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RECOMMENDED METHODS OF ANALYSIS AND SAMPLING

CXS 234-1999

Adopted in 1999.

This document is amended yearly. Last amendment: 2024*

* The most updated version of the method should be used, in application of ISO/IEC 17025. The present list of methods reflects the amendments adopted by the Forty-seventh Session of the Codex Alimentarius Commission in 2024.

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2. PART B – METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

All foods				
Commodity	Provision	Method	Principle	Туре
All foods	Acesulfame K, Aspartame	EN 12856	High performance liquid chromatography	II
All foods	Cyclamate	EN 12857	High performance liquid chromatography	II
All foods	Cyclamate	NMKL 123	Spectrophotometry	III
All foods	Saccharin	EN 12856	High performance liquid chromatography	
All foods (see also meat products)	Nitrates and/or nitrites	EN 12014-1	Part 1 – General considerations	N/A
Individual foods ⁱ	Sulphites	EN 1988-1 AOAC 990.28	Part 1 – Optimized Monier-Williams method	
Individual foods ⁱⁱ	Sulphites	EN 1988-2 NMKL 135	Part 2: Enzymatic method	III
Cereals, pulses and legumes and d	lerived products			
Commodity	Provision	Method	Principle	Туре
Certain pulses (soybeans)	Moisture	ISO 665	Gravimetry (oven drying at 103 °C)	I
Certain pulses except soybeans	Moisture	ISO 24557/AACC 44-17.01	Gravimetry (oven drying at 130 °C)	
Degermed maize (corn) meal and maize (corn) grits	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Degermed maize (corn) meal and maize (corn) grits	Fat, crude	AOAC 945.38F and 920.39C and ICC 11/1	Calculation from moisture and Gravimetry (ether extraction)	I

Hominy, fruit juice, seafood.
Wine, dried apples, lemon juice, potato flakes, sultanas, beer.

Commodity	Provision	Method	Principle	Туре
Degermed maize (corn) meal and maize (corn) grits	Moisture	ICC 110/1	Gravimetry (oven drying at 130 ºC – 133 ºC)	I
Degermed maize (corn) meal and maize (corn) grits	Particle size (granularity)	AOAC 965.22 ¹¹¹	Gravimetry (sieving)	Ι
Degermed maize (corn) meal and maize (corn) grits	Protein	ICC 105/2 and ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	Ι
Durum wheat semolina and durum wheat flour	Ash	AOAC 923.03 / ISO 2171 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (incineration at 550 °C)	Ι
Durum wheat semolina and durum wheat flour	Moisture	ISO 712 / ICC 110/1	Gravimetry (oven drying at 130 °C – 133 °C)	Ι
Durum wheat semolina and durum wheat flour	Protein	ICC 105/2 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	I
Instant noodles	Extraction of oil from instant noodles	See Appendix I, Part A	Gravimetry	I
Instant noodles	Acid value	See Appendix I, Part B	Titrimetry (ether extraction)	
Instant noodles	Moisture	See Appendix I, Part C	Gravimetry (oven drying at 105 °C)	I
Maize (corn)	Moisture	ISO 6540 / ICC 110/1	Gravimetry (oven drying at 130 °C – 133 °C)	Ι
Pearl millet flour	Ash	AOAC 923.03 / ISO 2171 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (incineration at 550 °C)	Ι
Pearl millet flour	Colour	ISO 16624	Diffuse reflectance colorimetry (specific colour grader)	I
Pearl millet flour	Fat, crude	AOAC 945.38F and 920.39C and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (ether extraction)	I
Pearl millet flour	Fibre, crude	ISO 5498 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (extraction and filtration)	I

ⁱⁱⁱ Sieve specifications as in ISO 3310/1.

Cereals, pulses and legum	•			
Commodity	Provision	Method	Principle	Туре
Pearl millet flour	Moisture	ISO 712 / ICC 110/1	Gravimetry (oven drying at 130 ºC– 133 ºC)	Ι
Pearl millet flour	Protein	ISO 20483 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	Ι
Quinoa	Moisture	ISO 712	Gravimetry (oven drying)	I
Quinoa	Protein	ISO 1871	Titrimetry (Kjeldahl digestion)	IV
Sorghum flour	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Sorghum flour	Colour	ISO 16624	Diffuse reflectance colorimetry (specific colour grader)	Ι
Sorghum flour	Fat, crude	AOAC 945.38F and 920.39C and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (ether extraction)	Ι
Sorghum flour	Fibre, crude	ICC 113 / ISO 6541 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (separation, incineration)	Ι
Sorghum flour	Moisture	ISO 712 / ICC 110/1	Gravimetry (oven drying at 130 °C – 133 °C)	Ι
Sorghum flour	Particle size (granularity)	AOAC 965.22 ^{iv}	Sieving	I
Sorghum flour	Protein	ICC 105/2 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	Ι
Sorghum flour	Tannins	ISO 9648 and ISO 712 / ICC 110/1	Calculation from moisture and spectrophotometry	Ι
Sorghum grains	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Sorghum grains	Fat, crude	AOAC 945.38F and 920.39C and ISO 6540	Calculation from moisture and gravimetry (ether extraction)	Ι

^{iv} Sieve specifications as in ISO 3310/1.

Cereals, pulses and legumes ar	nd derived products			
Commodity	Provision	Method	Principle	Туре
Sorghum grains	Moisture	ISO 6540	Gravimetry (oven drying at 130 °C – 133 °C)	Ι
Sorghum grains	Protein	ICC 105/2 and ISO 6540	Titrimetry, Kjeldahl digestion	I
Sorghum grains	Tannins	ISO 9648 and ISO 6540	Calculation from moisture and spectrophotometry	Ι
Soy protein products	Ash	AOAC 923.03 ISO 2171: (Method B)	Gravimetry	Ι
Soy protein products	Fat	ISO 734	Gravimetry (extraction)	I
Soy protein products	Fibre, crude	ISO 5498 and AOAC 925.09	Calculation from moisture and gravimetry (extraction and filtration)	Ι
Soy protein products	Moisture	AOAC 925.09	Gravimetry (vacuum oven 98 ºC – 100 ºC)	Ι
Soy protein products	Crude protein: excluding	AOCS Ba 4f-00	100 °C) Gravimetry (combustion)	IV
	added vitamins, minerals, amino acids and food additives	AACCI 46.30		IV
		ISO 16634-1		IV
Vegetable protein products	Ash	AOAC 923.03 ISO 2171 and AOAC 925.09	Calculation from moisture and gravimetry (incineration at 550 °C)	Ι
Vegetable protein products	Fat	ISO 734	Gravimetry (extraction)	Ι
Vegetable protein products	Fibre, crude	AACC 32-10.01 and AOAC 925.09	Calculation from moisture and gravimetry (ceramic fibre filtration)	Ι
Vegetable protein products	Moisture	AOAC 925.09	Gravimetry (vacuum oven at 98 C – 100 °C)	I
Vegetable protein products	Crude protein: excluding	AOCS Ba 4f-00	Gravimetry (combustion)	IV
	added vitamins, minerals, amino acids and food additives	AACCI 46.30		IV
		ISO 16634-1		IV

Cereals, pulses and legumes and d	erived products			
Commodity	Provision	Method	Principle	Туре
Wheat flour	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Wheat flour	Fat acidity	ISO 7305 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (extraction)	I
Wheat flour	Moisture	ISO 712: ICC 110/1	Gravimetry (oven drying at 130 °C and 133 °C)	I
Wheat flour	Particle size (granularity)	AOAC 965.22 ^v	Gravimetry (sieving)	I
Wheat flour	Protein	ICC 105/2 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	I
Wheat protein products including wheat gluten	Crude protein excluding added vitamins, minerals, amino acids and optional ingredients	Vital wheat gluten and devitalized wheat gluten	Calculation from moisture and titrimetry (Kjeldahl digestion)	I
		ISO 20483 and AOAC 925.09		
		Solubilized wheat protein ISO 20483 and AOAC 925.09	Calculation from moisture and titrimetry (Kjeldahl digestion)	Ι
Wheat protein products including Wheat gluten	Fibre, crude	AOAC 962.09 and AOAC 925.09	Calculation from moisture and gravimetry (ceramic fibre filtration)	Ι
Wheat protein products including wheat gluten	Moisture	AOAC 925.09	Gravimetry (vacuum oven at 98 °C – 100 °C)	Ι
Wheat protein products including Wheat gluten	Ash	AOAC 923.03 ISO 2171 and AOAC 925.09	Calculation from moisture and gravimetry (incineration at 550 °C)	I
Whole and decorticated pearl millet grains	Ash	AOAC 923.03 / ISO 2171 and ISO 712/ ICC 110/1	Calculation from moisture and gravimetry (incineration at 550 °C)	I
Whole and decorticated pearl millet grains	Fat, crude	AOAC 945.38F and 920.39C and ISO 712/ ICC 110/1	Calculation from moisture and gravimetry (ether extraction)	I

^v Sieve specifications as in ISO 3310/1.

Commodity	Provision	Method	Principle	Туре
Whole and decorticated pearl millet grains	Fibre, crude	ISO 5498 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (filtration through filter paper)	Ι
Whole and decorticated pearl millet grains	Moisture	ISO 712 ICC 110/1	Gravimetry (oven drying 130 ºC – 133 ºC)	Ι
Whole and decorticated pearl millet grains	Protein	ISO 20483 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	Ι
Whole maize (corn) meal	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Whole maize (corn) meal	Fat, crude	AOAC 945.38Fand 920.39C and ICC 110/1	Calculation from moisture and gravimetry (ether extraction)	I
Whole maize (corn) meal	Moisture	ICC 110/1 ISO 6540	Gravimetry (oven drying 130 ºC – 133 ºC)	I
Whole maize (corn) meal	Particle size (granularity)	AOAC 965.22 ^{vi}	Sieving	I
Whole maize (corn) meal	Protein	ICC 105/2 and ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	Ι

^{vi} Sieve specifications as in ISO 3310/1.

Commodity	Provision	Method	Principle	Туре
Chocolate and chocolate products	Cocoa butter	AOAC 963.15 IOCCC 14	Gravimetry (Soxhlet extraction)	Ι
Chocolate and chocolate products	Fat-free cocoa solids	AOAC 931.05	Oven evaporation and factor	Ι
Chocolate and chocolate products	Fat-free milk solids	IOCCC 17 or AOAC 939.02	Titrimetry, Kjeldahl digestion; after extraction of milk proteins	II
Chocolate and chocolate products	Fat, total	AOAC 963.15	Gravimetry (Soxhlet extraction)	Ι
Chocolate and chocolate products	Milkfat	IOCCC 5 AOAC 945.34; 925.41B; 920.80	Titrimetry/Distillation	Ι
Chocolate and chocolate products	Moisture	IOCCC 26 or AOAC 977.10 (Karl Fischer method); or AOAC 931.04 or IOCCC 1	Gravimetry	I
Chocolate and chocolate products	Non-cocoa butter vegetable fat	AOCS Ce 10/02 and described in the standard	Described in the standard	Ι
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Cocoa shell	AOAC 968.10 and 970.23	Spiral vessel count, stone cell count	Ι
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Fat	AOAC 963.15 or IOCCC 14	Gravimetry (Soxhlet extraction)	Ι
Cocoa butter	Free fatty acids	ISO 660 or AOCS Cd 3d-63	Titrimetry	I
Cocoa butter	Unsaponifiable matter	ISO 3596 or ISO 18609 or AOCS Ca 6b-53	Titrimetry after extraction with diethyl ether	Ι
Cocoa powders (cocoa) and dry cocoa-sugar mixtures	Moisture	IOCCC 26 or AOAC 977.10 (Karl Fischer method)	Gravimetry	Ι

Commodity	Provision	Method	Principle	Туре
Fats and oils (all)	Arsenic	AOAC 963.21 and AOAC 942.17	Kjeldahl flask digestion and colorimetry (molybdenum blue)	
Fats and oils (all)	Arsenic	AOAC 963.21 and AOAC 952.13	Kjeldahl flask digestion and colorimetry (diethyldithiorcarbamate)	
Fats and oils (all)	Arsenic	AOAC 986.15	Atomic absorption spectrophotometry (hydride generation)	II
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)	Moisture and volatile matter	ISO 662	Gravimetry, drying at 103 °C	Ι
Fats and oils (all)	Soap content	ISO 10539 / AOCS Cc 17-95	Titrimetry (colorimetric)	Ι
Fats and oils	Synthetic phenolic antioxidants	AOCS Ce 6a-2021	Liquid chromatography	II
Fats and oils	Synthetic phenolic antioxidants	AOAC 983.15	Liquid chromatography	
Fats and oils not covered by individual standards	Acidity: acid value	ISO 660 / AOCS Cd 3d-63	Titrimetry	Ι
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 / NMKL 158	Titrimetry (colorimetric)	I
Fat spreads and blended spreads	Total fat	ISO 17189 IDF 194	Gravimetry, direct determination of fat using solvent extraction	I
Fish oils	Fatty acid composition	AOCS Ce 2c-66 and AOCS Ce 1i-07 / AOCS Ce 1j-07	Preparation of methyl esters and gas chromatography	
Fish oils	Fatty acid composition	ISO 12966-2 and ISO 12966-4	Preparation of methyl esters and gas chromatography	

Fats and oils and related p	roducts			
Commodity	Provision	Method	Principle	Туре
Fish oils	Acidity: acid value	AOCS Ca 5a-40 / AOCS Cd 3d-63 / ISO 660 / NMKL 38	Titrimetry	Ι
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 12 2S (Krill oil – phospholipids),	Nuclear magnetic resonance spectroscopy	I
Fish oils	P-Anisidine value	European Pharmacopoeia 2.5.36/	Spectrophotometry	I
		AOCS Cd 18-90/		
		ISO 6885		
Fish oils	Triglycerides	AOCS Cd 11d-96	Liquid chromatography with evaporative light scattering detection	II
Fish oils	Triglycerides	European Pharmacopoeia 1352	Liquid chromatography with refractive index detection	III
Fish oils	Triglycerides	USP 40 NF37	Liquid chromatography with refractive index detection	
Fish oils	Vitamin A ^{vii}	European Pharmacopoeia Monograph on Cod Liver Oil (Type A), Monograph 01/2005:1192, with LC end-point 2.2.29	Liquid chromatography	
Fish oils	Vitamin A ^{vii}	EN 12823-1	Liquid chromatography	П
Fish oils	Vitamin D	EN 12821 (Determination of vitamin D by high performance liquid chromatography – Measurement of cholecalciferol (D3) or ergocalciferol (D2))	Liquid chromatography	III
Fish oils	Vitamin D ^{viii}	NMKL 167 / EN 12821	Liquid chromatography	П
Fish oils (calanus oil)	Wax content	AOCS Ch 8-02	Gas chromatography (FID)	IV

^{vii} The respective *Standard for fish oils* (CXS 329-2017) states that vitamin A is expressed as 'retinol equivalents' (RE) where RE takes into account the fact that different vitamers of vitamin A differ in activity. ISO/TR 23304:2021 "Food products – Guidance on how to express vitamins and their vitamers".

Commodity	Provision	Method	Principle	Туре
Named animal fats	Acidity: acid value	ISO 660 / AOCS Cd 3d-63	Titrimetry	Ι
Named animal fats	Fatty acid composition	ISO 12966-2 and ISO 12966-4	Preparation of methyl esters and gas chromatography	III
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	lodine value (IV)	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Titrimetry (Wijs)	Ι
Named animal fats	Peroxide value	AOCS Cd 8b-90 / ISO 3960 / NMKL 158	Titrimetry (colorimetric)	Ι
Named animal fats	Relative density	ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95	Pycnometry	Ι
Named animal fats	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
Named animal fats	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry (colorimetric)	Ι
Named animal fats	Unsaponifiable matter	ISO 3596 / ISO 18609 / AOCS Ca 6b-53	Gravimetry, drying at 103 ^o C and titrimetry (colorimetry)	Ι
Named animal fats	Titre	ISO 935	Thermometry	Ι
Named animal fats	Titre	AOCS Cc 12-59 ^{ix}	Thermometry	IV
Named vegetable oils	Acidity:	ISO 660 / AOCS Cd 3d-63 / AOCS Ca 5a-	Titrimetry	I
U U	Acid value	40		
Named vegetable oils	Free fatty acids	ISO 660 / AOCS Cd 3d-63 / AOCS Ca 5a- 40	Titrimetry	Ι
Named vegetable oils	Apparent density	ISO 6883 / AOCS Cc 10c-95	Pycnometry	Ι
Named vegetable oils	Baudouin test (modified Villavecchia or sesame seed oil test)	AOCS Cb 2-40	Colour reaction	I

^{ix} AOCS Cc 12-59 is the preferred method in certain regions. Due to difference in practical application of AOCS Cc 12-59 compared to ISO 935, it is listed as a Type IV method.

Commodity	Provision	Method	Principle	Туре
Named vegetable oils	Carotenoids, total	BS 684-2.20	Spectrophotometry	II
Named vegetable oils	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	Calculation from individual fatty acid composition (gas chromatography of methyl esters) and turbidity	Ι
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	Halphen test	AOCS Cb 1-25	Colorimetry	I
Named vegetable oils	Insoluble impurities	ISO 663	Calculation from total insoluble content in <i>n</i> -hexane or light petroleum.	Ι
			Gravimetry, drying at 103 °C	
Named vegetable oils	lodine value	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Titrimetry (Wijs)	I
Named vegetable oils	Moisture and volatile matter	ISO 662	Gravimetry, drying at 103 °C	I
Named vegetable oils	Peroxide value (PV)	AOCS Cd 8b-90 / ISO 3960 / NMKL 158	Titrimetry (colorimetric)	I
Named vegetable oils	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	
Named vegetable oils	Reichert-Meissi value and Polenske value	AOCS Cd 5-40	Calculation from soluble and insoluble volatile fatty acids. Titrimetry (colorimetric)	Ι
Named vegetable oils	Relative density	ISO 6883 / AOCS Cc 10c-95	Pycnometry	Ι
Named vegetable oils	Saponification value (SV)	ISO 3657 / AOCS Cd 3-25	Titrimetry (colorimetric)	Ι
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	Ι
Named vegetable oils	Sterol composition and total sterols	ISO 12228-1 / AOCS Ch 6-91	Thin-layer chromatography and gas chromatography	

Fats and oils and related products

Principle Provision Method Commodity Type Liquid chromatography with ISO 9936 / AOCS Ce 8-89 Tocopherol content Named vegetable oils Ш fluorescence detection Unsaponifiable matter Diethyl ether extraction and ISO 3596 / AOCS Ca 6b-53 Named vegetable oils gravimetry, drying at 103 °C and titrimetry (colorimetry) and correction for free fatty acids titrimetry (colorimetry) Hexane extraction and gravimetry. Unsaponifiable matter ISO 18609× Named vegetable oils IV drying at 103 °C and titrimetry (colorimetry) and correction for free fatty acids titrimetry (colorimetry)xi Separation and Gas chromatography 4α -desmethylsterol and total Olive oils and olive pomace oils COI/T.20/Doc. No 26 Ш sterol content (FID) 4α -desmethylsterol and total Separation and Gas chromatography Olive oils and olive pomace oils Ш ISO 12228-2 (part 2) sterol content (FID) Separation and Gas chromatography 4α -desmethylsterol and total Olive oils and olive pomace oils Ш AOCS Ch 6-91 sterol content (FID) Absorbency in ultraviolet Absorption in ultraviolet COI/T.20/Doc. No. 19 / ISO 3656 / AOCS Olive oils and olive pomace oils Т Ch 5-91 ISO 660 (Section 9.1) / AOCS Cd 3d-63 / Acidity, free (acid value) Titrimetrv Olive oils and olive pomace oils Т COI/T.20/Doc. No. 34 Alpha-tocopherol ISO 9936 HPLC (UV or fluorescence) Olive oils and olive pomace oils Ш Alpha-tocopherol AOCS Ce 8-89 HPLC (UV or fluorescence) Olive oils and olive pomace oils COI/T.20/Doc. No. 20 and COI/T.20/Doc. Analysis of triglycerides by HPLC Difference between the actual Olive oils and olive pomace oils and fatty acids by GC followed by and theoretical ECN 42 No. 33 triglyceride content calculation Ervthrodiol + uvaol COI/T.20/Doc. No. 26 Separation and gas chromatography Olive oils and olive pomace oils Ш (FID) Gas chromatography (FID) of methyl Fatty acid composition COI/T.20/Doc. No. 33 Olive oils and olive pomace oils Ш esters

^{*} Results obtained from ISO 18609 are systematically lower. In case of limitations due to climate or regulations that prohibit the use of diethyl ether, ISO 18609 can be used instead of the Type I method.

xⁱ The technique in ISO 18609 is gravimetric. The correction by titration and colorimetry is only when it is necessary to correct for free fatty acids.

Fats and oils and related products

Commodity	Provision	Method	Principle	Туре
Olive oils and olive pomace oils	Fatty acid composition	AOCS Ce 2-66 and AOCS Ch 2-91 / Ce 1h- 05	Gas chromatography (FID) of methyl esters	
Olive oils and olive pomace oils	Fatty acid composition	ISO 12966-2 and ISO 12966-4	Gas chromatography (FID) of methyl esters	
Olive oils and olive pomace oils	2-glyceryl monopalmitate percentage	COI/T.20/Doc. No. 23	Hydrolysis and derivatization gas chromatography (FID)	II
Olive oils and olive pomace oils	Fatty acid ethyl ester content	COI/T.20/Doc. No. 28	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Halogenated solvents, traces	ISO 16035	Headspace gas chromatography (ECD)	II
Olive oils and olive pomace oils	Insoluble impurities in light petroleum	ISO 663	Gravimetry	I
Olive oils and olive pomace oils	lodine value	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Wijs-titrimetry	1
Olive oils and olive pomace oils	Moisture and volatile matter	ISO 662	Gravimetry	
Olive oils and olive pomace oils	Organoleptic characteristics	COI/T.20/Doc. No. 15	Sensory Panel test	Ι
Olive oils and olive pomace oils	Peroxide value	ISO 3960 / AOCS Cd 8b-90 / NMKL 158	Titrimetry	I
Olive oils and olive pomace oils	Relative density	ISO 6883 / AOCS Cc 10c-95	Pycnometry	Ι
Olive oils and olive pomace oils	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
Olive oils and olive pomace oils	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry	I
Olive oils and olive pomace oils	Stigmastadienes	COI/T.20/Doc. no. 11	Gas chromatography (FID)	II
Olive oils and olive pomace oils	Stigmastadienes	AOCS Cd 26-96	Gas chromatography (FID)	III
Olive oils and olive pomace oils	Stigmastadienes	ISO 15788-2	HPLC	
Olive oils and olive pomace oils	Stigmastadienes	ISO 15788-1	Gas chromatography (FID)	
Olive oils and olive pomace oils	Trans fatty acids content	COI/T.20/Doc. No. 33	Gas chromatography (FID) of methyl esters	II
Olive oils and olive pomace oils	Trans fatty acids content	ISO 12966-2 and ISO 12966-4	Gas chromatography (FID) of methyl esters	

Fats and oils and related products	5			
Commodity	Provision	Method	Principle	Туре
Olive oils and olive pomace oils	Trans fatty acids content	AOCS Ce 2-66 and AOCS Ce 1h-05	Gas chromatography (FID) of methyl esters	
Olive oils and olive pomace oils	Unsaponifiable matter	ISO 3596 / AOCS Ca 6b-53	Gravimetry	I
Olive oils and olive pomace oils	Wax content	COI/T.20/Doc. No. 28	Gas chromatograph (FID)	II
Olive oils and olive pomace oils	Wax content	AOCS Ch 8-02	Gas chromatography (FID)	111

Table 1. Method performance criteria for iron and copper in olive oils and olive pomace oils

Commodity Provision				Method performance criteria					
		ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)		Recovery (%)	of methods that meet the criteria	Principle
Olive oils and olive pomace oils	Iron	3.0	1.8–4.2	0.3	0.6	27	80–110	ISO 8294 AOAC 990.05 AOCS Ca 17a- 18 ISO 21033	GF-AAS GF-AAS ICP-OES ICP-OES
Olive oils and olive pomace oils	Copper	0.1	0.03–0.17	0.01	0.02	44	80–110	ISO 8294 AOAC 990.05 AOCS Ca 17a- 18	GF-AAS GF-AAS ICP-OES

