified	Determination of Relative density IFU Method No 1A	Densitometry
	Not specified	Determination of Sodium, potassium, calcium, magnesium EN 1134 (1994) IFU Method No 33 (1984)	Atomic Absorption Spectroscopy

⁸ Benzoic acid and its salts; sorbic acid and its salts

	Soluble solids	AOAC 983.17 EN 12143 (1996) IFU Method No 8 (1991)	Indirect by refractometry
	Not specified	Determination of Sorbitol-D IFU Method No 62 (1995)	Enzymatic determination
	Not specified	Determination of Stable carbon isotope ratio in the pulp of fruit juices ENV 13070 (1998) Analytica Chimica Acta 340 (1997)	
	Not specified	Determination of Stable carbon isotope ratio of sugars from fruit juices ENV 12140 Analytica Chimica Acta.271 (1993)	Stable isotope mass spectrometry
	Not specified	Determination of Stable hydrogen isotope ratio of water from fruit juices ENV 12142 (1997)	Stable isotope mass spectrometry
	Not specified	Determination of Stable oxygen isotope ratio in fruit juice water ENV 12141(1997)	Stable isotope mass spectrometry
	Not specified	Determination of Starch AOAC 925.38 IFU Method No 73	
	Sucrose (permitted ingredients)	EN 12146 (1996) IFU Method No 56 (1985/1998)	Enzymatic determination
	Sucrose (permitted ingredients)	EN 12630 IFU Method No 67 (1996)	HPLC
	Not specified	Determination of Sugar -beet derviced syrups in frozen concentrated orange juice $\delta^{18}\text{O}$ Measurements in Water - AOAC 992.09	Oxygen isotope ratio analysis
	Not specified	Determination of Sulfates EN 1142 (1994) IFU Method No 36 (1987)	Precipitation/Gravimetry
	Suphites (additives and processing aids)	Determination of Sulphur dioxide by modified Powell EN 13196 not identical with IFU Method No Method No 7A (2000)	Titrimetry after distillation
	Suphites (additives and processing aids)	Determination of Sulphur dioxide by Optimized Monier Williams AOAC 990.28	Titrimetry after distillation

	Tartaric acid in grape juice (additives)	EN 12137 (1997) IFU Method No 65 (1995)	HPLC
	Not specified	Determination of Titrable acids, total EN 12147 (1995) IFU Method No Method No 3, (1968)	Titrimetry
	Not specified	Determination of Total dry matter EN 12145 (1996) IFU Method No 61 (1991)	
	Not specified	Determination of Total nitrogen EN 12135 (1997) IFU Method No 28 (1991)	Digestion/titration
	Not specified	Determination of Total solids AOAC 985.26	Microwave oven drying
	Not specified	Determination of Vitamin C AOAC 967.22	Microfluorometry
	Not specified	Determination of Vitamin C - CEN	DNA

Part II. SAMPLING

CODEX COMMITTEE ON FATS AND OILS (ALINORM 03/17, Appendix II)

Standard for Olive Oils and Olive-Pomace Oils

Sections 8.16 and Annex-Section 4.12 Sampling

According to ISO 661:1989 and ISO 5555:2001.