Fish and fishery products				
Commodity	Provision	Method	Principle	Туре
Canned tuna and bonito	Determination of presentation	See Appendix II		
Crackers from marine and freshwater fish, crustacean and molluscan shellfish	Crude protein	AOAC 2001.11	Titrimetry (Kjeldahl digestion)	IV
Crackers from marine and freshwater fish, crustacean and molluscan shellfish	Crude protein	Described in the standard		
Crackers from marine and freshwater fish, crustacean and molluscan shellfish	Moisture	Described in the standard		
Fish and fishery products, except raw bivalve molluscs (shucked)	Drained weight	See Appendix III	Gravimetry	Ι
Raw bivalve molluscs (shucked)	Drained weight	Described in the standard		
Fish and fishery products, except frozen abalone (covered by glaze), raw fresh chilled or frozen abalone, quick-frozen raw scallop products, raw bivalve molluscs	Net weight	See Appendix III	Gravimetry	I
Fish and fishery products	Washed drained weight	See Appendix III	Gravimetry	Ι
Fish and fishery products	Sensory and physical determinations	Described in the standard and see Appendix IV and CXG 31-1999	Sensory analysis, visual inspections, counting	Ι
Fish sauce	Total nitrogen	AOAC 978.02	Titrimetry (Kjeldahl digestion)	Ι
Fish sauce	Amino acid nitrogen	AOAC 920.04 and AOAC 920.03	Determining formaldehyde titration method Subtracting by ammoniacal nitrogen (magnesium oxide method)	I
Fish sauce	рН	NMKL 179	Potentiometry	II

Fish and fishery products										
Commodity	Provision	Method	Principle	Туре						
Fish sauce	рН	AOAC 981.12 The pH shall be measured in a sample of fish sauce diluted with water to 1:10 using a pH metre. The dilution of fish sauce is necessary because of the high ionic strength in the undiluted sauce	Potentiometry	IV						
Frozen abalone (covered by glaze)	Net weight	AOAC 963.18	Gravimetry							
Frozen fish and fishery products	Thawing and cooking procedures	See Appendix V	Thawing and heating	I						

Fish and fishery products

Commodity	Provision	Method	Principle	Туре
Quick-frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh	Proportion of fish fillet and minced fish	AOAC 988.09	Physical separation	I
Quick-frozen fish fillets	Gelatinous condition (determined as moisture)	AOAC 983.18 and AOAC 950.46A	Gravimetry	Ι
Quick-frozen finfish, uneviscerated and eviscerated	Gelatinous condition (determined as moisture)	AOAC 983.18 and AOAC 950.46A	Gravimetry	I
Quick-frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh	Gelatinous condition (determined as moisture)	AOAC 983.18 and AOAC 950.46A	Gravimetry	I
Quick-frozen fish sticks (fish fingers) and fish portions – breaded or in batter	Fish content (declaration)	AOAC 996.15 and calculation (See Appendix VI)	Gravimetry	I
Quick-frozen fish sticks (fish fingers) and fish portions-breaded and in batter	Proportion of fish fillet and minced fish	WEFTA Method – See Appendix VII	Gravimetry	I
Quick-frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter	Gelatinous condition (determined as moisture)	AOAC 983.18 and AOAC 950.46A	Gravimetry	I
Quick-frozen raw scallop products	Net weight	AOAC 963.18 and Appendix III	Gravimetry	
Raw bivalve molluscs	Net weight	AOAC 963.18 and Appendix III	Gravimetry	I
Raw fresh chilled or frozen abalone	Net weight	AOAC 963.18	Gravimetry	I
Salted Atlantic herring and salted sprat	Water content (determined as moisture)	AOAC 950.46B a)	Gravimetry	I
Salted Atlantic herring and salted sprat and sturgeon caviar	Determination of salt content	See Appendix VIII		
Live and raw bivalve molluscs	Determination of Escherichia coli	See Appendix IX		
Smoked fish, smoke-flavoured fish and smoke-dried fish	Determination of <i>Listeria</i> monocytogenes	See Appendix IX		
Smoked fish, smoke-flavoured fish and smoke-dried fish	Determination of Clostridium botulinum	See Appendix IX		

Fish and fishery products

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Commodity	Provision	Method	Principle	Туре
Salted fish and dried salted fish of the Gadidae family of fishes	Moisture	AOAC 937.07 and AOAC 950.46B (air- drying (a))	Gravimetry	I
Salted fish and dried salted fish of the Gadidae family of fishes	Salt saturation	See equation in footnotexii	Calculation	I
Salted fish and dried salted fish of the Gadidae family of fishes	Water content in the whole fish	See Appendix VIII	Gravimetry	I
Smoked fish, smoke-flavoured fish and smoke-dried fish	Water phase salt (salt determined as chloride expressed as sodium chloride)	AOAC 952.08 and sodium chloride see method criteria in Table 5 Water phase salt = (% salt x 100) / (% water + % salt)	Gravimetry and titrimetry and calculation	Ι
Smoked fish, smoke-flavoured fish and smoke-dried fish	Water activity	NMKL 168 ISO 18787	Electrometry	II
Live and raw bivalve molluscs	Paralytic shellfish toxicity	AOAC 959.08	Mouse bioassay	IV
Live and raw bivalve molluscs	Paralytic shellfish toxicity	AOAC 2011.27	Receptor binding assay	IV

 Table 2. Method performance criteria for histamine for fish and fishery products

Provision	ML (mg/100 g)	Minimum applicable range (mg/100 g)	LOD (mg/100 g)	LOQ (mg/100 g)	RSD _R (%) no more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
Histamine	10 (average)	8–12	1	2	16	90–107	AOAC 977.13 / NMKL 99, NMKL 196, ISO 19343	Fluorometry, HPLC-UV, HPLC- UV, HPLC-FLD
Histamine	20 (each unit)	16–24	2	4	14	90–107	AOAC 977.13 / NMKL 99, NMKL 196, ISO 19343	Fluorometry, HPLC-UV, HPLC- UV, HPLC-FLD

^{xii} The % salt saturation is calculated as follows:

* The solubility of sodium chloride in water is 36 g per 100 g water, and the constant is calculated as follows: 36 g sodium chloride / (100 g water + 36 g sodium chloride) x 100% = 26.4%

^{1. %} salt in water = (% salt content / (% salt content + % moisture)) x 100%

^{2. %} salt saturation = (% salt in water / 26.4 %*) x 100%

Determination of biotoxins in live and raw bivalve molluscs, live abalone and raw fresh chilled or frozen abalone

The method selected should be chosen on the basis of practicability and preference should be given to methods which have applicability for routine use.

Method performance criteria for determination of toxin analogues by chemical methods

Methods shall meet the numerical criteria listed in Table 3 and may either meet the minimum applicable range, or LOD and LOQ criteria listed.

Toxin group	Toxin	Minimum applicable range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD _R) (%) no more than	Recovery (%)	Examples of applicable methods that meet the criteria
STX group	Saxitoxin 0.05–0.2 0.01 0.02 (STX)	44	50–130	AOAC 2005.06 (HPLC-FLD) NMKL 182 (HPLC-FLD), EN			
	NEO	0.05–0.2	0.01	0.02	44	50–130	14526 (HPLC-FLD)
	dcSTX	0.05–0.2	0.01	0.02	44	50–130	AOAC 2011.02 (HPLC-FLD)
	GTX1	0.05–0.2	0.01	0.02	44	50–130	NMKL 197 (HPLC-FLD),
	GTX2	0.1–0.5	0.03	0.06	38	50–130	Turner <i>et al.</i> (2020) J.AOAC Int. Vol. 103, No. 2,
	GTX3 0.1–0.5 0.03	0.06	38	50–130	p533-62 (uHPLC-MSMS)		
	GTX4	0.05–0.2	0.01	0.02	44	50–130	
	GTX5	0.1–0.5	0.03	0.06	38	50–130	
	GTX6	0.1–0.5	0.03	0.06	38	50–130	-
	dcGTX2	0.1–0.5	0.03	0.06	38	50–130	
	dcGTX3	0.1–0.5	0.03	0.06	38	50–130	
	C1	0.1–0.5	0.03	0.06	38	50–130	-
	C2	0.1–0.5	0.03	0.06	38	50–130	
	C3	0.5–1.5	0.1	0.2	32	50–130	
	C4	0.5–1.5	0.1	0.2	32	50–130	-
OA group	OA	0.03–0.2	0.01	0.02	44	70–130	EU-harmonized SOP using HPLC-MSMS – see reference
	DTX1	0.03–0.2	0.01	0.02	44	60–115 70–130	below* For other methods see
	DTX2	0.1–0.5	0.03	0.06	38	60–115 70–130	references **
Domoic aid	DA	14–26	2	4	20	70–130	EN 14176 (HPLC-UV) AOAC 991.26 (HPLC-UV)

Table 3. Method performance criteria for determination of toxin analogues by chemical methods

Toxin group	Toxin	Minimum applicable range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD _R) (%) no more than	Recovery (%)	Examples of applicable methods that meet the criteria
AZA group	AZA1	0.03–0.2	0.01	0.02	44	70–130	EU-harmonized SOP using HPLC-MSMS – see reference
	AZA2	0.03–0.2	0.01	0.02	44	70–130	below*
	AZA3	0.03–0.2	0.01	0.02	44	70–130	For other methods see references**

* https://www.aesan.gob.es/en/CRLMB/docs/docs/metodos_analiticos_de_desarrollo/EU-Harmonized-SOP-LIPO-LCMSMS_Version5.pdf

** H.J. van den Top, A. Gerssen, P. McCarron, H.P. van Egmond. Quantitative determination of marine lipophilic toxins in mussels, oysters and cockles using liquid chromatography-mass spectrometry: interlaboratory validation study. *Food Additives & Contaminants*: Part A, 2011, Vol. 28, Iss. 12.

Total toxicity is estimated as the sum of the molar concentrations of detected analogues multiplied by the relevant specific toxicity equivalency factors (TEFs). Internationally scientifically validated TEFs must be used. The science behind TEFs is developing. Current internationally validated TEFs are available on the FAO website.

Methods should be validated and used for the relevant toxin analogues that may contribute to total toxicity. Currently known toxin analogues to consider are listed in Table 3.

Where toxin analogues that are not listed in Table 3 are determined the competent authority must assess the contribution of these analogues to total toxicity while conducting further investigations.

Table 4. Method performance criteria for screening and for determination of methylmercury*

Commodity	Provision	ML (mg/kg)	Min appl. range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (%) not more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
Tuna (all species)	methylmercury*	1.2	0.64–1.8	0.12	0.24	31	80–110	EN 16801 / NMKL 202 AOAC 977.15** NMKL 186** / AOAC 2013.06** / EN 15763**	GC-ICP/MS AAS-flame ICP-MS ICP-MS ICP-MS
Alfonsino	methylmercury*	1.5	0.82–2.2	0.15	0.30	30	80–110	AOAC 988.11 EN 16801 / NMKL 202 AOAC 977.15** NMKL 186** / AOAC 2013.06** / EN 15763**	GC-electron capture GC-ICP/MS AAS-flame ICP-MS ICP-MS ICP-MS

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Marlin (all species)	methylmercury*	1.7	0.95–2.5	0.17	0.34	30	80–110	AOAC 988.11 EN 16801 / NMKL 202 AOAC 977.15** NMKL 186** / AOAC 2013.06** / EN 15763**	GC-electron capture GC-ICP/MS AAS-flame ICP-MS ICP-MS ICP-MS
Shark (all species)	methylmercury*	1.6	0.88–2.3	0.16	0.32	30	8–110	AOAC 988.11 EN 16801 / NMKL 202 AOAC 977.15** NMKL 186** / AOAC 2013.06** / EN 15763**	GC-electron capture GC-ICP/MS AAS-flame ICP-MS ICP-MS ICP-MS
Orange roughy	Methylmercury*	0.8	0.40–1.2	0.08	0.16	33	80–110	AOAC 988.11 EN 16801 / NMKL 202 AOAC 977.15** NMKL 186** / AOAC 2013.06** / EN 15763**	GC-electron capture GC-ICP/MS AAS-flame ICP-MS ICP-MS ICP-MS
Pink cusk eel	Methylmercury*	1.0	0.52–1.5	0.10	0.20	32	80–110	AOAC 988.11 EN 16801 / NMKL 202 AOAC 977.15** NMKL 186** / AOAC 2013.06** / EN 15763**	GC-electron capture GC-ICP/MS AAS-flame ICP-MS ICP-MS ICP-MS

* Countries or importers may decide to use their own screening when applying the ML for methylmercury in fish by analysing total mercury in fish. If the total mercury concentration is below or equal to the ML for methylmercury, no further testing is required, and the sample is determined to be compliant with the ML. If the total mercury concentration is above the ML for methylmercury, follow-up testing shall be conducted to determine if the methylmercury concentration is above the ML. The ML also applies to fresh or frozen fish intended for further processing.

**Method applicable for determination of mercury and can be used for screening of methyl mercury, see *

Table 5. Method performance criteria for sodium chloride and for salt determined as chloride expressed as sodium chloride

Commodity	Provision	ML (%)	Min. appl. range (%)	LOD (%)	LOQ (%)	Precision (RSD _R) (%) no more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
Boiled dried salted	Sodium chloride and salt	15 (NaCl)	13.8–16.2	1.5	3.0	5.3	98–102	NMKL 178	Potentiometric titration
anchovies	determined as chloride expressed as sodium chloride	9.1 (Cŀ)	8.3–9.9	0.91	1.8	5.7	98–102	AOAC 971.27 AOAC 937.09	Potentiometric titration Titration
Fish sauce	Salt determined as chloride expressed as sodium chloride	20 (NaCl) Minimum limit	18	2.0	4.0	5.1	98–102	NMKL 178 AOAC 971.27	Potentiometric titration Potentiometric titration
		12 (Cŀ)	11	1.2	2.4	5.5	98–102	AOAC 976.18 AOAC 937.09	Titration Titration

Foods for special d	lietary uses			
Commodity	Provision	Method	Principle	Туре
Special foods	Ash	AOAC 942.05	Gravimetry	I
Special foods	Calcium	AOAC 984.27	ICP emission spectrometry	III
Special foods	Calories by calculation	Method described in CAC/VOL IX-Ed.1, Part III	Calculation method	
Special foods	Carbohydrates	Method described in CAC/VOL IX-Ed.1, Part III	Calculation	
Special foods	Chloride	AOAC 971.27 (Codex general method)	Potentiometry	II
Special foods	Dietary fibre, total	AOAC 985.29	Gravimetry (enzymatic digestion)	I
Special foods	Fat	CAC/RM 55	Gravimetry (extraction)	I
Special foods	Fat in foods not containing starch, meat or vegetable products	CAC/RM 1, B-2	Gravimetry	I
Special foods	Fill of containers	CAC/RM 46 (See Appendix X)	Weighing	I
Special foods	Folic acid	AOAC 944.12	Microbioassay	II
Special foods	Linoleate (in the form of glycerides)	AOAC 922.06; 969.33; 963.22	Acid hydrolysis, preparation of methyl esters and gas chromatography	II
Special foods	Linoleate (in the form of glycerides)	AOAC 922.06; 979.19	Acid hydrolysis and spectrophotometry	
Special foods	Loss on drying (milk-based)	AOAC 925.23 ISO 6731 IDF 21	Gravimetry	I
Special foods	Nicotinamide for foods not based on milk	AOAC 961.14	Colorimetry	II
Special foods	Nicotinamide for milk-based foods	AOAC 944.13	Microbioassay	
Special foods	Pantothenic acid/enriched foods	AOAC 945.74	Microbioassay	II
Special foods	Pantothenic acid/non-enriched foods	<i>The Analyst</i> 89 (1964):1, 3-6, ibid. 232 US Dept Agr., <i>Agr. Handbook</i> 97 (1965)	Microbioassay	IV

Foods for special diet	ary uses			
Commodity	Provision	Method	Principle	Туре
Special foods	Phosphorous	AOAC 986.24	Colorimetry (molybdovanadate)	II
Special foods	Protein efficiency ratio (PER)	AOAC 960.48	Rat bioassay	I
Special foods	Protein, crude	Method described in CAC/Vol IX-Ed. 1, Part III	Titrimetry, Kjeldahl digestion	I
Special foods	Riboflavin	AOAC 970.65	Fluorometry	II
Special foods	Sodium and potassium	ISO 8070 IDF 119	Flame atomic absorption spectrometry	II
Special foods	Sodium and potassium	AOAC 984.27	ICP emission spectrometry	III
Special foods	Vitamin A	AOAC 974.29	Colorimetry	IV
Special foods	Vitamin A in foods in which carotenes have been added as a source of vitamin A	AOAC 941.15	Spectrophotometry	III
Special foods	Vitamin B ₁₂	AOAC 952.20	Microbioassay	II
Special foods	Vitamin B ₆	AOAC 961.15	Microbioassay	II
Special foods	Vitamin C	AOAC 967.22	Microfluorometry	II
Special foods	Vitamin C	AOAC 967.21	Colorimetry (dichloroindophenol)	
Special foods	Vitamin D (D3, milk-based infant formula)	AOAC 992.26	Liquid chromatography	II
Special foods	Vitamin E	AOAC 971.30	Colorimetry	IV
Special foods	Vitamin E (milk-based infant formula)	AOAC 992.03	Liquid chromatography	II
Special foods	Sodium and potassium	ISO 8070 IDF 119	Flame atomic absorption spectrometry	II
Follow-up formula	Dietary fibre, total	AOAC 991.43	Gravimetry (enzymatic digestion)	Ι
Follow-up formula	lodine (milk-based formula)	AOAC 992.24	Ion-selective potentiometry	II

Commodity	Provision	Method	Principle	Туре
Follow-up formula	Pantothenic acid	AOAC 992.07	Microbioassay	II
		Measures total pantothenate (free pantothenic acid + CoA- + ACP-bound) and measured as D-pantothenic acid (or calcium D-pantothenate)		
Follow-up formula	Vitamin A	AOAC 974.29	Colorimetry	IV
Follow-up formula	Vitamin A (retinol isomers)	AOAC 992.04	HPLC	
Follow-up formula	Vitamin A (retinol) (above 500 IU/I milk after reconstitution)	AOAC 992.06	HPLC	111
Follow-up formula	Vitamin K	AOAC 2015.09 / ISO 21446	HPLC-FLD	

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Foods for special dietary uses

Commodity	Provision	Method	Principle	Туре
Foods with low-sodium content (including salt substitutes)	lodine	AOAC 925.56	Titrimetry	II
Foods with low-sodium content (including salt substitutes)	Silica (colloidal, calcium silicate)	AOAC 950.85N	Gravimetry	IV
Gluten-free foods	Gluten	Enzyme-Linked Immunoassay R5 Mendez (ELISA) Method	Immunoassay	Ι
		Eur J Gastroenterol Hepatol 2003; 15: 465-4	74	
Infant formula	Biotin	AOAC 2016.02 / ISO 23305	HPLC-UV	II
Infant formula	Biotin	EN 15607 (d-biotin) (Measures total D-biotin [free + D-biocytin])	HPLC- FLD	III
Infant formula	Calories (by calculation)	Method described in CAC/Vol IX-Ed.1, Part III ^{xiii}	Calculation	Ι
Infant formula	Calcium	AOAC 2015.06/ISO 21424 IDF 243	ICP-MS	II
Infant formula	Calcium	AOAC 2011.14/ISO 15151 IDF 229	ICP emission spectroscopy	
Infant formula	Calcium	ISO 8070 IDF 119	Flame atomic absorption spectrophotometry	
Infant formula	Calcium	AOAC 985.35	Flame atomic absorption spectroscopy	

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xiii Section 9 Calories by calculation – Section 9.2 Conversion factors

⁽a) protein 4 kcal per g

⁽b) carbohydrate 4 kcal per g

⁽c) fat 9 kcal per g

⁽d) monosaccharides 3.75 kcal per g

⁽e) specific food ingredients: See "Energy and Protein Requirements" (FAO Nutrition Meeting Report Series No. 52 or WHO Technical Report Series No. 522).

⁽f) other specific calorie conversion factors may be used where the formulation of the food and the nutrient content are known and where such specific conversion factors are physiologically more meaningful than the factors listed above.

Commodity	Provision	Method	Principle	Туре
Infant formula	Carnitine	AOAC 2015.10/ISO 21468	UHPLC-MS/MS	Ш
Infant formula	Chloride	AOAC 986.26	Potentiometry	
Infant formula	Chloride	AOAC 2016.03/ISO 21422 IDF 242	Potentiometry	II
Infant formula	Choline	AOAC 2015.10/ISO 21468	UHPLC-MS/MS	II
Infant formula	Choline	AOAC 999.14	Enzymatic colorimetric method with limitations on applicability due to choline and ascorbate concentration	111
Infant formula	Copper	AOAC 2015.06/ISO 21424 IDF 243	ICP-MS	Ш
Infant formula	Copper	AOAC 985.35	Flame atomic absorption spectroscopy	
Infant formula	Copper	AOAC 2011/14/ISO 15151 IDF 229	ICP emission spectroscopy	
Infant formula	Chromium (Section B of CXS 72-1981 only)	EN 14082	Graphite furnace atomic absorption after dry ashing	
Infant formula	Chromium (Section B of CXS 72-1981 only)	EN 14083	Graphite furnace AAS after pressure digestion	
Infant formula	Chromium (Section B of CXS 72-1981 only)	AOAC 2006.03	ICP emission spectroscopy	
Infant formula	Chromium (Section B of CXS 72-1981 only)	AOAC 2011.19/ISO 20649 IDF 235	ICP-MS	II
Infant formula	Crude protein ^{xiv}	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	I
Infant formula	Fatty acids (including trans fatty acid)	AOAC 996.06	Gas chromatography	111

xiv Determination of crude protein

The calculation of the protein content of infant formulas prepared ready for consumption may be based on N x 6.25, unless a scientific justification is provided for the use of a different conversion factor for a particular product. The value of 6.38 is generally established as a specific factor appropriate for conversion of nitrogen to protein in other milk products, and the value of 5.71 as a specific factor for conversion of nitrogen to protein in other soy products.

Commodity	Provision	Method	Principle	Туре
Infant formula	Fatty acids (including trans fatty acid)	AOCS Ce 1i-07	Gas chromatography	
Infant formula	Folic acid	AOAC 992.05	Microbioassay	
		(Measures free folic acid + free, unbound natural folates, aggregated, and measured as folic acid)		
		EN 14131		
		(Total folate (free + bound), aggregated and measured as folic acid)		
Infant formula	Folic acid	AOAC 2011.06	LC-MS/MS	II
Infant formula	lodine	AOAC 2012.15 / ISO 20647 IDF 234	ICP-MS	11
	(for milk-based formula)			
Infant formula	Iron	AOAC 2015.06 /	ICP-MS	
		ISO 21424 IDF 243		
Infant formula	Iron	AOAC 2011.14 / ISO 15151 IDF 229	ICP emission spectroscopy	
Infant formula	Iron ^{xv}	AOAC 985.35	Flame atomic absorption spectrophotometry	
Infant formula	Iron	AOAC 999.11 NMKL139	AAS after dry ashing	II
Infant formula	Magnesium	AOAC 2015.06 /	ICP-MS	
		ISO 21424 IDF 243		
Infant formula	Magnesium	AOAC 2011.14 / ISO 15151 IDF 229	ICP emission spectroscopy	
Infant formula	Magnesium	ISO 8070 IDF 119	Flame atomic absorption spectrophotometry	III
Infant formula	Magnesium	AOAC 985.35	Flame atomic absorption spectroscopy	

^{xv} General Codex methods are also available.

Foods for special dietary uses

Commodity	Provision	Method	Principle	Туре
nfant formula	Manganese	AOAC 2015.06 /	ICP-MS	
		ISO 21424 243		
Infant formula	Manganese	AOAC 2011.14 / ISO 15151 IDF 229	ICP emission spectroscopy	
Infant formula	Manganese	AOAC 985.35	Flame atomic absorption spectrophotometry	III
Infant formula	Melamine	ISO/TS 15495 IDF/RM 230	LC-MS/MS	IV
Infant formula	Molybdenum (Section B of CXS 72-1981 only)	EN 14083	Graphite furnace AAS after pressure digestion	III
Infant formula	Molybdenum (Section B of CXS 72-1981 only)	AOAC 2006.03	ICP emission spectroscopy	III
Infant formula	Molybdenum (Section B of CXS 72-1981 only)	AOAC 2011.19 / ISO 20649 IDF 235	ICP-MS	II
Infant formula	Myo-Inositol	AOAC 2011.18 / ISO 20637	LC-pulsed amperometry	
Infant formula	Niacin	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II
Infant formula	Niacin	AOAC 985.34 (niacin (preformed) and nicotinamide)	Microbioassay and turbidimetry	
Infant formula	Niacin	EN 15652	HPLC	III xvi
		(Free and bound and phosphorylated forms measured either as aggregate of nicotinic acid + nicotinamide, or as individual forms)		
Infant formula	Pantothenic acid	AOAC 2012.16 ISO 20639	UHPLC-MS/MS	II
Infant formula	Phosphorus	AOAC 2015.06 / ISO 21424 IDF 243	ICP-MS	II
Infant formula	Phosphorus	AOAC 2011.14 / ISO 15151 IDF 229	ICP emission spectroscopy	

^{xvi} When published as EN method.

Commodity	Provision	Method	Principle	Туре
Infant formula	Phosphorus	AOAC 986.24	Spectrophotometry (molybdovanadate)	
Infant formula	Riboflavin	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II
Infant formula	Riboflavin	AOAC 985.31 ^{xvii}	Fluorimetry	Ш
Infant formula	Riboflavin	EN 14152 (Measures natural and supplemental forms, free, bound and phosphorylated (FMN and FAD) aggregated and measured as riboflavin)	HPLC	
Infant formula	Selenium	AOAC 996.16 or AOAC 996.17	Continuous hydride generation flame atomic absorption spectrometry (HGAAS)	111
Infant formula	Selenium	EN 14627	Hydride generation atomic absorption spectrometry (HGAAS)	111
Infant formula	Selenium	AOAC 2006.03	ICP emission spectroscopy	
	Selenium	AOAC 2011.19 / ISO 20649 IDF 235	ICP-MS	
Infant formula	Sodium and potassium	AOAC 2015.06 / ISO 21424 243	ICP-MS	II
Infant formula	Sodium and potassium	AOAC 2011.14 / ISO 15151 IDF 229	ICP emission spectroscopy	
Infant formula	Sodium and potassium	ISO 8070 IDF 119	Flame atomic absorption spectrophotometry	
Infant formula	Thiamine	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II
Infant formula	Thiamine	AOAC 986.27 ^{xvii}	Fluorimetry	

^{xvii} Care should be taken in the application of the method due to spectral interference.

Foods for special dietary uses

Commodity	Provision	Method	Principle	Тур
nfant formula	Thiamine	EN 14122	HPLC with pre-or post-column	
		(Measures all vitamin B₁ forms (natural and added free, bound and phosphorylated) following extraction and conversion to thiamine)	derivatization to thiochrom	
nfant formula	Total amino acids (excluding taurine and tryptophan) for use according to Section 3.1.3 (a) notes 2) and 3) of CXS 72-1981	AOAC 2018.06 / ISO 4214 IDF 254 / AACC 07-50.01	UHPLC-UV	II
Infant formula	Total carbohydrates	AOAC 986.25	Determination by difference	
		AOAC 990.19 or		
	Moisture/total solids	AOAC 990.20	Gravimetry	
		ISO 6731 IDF 21	Gravinetry	
	Ash	AOAC 942.05	Gravimetry	
Infant formula	Total fat	AOAC 989.05	Gravimetry (Röse-Gottlieb)	
		ISO 23318 IDF 249		
nfant formula	Total fat	ISO 8262-1 IDF 124-1	Gravimetry (Weibull-Berntrop)	
	for milk-based infant formula (products not completely soluble in ammonia)			
Infant formula	Total fatty acids	AOAC 996.06	Gas chromatography	111
Infant formula	Total fatty acids	AOAC 2012.13 / ISO 16958 IDF231	Gas chromatography	II
Infant formula	Total nucleotides	AOAC 2011.20 ISO 20638	LC	
Infant formula	Total phospholipids	AOCS Ja7b-91	Gas chromatography with suitable extraction and preparation procedures	III

Foods for special dietary uses

Commodity	Provision	Method	Principle	Туре
Infant formula	Tryptophan For use according to Section 3.1.3 (a) notes 2 and 3 of CXS 72-1981	AOAC 2017.03	HPLC	II
Infant formula	Vitamin A	EN 12823-1 (all-trans-retinol and 13-cis- retinol)	HPLC	
		Vitamin A (both natural + supplemental ester forms) aggregated and quantified as individual retinol isomers (13-cis and all-trans)		
Infant formula	Vitamin A palmitate (retinyl palmitate), vitamin A acetate	AOAC 2012.10 ISO 20633	HPLC	II
	(retinyl acetate)			
Infant formula	Vitamin B12	AOAC 2014.02	LC-UV	
Infant formula	Vitamin C	AOAC 2012.22 / ISO/DIS 20635	HPLC-UV	II
Infant formula	Vitamin D	EN 12821	HPLC-UV	
		(D2 and/or D3 measured as single components. Hydroxylated forms not measured)		
		NMKL 167		
Infant formula	Vitamin D	AOAC 995.05	HPLC-UV	
		D2 and D3 measured		
Infant formula	Vitamin D	AOAC 2016.05 / ISO 20636	LC-MS	II
	Vitamin E	AOAC 992.03	HPLC	
Infant formula		Measures all rac-vitamin E (both natural + supplemental ester forms) aggregated and quantified as α-congeners		III

Foods for special dietary uses

Commodity	Provision	Method	Principle	Тур
nfant formula	Vitamin E	EN 12822	HPLC	11
		(Measures vitamin E (both natural + supplemental ester forms) aggregated and quantified as individual tocopherol congeners (α , β , γ , δ)		
Infant formula	Vitamin E	AOAC 2012.10 / ISO 20633	HPLC	II
Infant formula	Vitamin B ₆	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC- MS/MS	II
Infant formula	Vitamin B ₆	AOAC 985.32	Microbioassay	
Infant formula	Vitamin B ₆	EN 14166	Microbioassay	
		(Aggregates free and bound pyridoxal, pyridoxine and pyridoxamine and measures as pyridoxine)		
Infant formula	Vitamin B ₆	AOAC 2004.07		
		EN 14164	HPLC	
		(Free and bound phosphorylated forms (pyridoxal, pyridoxine and pyridoxamine) converted and measured as pyridoxine)		
Infant formula	Vitamin B ₁₂	AOAC 986.23	Turbidimetric method	
		(Measures total vitamin B ₁₂ as cyanocobalamin)		
Infant formula	Vitamin B ₁₂	AOAC 2011.10 / ISO 20634	HPLC	11
Infant formula	Vitamin K	AOAC 2015.09 / ISO 21446	HPLC-FLD	II
Infant formula	Zinc	AOAC 2015.06 /	ICP-MS	
		ISO 21424 IDF 243		
Infant formula	Zinc	AOAC 2011.14 / ISO 15151 IDF 229	ICP emission spectroscopy	
Infant formula	Zinc	AOAC 985.35	Flame atomic absorption spectroscopy	III

Table 6. Methods of analysis for dietary fibre: Guidelines for use of nutrition and health claims (CXG 23-1997): Table of conditions for claims

Standard	Provisions	Method	Principle	Туре
General meth	ods that do not measure the lower molecular weight fraction (i.e. monom	heric units $< = 9$) ⁽²⁾		÷
All foods (1)	Method applicable for determining dietary fibres that do not include	AOAC 985.29	Enzymatic gravimetry	Type I
	the lower molecular weight fraction (4)	AACC Intl 32-05.01		
All foods (1)	Method applicable for determining dietary fibres that do not include	AOAC 991.43	Enzymatic gravimetry	Туре І
	the lower molecular weight fraction and also includes determination for soluble and insoluble dietary fibres (4)	AACC Intl 32-07.01		
	To soluble and insoluble dietary libres (4)	NMKL 129		
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction, in foods and food products containing more than 10% dietary fibres and less than 2% starch (e.g. fruits) (4)	AOAC 993.21	Gravimetry	Туре І
All foods (1)	Method applicable for determining dietary fibres that do not include	AOAC 994.13	Enzymatic GC/	Type I
	the lower molecular weight fraction. Provides sugar residue composition of dietary fibre polysaccharides, as well as content of	AACC Intl 32- 25.01	colorimetry gravimetry	
	Klason lignin (4)	NMKL 162		
All foods (1)	Insoluble dietary fibres in food and food products (4)	AOAC 991.42 (Specific for insoluble fibre)	Enzymatic gravimetry	Туре І
		AACC Intl 32-20.01		
All foods (1)	Soluble dietary fibres in food and food products (4)	AOAC 993.19 (Specific for soluble fibre)	Enzymatic gravimetry	Type I
General meth	nods that measure both the higher (monomeric units > 9) and the lo	wer molecular weight fraction (m	onomeric units <=9) ⁽²⁾	
All foods (1)	Method applicable for determining the content of dietary fibres of	AOAC 2001.03	Enzymatic gravimetry	Type I
	higher and lower molecular weight, in food where resistant starches are not present	AACC Intl 32-41.01	and Liquid chromatography	
All foods (1)	Method applicable for determining the content of dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starch	ICC Standard No. 185 / AOAC 2017.16 / AACC 32-60-01	Enzymatic gravimetry High pressure liquid chromatography	Туре І
All foods (1)	Method applicable for determining the content of insoluble and soluble dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches	AACC Intl 32-50.01 AOAC 2011.25	Enzymatic gravimetry High Pressure Liquid Chromatography	Туре І

Standard	Provisions	Method	Principle	Туре
General meth	ods that do not measure the lower molecular weight fraction (i.e. mono	meric units $< = 9$) ⁽²⁾		
Methods that	measure individual specific components (monomeric units: the	whole range for each type of c	omponents is covered) ⁽²⁾	
All foods (1)	$(1\rightarrow 3)(1\rightarrow 4)$ Beta-D-Glucans	AOAC 995.16	Enzymatic	Type II
		AACC Intl 32-23.01		
All foods (1)	Fructans (oligofructoses, inulin, hydrolysed inulin, polyfructoses,	AOAC 997.08	Enzymatic & HPAEC-	Type II
	fructooligosaccharides)	AACC Intl 32-31.01	PAD	
	(applicable to added fructans)			
All foods (1)	Fructans (oligofructoses, inulin, hydrolysed inulin, polyfructoses,	AOAC 999.03	Enzymatic &	Type III
	fructooligosaccharides)	AACC Intl 32-32.01	colorimetric	
	(not applicable highly depolymerized fructans)			
All foods (1)	Polydextrose	AOAC 2000.11	HPAEC-PAD	Type II
		AACC Intl 32-28.01		
All foods (1)	Trans-galacto-oligo saccharides	AOAC 2001.02	HPAEC-PAD	Type II
		AACC Intl 32-33.01		
All foods (1)	Resistant starch (Recommended for RS3)	AOAC 2002.02	Enzymatic	Type II
		AACC Intl 32-40.01		

Other met	hods ⁽²⁾ that have not been subjected to interlaboratory evaluation	on under AOAC international guidelines	3	
Yeast cell wall	Insoluble glucans and mannans of yeast cell wall (for yeast cell wall only)	Eurasyp (European association for specialty yeast product) – LM Bonanno. Biospringer. 2004 – online version: http://www.eurasyp.org/public.techniq ue.home.screen	Chemical & HPAEC-PAD	Туре IV
All foods	Fructo-oligosaccharides (monomeric units < 5)	Ouarné <i>et al.</i> 1999 in <i>Complex</i> <i>Carbohydrates in Foods</i> . Edited by S. Sungsoo, L. Prosky & M. Dreher. Marcel Dekker Inc, New York	HPAEC-PAD	Туре IV

All foods	Non-starch polysaccharides (NSP) (3)	Englyst H.N., Quigley M.E., Hudson G. 1994. Determination of dietary fibre as non-starch polysaccharides with gas-liquid chromatographic high- performance liquid chromatographic or spectrophotometric measurement of constituent sugars – <i>Analyst</i> 119, 1497–1509	Gas-liquid chromatography	Type IV
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⁽¹⁾ Users should consult the description of each method for the food matrices that were the subject of interlaboratory study in the Official Methods of Analysis of AOAC International.
 ⁽²⁾ Two issues are left for national authorities: to include monomeric units 3–9 and which isolated or synthetic compounds have physiological benefit. (Refer to the *Guidelines on nutrition labelling* (CXG 2- 1985).
 ⁽³⁾ Quantitation lost for resistant starch. Refer to specific methods.
 ⁽⁴⁾ Quantitation lost for inulin, resistant starch, polydextrose and resistant maltodextrins. Refer to specific methods.

Fruit juices and nectars				
Commodity	Provisions	Method	Principle	Туре
Fruit juices and nectars	Ascorbic acid-L (additives)	IFUMA 17A	HPLC	II
Fruit juices and nectars	Ascorbic acid-L (additives)	ISO 6557-1	Fluorescence spectrometry	IV
Fruit juices and nectars	Ascorbic acid-L (additives)	AOAC 967.21 IFUMA 17	Indophenol method	
		ISO 6557-2		
Fruit juices and nectars	Carbon dioxide (additives and processing aids)	IFUMA 42	Titrimetry (back-titration after precipitation)	IV
Fruit juices and nectars	Cellobiose	IFUMA 4	Capillary gas chromatography	IV
Fruit juices and nectars	Citric acid ^{xviii} (additives)	AOAC 986.13	HPLC	II

^{xviii} All juices except citrus based juices.

Commodity	Provisions	Method	Principle	Туре
Fruit juices and nectars	Citric acid ^{xix} (additives)	EN 1137 IFUMA 22	Enzymatic determination	III
Fruit juices and nectars	Glucose and fructose	EN 12630	HPLC	
	(permitted ingredients)	IFUMA 67 NMKL 148		
Fruit juices and nectars	Glucose-D and fructose-D	EN 1140	Enzymatic determination	11
	(permitted ingredients)	IFUMA 55		
Fruit juices and nectars	HFCS and HIS in apple juice (permitted ingredients)	Determination of HFCS and HIS by Capillary GC method	CAP GC method	IV
		JAOAC 84, 486 (2001)		
Fruit juices and nectars	Malic acid (additives)	AOAC 993.05	Enzymatic determination and HPLC	
Fruit juices and nectars	Malic acid-D	EN 12138 IFUMA 64	Enzymatic determination	II
Fruit juices and nectars	Malic acid-D in apple juice	AOAC 995.06	HPLC	II
Fruit juices and nectars	Malic acid-L	EN 1138	Enzymatic determination	II
		IFUMA 21		
Fruit juices and nectars	Pectin (additives)	IFUMA 26	Precipitation/photometry	
Fruit juices and nectars	Benzoic acid and its salts;	IFUMA 63	HPLC	II
	sorbic acid and its salts	NMKL 124		
Fruit juices and nectars	Benzoic acid and its salts	ISO 5518, ISO 6560	Spectrometry	

xix All juices except citrus based juices.

Fruit juices and nectars				
Commodity	Provisions	Method	Principle	Туре
Fruit juices and nectars	Preservatives in fruit juices (sorbic acid and its salts)	ISO 5519	Spectrometry	III
Fruit juices and nectars	Quinic, malic and citric acid in cranberry juice cocktail and apple juice (permitted ingredients and additives)	Determination of quinic, malic and citric acid in cranberry juice cocktail and apple juice AOAC 986.13	HPLC	111
Fruit juices and nectars	Saccharin	NMKL 122	Liquid chromatography	II
Fruit juices and nectars	Soluble solids	AOAC 983.17 EN 12143 IFUMA 8	Indirect by refractometry	I
		ISO 2173		
Fruit juices and nectars	Sucrose (permitted ingredients)	EN 12146 IFUMA 56	Enzymatic determination	III
Fruit juices and nectars	Sucrose (permitted ingredients)	EN 12630 IFUMA 67 NMKL 148	HPLC	II
Fruit juices and nectars	Sulphur dioxide (additives)	Optimized Monier-Williams AOAC 990.28	Titrimetry after distillation	II
		IFUMA 7A NMKL 132		
Fruit juices and nectars	Sulphur dioxide (additives)	NMKL 135	Enzymatic determination	
Fruit juices and nectars	Sulphur dioxide (additives)	ISO 5522, ISO 5523	Titrimetry after distillation	
Fruit juices and nectars	Tartaric acid in grape juice (additives)	EN 12137 IFUMA 65	HPLC	II
Fruit juices and nectars	Total nitrogen	EN 12135 IFUMA 28	Digestion/titration	I

Commodity	Provisions	Method	Principle	Тур
Fruit juices and nectars	Sections 3.2 Quality criteria	Determination of acetic acid	Enzymatic determination	II
	and 3.3 Authenticity of CXS 247-2005 ^{xx}	EN 12632; IFUMA 66		
Fruit juices and nectars		Determination of alcohol (ethanol)	Enzymatic determination	II
		IFUMA 52		
Fruit juices and nectars		Detection of anthocyanins	HPLC	I
		IFUMA 71		
Fruit juices and nectars		Determination of ash in fruit products	Gravimetry	Ι
		AOAC 940.26; EN 1135; IFUMA 9		
Fruit juices and nectars		Detection of beet sugar in fruit juices	Deuterium NMR	II
		AOAC 995.17		
Fruit juices and nectars		Determination of benzoic acid as a marker in orange juice	HPLC	III
		AOAC 994.11		
Fruit juices and nectars		Determination of C ¹³ /C ¹² ratio of ethanol derived from fruit juices	Stable isotope mass spectrometry	II
		JAOAC 79, No. 1, 1996, 62-72		
Fruit juices and nectars		Determination of carbon stable isotope ratio of apple juice	Stable isotope mass spectrometry	II
		AOAC 981.09– JAOAC 64, 85 (1981)		
Fruit juices and nectars		Determination of carbon stable isotope ratio of orange juice	Stable isotope mass spectrometry	II
		AOAC 982.21		

XX 3.4 Verification of composition, quality and authenticity

The verification of a sample's authenticity/quality can be assessed by comparison of data for the sample, generated using appropriate methods included in the standard, with that produced for fruit of the same type and from the same region, allowing for natural variations, seasonal changes and for variations occurring due to processing.

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 9 (Methods of analysis and sampling).

Fruit juices and nectars

Commodity	Provisions	Method	Principle	Тур
Fruit juices and nectars		Determination of carotenoid, total/individual groups	Spectrophotometry	I
		EN 12136; IFUMA 59		
Fruit juices and nectars		Determination of centrifugable pulp	Centrifugation/% value	I
		EN 12134; IFUMA 60		
Fruit juices and nectars		Determination of chloride (expressed as sodium chloride) EN 12133 IFUMA 37	Electrochemical titrimetry	111
Fruit juices and nectars		Determination of chloride in vegetable juice AOAC 971.27 (Codex general method) ISO 3634	Titration	II
Fruit juices and nectars		Determination of essential oils (Scott titration) AOAC 968.20 - IFUMA 45 ^{xxi}	(Scott) distillation, titration	I
Fruit juices and nectars		Determination of essential oils (in citrus fruit) (volume determination) ^{xxi} ISO 1955	Distillation and direct reading of the volume determination	I
Fruit juices and nectars		Determination of fermentability IFUMA 18	Microbiological method	I
Fruit juices and nectars		Determination of formol number EN 1133 IFUMA 30	Potentiometric titration	I
Fruit juices and nectars		Determination of free amino acids EN 12742 IFUMA 57	Liquid chromatography	II
Fruit juices and nectars		Determination of fumaric acid IFUMA 72	HPLC	II
Fruit juices and nectars		Determination of glucose fructose and saccharose EN 12630 IFUMA 67 NMKL 148	HPLC	II
Fruit juices and nectars		Determination of gluconic acid IFUMA 76	Enzymatic determination	II

^{xxi} Because there is no numerical value in the standard, duplicate Type I methods have been included which may lead to different results.

Fruit juices and nectars

CommodityProvisionsMethodFruit juices and nectarsDetermination of gly
IFUMA 77Fruit juices and nectarsDetermination of he
EN 12148 IFUMA 5Fruit juices and nectarsDetermination of hy
IFUMA 69Fruit juices and nectarsDetermination of hy
IFUMA 69Fruit juices and nectarsDetermination of hy
IFUMA 69Fruit juices and nectarsDetermination of hy
ISO 7466Fruit juices and nectarsDetermination of hy
ISO 7466

Fruit juices and nectars

Method	Principle	Туре
Determination of glycerol IFUMA 77	Enzymatic determination	II
Determination of hesperidin and naringin EN 12148 IFUMA 58	HPLC	II
Determination of hydroxymethylfurfural IFUMA 69	HPLC	II
Determination of hydroxymethylfurfural ISO 7466	Spectrometry	111
Determination of isocitric acid-D IFUMA 54	Enzymatic determination	II
Determination of Lactic acid- D and L EN 12631 IFUMA 53	Enzymatic determination	II
Determination of L-malic/total malic acid ratio in apple juice AOAC 993.05	Enzymatic determination and HPLC	II
Determination of naringin and neohesperidin in orange juice AOAC 999.05	HPLC	III
Determination of pH value	Potentiometry	
NMKL 179 EN 1132 IFUMA 11 ISO 1842		II IV
Determination of phosphorus/phosphate EN 1136 IFUMA No 50	Photometric determination	II
Determination of proline by photometry – non- specific determination EN 1141 IFUMA 49	Photometry	I
Determination of relative density EN 1131 (1993); IFUMA 01 & IFU Method No General sheet (1971)	Pycnometry	II
Determination of relative density IFUMA 01A	Densitometry	III
Determination of sodium, potassium, calcium, magnesium in fruit juices EN 1134 IFUMA 33	Atomic absorption spectroscopy	II
Determination of sorbitol-D IFUMA62	Enzymatic determination	II

Fruit juices and nectars

Commodity	Provisions	Method	Principle	Тур
Fruit juices and nectars		Determination of stable carbon isotope ratio in the pulp of fruit juices ENV 13070 Analytica Chimica Acta 340 (1997)	Stable isotope mass spectrometry	II
Fruit juices and nectars		Determination of stable carbon isotope ratio of sugars from fruit juices ENV 12140 Analytica Chimica Acta 271 (1993)	Stable isotope mass spectrometry	II
Fruit juices and nectars		Determination of stable hydrogen isotope ratio of water from fruit juices ENV 12142	Stable isotope mass spectrometry	II
Fruit juices and nectars		Determination of stable oxygen isotope ratio in fruit juice water ENV 12141	Stable isotope mass spectrometry	II
Fruit juices and nectars		Detection of starch AOAC 925.38 IFUMA 73	Colorimetric	Ι
ruit juices and nectars		Determination of sugar beet derived syrups in frozen concentrated orange juice δ^{18} O Measurements in water AOAC 992.09	Oxygen isotope ratio analysis	I
ruit juices and nectars		Determination of titrable acids, total EN 12147 IFUMA 03 ISO 750	Titrimetry	I
ruit juices and nectars		Determination of total dry matter (vacuum oven drying at 70 °C) ^{xxii} EN 12145 IFUMA 61	Gravimetric determination	Ι
ruit juices and nectars		Determination of total solids (microwave oven drying) ^{xxii} AOAC 985.26	Gravimetric determination	Ι
Fruit juices and nectars		Determination of vitamin C (dehydro-ascorbic acid and ascorbic acid) AOAC 967.22	Microfluorometry	

^{xxii} Because there is no numerical value in the standard, duplicate Type I methods have been included which may lead to different results.

Commodity	Provisions	Method	Principle	Туре
Milk and milk products	Melamine	ISO 23970 IDF 252	LC-MS/MS	
Blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	I
Blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat (MSNF) ^{xxiii}	ISO 6731 IDF 21 and ISO 23318 IDF 249	Calculation from total solids content and fat content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb)	I
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ^{xxiiiiii}	ISO 6731 IDF 21 and ISO 23318 IDF 249 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ^{xxiiiiii}	ISO 6731 IDF 21 and ISO 23318 IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	I

^{xxiii} Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.

Commodity	Provisions	Method	Principle	Туре
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat (MSNF) ^{xxiv}	ISO 6731 IDF 21 and ISO 23318 IDF 249	Calculation from total solids content and fat content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb)	I
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ^{xxiv}	ISO 6731 IDF 21 and ISO 23318 IDF 249 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ^{xxiv}	ISO 6731 IDF 21 and ISO 23318 IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Total fat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	I
Blend of skimmed milk and vegetable fat in powdered form	Water ^{xxv} (moisture)	Described in Appendix XI ^{xxvi}	Gravimetry, drying at 102 °C	IV
Blend of skimmed milk and vegetable fat in powdered form	Water ^{xxv} (moisture)	ISO 5537 IDF 26	Gravimetry, drying at 87 °C	I
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF ^{xxiv}	ISO 5537 IDF 26 and ISO 23318 IDF 249 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 87 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV

 ^{xxiv} Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.
 ^{xxv} Water content excluding the crystallized water bound to lactose (generally known as moisture content).
 ^{xxvi} Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method described in Appendix XI is listed as Type IV.

Commodity	Provisions	Method	Principle	Туре
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF ^{xxvii}	Described in Appendix XI ^{xxviii} and ISO 23318 IDF 249 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF ^{xxvii}	ISO 5537 IDF 26 and and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 87 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF ^{xxvii}	Described in Appendix XI ^{xxviiiiii} and ISO 23318 IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Total fat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water ^{xxix} (moisture)	ISO 5537 IDF 26	Gravimetry, drying at 87 °C	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water ^{xxix} (moisture)	Described in Appendix XI ^{xxviii}	Gravimetry, drying at 102 °C	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF ^{xxvii}	ISO 5537 IDF 26 and ISO 1736 IDF 9 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 87 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV

^{xxvii} Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose. ^{xxviii} Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method as described in Appendix XI is listed as Type IV. ^{xxix} Water content excluding the crystallized water bound to lactose (generally known as moisture content).

Commodity	Provisions	Method	Principle	Туре
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF ^{xxx}	ISO 5537 IDF 26 and ISO 23318 IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 87 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF ^{xxx}	Described in Appendix XI ^{xxxi} and ISO 23318 IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	Ι
Blend of sweetened condensed skimmed milk and vegetable fat	Sucrose	ISO 2911 IDF 35	Polarimetry	IV
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk solids-not-fat (MSNF) ^{xxx}	ISO 6734 IDF 15 and ISO 23318 IDF 249 and ISO 2911 IDF 35	Calculation from total solids content, fat content and sucrose content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry	IV
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF ^{xxx}	ISO 6734 IDF 15 and ISO 23318 IDF 249 and ISO 2911 IDF 35 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content, sucrose content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry and titrimetry (Kjeldahl)	IV

^{xxx} Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose. ^{xxi} Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method as described in Appendix XI is listed as Type IV.

	Provisions	Method	Principle	Туре
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF ^{xxxii}	ISO 6734 IDF 15 and ISO 23318 IDF 249 and ISO 2911 IDF 35 and AOAC 991.20	Calculation from total solids content, fat content, sucrose content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry and titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	Ι
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk solids-not-fat (MSNF) ^{xxxii}	ISO 6734 IDF 15 and ISO 23318 IDF 249 and ISO 2911 IDF 35	Calculation from total solids content, fat content and sucrose content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF ^{xxxii}	ISO 6734 IDF 15 and ISO 23318 IDF 249 and ISO 2911 IDF 35 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content, sucrose content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry and titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF ^{xxxii}	ISO 6734 IDF 15 and ISO 23318 IDF 249 and ISO 2911 IDF 35 and AOAC 991.20	Calculation from total solids content, fat content, sucrose content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry and titrimetry (Kjeldahl)	IV
Butter	Milk solids-not-fat (MSNF)xxxii	ISO 3727-2 IDF 80-2	Gravimetry	I

^{xxxii} Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.

Milk and milk products				
Commodity	Provisions	Method	Principle	Туре
Butter	Milkfat (total fat)	ISO 17189 IDF 194	Gravimetry (direct determination of fat using solvent extraction)	I
Butter	Milk fat purity	ISO 17678 IDF 202	Calculation from determination of triglycerides by gas chromatography– FID	I
Butter	Salt	ISO 1738 IDF 12/	Titrimetry (Mohr: determination of	111
		AOAC 960.29	chloride, expressed as sodium chloride)	
Butter	Salt	ISO 15648 IDF 179	Potentiometry (determination of chloride, expressed as sodium chloride)	II
Butter	Water ^{xxxiii}	ISO 3727-1 IDF 80-1	Gravimetry	I
Cheese	Milkfat	ISO 23319 IDF 250	Gravimetry (Schmid-Bondzynski- Ratzlaff)	Ι
Cheese	Moisture	ISO 5534 IDF 4	Gravimetry, drying at 102 °C	I
Cheese (and cheese rind)	Natamycin	ISO 9233-1 IDF 140-1	Molecular absorption spectrophotometry	
Cheese (and cheese rind)	Natamycin	ISO 9233-2 IDF 140-2	HPLC-UV	11
Cheese	Propionic acid	ISO/TS 19046-11 IDF/RM 233-1	Gas chromatography– FID	IV
Cheese	Propionic acid	ISO/TS 19046-2I IDF/RM 233-2	Ion exchange chromatography-UV	IV
Cheese	Sodium chloride	ISO 5943 IDF 88	Potentiometry (determination of chloride, expressed as sodium chloride)	11
Cheeses, individual	Dry matter (total solids)xxxiv	ISO 5534 IDF 4	Gravimetry, drying at 102 °C	Ι
· · · · · · · · · · · · · · · · · · ·				

^{xxxiii} Water content excluding the crystallized water bound to lactose (generally known as moisture content). ^{xxxiv} Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.

Milk and milk products				
Commodity	Provisions	Method	Principle	Туре
Cheeses, individual	Milkfat in dry matter	ISO 5534 IDF 4 ISO 23319 IDF 250	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Schmid-Bondzynski- Ratzlaff)	I
Cheeses in brine	Milkfat in dry matter	ISO 5534 IDF 4 ISO 23319 IDF 250	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Schmid-Bondzynski- Ratzlaff)	I
Cottage cheese	Fat-free dry matter	ISO 5534 IDF 4 and ISO 23319 IDF 250	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Schmid-Bondzynski- Ratzlaff)	I
Cottage cheese (for samples containing lactose over 5% or with non-dairy ingredients)	Milkfat in dry matter	ISO 5534 IDF 4 and ISO 8262-3 IDF 124-3	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Weibull-Berntrop)	I
Cottage cheese (for samples containing lactose up to 5%)	Milkfat in dry matter	ISO 5534 IDF 4 and ISO 23319 IDF 250	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Schmid-Bondzynski- Ratzlaff)	I
Cottage cheese (for samples containing lactose up to 5%)	Milkfat	ISO 23319 IDF 250	Gravimetry (Schmid-Bondzynski- Ratzlaff)	Ι
Cottage cheese (for samples containing lactose over 5% or with non-dairy ingredients)	Milkfat	ISO 8262-3 IDF 124-3	Gravimetry (Weibull-Berntrop)	I
Cheese, unripened, including fresh cheese	Milk protein	ISO 8968-1 IDF 20-1	Titrimetry, Kjeldahl	Ι
Cream and prepared creams	Milk protein	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	1

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Commodity	Provisions	Method	Principle	Тур
Cream	Milkfat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	I
Cream	Solids ^{xxxv}	ISO 6731 IDF 21	Gravimetry (drying at 102 °C)	Ι
Creams lowered in milkfat content	Milkfat	ISO 23318 IDF 249 / AOAC 995.19	Gravimetry (Röse-Gottlieb)	
Creams, whipped creams and fermented creams	Milk solids-not-fat (MSNF)xxxv	ISO 3727-2 IDF 80-2	Gravimetry	Ι
Cream cheese	Dry matter	ISO 5534 IDF 4	Gravimetry drying at 102 °C (forced air oven)	Ι
Cream cheese	Moisture on fat-free basis	ISO 5534 IDF 4	Calculation from fat content and moisture content,	Ι
		ISO 23319 IDF 250	gravimetry drying at 102 °C (forced air oven), gravimetry (Schmid-Bondzynski- Ratzlaff)	
Dairy fat spreads	Milk fat purity	ISO 17678 IDF 202	Calculation from determination of triglycerides by gas chromatography– FID	Ι
Dairy fat spreads	Milkfat (total fat)	ISO 17189 IDF 194	Gravimetry Gravimetry (direct determination of fat using solvent extraction)	Ι
Dairy permeate powders	Lactose	ISO 22662 IDF 198	High performance liquid chromatography	II
Dairy permeate powders	Milkfat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	I
Dairy permeate powders	Nitrogen	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	Ι
Dairy permeate powders	Moisture ^{xxxvi}	ISO 5537 IDF 26	Gravimetry (drying at 87 °C)	
Dairy permeate powders	Ash	NMKL 173 / AOAC 930.30	Gravimetry (ashing at 550 °C)	
Edible casein products	Free acidity	ISO 5547 IDF 91	Titrimetry (aqueous extract)	

^{xxxv} Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.

Milk and milk products				
Commodity	Provisions	Method	Principle	Туре
Edible casein products (caseins obtained by rennet precipitation and of caseinates, with the exception of ammonium caseinate)	Ash (including P_2O_5)	ISO 5545 IDF 90	Gravimetry (ashing at 825 °C)	I

Commodity	Provisions	Method	Principle	Туре
Edible casein products (acid caseins, of ammonium caseinates, of their mixtures with rennet casein and with caseinates, and of caseins of unknown type)	Ash (including P ₂ O ₅)	ISO 5544 IDF 89	Gravimetry (ashing at 825 °C)	Ι
Edible casein products	Lactose	ISO 5548 IDF 106	Photometry (phenol and H_2SO_4)	IV
Edible casein products	Milkfat (total fat)	ISO 23319 IDF 250	Gravimetry (Schmid-Bondzynski- Ratslaff)	I
Edible casein products	рН	ISO 5546 IDF 115	Electrometry	II
Edible casein products	Milk protein (total N x 6.38 in	ISO 5550 IDF 78 and	Calculation from dry matter content	I
	dry matter)	ISO 8968-1 IDF 20-1	and protein content	
			Gravimetry, drying at 102 °C and	
			titrimetry (Kjeldahl)	
Edible casein products	Sediment (scorched particles)	ISO 5739 IDF 107	Visual comparison with standard discs, after filtration	IV
Edible casein products	Water ^{xxxvii}	ISO 5550 IDF 78	Gravimetry (drying at 102 °C)	I
Emmental	Calcium > = 800 mg/100 g	ISO 8070 IDF 119	Flame atomic absorption	
Emmental	Calcium > = 800 mg/100 g	AOAC 2015.06 / ISO 21424 IDF 243	ICP mass spectrometry	II
Emmental	Calcium > = 800 mg/100 g	AOAC 2011.14 / ISO 15151 IDF 229	ICP emission spectroscopy	III
Emmental	Propionic acid	ISO/TS 19046-11 IDF/RM 233-1	Gas Chromatography -FID	IV
Emmental	Propionic acid	ISO/TS 19046-2I IDF/RM 233-2	Ion exchange chromatography - UV	IV
Evaporated milks	Milkfat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	I

xxxvii Water content excluding the crystallized water bound to lactose (generally known as moisture content).

Milk and milk products				
Commodity	Provisions	Method	Principle	Туре
Evaporated milks	Milk protein in MSNF ^{xxxviii}	ISO 6731 IDF 21 and ISO 23318 IDF 249 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	I
Evaporated milks	Milk SOlidS ^{xxxviiiError!} Bookmark not defined.	ISO 6731 IDF 21	Gravimetry, drying at 102 °C	I
Fermented milks	Colony-forming units of yeasts and/or moulds	ISO 6611 IDF 94	Colony count at 25 °C	IV
Fermented milks	Dry matter (total solids)xxxviiixviii	ISO 13580 IDF 151	Gravimetry, drying at 102 °C	I
Fermented milks	Total acidity expressed as percentage of lactic acid	ISO/TS 11869 IDF/RM 150	Potentiometry, titration to pH 8.30	IV
Fermented milks	Lactobacillus acidophilus	ISO 20128 IDF 192	Colony count at 37 °C	I
Fermented milks Yoghurt and yoghurt products	Quantification of <i>Lactobacillus</i> delbrueckii subsp. bulgaricus and Streptococcus thermophilus	ISO 7889 IDF 117	Colony count at 37 °C	I
Fermented milks	Identification of Lactobacillus	ISO 9232 IDF 146	Test for strain identification	I
Yoghurt and yoghurt products	delbrueckii subsp. bulgaricus and Streptococcus thermophilus			
Fermented milks	Sum of microorganisms constituting the starter culture (bacteria in fermented milk deriving (or originating) from starter culture)	ISO 27205 IDF 149 (Annex A)	Colony count at 25 °C, 30 °C, 37 °C and 45 °C according to the starter organism in question	I
Fermented milks	Milkfat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	I

xxxviii Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.

Milk and milk products				
Commodity	Provisions	Method	Principle	Туре
Fermented milks	Milk protein	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	I
Milk powders and cream powders	Acidity, titratable	ISO 6091 IDF 86	Titrimetry, titration to pH 8.4	I
Milk powders and cream powders	Milkfat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	
Milk powders and cream powders	Milk protein	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	I
Milk powders and cream powders	Scorched particles	ISO 5739 IDF 107	Visual comparison with standard discs, after filtration	IV
Milk powders and cream powders	Scorched particles	ADPI Scorched Particles, 2016	Visual comparison with standard discs, after filtration	IV
Milk powders and cream powders	Solubility index	ISO 8156 IDF 129	Centrifugation	1
Milk powders and cream powders	Water ^{xxxix} (moisture)	ISO 5537 IDF 26	Gravimetry (drying at 87 °C)	1
Milk powders and cream powders	Water ^{xxxix} (moisture)	Described in Appendix XI ^{xI}	Gravimetry (drying at 102 °C)	IV
Milk fat products	Fatty acids, free (expressed as oleic acid)	ISO 1740 IDF 6	Titrimetry	Ι
Milk fat products	Milkfat purity	ISO 17678 IDF 202	Calculation from determination of triglycerides by gas chromatography– FID	Ι
Milk fat products (anhydrous milkfat)	Peroxide value (expressed as meq. of oxygen/kg fat)	ISO 3976 IDF 74	Photometry	Ι
Milk fat products	Water ^{xxxix}	ISO 5536 IDF 23	Titrimetry (Karl Fischer)	
Mozzarella	Milkfat in dry matter – with high moisture	ISO 5534 IDF 4 and ISO 23319 IDF 250	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Schmid-Bondzynski- Ratzlaff)	I
Mozzarella	Milkfat in dry matter – with low moisture	ISO 5534 IDF 4 and ISO 23319 IDF 250	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Schmid-Bondzynski- Ratzlaff)	I
Sweetened condensed milk	Milkfat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	Ι

^{xxxix} Water content excluding the crystallized water bound to lactose (generally known as moisture content). ^{xI} Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method described in Appendix XI is listed as Type IV.

Commodity	Provisions	Method	Principle	Туре
Sweetened condensed milks (for products sweetened with sucrose only)	Milk protein in MSNF ^{xli}	ISO 6734 IDF 15 and ISO 23318 IDF 249 and ISO 2911 IDF 35 and ISO 8968-1 IDF 20-1	Calculation from total solids content, fat content, sucrose and protein content, gravimetry, drying at 102 °C and polarimetry, gravimetry (Röse-Gottlieb), titrimetry (Kjeldahl)	I
Sweetened condensed milks	Solids ^{xli}	ISO 6734 IDF 15	Gravimetry, drying at 102 °C	I
Whey cheeses by coagulation	Milkfat	ISO 23319 IDF 250	Gravimetry (Schmid-Bondzynski- Ratzlaff)	I
Whey cheeses by coagulation	Milkfat in dry matter	ISO 23319 IDF 250 and ISO 5534 IDF 4	Calculation from fat content and dry matter content,	Ι
			gravimetry (Schmid-Bondzynski-Ratzlaff),	
			gravimetry, drying at 102 °C	
Whey cheeses by concentration carbohydrate contents below 5%)	Milkfat (total fat)	ISO 23318 IDF 59249	Gravimetry (Röse-Gottlieb)	I
Whey cheeses by concentration (does not dissolve completely in the ammonia, contains fats and fatty acid FFA) in significant quantities or carbohydrate content > 5%)	Milkfat (total fat)	ISO 8262-3 IDF 124-3	Gravimetry (Weibull-Berntrop)	1
Whey cheeses by concentration	Milkfat in dry matter	ISO 23318 IDF 249 and	Calculation from fat content and dry	I
for carbohydrate content under 5%)	(total fat in dry matter)	ISO 2920 IDF 58	matter content, gravimetry (Röse-Gottlieb) gravimetry, drying at 88 °C	
Whey cheeses by concentration (does no dissolve completely in the ammonia, contains FFA in significant quantities, or carbohydrate content >5%)	Milkfat in dry matter (total fat in dry matter)	ISO 8262-3 IDF 124-3 and ISO 2920 IDF 58	Calculation from fat content and dry matter contents, gravimetry (Weibull-Berntrop) gravimetry, drying at 88 °C	I

x^{li} Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.

Provisions	Method	Principle	Туре
Ash	ISO 5545 IDF 90	Gravimetry (ashing at 825 M °C)	IV
Lactose	ISO 5765-1/2 IDF 79-1/2	Enzymatic method: Part 1– Glucose moiety or Part 2– Galactose moiety	II
Milkfat	ISO 23318 IDF 249	Gravimetry (Röse-Gottlieb)	
Milk protein	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	
Water ^{xlii} (moisture)	ISO 5537 IDF 26	Gravimetry (drying at 87 °C)	<u> </u>
· · ·	Ash Lactose Milkfat Milk protein	Ash ISO 5545 IDF 90 Lactose ISO 5765-1/2 IDF 79-1/2 Milkfat ISO 23318 IDF 249 Milk protein ISO 8968-1 IDF 20-1	Ash ISO 5545 IDF 90 Gravimetry (ashing at 825 M °C) Lactose ISO 5765-1/2 IDF 79-1/2 Enzymatic method: Part 1– Glucose moiety or Part 2– Galactose moiety Milkfat ISO 23318 IDF 249 Gravimetry (Röse-Gottlieb) Milk protein ISO 8968-1 IDF 20-1 Titrimetry (Kjeldahl)

xlii Water content excluding the crystallized water bound to lactose (generally known as moisture content).

Table 7. Numeric performance criteria for methods of analysis for copper and iron in milk fat products

Commodity	Provision	ML (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	RSDR (%)	Recovery	Minimum applicable range		Examples of applicable	Principle	
							Minimum	Maximum	methods that meet the criteria	Thicipie	
Milk fat		0.05 0.010			44.0	60–115%	0.028	0.028 0.072	AOAC 2015.06 / ISO 21424 IDF 243	ICP mass spectrometry	
products	Copper		0.010	0.020					ISO 5738 IDF 76	Photometry, (diethyldithiocarbamate)	
									AOAC 960.40	Photometry, (diethyldithiocarbamate)	
Milk fat products	Iron	0.2	0.020	0.040	40.8	80–110%	0.08	0.32	AOAC 2015.06 / ISO 21424 IDF 243	ICP mass spectrometry	

Commodity	Provision	ML (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	RSDR (%)	Recovery (%)	Minin applicabl Minimum N	e range	Examples of applicable methods that meet the criteria	Principle
Edible casein products	Copper	5	0.50	1.0	25.1	80–110	3.1	6.9	AOAC 2015.06 / ISO 21424 IDF 243 AOAC 2011.14 / ISO 15151 IDF 229 AOAC 985.35 ISO 5738 IDF 76	ICP mass spectrometry ICP emission spectroscopy Atomic absorption spectrophotometry Colorimetry (diethyldiethiocarbamate)
Edible casein products	Iron	20	2.0	4.0	20.4	80–110	13,9	26.1	AOAC 2015.06 / ISO 21424 IDF 243 AOAC 2011.14 / ISO 15151 IDF 229	ICP mass spectrometry ICP emission spectroscopy
	Iron (in roller dried caseinates)	50	5.0	10.0	17.8	90–107	36.7	63.3	AOAC 2015.06 / ISO 21424 IDF 243 AOAC 2011.14 / ISO 15151 IDF 229	ICP mass spectrometry ICP emission spectroscopy

Table 8. Numeric performance criteria for copper and iron in edible casein products

Commodity	Provisions	Method	Principle	Туре
Natural mineral waters	Calcium	ISO 7980	Atomic absorption spectrophotometry	
Natural mineral waters	Chloride	<i>Examination of Water Pollution Control.</i> WHO Pergamon Press (1982) Vol. 2, pp. 205-208		II
Natural mineral waters	Chloride	AOAC 973.51	Titrimetry (mercuric nitrate)	
Natural mineral waters	Chloride	ISO 9297	Titrimetry	
Natural mineral waters	Iron, dissolved	ISO 6332	Spectrophotometry	II
Natural mineral waters	Magnesium	ISO 6059	Titrimetry	II
Natural mineral waters	Magnesium	ISO 7980	Atomic absorption spectrophotometry	
Natural mineral waters	Phenols	ISO 6439	Spectrophotometry	I
Natural mineral waters	Potassium	<i>Examination of Water Pollution Control.</i> WHO Pergamon Press (1982) Vol. 2, pp. 142-145		II
Natural mineral waters	Sodium	<i>Examination of Water Pollution Control.</i> WHO Pergamon Press (1982) Vol. 2 pp. 148-151		II
Natural mineral waters	Sodium	<i>Examination of Water Pollution Control.</i> WHO Pergamon Press (1982) Vol. 2, pp. 151-152		
Natural mineral waters	Sulphates	ISO 9280	Gravimetry	
Natural mineral waters	Sulphide	Handb. Spurenanal. 1974		IV

Provision	ML	Min. applicable	LOD	LOQ	Precision RSDR (%)	Recovery	Suggested methods	Principle
	(mg/L)	range (mg/L)	(mg/L)	(mg/L)	no more than	(%)	meeting the criteria	
Antimony	0.005	0.0028	0.001	0.002	44	80–110	ISO 17294-2	ICP-MS
							ISO 15586	GF-AAS
							EPA 200.8	ICP-MS
Arsenic	0.01	0.0056	0.002	0.004	44	90–107	ISO 17294-2	ICP-MS
							ISO 15586	GF-AAS
							ISO 11969	AAS (Hydride)
							EPA 200.8	ICP-MS
Barium	0.7	0.35	0.07	0.14	34	95–105	ISO 11885	ICP-OES
							ISO 17294-2 EPA 200.8	ICP-MS
							LI A 200.0	ICP-MS
Borate	5	3.1	0.5	1	25	97–103	ISO 9390	Spectrophotometry
							ISO 11885 ISO 17294-2	ICP-OES ^{xiiii}
							130 17294-2	ICP-MS ^{xliii}
Cadmium	0.003	0.0017–0.0043	0.0006	0.0012	44	40–120	ISO 17294-2	ICP-MS
							EPA 200.8	ICP-MS
							EN 17851	ICP-MS
							EN 14083	GF-AAS
Chromium	0.05	0.028	0.01	0.02	44	90–107	ISO 11885	ICP-OES
							ISO 17294-2	ICP-MS
							ISO 15586ISO 18412 (Cr	GF-AAS
							VI)	Photometric
							ISO 23913 (Cr VI)	CIA, spectrophotometry
							ISO 9174 (Section 4)	AAS
							EPA 200.8	ICP-MS
Copper	1	0.52	0.1	0.2	32	97–103	ISO 11885	ICP-OES
							ISO 17294-2	ICP-MS

^{xliii} Total boron is determined.

Provision	ML (mg/L)	Min. applicable range (mg/L)	LOD (mg/L)	LOQ (mg/L)	Precision RSDR (%) no more than	Recovery (%)	Suggested methods meeting the criteria	Principle
							ISO 15586	GF-AAS
							ISO 8288	Flame-AAS
							EPA 200.8	ICP-MS
Cyanide	0.07	0.039	0.014	0.028	44	90–107	ISO 14403	CFA
							ISO 6703-1	Photometric, trimetric
Fluoride	1.0	0.52	0.1	0.2	32	97–103	ISO 10304-1	LC of ions
							ISO 10359-1 (dissolved fluoride)	Electrochemical probe
							ISO 10359-2 (inorganic bound)	Digestion, distillation
Lead	0.01	0.006-0.014	0.002	0.004	44	60–115	ISO 17294-2	ICP-MS
							ISO 15586	GF-AAS
							EPA 200.8	ICP-MS
Manganese	0.4	0.18	0.04	0.08	37	95–105	ISO 11885I	ICP-OES
							SO 17294-2	ICP-MS
							ISO 15586	GF-AAS
							EPA 200.8	ICP-MS
Mercury	0.001	0.00056	0.0002	0.0004	44	80–110	EN 1483	AAS
							ISO 17852	Enrichment by amalgamation (III)
							ISO 5666	AFS
							ISO 16590	AAS after tin (II) chloride
							EPA 200.8	reduction
								Enrichment by amalgamation (III)
								ICP-MS
Nickel	0.02	0.011	0.004	0.008	44	90–107	ISO 17294-2	ICP-MS
							ISO 15586	GF-AAS
							EPA 200.8	ICP-MS
Nitrate	50	37	5	10	18	98–102	ISO 10304-1	LC of ions
							ISO 13395	CFA, FIA, spectrophotometry
							ISO 7890-3	

Provision		Min. applicable	LOD	LOQ	Precision RSDR (%)	Recovery	Suggested methods meeting the criteria	Principle
	(mg/L)	range (mg/L)	(mg/L)	(mg/L)	no more than	(%)	meeting the chiefia	
Nitrite	0.1	0.03	0.01	0.02	44	95–105	ISO 10304-1	LC of ions UV
							ISO 13395	CFA, FIA, spectrophotometry
							ISO 6777	
Selenium	0.01	0.0056	0.002	0.004	44	90–107	ISO 17294-2	ICP-MS
							ISO 15586	GF-AAS
							ISO 9965	AAS (hydride)
							EPA 200.8	ICP-MS

Table 9b. Performance characteristics of suggested methods

Provision	ML	Applicable range-from:	LOD	RSDR (%)	Recovery (%)	Suggested methods	Principle
Surface active agents	-	0.05–5.0 mg/L	0.05 mg/l	< 44	70–100	ISO 16265	CFA
Mineral oil (hydrocarbon index)	-	>0.1 mg/L		< 41	71–102	ISO 9377-2	GC
РСВ	-	>15 ng/L		< 20	70–130	AOAC 990.06	GC ECD
Pesticide (organochlorine)	-	> 15 ng/L		< 20	70–130	AOAC 990.06	GC ECD
PAH	-	0.005 µg/L		< 10	80–110	ISO 17993	HPLC FD
		0.04 µg/L		< 18	80–110	ISO 7981-1	TLC
		0.005 µg/L		< 19	80–100	ISO 7981-2	HPLC

Processed fruits and vegetables									
Commodity	Provision	Method	Principle	Туре					
Processed fruits and vegetables (except jams, jellies, marmalades, pickled cucumbers, mango chutney, coconut milk and coconut cream)	Benzoic acid	NMKL 124	Liquid chromatography (UV)	11					

Processed fruits and vegetables										
Commodity	Provision	Method	Principle	Туре						
Processed fruits and vegetables (except jams, jellies, marmalades, pickled cucumbers, mango chutney, coconut milk and coconut cream)	Benzoic acid	AOAC 983.16	Gas chromatography	111						
Processed fruits and vegetables (canned strawberries, pickled cucumbers, preserved tomatoes, canned citrus fruits, certain canned vegetables)	Calcium	AOAC 968.31	Complexometry/Titrimetry	II						
Processed fruits and vegetables	Drained weight	AOAC 968.30 (Codex general method)	Gravimetry (sieving)	I						
Processed fruits and vegetables	Fill of glass containers	ISO 8106	Gravimetry	I						

Processed fruits and v	Processed fruits and vegetables								
Commodity	Provision	Method	Principle	Туре					
Processed fruits and vegetables	Fill of metal containers	ISO 90-1	Gravimetry	I					
Processed fruits and vegetables	Packing medium canned berry fruits (raspberry, strawberry)	AOAC 932.12 ISO 2173	Refractometry	I					
Processed fruits and vegetables (pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney and aqueous coconut products)	рН	ISO 1842	Potentiometry	IV					
Canned bamboo shoots	рН	AOAC 981.12	Potentiometry	IV					
Processed fruits and vegetables (pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney and aqueous coconut products)	рН	AOAC 981.12	Potentiometry	111					

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Processed fruits and vegetables									
Commodity	Provision	Method	Principle	Туре					
Processed fruits and vegetables (pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney and aqueous coconut products)	рН	NMKL 179	Potentiometry	II					
Processed fruits and vegetables (pickled cucumbers, processed tomato concentrates, preserved tomatoes, canned applesauce, jams, jellies and marmalades, mango chutney and certain canned fruit)	Soluble solids (packing medium)	ISO 2173	Refractometry	Ι					
Processed fruits and vegetables (except jams, jellies, marmalades, pickled cucumbers)	Sorbates	AOAC 983.16	Gas chromatography (Flame ionization)	111					
Processed fruits and vegetables (except jams, jellies, marmalades, pickled cucumbers)	Sorbates	NMKL 124	Liquid chromatography (UV)	11					

Processed fruits and	vegetables			
Commodity	Provision	Method	Principle	Туре
Processed fruits and vegetables	Total solids	AOAC 920.151	Gravimetry	I
Aqueous coconut products	Total fats	ISO 23318 IDF 249	Gravimetry (Röse- Gottlieb)	Ι
Aqueous coconut products	Total solids	ISO 6731 IDF 21	Gravimetry	I
Aqueous coconut products	Non-fat solids	ISO 23318 IDF249 and ISO 6731 IDF 21	Calculation: Gravimetry (Röse- Gottlieb) Gravimetry	I
Aqueous coconut products	Moisture	ISO 6731 IDF 21	Gravimetry	I
Canned apple sauce	Fill of glass containers	ISO 8106	Gravimetry	Ι
Canned apple sauce	Fill of metal containers	ISO 90-1	Gravimetry	I
Canned apple sauce	Soluble solids (packing medium)	ISO 2173 (Codex general method for processed fruits and vegetables)	Refractometry	I
Canned green beans and wax beans	Tough strings	See Appendix XII	Stretching	Ι
Canned green peas	Fill of glass containers	ISO 8106	Gravimetry	I
Canned green peas	Fill of metal containers	ISO 90-1	Gravimetry	I
Canned green peas	Types of peas, distinguishing	See Appendix XIII	Visual examination	I
Canned mangoes	Soluble solids (packing medium)	AOAC 932.14C	Brix spindle method (refractometry)	

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Processed fruits and vegetables					
Commodity	Provision	Method	Principle	Туре	
Canned mushrooms	Drained weight	AOAC 968.30	Gravimetry (sieving)	I	
Canned palmito	Mineral impurities	ISO 762 Gravimetry		Ι	
Canned stone fruits	Drained weight	AOAC 968.30	Gravimetry (sieving)	Ι	
Canned stone fruits	Soluble solids (packing medium)	ISO 2173 Refractometry		I	
Canned strawberries	Calcium	AOAC 968.31	Complexometric titrimetr		
Canned strawberries	Mineral impurities	ISO 762	Gravimetry		
Certain canned citrus fruits	Calcium	NMKL 153	Atomic absorption spectrophotometry (flame)	II	
Certain canned citrus fruits	Calcium	AOAC 968.31	Complexometry titrimetry		
Citrus marmalade	Calcium	AOAC 968.31	Complexometric titrimetry		
Dates	Identification of defects	See Appendix XIV	Visual examination	I	
Dates	Moisture AOAC 934.06 Gravimetry (vacu oven)		Gravimetry (vacuum oven)	I	
Desiccated coconut	Total acidity of the extracted oil	ISO 660 or AOCS Cd 3d-63	Titrimetry		
Desiccated coconut	Ash	AOAC 950.49	Gravimetry (ashing)	I	
Desiccated coconut Extraneous Described in Appendix vegetable matter		Described in Appendix XV	Counting extraneous material with the naked eye	IV	

Processed fruits and v	•				
Commodity	Provision	Method	Principle	Туре	
Desiccated coconut Moisture		AOAC 925.40 Gravimetry (lo drying)			
Desiccated coconut	Oil content	AOAC 948.22	Gravimetry	Ι	
Dried apricots	Identification of defects	See Appendix XVI	Visual inspection (gravimetry)	Ι	
Dried apricots	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	I	
Dried apricots	Sulphur dioxide	AOAC 963.20	Colorimetry	Ш	
Jams, jellies and marmalades	Fill of glass containers	ISO 8106	Gravimetry	I	
Jams, (fruit preserves) and jellies and marmalades	Soluble solids	ISO 2173	Refractometry	I	
Mango chutney	Ash insoluble in HCl	ISO 763	Gravimetry	Ι	
Pickled cucumbers	Acidity, total	AOAC 942.15	Titrimetry	Ι	
Pickled cucumbers	Drained weight	AOAC 968.30	Gravimetry	Ι	
Pickled cucumbers	Mineral impurities	ISO 762	Gravimetry	Ι	
Pickled cucumbers	Salt (NaCl)	AOAC 971.27 (Codex general method)	Potentiometry	II	
Pickled cucumbers	Volume fill by displacement	See Appendix XVII	Displacement	Ι	
Preserved tomatoes	Calcium	AOAC 968.31	Complexometric titrimetry	III	
Preserved tomatoes Calcium NMKL 153		NMKL 153	Atomic absorption spectrophotometry (flame)	II	

Processed fruits and vegetables						
Commodity	Provision	Method	Principle	Туре		
Preserved tomatoes Drained weight						
Preserved tomatoes	Mould count	AOAC 965.41	Howard mould count	Ι		
Processed tomato concentrates	Lactic acid	EN 12631	Spectrometry (enzymatic determination)	II		
Processed tomato concentrates	Mineral impurities (sand)	ISO 762	Gravimetry	IV		
Processed tomato concentrates	Mould count	AOAC 965.41	Howard mould count	Ι		
Processed tomato concentrates	Natural tomato soluble solids	AOAC 970.59	Refractometry	Ι		
Processed tomato concentrates	Sodium chloride	AOAC 971.27 (Codex general method)	Potentiometry	II		
Processed tomato concentrates	Tomato soluble solids	AOAC 970.59	Refractometry	I		
Raisins	Mineral impurities	ISO 762	Ashing	Ι		
Raisins	Mineral oil	CAC/RM 52	Extraction and separation on alumina	II		
Raisins	Moisture	AOAC 972.20	Electrical conductance	I		
Raisins	Sorbitol	AOAC 973.28	Gas chromatography (flame ionization)	II		
Raisins	Sulphur dioxide	AOAC 963.20	Colorimetry	II		
Table olives	Drained weight	AOAC 968.30 (Codex general method for processed fruits and vegetables)	Gravimetry (sieving)	I		
Table olives	Fill of glass containers	ISO 8106	Gravimetry			

Processed fruits and vegetables					
Provision Method		Principle	Туре		
Fill of metal containers	ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)	Gravimetry	Ι		
pH of brine	NMKL 179 (Codex general method for processed fruits and vegetables)	Potentiometry			
pH of brine	AOAC 981.12 (Codex general method for processed fruits and vegetables)	Potentiometry			
pH of brine	ISO 1842	Potentiometry	IV		
Salt in brine	AOAC 971.27 NMKL 178 (Codex general method)	Potentiometry	II		
	ProvisionFill of metal containerspH of brinepH of brinepH of brinepH of brinepH of brine	ProvisionMethodFill of metal containersISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)pH of brineNMKL 179 (Codex general method for processed fruits and vegetables)pH of brineAOAC 981.12 (Codex general method for processed fruits and vegetables)pH of brineISO 1842	ProvisionMethodPrincipleFill of metal containersISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)GravimetrypH of brineNMKL 179 (Codex general method for processed fruits and vegetables)PotentiometrypH of brineAOAC 981.12 (Codex general method for processed fruits and vegetables)PotentiometrypH of brineISO 1842Potentiometry		

 Table 10. Numeric performance criteria for benzoic acid, calcium, sorbates and tin in processed fruits and vegetables

Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria
Jams, jellies and marmalades	Benzoic Acid	1 000	830–1 170	100	200	11.3	95–105	ISO 5518, NMKL 124, AOAC 983.16
Pickled cucumbers	Benzoic Acid	1 000	830–1 170	100	200	11.3	95–105	NMKL 124, AOAC 983.16
Mango chutney	Benzoic Acid	250	197–302	25	50	13.9	90–107	ISO 5518, NMKL 124, AOAC 983.16
Coconut milk and coconut cream	Benzoic Acid	1 000	830–1 170	100	200	11.3	95–105	ISO 5518, NMKL 124, AOAC 983.16
Jams, jellies and marmalades	Sorbates	1 000	830–1 170	100	200	11.3	95–105	NMKL 124, AOAC 983.16
Pickled cucumbers	Sorbates	1 000	830–1 170	100	200	11.3	95–105	NMKL 124, AOAC 983.16

Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria
Processed fruits and vegetables	Tin	250	197–302	25	50	13.9	90–107	AOAC 980.19, NMKL 126, NMKL 191
Table olives	Tin	250	197–302	25	50	13.9	90–107	NMKL 190, EN 15764, NMKL 126, NMKL 191

Commodity	Provisions	Method	Principle	Туре
Meat products	Nitrates and/or nitrites	EN 12014-3	Spectrometric determination of nitrate and nitrite content of meat products after enzymatic reduction of nitrate to nitrite	111
Meat products	Nitrates and/or nitrites	EN 12014-4 NMKL 165	lon exchange chromatographic method	III
Processed meat and poultry products	Fat	ISO 1443	Gravimetry	Ι
Processed meat and poultry products	Lead	AOAC 934.07	Colorimetry (dithizone)	II
Processed meat and poultry products	Nitrates	ISO 3091	Colorimetry (cadmium reduction)	II
Processed meat and poultry products	Nitrites	ISO 2918	Colorimetry	IV
Processed meat and poultry products	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Processed meat and poultry products	Nitrogen/protein	ISO 937	Titrimetry	II
Bouillons and consommés (soups and broths)	Amino nitrogen	AIIBP Method No 2/7	Volumetry (modified Van Slyke)	II
Bouillons and consommés (soups and broths)	Creatinine	AIIBP Method No 2/5	HPLC	II
Bouillons and consommés (soups and broths)	Nitrogen, total	AOAC 928.08	Kjeldahl	II
Bouillons and consommés (soups and broths)	Sodium chloride	AIIBP Method No 2/4	Potentiometric titration (chloride expressed as sodium chloride)	II
Canned corned beef	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Canned corned beef	Nitrites, potassium and/or sodium salt	AOAC 973.31 (Codex general method)	Colorimetry	II

Commodity	Provisions	Method	Principle	Туре
Canned corned beef	Nitrites, potassium and/or sodium salt	ISO 2918	Colorimetry	IV
Canned corned beef	Tin (products in tinplate and other containers)	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured chopped meat	Fat	ISO 1443	Gravimetry (extraction)	I
Cooked cured chopped meat	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured chopped meat	Nitrites	AOAC 973.31 (Codex general method)	Colorimetry	II
Cooked cured chopped meat	Nitrites	ISO 2918	Colorimetry	IV
Cooked cured chopped meat	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured ham	Fat	ISO 1443	Gravimetry (extraction)	I
Cooked cured ham	Gelatin, added	Described in the standard	Calculation	I
Cooked cured ham	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured ham	Nitrites	AOAC 973.31 (Codex general method)	Colorimetry	II
Cooked cured ham	Nitrites	ISO 2918	Colorimetry	IV
Cooked cured ham	Protein (conversion factor 6.25)	ISO 937	Titrimetry, Kjeldahl digestion	II
Cooked cured ham	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured pork shoulder	Fat	ISO 1443	Gravimetry (extraction)	I
Cooked cured pork shoulder	Gelatin, added	Described in the standard	Calculation	Ι
Cooked cured pork shoulder	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II

Commodity	Provisions	Method	Principle	Туре
Cooked cured pork shoulder	Nitrites	AOAC 973.31 (Codex general method)	Colorimetry	II
Cooked cured pork shoulder	Nitrites	ISO 2918	Colorimetry	IV
Cooked cured pork shoulder	Protein	ISO 937	Titrimetry, Kjeldahl digestion	II
Cooked cured pork shoulder	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Luncheon meat	Fat	ISO 1443	Gravimetry (extraction)	I
Luncheon meat	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Luncheon meat	Nitrites, potassium and/or sodium salt	AOAC 973.31 (Codex general method)	Colorimetry	II
Luncheon meat	Nitrites, potassium and/or sodium salt	ISO 2918	Colorimetry	IV
Luncheon meat	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II

Quick-frozen fruits and vegetables						
Commodity	Provisions	Method	Principle	Туре		
Quick-frozen fruits and vegetables(non-glazed)	Net weight	AOAC 963.26	Weighing	I		
Quick-frozen fruits and vegetables	Thawing procedure	See Appendix XVIII	Thawing	I		
Quick-frozen fruits and vegetables: berries, leek and carrot	Mineral impurities	AOAC 971.33	Gravimetry	I		
Quick-frozen fruits and vegetables: berries, whole kernel corn and corn-on-the-cob	Soluble solids, total	AOAC 932.12	Refractometry	I		

Commodity	Provisions	М	lethod	Principle Type	
Quick-frozen fruits and vegetables: peaches and berries	Drained fruit/drained A berries	AOAC 95	3.15	Draining I	
Quick-frozen fruits and vegetables: vegetables	Cooking procedure S	See Appe	endix XIX	Cooking I	
Quick-frozen French-fried potatoes	Moisture A	AOAC 98	4.25	Gravimetry (convection oven) I	
Quick-frozen green and wax beans	Tough strings S	See Appe	endix XII	Stretching I	
Quick-frozen peas	Solids, alcohol S insoluble	See Appe	endix XX	Gravimetry I	
Quick-frozen spinach	Dry matter, sodium S chloride-free	See Appe	endix XXI	Weighing I	
Spices and culinary herbs					
Commodity	Provisions		Method	Principle	Тур
Allspice, juniper berry and star anise	Moisture		ISO 939	Distillation	I
Allspice, juniper berry and star anise	Total ash on dry basis		ISO 939 and ISO 928	Calculation from moisture and ash (at 550°C), distillation and gravimetry	I
Allspice, juniper berry and star anise	Acid-insoluble ash on dry l	basis	ISO 939 and ISO 930	Calculation from moisture and ash (at 550°C), distillation and gravimetry	I
Allspice, juniper berry and star anise	Volatile oils on dry basis		ISO 939 and ISO 6571	Calculation from moisture and volatile oils, distillation and distillation	I
Allspice, juniper berry and star anise	Extraneous matter		ISO 927	Visual examination followed by gravimetry	I
Allspice, juniper berry and star anise	Foreign matter		ISO 927	Visual examination followed by gravimetry	I
Allspice, juniper berry and star anise	Mould visible		ISO 927	Visual examination followed by gravimetry	I
Allspice, juniper berry and star anise	Mammalian and other exc (whole spice)	reta	MPM V-8 Spices, Condiments, Flavors and Crude Drugs MPM:	Visual examination followed by gravimetry	IV

Commodity	Provisions	Method	Principle	Туре
Allspice, juniper berry and star anise	Whole dead insects and live insects	ISO 927	Visual examination (counting)	I
Allspice, juniper berry and star anise	Insect fragments (for whole spices)	ISO 927	Visual examination (counting)	I
Allspice, juniper berry and star anise	Insect fragments (for powdered/pieces)	AOAC 975.49	Flotation	Ι
Allspice, juniper berry and star anise	Insect defiled	ISO 927	Visual examination followed by gravimetry	I
Allspice, juniper berry and star anise	Rodent hair	AOAC 965.40	Flotation	I
Cloves	Moisture	ISO 939	Distillation	I
Cloves	Volatile oil on dry basis	ISO 939 and ISO 6571	Calculation from moisture and volatile oils, distillation and distillation	I
Cloves	Total ash on dry basis	ISO 939 and ISO 928	Calculation from moisture and ash (at 550 ^o C), distillation and gravimetry	I
Cloves	Acid-insoluble ash on dry basis	ISO 939 and ISO 930	Calculation from moisture and ash (at 550 ^o C), distillation and gravimetry	Ι
Cloves	Extraneous matter	ISO 927	Visual examination followed by gravimetry	Ι
Cloves	Foreign matter	ISO 927	Visual examination followed by gravimetry	I
Cloves	Insect damage	ISO 927	Visual examination followed by gravimetry	I
Cloves	Insects/insect fragments	ISO 927	Visual examination (counting)	I
Cloves	Crude fibre	ISO 5498	Gravimetry	I
Cloves	Mould visible (for whole)	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) <u>https://www.fda.gov/food/laborat</u> ory-methods-food/mpm-v-8- spices-condiments-flavors-and- crude-drugs#v32	Visual examination followed by gravimetry	IV

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Cloves	Live insects	ISO 927	Visual examination (counting)	I
Cloves	Mammalian or/and other excreta (for whole)	MPM V-8 Spices, Condiments, Flavors and Crude Drugs A, General methods for spices, herbs and botanicals (v 32)	Visual examination followed by gravimetry	IV
		https://www.fda.gov/food/laborat ory-methods-food/mpm-v-8- spices-condiments-flavors-and- crude-drugs#v32		
Cumin	Moisture	ISO 939	Distillation	I
Cumin	Total ash	ISO 928	Gravimetry	I
Cumin	Acid-insoluble ash	ISO 930	Gravimetry	I
Cumin	Volatile oils	ISO 6571	Distillation/Volumetric	I
Cumin	Extraneous vegetable matter	ISO 927	Visual examination/Gravimetry	I
Cumin	Foreign matter	ISO 927	Visual examination/Gravimetry	I

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Cumin	Insect damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA)	Visual examination	IV
		http://www.fda.gov/Food/FoodSc ienceResearch/LaboratoryMetho ds/ucm084394.htm#v-32		
Cumin	Mammalian excreta	Macroanalytical Procedure Manual USFDA Technical Bulletin V.39 B (for whole)	Visual examination	IV
Cumin	Mammalian excreta	AOAC 993.27 (for ground)	Enzymatic detection method	IV
Cumin	Mould damage	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual, FDA) <u>http://www.fda.gov/Food/FoodSc</u> <u>ienceResearch/LaboratoryMetho</u> <u>ds/ucm084394.htm#v-32</u>	Visual examination	IV
Dried basil	Moisture	ISO 939	Distillation	I
Dried basil	Total ash on dry basis	ISO 939 and ISO 928	Calculation from moisture and ash at (at 550 °C), distillation and gravimetry	I
Dried basil	Acid-insoluble ash on dry basis	ISO 928 and ISO 930	Calculation from moisture and ash at (at 550 $^{\circ}$ C), distillation and gravimetry	I
Dried basil	Volatile oil on dry basis	ISO 939 and ISO 6571	Calculation from moisture and volatile oils, distillation and distillation	Ι
Dried basil	Extraneous matter	ISO 927	Visual examination followed by gravimetry	I
Dried basil	Foreign matter	ISO 927	Visual examination followed by gravimetry	I

Spices and culinary herbs				
Commodity	Provisions	Method	Principle 1	Гуре
Dried basil	Insect damage (whole leaves)	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) <u>https://www.fda.gov/food/laborat</u> <u>ory-methods-food/mpm-v-8-</u> <u>spices-condiments-flavors-and- crude-drugs#v32</u>	Visual examination followed by gravimetry	IV
Dried basil	Insects / insect fragments	ISO 927	Visual examination (counting)	I
Dried basil	Mould damage (for whole leaves)	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) <u>https://www.fda.gov/food/laborat</u> ory-methods-food/mpm-v-8- <u>spices-condiments-flavors-and- crude-drugs#v32</u>	Visual examination followed by gravimetry	IV
Dried basil	Mammalian excreta and other excreta (for whole leaves)	Method V-8 Spices, Condiments, Flavors and Crude Drugs A. General methods for spices, herbs and botanicals (V 32) (<u>https://www.fda.gov/food/laborat</u> <u>ory-methods-food/mpm-v-8-</u> <u>spices-condiments-flavors-and- crude-drugs#v32</u>	Visual examination followed by gravimetry	IV
Dried or dehydrated chilli pepper and paprika	Moisture	ISO 939	Distillation	I
Dried or dehydrated chilli pepper and paprika	Total ash on dry basis	ISO 939 and ISO 928	Calculation from moisture and ash (at 550 °C) distillation and gravimetry	, I
Dried or dehydrated chilli pepper and paprika	Acid-insoluble ash on dry basis	ISO 939 and ISO 930	Calculation from moisture and ash (at 550 °C) distillation and gravimetry	, I

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Dried or dehydrated chilli pepper and paprika	Pungency, Scoville Heat Units	ISO 3513	Sensory evaluation	I
Dried or dehydrated chilli pepper and paprika	Colour value	ISO 7541	Spectrophotometry	Ι
Dried or dehydrated chilli pepper and paprika	Mammalian excreta (whole)	ISO 927	Visual examination followed by gravimetry	I
Dried or dehydrated chilli pepper and paprika	Mould damage (for whole chilli peppers)	MPM V-8 Spices, Condiments, Flavors and Crude Drugs A. General methods for spices, herbs and botanicals (V.32)	Visual examination followed by gravimetry	IV
Dried or dehydrated chilli pepper and paprika	Mould damage (for ground)	AOAC 945.94	Visual examination (Howard mould count)	I
Dried or dehydrated chilli pepper and paprika	Insect damage (for whole chilli peppers)	MPM V-8 Spices, Condiments, Flavors and Crude Drugs A. General methods for spices, herbs and botanicals (V.32)	Visual examination followed by gravimetry	IV
Dried or dehydrated chilli pepper and paprika	Extraneous matter	ISO 927	Visual examination followed by gravimetry	Ι
Dried or dehydrated chilli pepper and paprika	Foreign matter	ISO 927	Visual examination followed by gravimetry	I
Dried or dehydrated chilli pepper and paprika	Live insects	ISO 927	Visual examination (counting)	Ι
Dried or dehydrated chilli pepper and paprika	Insect fragments	ISO 927	Visual examination (counting)	Ι
Dried or dehydrated chilli pepper and paprika	Rodent hair (ground chilli)	AOAC 978.22	Flotation	I
Dried or dehydrated chilli pepper and paprika	Rodent hair (ground paprika)	AOAC 977.25 B	Microscopic examination	I
Dried or dehydrated ginger	Moisture	ISO 939	Distillation	I

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Dried or dehydrated ginger	Total ash on dry basis	ISO 939 and ISO 928	Calculation from moisture and ash (at 600 °C), distillation and gravimetry	
Dried or dehydrated ginger	Acid-insoluble ash on dry basis	ISO 939 and ISO 930	Calculation from moisture and ash (at 600 °C), distillation and gravimetry	
Dried or dehydrated ginger	Volatile oil on dry basis	ISO 939 and ISO 6571	Calculation from moisture and ash (at 600 °C), distillation and distillation	
Dried or dehydrated ginger	Extraneous matter	ISO 927	Visual examination followed by gravimetry	
Dried or dehydrated ginger	Foreign matter	ISO 927	Visual examination followed by gravimetry	
Dried or dehydrated ginger	Insect damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8 Spices	Visual examination followed by gravimetry	IV
Dried or dehydrated ginger	Whole dead insect	ISO 927	Visual examination	I
Dried or dehydrated ginger	Mammalian/other excreta (for whole)	Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B	Visual examination followed by gravimetry	IV
		https://www.fda.gov/food/laborator y-methods-food/mpm-v-8-spices- condiments-flavors-and-crude- drugs#v32		
Dried or dehydrated ginger	Mould visible	ISO 927	Visual examination followed by gravimetry	I
Dried or dehydrated ginger	Live insects	ISO 927	Visual examination	I
Dried or dehydrated ginger	Calcium (as oxide) on dry basis	ISO 939 and ISO 928 and ISO 1003-Annex A	Calculation from moisture and ash (at 600 °C), and titrimetry	IV
Dried or dehydrated ginger	Sulphur dioxide	AOAC 990.28	Distillation followed by titrimetry	IV
Dried oregano	Moisture	ISO 939	Distillation	
Dried oregano	Total ash (dry weight basis)		Calculation from moisture and ash Distillation and gravimetry	

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Dried oregano	Acid-insoluble ash (dry weight basis)	ISO 939 and ISO 930	Calculation from moisture and ash Distillation and gravimetry	I
Dried oregano	Volatile oils (dry weight basis)	ISO 939 and ISO 6571	Calculation from moisture and volatile oils distillation and distillation	
Dried oregano	Extraneous matter	ISO 927	Visual examination followed by gravimetry	
Dried oregano	Foreign matter	ISO 927	Visual examination followed by gravimetry	

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Dried oregano	Mammalian excreta other excreta	Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (for whole) <u>https://www.fda.gov/food/laborat</u> <u>ory-methods-food/mpm-v-8-</u> <u>spices-condiments-flavors-and- crude-drugs#v32</u>	Visual examination	IV
Dried oregano	Whole dead insect	ISO 927	Visual examination	IV
Dried oregano	Whole dead insect	MPM V-8 Spices, Condiments, Flavors and Crude Drugs A. General methods for spices, herbs and botanicals (V 32) <u>https://www.fda.gov/food/laborat</u> <u>ory-methods-food/mpm-v-8-</u> <u>spices-condiments-flavors-and- crude-drugs#v32</u>	Visual examination	IV
Dried oregano	Mould visible	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) <u>https://www.fda.gov/food/laborat</u> <u>ory-methods-food/mpm-v-8-</u> <u>spices-condiments-flavors-and- crude-drugs#v32</u>	Visual examination	IV
Dried oregano	Insect damage	ISO 927	Visual examination	I
Nutmeg	Piece of mace	ISO 927	Visual examination followed by gravimetry	I
Nutmeg	Moisture	ISO 939	Distillation	I
Nutmeg	Total ash on dry basis	ISO 939 and ISO 928	Calculation from moisture and ash (at 550 °C), distillation and gravimetry	I

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Nutmeg	Acid-insoluble ash on dry basis	ISO 939 and ISO 930	Calculation from moisture and ash (at 550 °C), distillation and gravimetry	Ι
Nutmeg	Water-insoluble ash on dry basis	ISO 939 and ISO 929	Calculation from moisture and ash (at 550 °C), distillation and gravimetry	Ι
Nutmeg	Volatile oil content on dry basis	ISO 939 and ISO 6571	Calculation from moisture and volatile oils, distillation and distillation	Ι
Nutmeg	Extraneous matter	ISO 927	Visual examination followed by gravimetry	Ι
Nutmeg	Foreign matter	ISO 927	Visual examination followed by gravimetry	Ι
Nutmeg	Mould visible	ISO 927	Visual examination followed by gravimetry	Ι
Nutmeg	Insect defiled/infested	ISO 927	Visual examination followed by gravimetry	Ι
Nutmeg	Dead insect, insect fragments, rodent contamination (hair)	ISO 927	Visual examination (counting)	Ι
Nutmeg	Live insects	ISO 927	Visual examination (counting)	I
Nutmeg	Mammalian and/or other excreta (for whole and broken)	Microanalytical Procedure Manual, USFDA, Technical Bulletin V.41	Visual examination followed by gravimetry	IV
		https://www.fda.gov/food/laborat ory-methods-food/mpm-v-8- spices-condiments-flavors-and- crude-drugs#v32		
Saffron	Moisture	ISO 363202	Gravimetry (drying at 103 °C)	I

Spices and culinary herb	S			
Commodity	Provisions	Method	Principle	Туре
Saffron	Total ash on dry basis	ISO 3632-2 and ISO 928	Calculation from moisture and ash (at 550 °C), gravimetry and gravimetry	I
Saffron	Acid-insoluble ash on dry basis	ISO 3632-2 and ISO 928 and ISO 930	Calculation from moisture and ash (at 550 °C), gravimetry and gravimetry	Ι
Saffron	Soluble extract in cold water on dry basis	ISO 3632-2 and ISO 941	Calculation from moisture and soluble extract, gravimetry and extraction	Ι
Saffron	Taste strength (expressed as picrocrocin) $A_{1cm}^{1\%}$ 257 nm	ISO 3632-2	Absorbance	Ι
Saffron	Aroma strength (expressed as safranal) $A \frac{1\%}{1 cm}$ 330 nm	ISO 3632-2	Absorbance	Ι
Saffron	Colouring strength (expressed as crocin) $A_{1cm}^{1\%}$ 440 nm	ISO 3632-2	Absorbance	Ι
Saffron	Extraneous matter	ISO 3632-2	Visual examination followed by gravimetry	Ι
Saffron	Foreign matter	ISO 3632-2	Visual examination followed by gravimetry	Ι
Saffron	Insect damage	ISO 927	Visual examination followed by gravimetry	Ι
Saffron	Whole dead insects/insect fragments	ISO 927	Visual examination (counting)	Ι
Saffron	Mould visible	ISO 927	Visual examination followed by gravimetry	I
Saffron	Mammalian excreta (whole spice)	MPM V-8 Spices, Condiments, Flavors and Crude Drugs MPM: V-8 Spices, Condiments, Flavors and Crude Drugs FDA	Visual examination followed by gravimetry	I

Spices and culinary herbs	5			
Commodity	Provisions	Method	Principle	Туре
Saffron	Rodent filth	ISO 927	Visual examination followed by gravimetry	I
Thyme	Moisture	ISO 939	Distillation	Ι
Thyme	Total ash	ISO 928	Gravimetry	I
Thyme	Acid-insoluble ash	ISO 930	Gravimetry	I
Thyme	Volatile oils	ISO 6571	Distillation/Volumetric	I
Thyme	Extraneous vegetable matter	ISO 927	Visual examination/Gravimetry	I
Thyme	Foreign matter	ISO 927	Visual examination/Gravimetry	I
Thyme	Insect damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA) http://www.fda.gov/Food/FoodSc ienceResearch/LaboratoryMetho	Visual examination	IV
Thyme	Mammalian excreta	Macroanalytical Procedure Manual USFDA Technical Bulletin V.39 B (for whole)	Visual examination	IV
Thyme	Mammalian excreta	AOAC 993.27 (for ground)	Enzymatic detection method	IV
Thyme	Mould damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA)	Visual examination	IV
		http://www.fda.gov/Food/FoodSc ienceResearch/LaboratoryMetho ds/ucm084394.htm#v-32		
Turmeric	Moisture	ISO 939	Distillation	Ι

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Turmeric	Total ash on dry basis	ISO 939 and ISO 928	Calculation from moisture and ash at (at 550 °C), distillation and gravimetry	I
Turmeric	Acid-insoluble ash on dry basis	ISO 939 AND iso 930	Calculation from moisture and ash at (at 550 °C), distillation and gravimetry	I
Turmeric	Extraneous matter	ISO 927	Visual examination followed by gravimetry	I
Turmeric	Foreign matter	ISO 927	Visual examination followed by gravimetry	I
Turmeric	Insect defiled	ISO 927	Visual examination followed by gravimetry	Ι
Turmeric	Whole insects live/dead (for whole)	ISO 927	Visual examination (counting)	I
Turmeric	Whole insects live/dead (for powdered/pieces)	AOAC 975.49	Flotation	I
Turmeric	Mammalian or/and other excreta (whole)	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual) MPM: V-8 Spices <u>https://www.fda.gov/food/laborat</u> <u>ory-methods-food/mpm-v-8-</u> <u>spices-condiments-flavors-and-</u> <u>crude-drugs</u>	Visual examination followed by gravimetry	IV
Turmeric	Mould visible	ISO 927	Visual examination followed by gravimetry	Ι
Black and white pepper	Bulk density	ISO 959-1 Annex B (black) ISO 959-2 Annex A (white)	Gravimetry	IV
Black pepper	Light berries	ISO 959-1 Annex A (black)	Flotation	IV
Black, white and green pepper	Extraneous vegetable matter	ISO 927	Visual examination/Gravimetry	I
Black, white and green pepper	Foreign matter	ISO 927	Visual examination/Gravimetry	I

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Black, white and green pepper	Black berries	Physical separation and weighing	Visual examination	IV
		ISO 959-2		
Black, white and green pepper	Broken berries	Physical separation and weighing	Visual examination	IV
		ISO 959-2		
Black, white and green pepper	Mouldy berries	Macroanalytical Procedure Manual USFDA Technical Bulletin V.39 B	Visual examination	IV
Black, white and green pepper	Insect damage	Macroanalytical Procedure Manual USFDA Technical Bulletin V.39 B	Visual examination	IV
Black, white and green pepper	Pinheads or broken berries	Physical separation and weighing ISO 959-1	Visual examination	IV
Black, white and green pepper	Mammalian excreta	Macroanalytical Procedure Manual USFDA Technical Bulletin V.39 B (for pepper whole)	Visual examination (for whole pepper)	IV
Black, white and green pepper	Mammalian excreta	AOAC 993.27 (for ground pepper)	Enzymatic detection method (for ground pepper)	I
Black, white and green pepper	Moisture	ISO 939	Distillation	I
Black, white and green pepper	Total ash	ISO 928	Gravimetry	I
Black, white and green pepper	Non-volatile ether extract	ISO 1108	Soxhlet extraction	I
Black, white and green pepper	Volatile oils	ISO 6571	Distillation	I
Black, white and green pepper	Piperine content	ISO 5564	Spectrophotometry	I
-				

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Black, white and green pepper	Acid-insoluble ash	ISO 930	Gravimetry	I
Black, white and green pepper	Crude fibre	ISO 5498	Gravimetry	I
Small cardamom	Moisture	ISO 939	Distillation	I
Small cardamom	Total ash, on dry basis	ISO 939 and ISO 928	Calculation from moisture and ash (at 550 °C), distillation and gravimetry	I
Small cardamom	Acid-insoluble ash, on dry basis	ISO 939 and ISO 930	Calculation from moisture and ash (at 550 °C), distillation and gravimetry	I
Small cardamom	Volatile oil on dry basis	ISO 939 and ISO 6571	Calculation from moisture and volatile oils, distillation and distillation	I
Small cardamom	Extraneous matter	ISO 927	Visual examination followed gravimetry	Ι
Small cardamom	Foreign matter	ISO 927	Visual examination followed gravimetry	Ι
Small cardamom	Insect defiled/infested	ISO 927	Visual examination followed gravimetry	I
Small cardamom	Immature and shrivelled capsules	ISO 882-1 and ISO 927	Visual examination followed gravimetry	I
Small cardamom	Mammalian or/and other excreta (for whole)	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual MPM: V-8 Spices	Visual examination followed gravimetry	IV
		https://www.fda.gov/food/laborat ory-methods-food/mpm-v-8- spices-condiments-flavors-and- crude-drugs		
Small cardamom	Mould visible	ISO 927	Visual examination followed gravimetry	I

Spices and culinary herbs				
Commodity	Provisions	Method	Principle	Туре
Small cardamom	Empty and malformed capsules	ISO 882-1	Visual examination (counting)	I
Small cardamom	Whole insects live/dead (for whole)	ISO 927	Visual examination (counting)	I
Small cardamom	Whole insects live/dead (for powdered/pieces)	AOAC 975.49	Flotation	I

Sugars and honey				
Commodity	Provisions	Method	Principle	Туре
Honey	Acidity	MAFF Validated Method V19	Titrimetry	I
		J. Assoc. Public Analysts (1992) 28 (4) 171-175		
Honey	Diastase activity	IHC Method for determination of diastase activity with Phadebas, 2009 except that the incubation time should be increased from 15 to 30 minutes		IV
Honey	Moisture	AOAC 969.38B or MAFF Validated Method V21	Refractometry	I
Honey	Sample preparation	AOAC 920.180	-	-
Honey	Solids, water-insoluble	MAFF Validated Method V22 <i>J. Assoc. Public Analysts</i> (1992) 28(4) 189-193	Gravimetry	I
Honey	Sugars added (for sugar profile)	AOAC 998.18	Carbon isotope ratio mass spectrometry	I
Honey	Sugars added: detection of corn and cane sugar products	AOAC 978.17	Carbon isotope ratio mass spectrometry	I

Sugars and honey				
Commodity	Provisions	Method	Principle	Туре
Sugars (dextrose anhydrous and dextrose monohydrate)	D-Glucose	ISO 5377	Titrimetry	Ι
Sugars (dextrose anhydrous and dextrose monohydrate)	Solids, total	ISO 1741	Gravimetry (vacuum oven)	Ι

Sugars and honey				
Commodity	Provisions	Method	Principle	Туре
Sugars (dextrose anhydrous and dextrose monohydrate, dried glucose syrup, glucose syrup, powdered dextrose, lactose)	Sulphated ash	ISO 5809	Single sulphonation	Ι
Sugars (dextrose anhydrous and dextrose monohydrate)	Sulphur dioxide	ISO 5379	Acidimetry and nephelometry	IV
Sugars (fructose)	рН	ICUMSA GS 1/2/3/4/7/8-23	Potentiometry	I
Sugars (fructose)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	I
Sugars (fructose)	D-Fructose	ISO 10504	Liquid chromatography (refractive index detection)	II
Sugars (fructose)	D-Glucose	ISO 10504	Liquid chromatography (refractive index detection)	II
Sugars (fructose)	Loss on drying	ISO 1742	Gravimetry	Ι
Sugars (fructose)	Sulphur dioxide	ISO 5379	Acidimetry and nephelometry	IV
Sugars (glucose syrup and dried glucose syrup)	Reducing sugar	ISO 5377	Titrimetry	Ι
Sugars (glucose syrup and dried glucose syrup)	Solids, total	ISO 1742	Gravimetry (vacuum oven)	Ι
Sugars (glucose syrup and dried glucose syrup)	Sulphur dioxide	ISO 5379	Acidimetry and nephelometry	IV
Sugars (lactose)	Lactose, anhydrous	ICUMSA GS 4/3-3	Titrimetry	П
Sugars (lactose)	Loss on drying	USP General Chapter 731	Gravimetry (drying at 120 °C for 16 h)	Ι
Sugars (lactose)	рН	ICUMSA GS 1/2/3/4/7/8-23	Potentiometry	I
Sugars (plantation and mill white sugar)	Colour	ICUMSA GS 9/1/2/3-8	Photometry	Ι
Sugars (plantation or mill white sugar)	Conductivity ash	ICUMSA GS 1/3/4/7/8-13	Conductimetry	I

Sugars and honey				
Commodity	Provisions	Method	Principle	Туре
Sugars (plantation or mill white sugar)	Invert sugar	ICUMSA GS 1/3/7-3	Titrimetry (Lane & Eynon)	Ι
Sugars (plantation or mill white sugar)	Loss on drying	ICUMSA GS 2/1/3-15	Gravimetry	I
Sugars (plantation or mill white sugar)	Polarization	ICUMSA GS 1/2/3-1	Polarimetry	II
Sugars (plantation or mill white sugar)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II
Sugars (powdered sugar and powdered dextrose)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II
Sugars (powdered sugar)	Colour	ICUMSA GS 2/3-9	Photometry	Ι
Sugars (powdered sugar)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	I
Sugars (powdered sugar)	Invert sugar	ICUMSA GS 2/3-5 after filtration if necessary to remove any anticaking agents	Titrimetry	I
Sugars (powdered sugar)	Loss on drying	ICUMSA GS 2/1/3-15	Gravimetry	I
Sugars (powdered sugar)	Polarization	ICUMSA GS 2/3-1 after filtration if necessary to remove any anticaking agents	Polarimetry	II
Sugars (raw cane sugar)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II
Sugars (soft white sugar and soft brown sugar)	Conductivity ash	ICUMSA GS 1/3/4/7/8-13	Conductimetry	Ι
Sugars (soft white sugar and soft brown sugar)	Invert sugar	ICUMSA GS 4/3-3 (applicable at levels >10% m/m)	Titrimetry (Lane and Eynon)	Ι
Sugars (soft white sugar and soft brown sugar)	Invert sugar	ICUMSA GS 1/3/7-3 (applicable at levels <10% m/m)	Titrimetry (Lane and Eynon)	
Sugars (soft white sugar and soft brown sugar)	Loss on drying	ICUMSA GS 2/1/3-15	Gravimetry	Ι

Sugars and honey				
Commodity	Provisions	Method	Principle	Туре
Sugars (soft white sugar and soft brown sugar)	Sucrose plus invert sugar	ICUMSA GS 4/3-7	Titrimetry	I
Sugars (soft brown sugar)	Sulphated ash	ICUMSA GS 1/3/4/7/8-11	Gravimetry	Ι
Sugars (soft white sugar and soft brown sugar)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II
Sugars (soft white sugar)	Colour	ICUMSA GS 2/3-9	Photometry	I
Sugars (white sugar)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	I
Sugars (white sugar)	Invert sugar	ICUMSA GS 2/3-5	Titrimetry	I
Sugars (white sugar)	Loss on drying	ICUMSA GS 2/1/3-15	Gravimetry	I
Sugars (white sugar)	Polarization	ICUMSA GS 2/3-1	Polarimetry	II
Sugars (white sugar)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II
Miscellaneous products				
Commodity	Provisions	Method	Principle	Туре
Chilli sauce	рН	NMKL 179 (Codex general method) / AOAC 981.12	Potentiometry	II
Chilli sauce	рН	AOAC 981.12 (Codex general method)	Potentiometry	III
Chilli sauce	Fill of containers	CAC/RM 46 (see Appendix X) (Codex general method) (for glass container)	Gravimetry	I
Cooked rice wrapped in plant leaves	Peroxide value	ISO 3960 / AOCS Cd 8b-90 Extraction of oils from product (see Appendix XXII)	Titrimetry	IV
Date paste	Moisture	AOAC 934.06	Gravimetry	
Date paste	Mineral impurities	ISO 762	Gravimetry	I
Date paste	Ash	AOAC 940.26	Gravimetry	

Miscellaneous products				
Commodity	Provisions	Method	Principle	Туре
Date paste	Acid soluble ash	AOAC 900.02D	Gravimetry, calculation	Ι
Dried fruits	Identification of defects	Described in the standard	Visual examination	Ι
Dried fruits (except prunes and raisins)	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	Ι
Dried meat	Chloride as sodium chloride (≥ 1.0%)	ISO 1841-1	Titrimetry (Volhard method)	III
Dried meat	Chloride as sodium chloride (≥ 0.25%)	ISO 1841-2	Titrimetry (potentiometry)	II
Dried meat	Ash	ISO 936	Gravimetry	Ι
Dried meat	Water activity	ISO 18787	Electrometry	П
Dried meat	Moisture content	AOAC 950.46B	Gravimetry	Ι
Dried meat	Protein* (*nitrogen-to-protein conversion factor = 6.25)	ISO 937	Calculation and titrimetry	I
Dried meat	Total fat	ISO 1443	Gravimetry	I
Edible cassava flour	Ash	ISO 2171 and ISO 712	Calculation from moisture and gravimetry (incineration at 550 °C)	I
Edible cassava flour	Fibre, crude	ISO 5498	Gravimetry	Ι
Edible cassava flour	Moisture	ISO 712	Gravimetry (oven drying at 98 – 100 °C)	I
Edible cassava flour	Particle size	ICC Recommendation 207	Sieving and gravimetry	I
Fermented noni fruit juice	Brix value (soluble solids)	AOAC 983.17 / EN 12143 / IFUMA 8 / ISO 2173	Refractometry	IV
Fermented noni fruit juice	Ethanol	AOAC 2017.07	Enzymatic determination	IV

Miscellaneous products				
Commodity	Provisions	Method	Principle	Туре
Fermented noni fruit juice	Ethanol	IFUMA 52	Enzymatic determination	IV
Fermented noni fruit juice	Ethanol	AOAC 2016.12	Headspace GC-FID	IV
Fermented noni fruit juice	Identification of scopoletin	Method described in Appendix XXIII, Part A	Solid-phase extraction and thin layer chromatography	IV
Fermented noni fruit juice	Identification of deacetylasperulosidic acid	Method described in Appendix XXIII, Part B	Thin layer chromatography	IV
Fermented noni fruit juice	pH value	NMKL 179 / AOAC 981.12	Potentiometry	Ш
Fermented soybean paste	Total nitrogen	AOAC 984.13	Kjeldahl	I
Fermented soybean paste	Amino nitrogen	AOAC 920.154 on the conditions specified in the standard ^{xliv}	Volumetry	Ι
Fermented soybean paste	Moisture	AOAC 934.01	Gravimetry	I
		(≤ 70°C, ≤ 50 mm Hg)		
Food-grade salt	Arsenic	EuSalt/AS 015	ICP-OES	IV
Food-grade salt	Cadmium	EuSalt/AS 015	ICP-OES	III
Food-grade salt	Cadmium	EuSalt/AS 014	Atomic absorption spectrophotometry	IV
Food-grade salt	Calcium and magnesium	ISO 2482	Complexometric titrimetry	II

xliv Section 9.2 Determination of amino nitrogen

Preparation of test samples: Weigh 2 g of sample into a 250 ml beaker and mix the sample with 100 ml of cold (15 °C) NH₃-free H₂O and then stir the mixture for 60 min. Next, decant the mixture through a quantitative filter and collect the filtrate in a 100 ml volumetric flask.

End-point – A pH metre shall be used to determine the end-point instead of optical verification of colours.

Miscellaneous products				
Food-grade salt	Calcium and magnesium	EuSalt/AS 009	Flame atomic absorption spectrometry	
Food-grade salt	Calcium and magnesium	EuSalt/AS 015	ICP-OES	III
Food-grade salt	Copper	EuSalt/AS 015	ICP-OES	III
Food-grade salt	Insoluble matter	ISO 2479	Gravimetry	II
Miscellaneous products				
Commodity	Provisions	Method	Principle	Туре
Food-grade salt	lodine	EuSalt/AS 002	Titrimetry using sodium thiosulphate	П
Food-grade salt	lodine	EuSalt/AS 019	ICP-OES	III
Food-grade salt	lodine	WHO/UNICEF/ICCIDD method ^{xlv} Only applicable to a product which has been fortified with iodate	Titrimetry using sodium thiosulphate	IV
Food-grade salt	Loss on drying	ISO 2483	Gravimetry (drying at 110 °C)	
Food-grade salt	Mercury	EuSalt/AS 012	Cold vapour atomic absorption spectrophotometry	IV
Food-grade salt	Potassium	EuSalt/AS 008	Flame atomic absorption spectrophotometry	II
Food-grade salt	Potassium	EuSalt/AS 015	ICP-OES	III
Food-grade salt	Sodium chloride	Described in the standard	Calculation	I
Food-grade salt	Sulphate	ISO 2480	Gravimetry	II
Food-grade salt	Sulphate	EuSalt/AS 015	ICP-OES	

xiv Assessment of iodine deficiency disorders and monitoring their elimination. A guide for programme managers. Third edition, Annex 1: Titration method for determining salt iodate and salt iodine content. World Health Organization, Geneva, 2007. The report is available from http://www.who.int/nutrition/publications/micronutrients/iodine_deficiency/WHO_NHD_01.1/en/index.html

Miscellaneous products				
Food-grade salt	Sulphate	EuSalt/AS 018	lon chromatography	III
Foul medames	Sample preparation	AOAC 945.68		_
Foul medames	Salt content	AOAC 971.27	Potentiometry	II
		NMKL 178		
Foul medames	Drained weight	AOAC 968.30	Sieving	I

Miscellaneous products				
Commodity	Provisions	Method	Principle	Туре
Gari	Ash	ISO 2171 and ISO 712	Calculation from moisture and Gravimetry (incineration at 550 °C)	I
Gari	Fibre, crude	ISO 5498 and ISO 712	Gravimetry (separation)	I
Gari	Moisture	ISO 712 Gravimetry (oven drying at 130 – 133 °C)		Ι
Gari	Particle size	ICC Recommendation 207	Sieving and gravimetry	I
Gari	Total acidity	ISO 7305 and ISO 712	Titrimetry (ethanol extraction)	I
Ginseng products	Moisture	AOAC 925.45 B (dried Gravimetry ginseng) Quantity of sample: 2 g		Ι
Ginseng products	Moisture	AOAC 925.45 D (ginseng extract) Quantity of sample: 1.5 g (mixing with 20 g of sea sand)	Gravimetry	Ι
Ginseng products	Solids	AOAC 925.45 B (dried ginseng) calculated by subtracting the content of water from 100% Quantity of sample: 2 g	Calculation	I
Ginseng products	Ash	AOAC 923.03 AACC Intl 08-01.01	Gravimetry	Ι
Ginseng products	Water-insoluble solids	Described in the standard (Annex I)	Gravimetry	Ι
Ginseng products	Water-saturated n-butanol extracts	Described in the standard (Annex II)	Gravimetry	I
Ginseng products	Identification of ginsenosides Rb1 and Rf	Described in the standard (Annex III)	TLC or HPLC	IV
Gochujang	Capsaicin	Journal of AOAC International Vol. 91 No. 2, 2008, pp 387-391	HPLC-Fluorescence	IV

Miscellaneous products	Provisions	Method	Principle	Tuna
<i>Commodity</i> Gochujang	Capsaicin	Journal of AOAC International Vol. 91 No. 2, 2008, pp 387-391	Gas chromatography-FID	<i>Type</i> I∨
Gochujang	Crude protein	AOAC 984.13	Titrimetry, Kjeldahl	I
		(Nitrogen conversion factor: 6.25)		
Gochujang	Moisture	AOAC 945.43	Gravimetry	I
Guideline level for acrylonitrile	Acrylonitrile	AOAC 985.13	Gas chromatography	II
Guideline levels for vinyl chloride monomer	Vinyl chloride monomer	ISO 6401	Gas chromatography	II
Guideline levels for vinyl chloride monomer	Vinyl chloride monomer	Commission Directive 81/432/EEC O.J. No. L.167, p. 6, 24.6.81	Gas chromatography ("headspace")	
Guidelines for nutrition labelling	Polyunsaturated fatty acids	AOCS Ce 1h-05 ^{xlvi}	Gas-liquid chromatography	П
Guidelines for nutrition labelling	Saturated fat	AOAC 996.06; or AOCS Ce 1h-05	Gas-liquid chromatography	II
Guidelines for nutrition labelling	Saturated fatty acids	AOCS Ce 1h-05	Gas-liquid chromatography	II
Harissa	Acidity	ISO 750	Titrimetry	Ι
Harissa	Acid-insoluble ash	ISO 763	Gravimetry	I
Harissa	Dry extract – soluble solids	ISO 2173	Refractometry	I
Halwa tehenia	Acidity	AOAC 924.53, AOAC 942.15	Titrimetry	IV

¹⁰²

 $^{^{\}mbox{xivi}}\mbox{Can}$ also be used to measure trans unsaturated fatty acids.

Commodity	Provisions	Method	Principle	Туре
Halwa tehenia	Ash	AOAC 900.02 AACC Intl 8.14.01	Gravimetry	I
Halwa tehenia	Fat	AOAC 963.15	Gravimetry	I
Halwa tehenia	Moisture	AOAC 925.45 AACC Intl 44.60.01	Gravimetry	I
Halwa tehenia	Sugars	ISI 28-1e ^{xlvii}	Titrimetry	IV
Humus with tehenia	Salt content	AOAC 971.27 NMKL 178	Potentiometry	II
Humus with tehenia	Total acidity	AOAC 925.53	Titrimetry	I

xlviihttp://www.starch.dk/isi/methods/28luff.htm

Miscellaneous products				
Commodity	Provisions	Method	Principle	Туре
Kava products for use as a beverage when mixed with water	Moisture	AOAC 925.45	Gravimetry	I
Mixed zaatar	Sodium chloride	ISO 939 and	Calculation by moisture and ash Distillation and	I
	(dry weight basis)	AOAC 971.27	titrimetry	
Mixed zaatar	Moisture	ISO 939	Distillation	I
Mixed zaatar	Acid-insoluble ash (dry weight basis)	ISO 939 and AOAC 941.12 (corrected for moisture by ISO 930)	Calculation by moisture and ash Distillation and gravimetry, Furnace, 550 ºC	I
Mixed zaatar	Extraneous matter	ISO 927	Visual examination Gravimetry	I
Mixed zaatar	Foreign matter	ISO 927	Visual examination Gravimetry	I
Mixed zaatar	Insects/Insect fragments	ISO 927	Visual examination	IV
Mixed zaatar	Insects/Insect fragments	AOAC 969.44	Visual examination	IV
Mixed zaatar	Insects/Insect fragments	AOAC 975.49	Visual examination	IV
Mixed zaatar	Mould damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA, Technical Bulletin Number 5)	Visual examination	IV

Miscellaneous products				
Commodity	Provisions	Method	Principle	Туре
Mixed zaatar	Mammalian excreta	Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (for whole)	Visual examination	IV
Mixed zaatar	Mammalian excreta	AOAC 993.27 (for ground)	Enzymatic detection method	IV
Non-fermented soybean products	Moisture content	AOAC 925.09 AACCI 44- 40.01	Gravimetry (vacuum oven)	l
Non-fermented soybean products	Protein content	NMKL 6 or AACCI 46-16.01 or AOAC 988.05 or AOCS Bc 4-91 or AOCS Ba 4d-90 (Nitrogen factor 5.71)	Titrimetry, Kjeldahl digestion	I
Sago flour	Moisture content	ISO 712	Gravimetry	I
Sago flour	Ash (inorganic extraneous matter)	ISO 2171	Gravimetry	I
Sago flour	Acidity	AOAC 939.05	Titrimetry	l
Sago flour	Crude fibre	ISO 6541	Gravimetry	l
Sago flour	Starch	AOAC 920.44	Gravimetry	l

Miscellaneous products				
Commodity	Provisions	Method	Principle	Туре
Soybean products fermente	d with <i>Bacillus species</i>			
Natto	Lipid content 4 g quantity of samples	AOAC 963.15	Gravimetry (Soxhlet)	I
Natto	Moisture content	AOAC 925.09	Gravimetry	I
Natto	Protein content (*nitrogen-to-protein conversion factor = 5.71)	AOAC 988.05	Titrimetry (Kjeldahl)	I
Cheonggukjang	Moisture content	AOAC 934.01	Gravimetry	I
Cheonggukjang	Protein content (*nitrogen-to-protein conversion factor = 5.71)	AOAC 988.05	Titrimetry (Kjeldahl)	I
Cheonggukjang	Lipid content 5 g quantity of samples	AOAC 963.15	Gravimetry (Soxhlet)	I
Thua Nao	Moisture content	AOAC 925.09	Gravimetry	I
Thua Nao	Protein content (*nitrogen-to-protein conversion factor = 5.71)	AOAC 988.05	Titrimetry (Kjeldahl)	I
Tehena	Moisture content	ISO 934	Gravimetry	I
Tehena	Protein content	ISO 1871	Titrimetry, Kjeldahl	I
Tehena	Total ash	ISO 6884	Gravimetry	I
Tehena	Acid-insoluble ash	ISO 735	Gravimetry	I
Tehena	Total acidity	ISO 729	Titrimetry	I

Miscellaneous products					
Commodity	Provisions	Method	Principle	Туре	
Tehena	Sesame oil	AOCS Cb 2-40 (Baudouin test)	Colour reaction	I	
Tempe	Moisture content	AOAC 925.09 AACCI 44- 40.01	Gravimetry (vacuum oven)	Ι	
Tempe	Protein content	NMKL 6 or AOAC 988.05 or AACCI 46-16.01 (Nitrogen factor 5.71)	Titrimetry, Kjeldahl digestion	I	
Tempe	Lipid content	AOAC 963.15	Gravimetry (Soxhlet extraction)	I	
Tempe	Crude fibre	ISO 5498 or AOAC 962.09 or AACCI 32-10.01	Gravimetry	Ι	
Laver products	Moisture content	AOAC 925.45B	Gravimetry, drying at atmospheric pressure	IV	
Laver products	Acidity: acid value for the extracted oil	See Appendix XXIV and	Extraction of oil	I	
		ISO 660 AOCS Cd 3d-63	Titrimetry		
Laver products	Moisture content	AOAC 925.45	Gravimetry, drying at atmospheric pressure	Ι	
Unrefined shea butter	Moisture content	ISO 662	Gravimetry	I	
Unrefined shea butter	Free fatty acid content acid value and acidity	ISO 660	Titrimetry	I	
		AOCS Cd 3d-63			
Unrefined shea butter	Relative density	AOCS Cc 10c-95/	Pycnometry	I	
		ISO 6883			

Miscellaneous products						
Commodity	Provisions	Method	Principle	Туре		
Unrefined shea butter	Saponification value	ISO 3657	Titrimetry	I		
		AOCS Cd 3d-25				
Unrefined shea butter	lodine value	AOAC 993.20/	Wijs-titrimetry	I		
		ISO 3961/				
		AOCS Cd 1d-92/				
		NMKL 39				
Unrefined shea butter	Peroxide value	AOCS Cd 8b-90/	Titrimetry	I		
		ISO 3960/				
		NMKL 158				
Unrefined shea butter	Unsaponifiable matter	ISO 3596/	Gravimetry			
		AOCS Ca 6a-40				
Unrefined shea butter	Insoluble impurities content	ISO 663/	Gravimetry	I		
		AOCS Ca 3a-46				
Unrefined shea butter	Melting point	ISO 6321	Open ended capillary tube	I		
		AOCS Cc 3b-92				

IRRADIATED FOODS

Commodity	Provision	Method	Principle	Туре
Food containing fat (e.g. raw meat and chicken, cheese, fruits)	Detection of irradiated food – Detection of radiation-induced hydrocarbons	EN 1784	Gas chromatographic analysis of hydrocarbons	II
Food containing fat (e.g. raw meat and chicken, liquid whole egg)	Detection of irradiated food – Detection of radiation-induced 2- alkylcyclobutanones	EN 1785	Gas chromatographic mass spectrometric analysis of 2- alkylcyclobutanones	111
Food containing bone	Detection of irradiated food – Radiation-induced Electron Spin Resonance (ESR) signal attributed to hydroxyapatite (principal component of bones)	EN 1786	ESR spectroscopy	II
Food containing cellulose (e.g. nuts and spices)	Detection of irradiated food – Radiation-induced Electron Spin Resonance (ESR) signal attributed to crystalline cellulose	EN 1787	ESR spectroscopy	II
Food containing silicate minerals (e.g. herbs, spices, their mixtures and shrimps)	Detection of irradiated foods – Thermoluminescence glow ratio used to indicate the irradiation treatment of the food	EN 1788	Thermoluminescence	II
Food containing silicate minerals (e.g. shellfish, herbs, spices, seasonings)	Detection of irradiated foods – Measurement of photo- stimulated luminescence intensity	EN 13751	Photo-stimulated luminescence	111
Food containing crystalline sugar (e.g. dried fruits and raisins)	Detection of irradiated food – Radiation-induced Electron Spin Resonance (ESR) signal attributed to crystalline sugar	EN 13708	ESR spectroscopy	II

Table 11. Numeric performance criteria for lead and cadmium in foods

					Met	hod perform	ance criteria	ı	
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Infant formula, formula for special medical purposes intended for infants and follow-up formula	lead	0.01	0.006–0.014	0.002	0.004	44	60–115	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851	ICP-MS ICP-MS ICP-MS ICP-MS
Milk	lead	0.02	0.011–0.029	0.004	0.008	44	60–115	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, EN 14083, NMKL 186	ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS ICP-MS
Secondary milk products (including butter, edible casein products and whey powders)	lead	0.02	0.011–0.029	0.004	0.008	44	60–115	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083, AOAC 999.11 (edible casein)	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS AAS or GF-AAS

		Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Fruit juices, except juices exclusively from berries and other small fruits	lead	0.03	0.017–0.043	0.006	0.012	44	60–115	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 15763, EN 17851, EN 14083, NMKL 186, NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS ICP-MS GF-AAS
Fat spreads and blended spreads	lead	0.04	0.022–0.058	0.008	0.016	44	60–115	EN 15763, EN 17851. NMKL 186 NMKL 161	ICP-MS ICP-MS ICP- MS GF-AAS
Grape juice	lead	0.04	0.022–0.058	0.008	0.016	44	60–115	EN 15763, EN 17851, NMKL 186 EN 14083, NMKL 161	ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028–0.072	0.010	0.020	44	60–115	EN 15763, EN 17851, NMKL 186, EN 14083, NMKL 161	ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS
Fruit juices obtained exclusively from berries and other small fruits, except grape juice	lead	0.05	0.028–0.072	0.010	0.020	44	60–115	FDA Method 4.7 Ver.1.2, AOAC 2013:06,	ICP-MS ICP-MS ICP-MS ICP-MS

			Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle	
								EN 15763, EN 17851, NMKL 186, EN 14083. NMKL 161	ICP-MS GF-AAS GF-AAS	
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028–0.072	0.010	0.020	44	60–115	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083. NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Preserved tomatoes	lead	0.05	0.028–0.072	0.010	0.020	44	60–115	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 186 EN 14083. NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Edible fats and oils (including fats and oils (all); named vegetable oils; olive oils and olive pomace oils)	lead	0.08	0.045–0.115	0.016	0.032	44	60–115	AOAC 994.02, AOCS Ca 17a-18,	GF-AAS ICP-OES GF-AAS ICP-MS ICP-MS	

			Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle	
								ISO 12193, EN 17851, NMKL 186	ICP-OES	
								ISO 21033		
Berries and other small fruits, except cranberry, currant and elderberry	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161, EN 14083	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS	
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 161 EN 14083	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS	
Bulb vegetables	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS	

					Met	hod perform	ance criteria	ı	
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
								NMKL 186 EN 14083. NMKL 161	GF-AAS
Canned fruits	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS
Canned vegetables	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS
Fruits, except cranberry, currants and elderberry	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851,	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS

			Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle	
								NMKL 186 EN 14083 NMKL 161		
Legume vegetables	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Meat and fat of poultry	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Meat of cattle, pigs and sheep	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2 AOAC 2013.06, EN 15763, EN 17851,	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	

			Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle	
								NMKL 186 EN 14083 NMKL 161		
Pickled cucumbers (cucumber pickles)	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Poultry, edible offal of	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Pulses	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	

					ance criteria				
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Root and tuber vegetables	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS
Wine from grapes harvested after July 2019	lead	0.1	0.03–0.17	0.01	0.02	44	80–110	EN 15763, OIV-MA- AS323-07.	ICP-MS ICP-MS
Fortified/liqueur wine from grapes harvested after 2019	lead	0.15	0.05–0.25	0.015	0.03	43	80–110	EN 15763, OIV-MA- AS323-07.	ICP-MS ICP-MS
Pig, edible offal of	lead	0.15	0.05–0.25	0.015	0.03	43	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS
Cattle, edible offal of	lead	0.2	0.08–0.32	0.02	0.04	41	80–110	FDA Method 4.7 Ver.1.2, AOAC	ICP-MS ICP-MS

			Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle	
								2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.08–0.32	0.02	0.04	41	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Cranberry	lead	0.2	0.08–0.32	0.02	0.04	41	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS. ICP-MS GF-AAS GF-AAS	
Currants	lead	0.2	0.08–0.32	0.02	0.04	41	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06,	ICP-MS ICP-MS ICP-MS ICP-MS	

					Met	hod perform	ance criteria	1	
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
								EN 15763, EN 17851, NMKL 186 EN 14083. NMKL 161	ICP-MS GF-AAS GF-AAS
Elderberry	lead	0.2	0.08–0.32	0.02	0.04	41	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS
Wine (wine and fortified/ liqueur wine) made from grapes harvested before July 2019	lead	0.2	0.08–0.32	0.02	0.04	41	80–110	EN 15763, OIV-MA- AS323-07	ICP-MS ICP-MS.
Fish	lead	0.3	0.13–0.47	0.03	0.06	38	80–110	AOAC 999.11, FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851	GF-AAS ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS

			Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle	
								NMKL 186 EN 14083 NMKL 161		
Fresh farmed mushrooms (common mushrooms (<i>Agaricus</i> <i>bisporous</i>), shiitake mushrooms (<i>Lentinula</i> <i>edodes</i>), and oyster mushrooms (<i>Pleurotus</i> <i>ostreatus</i>))	lead	0.3	0.13–0.47	0.03	0.06	38	80–110	EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Leafy vegetables, except spinach	lead	0.3	0.13–0.47	0.03	0.06	38	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Jams, jellies and marmalades	lead	0.4	0.18–0.62	0.04	0.08	37	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	

					Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle			
Mango chutney	lead	0.4	0.18–0.62	0.04	0.08	37	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS			
Table olives	lead	0.4	0.18–0.62	0.04	0.08	37	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013.06, EN 15763, EN 17851, NMKL 186 EN 14083, AOAC 999.11 NMKL 139 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS AAS (Flame Absorption) GF-AAS			
Salt, food grade	lead	1	0.5–1.5	0.1	0.2	32	80–110	EUsalt/AS 015, EN 17851, EN 14083	ICP-OES ICP-MS GF-AAS			
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.03–0.07	0.01	0.02	44	60–115	FDA Method 4.7 Ver.1.2, AOAC 2013:06,	ICP-MS ICP-MS ICP-MS ICP-MS			

				Method performance criteria							
Commodity Provis	Provision	ML Provision (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle		
								EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS GF-AAS GF-AAS		
Bulb vegetables	cadmium	0.05	0.03–0.07	0.01	0.02	44	60–115	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083.	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS		
Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.03–0.07	0.01	0.02	44	60–115	NMKL 161 FDA Method 4.7 Ver.1.2 AOAC 2013:06, EN 15763, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS		
Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice	cadmium	0.1	0.03–0.17	0.01	0.02	44	80–110	ISO 23637, EN 17851, NMKL 186 EN 14083 NMKL 161	GF-AAS ICP-MS ICP-MS GF-AAS GF-AAS		

					Met	hod perform	ance criteria	l	
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Legume vegetables	cadmium	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS
Pulses, except soya bean (dry)	cadmium	0.1	0.03–0.17	0.01	0.02	44	80–110	EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS
Root and tuber vegetables, except celeriac	cadmium	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS
Stalk and stem vegetables	cadmium	0.1	0.03–0.17	0.01	0.02	44	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851,	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS

			Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle	
								NMKL 186 EN 14083 NMKL 161	GF-AAS	
Leafy vegetables	cadmium	0.2	0.08–0.32	0.02	0.04	41	80–110	FDA Method 4.7 Ver.1.2, AOAC 2013:06, EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.08–0.32	0.02	0.04	41	80–110	ISO 23637, EN 17851, NMKL 186 EN 14083 NMKL 161	GF-AAS ICP-MS ICP-MS GF-AAS GF-AAS	
Chocolate containing or declaring < 30% total cocoa solids on a dry matter basis	cadmium	0.3	0.13–0.47	0.03	0.06	38	80–110	EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS	
Rice, polished	cadmium	0.4	0.18–0.62	0.04	0.08	37	80–110	ISO 23637 EN 17851,	GF-AAS ICP-MS	

			Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle	
								NMKL 186 EN 14083 NMKL 161	ICP-MS GF-AAS GF-AAS	
Salt, food grade	cadmium	0.5	0.23–0.77	0.05	0.10	36	80–110	EUsalt/AS 015, EN 17851, EN 14083.	ICP-OES ICP-MS GF-AAS	
Chocolate containing or declaring \geq 30% to < 50% total cocoa solids on a dry matter basis	cadmium	0.7	0.35–1.05	0.07	0.14	34	80–110	EN 15763, EN 17851 NMKL 186 EN 14083	ICP-MS ICP-MS ICP-MS GF-AAS	
Chocolate containing or declaring ≥ 50% to < 70% total cocoa solids on a dry matter basis, including sweet chocolate, gianduja chocolate, semi – bitter table chocolate, vermicelli chocolate/ chocolate flakes, and bitter table chocolate	cadmium	0.8	0.40–1.20	0.08	0.16	33	80–110	EN 15763, EN 17851, NMKL 186 EN 14083	ICP-MS ICP-MS ICP-MS GF-AAS	

				Method performance criteria							
Commodity	Provision	ML (mg/kg)	Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) no more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle		
Chocolate containing or declaring ≥ 70% total cocoa solids on a dry matter basis, including sweet chocolate, gianduja chocolate, semi – bitter table chocolate, vermicelli chocolate/ chocolate flakes, and bitter table	cadmium	0.9	0.46–1.34	0.09	0.18	33	80–110	EN 15763 EN 17851 NMKL 186 EN 14083	ICP-MS ICP-MS ICP-MS GF-AAS		
Cephalopods	cadmium	2	1.1–2.9	0.2	0.4	29	80–110	EN 15763, EN 17851, NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS		
Marine bivalve molluscs (clams, cockles and mussels), except oysters and scallops	cadmium	2	1.1–2.9	0.2	0.4	29	80–110	EN 15763 EN 17851 NMKL 186 EN 14083 NMKL 161	ICP-MS ICP-MS ICP-MS GF-AAS GF-AAS		

PART B – METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

Commodity categories	Method of sampling	Notes
Cereals, pulses and legumes and derived products		
Wheat protein products including wheat gluten	ISO 13690	
Fats and oils		
Olive oils and olive pomace oils	ISO 661 and ISO 5555	
Fish oils	ISO 5555	
Milk and milk products		
Milk products	ISO 707 IDF 50	General instructions for obtaining a sample from a bulk
Milk products	ISO 5538 IDF 113	Inspection by attributes
Milk products	ISO 3951-1	Inspection by variables
Processed fruits and vegetables		
Desiccated coconut	Described in the standard	
Certain canned vegetables, jams and jellies	Described in the standard	
Chilli sauce	Described in the standard	
Table olives	Described in the standard	

APPENDIX I

PART A - EXTRACTION OF OIL FROM INSTANT NOODLES

1. Extraction of oil from instant noodles

1.1 Apparatus

- (a) Rotary evaporator
- (b) Water bath

1.2 Preparation of test sample

Remove instant noodles from package and leave garnishing and seasoning in package. Transfer the noodles to plastic bag to prevent moisture change and then break these into small fragments with hands or wooden hammer. Select broken noodles in the size range of 2.36 mm to 1.7 mm by using two sieves with 2.36 mm and 1.7 mm openings and mix well. Use these noodles for the test sample. If the noodles are too thin to screen with sieves, cut them into 1 cm to 2 cm lengths, mix well and use these cut noodles for the test sample.

1.3 Extraction

Weigh 25 g test portion into 200 MI Erlenmeyer flask. Add 100 MI petroleum ether to the flask after replacing air in flask by N2 gas. Stopper flask and leave for 2 hours. Decant supernatant through filter paper into separating funnel. Add 50 MI petroleum ether to residue and filtrate supernatant through filter paper into the separating funnel. Add 75 MI water to the separating funnel and shake well. Allow layers to separate and drain the lower aqueous layer. Add water, shake and remove aqueous layer again as done previously. Decant the petroleum ether layer after dehydration with Na2SO4 into pear-shaped flask. Evaporate petroleum ether in the flask on rotary evaporator at not over 40 °C. Spray N2 gas on extract in the flask to remove all petroleum ether.

PART B – DETERMINATION OF ACID VALUE

2. Determination of acid value

2.1 Definition and principle

Acid value of oil from fried instant noodles = mg KOH required to neutralize 1 g oil. Oil extracted from noodle is dissolved in alcohol-ether mixture and titrated with alcoholic KOH standard solution.

2.2 Apparatus

Air-tight desiccator: silica gel heated at 150 °C is satisfactory drying agent.

2.3 Reagents

(a) Alcoholic potassium hydroxide standard solution: 0.05 mol/L. Dissolve 3.5 g potassium hydroxide in equal volume of water (CO₂-free) and add ethanol (95%) to 1 L. After mixing, let solution stand for several days keeping the solution CO₂-free. Use supernatant after standardization.

Standardization:

Weigh required quantity of amidosulfuric acid (certified reference material for volumetric analysis) and place it into desiccator (< 2.0 kPa) for 48 hours. Next, accurately weigh 1 g to 1.25 g (recording the weight to 0.1 mg), dissolve in water (CO₂-free), and dilute to 250 mL. Put 25 mL solution into Erlenmeyer flask, add 2 to 3 drops of bromothymol blue indicator and titrate with 0.05 mol/L alcoholic potassium hydroxide solution until colour of solution change to faint blue.

Calculation:

Factor of molarity = (g amidosulfuric acid x purity x 25) / 1.2136 / mL KOH

- (b) Alcohol-ether mixture: equal volumes ethanol (99.5%) and ether.
- (c) Phenolphthalein solution: 1% in alcohol.

2.4 Titration

Before sampling, liquefy extracted oil using water bath. Weigh 1 g to 2 g liquefied test portion into Erlenmeyer flask. Add 80 mL alcohol-ether mixture and a few drops of phenolphthalein solution. Titrate with 0.05 mol/L alcoholic KOH until faint pink colour appears and retain for more than 30 s. Perform blank test using only alcohol-ether mixture and phenolphthalein solution.

2.5 Calculation

Calculate using following equation:

Acid value [mg/g] = (mL test portion – mL blank) × factor of molarity × 2.806 / g test portion

2.5.1 *Definition and principle*

Acid value of oil from fried instant noodles = mg KOH required to neutralize 1 g oil. Oil extracted from noodle is dissolved in alcohol-ether mixture and titrated with alcoholic KOH standard solution.

2.5.2 Apparatus

Air-tight desiccator: silica gel heated at 150 °C is satisfactory drying agent.

2.5.3 Reagents

(d) Alcoholic potassium hydroxide standard solution: 0.05 mol/L. Dissolve 3.5 g potassium hydroxide in equal volume of water (CO₂-free) and add ethanol (95%) to 1 L. After mixing, let solution stand for several days keeping the solution CO₂-free. Use supernatant after standardization.

Standardization:

Weigh required quantity of amidosulfuric acid (certified reference material for volumetric analysis) and place it into desiccator (< 2.0 kPa) for 48 hour. Next, accurately weigh 1 g to 1.25 g (recording the weight to 0.1 mg), dissolve in water (CO₂-free), and dilute to 250 mL. Put 25 mL solution into Erlenmeyer flask, add 2 to 3 drops of bromothymol blue indicator and titrate with 0.05 mol/L alcoholic potassium hydroxide solution until colour of solution change to faint blue.

Calculation:

Factor of molarity = (g amidosulfuric acid x purity x 25) / 1.2136 / mL KOH

- (e) Alcohol-ether mixture: equal volumes ethanol (99.5%) and ether.
- (f) Phenolphthalein solution: 1% in alcohol.

2.5.4 Titration

Before sampling, liquefy extracted oil using water bath. Weigh 1 g to 2 g liquefied test portion into Erlenmeyer flask. Add 80 mL alcohol-ether mixture and a few drops of phenolphthalein solution. Titrate with 0.05 mol/L alcoholic KOH until faint pink colour appears and retain for more than 30 s. Perform blank test using only alcohol-ether mixture and phenolphthalein solution.

2.5.5 Calculation

Calculate using following equation:

Acid value [mg/g] = (mL test portion – mL blank) × factor of molarity × 2.806 / g test portion

PART C- DETERMINATION OF MOISTURE

3. Determination of moisture

3.1 Apparatus

- (a) Aluminium dish: diameter \geq 55 mm, height \geq 15 mm, and with inverted tight-fitting lid.
- (b) Air oven: with control accuracy ± 1 °C.
- (c) Air-tight desiccator: silica gel heated at 150 °C is satisfactory drying agent.

3.2 Preparation of test sample

Remove instant noodles from package and leave garnishing and seasoning in package. Transfer the noodles to plastic bag to prevent moisture change and then break these into small fragments with hands or wooden hammer. Select broken noodles in the size range of 2.36 mm to 1.7 mm by using two sieves with 2.36 mm and 1.7 mm openings (mesh size 12–8) and mix well. Use these noodles for test sample. If noodles are too thin to screen with sieves, cut them into 1 cm to 2 cm lengths, mix well and use these cut noodles for test sample.

3.3 Determination

3.3.1 Fried noodles

In cooled and weighed dish (with lid), previously heated to 105 °C, weigh ca 2 g well-mixed test portion to 1 mg. Uncover test portion and dry dish, lid, and contents 2 h in oven provided with opening for ventilation and maintained at 105 °C. (The 2 h drying period begins when oven temperature is actually 105 °C.) After drying period, cover dish while still in oven, transfer to desiccator, and weigh to 1 mg soon after reaching room temperature. Report loss in weight as moisture (indirect method).

3.3.2 Non-fried noodles

For non-fried noodles, follow the directions for fried noodles, but dry test portion for 4 h.

3.4 Calculation

Calculate using the following equation:

Moisture (%) = {(g test portion before drying – g test portion after drying) / g test portion before drying}×100

APPENDIX II

DETERMINATION OF PRESENTATION IN CANNED TUNA AND BONITO

The presentation of all sample units shall be determined by the following procedure.

- (i) Open the can and drain the contents, following the procedures outlined in Appendix III determination of drained weight.
- (ii) Remove and place the contents onto a tared 1.2 cm mesh screen equipped with a collecting pan.
- (iii) Separate the fish with a spatula being careful not to break the configuration of the pieces. Ensure that the smaller pieces of fish are moved to the top of a mesh opening to allow them to fall through the screen onto the collecting pan.
- (iv) Segregate the material on the pan according to flaked, grated (shredded) or paste and weigh the individual portions to establish the weight of each component.
- (v) If declared as a "chunk" pack weigh the screen with the fish retained and record the weight. Subtract the weight of the sieve from this weight to establish the weight of solid and chunk fish.
- (vi) If declared as "solid" pack remove any small pieces (chunks) from the screen and reweigh. Subtract the weight of the sieve from this weight to establish the weight of "solid" fish.

Calculations

(i) Express the weight of flaked, grated (shredded and paste) as a percentage of the total drained weight of fish.

% flakes =
$$\frac{\text{Weight of flakes}}{\text{Total weight of drained fish}} \times 100$$

(ii) Calculate the weight of solid and chunk fish retained on the screen by difference and express as a % of the total drained weight of fish.

% solid chunk fish = $\frac{\text{Weight of solid \& chunk fish}}{\text{Total weight of drained fish}} \times 100$

(iii) Calculate the weight of solid fish retained on the screen by difference and express as a % of the total drained weight of the fish.

% of solid fish = $\frac{\text{Weight of solid fish}}{\text{Total weight of drainedfish}} \times 100$

DETERMINATION OF NET WEIGHT, DRAINED WEIGHT, AND WASHED DRAINED WEIGHT IN FISH AND FISHERY PRODUCTS

Commodity	Provision	Method	
	Net weight	Net content	ts of all sample units shall be determined by the following procedure:
		(i)	Weigh the unopened container.
		(ii)	Open the container and remove the contents.
		(iii)	Weigh the empty container, (including any ends) after removing excess liquid and adhering meat.
		(iv)	Subtract the weight of the empty container from the weight of the unopened container.
Canned salmon		(v)	The resultant figure will be the net content.
Canned shrimps or prawns	Drained weight	The drained	d weight of all sample units shall be determined by the following procedure:
Canned tuna and bonito		(i)	Maintain the container at a temperature between 20 °C and 30 °C for a minimum of 12 hours prior to examination.
Canned crab meat		(ii)	Open and tilt the container to distribute the contents on a pre-weighed circular sieve
Canned sardines and sardine-type			which consists of wire mesh with square openings of 2.8 mm x 2.8 mm.
 Canned finfish		(iii)	Remove any wrapping material and incline the sieve at an angle of approximately 17–20° and allow sample to drain for two minutes, measured from the time the product is poured into the sieve.
		(iv)	Weigh the sieve containing the drained sample.
		(v)	The weight of drained sample is obtained by subtracting the weight of the sieve from the weight of the sieve and drained product.
Canned sardines and sardine-type products	Washed drained weight (for packs	(i)	Maintain the container at a temperature between 20 °C and 30 °C for a minimum of 12 hours prior to examination.
Canned finfish	with sauces)	(ii)	Open and tilt the container and wash the covering sauce and then the full contents with hot tap water (approx. 40 $^{\circ}$ C), using a wash bottle (e.g. plastic) on the tared circular sieve.
		(iii)	Wash the contents of the sieve with hot water until free of adhering sauce; where necessary separate optional ingredients (spices, vegetables, fruits) with pincers.

Commodity	Provision	Method
		Incline the sieve at an angle of approximately 17–20° and allow the fish to drain two minutes, measured from the time the washing procedure has finished.
		(iv) Remove adhering water from the bottom of the sieve by use of paper towel. Weigh the sieve containing the washed drained fish.
		(v) The washed drained weight is obtained by subtracting the weight of the sieve from the weight of the sieve and drained product
Quick-frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter	Net weight	The net weight (exclusive of packaging material) is determined on each whole primary container of each sample representing a lot and shall be determined in the frozen state.
Salted fish and dried salted fish of the Gadidae family of fishes	Net weight	The net weight (excluding packaging material and excess salt) of each sample unit in the sample lot shall be determined.
Dried shark fins	Net weight	The net weight (exclusive of packaging material) of each sample unit in the sample lot shall be determined.
Salted Atlantic herring and salted sprat	Net weight	The net weight (excluding packaging material) of each sample unit in the sample lot shall be determined. Remove the herring from the container (barrel) and put it on an appropriate sieve. Allow to drain for 5 min and remove adhering salt crystals. Weigh the herring and calculate net weight.
Sturgeon caviar	Net weight	The net weight (excluding packaging material) of each sample unit in the sample lot shall be determined by deducting the weight of the empty container from the total weight.
Smoked fish, smoke-flavoured fish and smoke-dried fish	Net weight	The net weight is determined as the weight of the product, exclusive of packaging material, interleaving material, etc.
Raw bivalve molluscs	Net weight	(i) Weigh the unopened container.
		(ii) Open the container and remove the contents.
		(iii) Dry the empty container and weigh.
		(iv) Subtract the weight of the empty container from the weight of the unopened container.
		The resultant figure will be the total net weight.

Commodity	Provision	Method
	Net weight of frozen products not covered by glaze	The net weight (exclusive of packaging material) of each sample unit representing a lot shall be determined in the frozen state.
	Net weight of products covered by glaze	Additionally, AOAC 963.26– Net weight of products with water added that is inside a "block-frozen" product.
(Live abalone and) raw fresh chilled or	Net weight	(i) Remove any frost and ice from outside of package.
frozen abalone for direct consumption or for further processing		(ii) Weigh the unopened container.
		(iii) Open the container and remove the contents.
		(iv) Dry the empty container and weigh.
		(v) Subtract the weight of the empty container from the weight of the unopened container.
		The resultant figure will be the total net weight.
	Net weight of frozen products not covered by glaze	The net weight (exclusive of packaging material) of each sample unit representing a lot shall be determined in the frozen state.
Quick-frozen finfish, uneviscerated and eviscerated	Net weight of frozen products	The net weight (exclusive of packaging material) of each sample unit representing a lot shall be determined in the frozen state.
Quick-frozen shrimps or prawns	not covered by glaze	
Quick-frozen lobsters	-	
 Quick-frozen blocks of fish fillets, minced fish flesh and mixtures of fillets and minced fish flesh 		
Quick-frozen fish fillets		
Quick-frozen raw squid		

Commodity	Provision	Method
Quick-frozen shrimps or prawns	Net weight of products covered	(1) Open the package with quick-frozen shrimps or prawns immediately after removal from low temperature storage.
	by glaze	 (i) For the raw product, place the contents in a container into which fresh water at room temperature is introduced from the bottom at a flow of approximately 25 litres per minute.
		(ii) For the cooked product place the product in a container containing an amount of fresh potable water of 27 °C (80 ° F) equal to 8 times the declared weight of the product. Leave the product in the water until all ice is melted. If the product is block frozen, turn block over several times during thawing. The point at which thawing is complete can be determined by gently probing the block apart.
		(2) Weigh a dry clean sieve with woven wire cloth with nominal size of the square aperture 2.8 mm (ISO Recommendation R565) or alternatively 2.38 mm (US No. 8 Standard Screen).
		 (i) If the quantity of the total contents of the package is 500 g (1.1 lbs) or less, use a sieve with a diameter of 20 cm (8 inches).
		(ii) If the quantity of the total contents of the package is more than 500 g (1.1 lbs) use a sieve with a diameter of 30 cm (12 inches).
		(3) After all glaze that can be seen or felt has been removed and the shrimps or prawns separate easily, empty the contents of the container on the previously weighed sieve. Incline the sieve at an angle of about 20° and drain for two minutes.
		(4) Weigh the sieve containing the drained product. Subtract the mass of the sieve; the resultant figure shall be considered to be the net content of the package.
Quick-frozen lobsters	Net weight of products covered by glaze	(1) As soon as the package is removed from frozen temperature storage, open immediately and place the contents under a gentle spray of cold water until all ice glaze that can be seen or felt is removed. Remove adhering water by the use of paper towel and weigh the product.
	(alternate methods)	(2) The pre-weighed glazed sample is immersed into a water bath by hand, until all glaze is removed, which preferably can be felt by the fingers. As soon as the surface becomes rough, the still frozen sample is removed from the water bath and dried by use of a paper towel before estimating the net product content by second weighing. By this procedure thaw drip losses and/or re-freezing of adhering moisture can be avoided.
		(i) As soon as the package is removed from frozen temperature storage, place the product in a container containing an amount of fresh potable water of 27 °C (80 °F) equal to 8 times the declared weight of the product. Leave the product in the water until all ice is melted. If

Commodity	Provision	Method					
		the product is block frozen, turn block over several time during thawing. The point at which thawing is complete can be determined by gently probing the block.					
		 (ii) Weigh a dry clean sieve with woven wire cloth with nominal size of the square aperture 2.8 mm (ISO Recommendation R565) or alternatively 2.38 mm (U.S. No. 8 Standard Screen.) (a) If the quantity of the total contents of the package is 500 g (1.1 lbs) or less, use a sieve with a diameter of 20 cm (8 inches). (b) If the quantity of the total contents of the package is more than 500 g (1.1 lbs) use a sieve with a diameter of 30 cm (12 inches). 					
		(iii) After all glaze that can be seen or felt has been removed and the lobsters separate easily, empty the contents of the container on the previously weighed sieve. Incline the sieve at an angle of about 20° and drain for two minutes.					
		(iv) Weigh the sieve containing the drained product. Subtract the mass of the sieve; the resultant figure shall be considered to be part of the net content of the package.					
Quick-frozen blocks of fish fillets, minced fish flesh and mixtures of fillets and minced fish flesh	Net weight of products covered by glaze	As soon as the package is removed from frozen temperature storage, open immediately and place the contents under a gentle spray of cold water until all ice glaze that can be seen or felt is removed. Remove adhering water by the use of paper towel and weigh the product.					
		An alternate method is outlined in the annex.					
Quick-frozen fish fillets	Determination of net weight of	As soon as the package is removed from low temperature storage, open immediately and place the contents under a gentle spray of cold water.					
	products covered by glaze	Agitate carefully so that the product is not broken.					
	by glaze	Spray until all ice glaze that can be seen or felt is removed.					
		Remove adhering water by the use of paper towel and weight the product in a tared pan					
Quick-frozen raw scallop products	Net weight	(i) AOAC 963.18.					
		(ii) Block frozen products: AOAC 967.13. The block-frozen scallops shall be thawed inside waterproof bags to prevent contact with, and absorption of, the water used to thaw the product					

ANNEX TO APPENDIX III

METHOD FOR THE DETERMINATION OF NET CONTENT OF FROZEN FISH BLOCKS COVERED BY GLAZE

Glazing is not used for quick-frozen blocks of white fish. Only quick-frozen blocks of herring, mackerel and other brown (fat) fish are glazed, which are destined for further processing (canning, smoking). For such blocks the following procedure may be applicable (tested with block-frozen shrimps).

1. PRINCIPLE

The pre-weighed glazed sample is immersed into a water bath by hand till all glaze is removed (as felt by fingers). As soon as the surface becomes rough, the still frozen sample is removed from the water bath and dried by use of a paper towel before estimating the net product content by repeated weighing. By this procedure thaw drip losses and/or re-freezing of adhering moisture can be avoided.

2. EQUIPMENT

Balance – sensitive to 1 g

Water bath, preferably with adjustable temperature

Circular sieve with a diameter of 20 cm and 1-3 mm mesh apertures (ISO R 565)

Paper or cloth towels with smooth surface

A freezer box should be available at the working place

3. PREPARATION OF SAMPLES AND WATER BATH

The product temperature should be adjusted to -18/-20 °C to achieve standard deglazing conditions (especially necessary if a standard deglazing period shall be defined in case of regular shaped products).

After sampling from the low temperature store remove, if present, external ice crystals or snow from the package with the frozen product.

The water bath shall contain an amount of fresh potable water equal to about 10 times of the declared weight of the product; the temperature should be adjusted on about 15 °C to 35 °C.

4. DETERMINATION OF GROSS-WEIGHT "A"

After removal of the package, the weight of the glazed product is determined: In case of single fish fillets, single weights are recorded (A 1-A n). The weighed samples are placed intermediately into the freezer box.

5. REMOVAL OF GLAZE

The pre-weighed samples/sub-samples are transferred into the water bath and kept immersed by hand. The product may be carefully agitated, till no more glaze can be felt by the fingertips on the surface of the product: change from slippery to rough. Needed time, depending on size/shape and glaze content of the product, 10 to 60 sec (and more in case of higher glaze contents or if frozen together).

For block-frozen products in consumer packs (also for single glaze products, which are frozen together during storage) the following (preliminary) procedure may be applicable: The pre-weighed block or portion is transferred onto a suitable sized sieve and immersed into the water bath. By slight pressure of the fingers separating deglazed portions are removed fractionally. Short immersing is repeated, if glaze residues are still present.

6. DETERMINATION OF NET WEIGHT "B"

The deglazed sample/subsample, after removal of adhering water by use of a towel (without pressure) is immediately weighed. Single net-weights of sub-samples are summed up: B₁-n.

7. DETERMINATION OF GLAZE-WEIGHT "C"

Grossweight "A" – Net weight "B" = Glaze weight "C"

8. CALCULATION OF PERCENTAGE PROPORTIONS

% net content of the product "F"= $\frac{"B"}{"A"} \times 100$

% glaze – related to the gross weight of the product "G" = $\frac{"C"}{"A"} \times 100$

% glaze – related to the net weight of the product "H" = $\frac{"C"}{"B"} \times 100$

APPENDIX IV

SENSORY AND PHYSICAL EXAMINATION OF FISH AND FISHERY PRODUCTS

Canned salmon, canned crab meat, canned shrimps or prawns, canned tuna and bonito, canned finfish, canned sardine and sardine-type products

- 1. Complete external can examination for the presence of container integrity defects or can ends which may be distorted outward.
- 2. Open can and complete weight determination according to defined procedures in Appendix III.

3. For canned salmon and canned crab meat

• Examine product for discolouration, foreign and objectionable matter.

4. For canned shrimps or prawns

- Carefully remove the product and examine for size designation in accordance with the procedure in Section 7.2.2 of CXS 37-1991.²
- Examine product for discolouration, foreign and objectionable matter.

5. For canned tuna and bonito

- Examine the product for discolouration.
- Carefully remove the product and determine the presentation according to the defined procedures in Appendix III.
- Examine product for discolouration, foreign matter and struvite crystals.

6. For canned finfish

- Examine the product for the form of presentation.
- Examine product for discolouration, foreign and objectionable matter.

7. For canned sardines and sardine-type products

- Carefully remove product and examine for discolouration, foreign matter and struvite crystals.
- 8. The presence of hard bone in canned salmon, canned tuna and bonito, canned finfish, canned sardines and sardine-type products is an indicator of underprocessing and will require an evaluation for sterility.
- 9. Assess odour, flavour and texture in accordance with the *Guidelines for the sensory evaluation of fish and shellfish in laboratories* (CXG 31-1999).³

Quick-frozen finfish, uneviscerated and eviscerated, quick-frozen shrimps or prawns, quick-frozen lobsters, quick-frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh, quick-frozen fish fillets, quick-frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter, quick-frozen raw squid, fresh and quick-frozen raw scallop products

- 1. **For quick-frozen fish sticks (fish fingers), fish portions and fish fillets– breaded or in batter**, the sample used for sensory evaluation should not be the same as that used for other examinations.
- 2. Complete net weight determination, according to defined procedures in Appendix III (de-glaze as required).

3. For quick-frozen finfish, uneviscerated and eviscerated

- Examine the frozen sample unit for the presence of deep dehydration by measuring those areas or counting instances which can only be removed with a knife or other sharp instrument. Measure the total surface area of the sample unit, and calculate the percentage affected.
- Thaw and individually examine each fish in the sample unit for the presence of foreign matter.
- Examine each fish using the criteria outlined in the respective standard. Flesh odours are examined by tearing or making a cut across the back of the neck such that the exposed surface of the flesh can be evaluated.

4. For quick-frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh

- Examine the frozen block or fillets for the presence of dehydration by measuring those areas which can only be removed with a knife or other sharp instrument. Measure the total surface area of the sample unit, and calculate the percentage affected.
- Thaw and individually examine each block in the sample unit for the presence of foreign matter, bone where applicable, odour and textural defects.

5. For quick-frozen fish fillets

- Examine the frozen block or fillets for the presence of dehydration by measuring those areas which can only be removed with a knife or other sharp instrument. Measure the total surface area of the sample unit, and calculate the percentage affected.
- Thaw and individually examine each block in the sample unit for the presence of foreign matter, parasites, bone where applicable, odour and flesh abnormality defects.

6. For quick-frozen shrimps and prawns

- Examine the frozen shrimp in the sample unit or the surface of the block for the presence of dehydration. Determine the percentage of shrimp or surface area affected.
- Thaw using the procedure described in Appendix V and individually examine each shrimp in the sample unit for the presence of foreign matter and presentation defects. Determine the weight of shrimp affected by presentation defects.
- Examine product for count declarations in accordance with procedures in CXS 92-1981.⁴
- Assess the shrimp for odour and discolouration as required.

7. For quick-frozen lobsters

- Examine the frozen lobster for the presence of deep dehydration. Determine the percentage of lobster affected.
- Thaw using the procedure described in Appendix V and individually examine each sample unit for the presence of foreign and objectionable matter.
- Examine product count and weight declarations in accordance with procedures in CXS 95-1981.5
- Assess the lobster for odour and discolouration as required.

8. For quick-frozen fish sticks (fish fingers), fish portions and fish fillets- breaded or in batter

- Complete fish core determination on one set of the sample units according to defined procedures in Appendix VI.
- Complete the estimation of the proportion of fillets and minced flesh, if required.
- Cook the other set of sample units and examine for odour, flavour, texture, foreign matter and bones.

9. For quick-frozen raw squid

• Examine the frozen squid for the presence of deep dehydration by measuring those areas which can only be removed with a knife or other sharp instrument. Measure the total surface area of the sample unit, and determine the percentage affected using the following formula;

 $\frac{\text{area affected}}{\text{total surface area}} \times 100\% = \%$ affected by deep dehydration

- Thaw and individually examine each squid in the sample unit for the presence of foreign matter and colour.
- Examine each squid using the criteria outlined in CXS 191-1995.⁶ Flesh odours are examined by making a cut parallel to the surface of the flesh so that the exposed surface can be evaluated.

10. For fresh and quick-frozen raw scallop products

• Examine the frozen scallop product in the sample unit or the surface of the block for the presence of dehydration. Determine the percentage of scallop meat or surface area affected.

- Thaw using the procedure described in Appendix III and individually examine each scallop product in the sample unit for the presence of foreign matter, objectionable matter and presentation defects.
- Determine the weight of scallop product affected by presentation defects.
- Examine product for pieces and count declarations in accordance with procedures in CXS 315-2014.7
- Assess the scallop product for odour and parasites as required.
- A small portion of the sample unit (100 g to 200 g) is cooked without delay and the odour/flavour/texture and presence of sand is determined. If necessary, additional portions may be cooked and examined for confirmation.
- 11. In cases where a final decision regarding the odour or texture cannot be made in the thawed uncooked state:
 - In quick-frozen finfish, uneviscerated and eviscerated, a small portion of the flesh (approximately 200 g) is sectioned from the product and the odour, flavour or texture confirmed without delay by using one of the cooking methods defined as follows: AOAC Methods "Moisture in Meat and Meat Products, Preparation of Sample Procedure"; 883.18 and "Moisture in Meat" (Method A); 950.46; AOAC 1990.
 - In quick-frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh, a small portion of the disputed material (approximately 200 g) is sectioned from the block and the odour and flavour confirmed without delay by using one of the cooking methods defined in Appendix V.
 - In quick-frozen raw squid, a portion of the sample unit is sectioned off and the odour, flavour and texture confirmed without delay by using one of the cooking methods defined in Appendix V.
- 12. In cases where a final decision regarding the odour cannot be made in the thawed uncooked state:
 - In quick-frozen fish fillets, a small portion of the disputed material (approximately 200 g) is sectioned from the sample unit and the odour and flavour confirmed without delay by using one of the cooking methods defined in Appendix V.
- 13. In cases where a final decision on gelatinous condition cannot be made in the thawed uncooked state:
 - In quick-frozen finfish, uneviscerated and eviscerated, the disputed material is sectioned from the product and gelatinous condition confirmed by cooking as defined in Appendix IV or by using one of the cooking methods defined as follows to determine if greater than 86 percent moisture is present in any fish: AOAC Methods "Moisture in Meat and Meat Products, Preparation of Sample Procedure"; 883.18 and "Moisture in Meat" (Method A); 950.46; AOAC 1990. If a cooking evaluation is inconclusive, then one of the cooking methods defined as follows would be used to make the exact determination of moisture content: AOAC Methods "Moisture in Meat" (Method A); 950.46; AOAC 1990. If a cooking evaluation of Sample Procedure"; 883.18 and "Moisture in Meat" (Method A); 950.46; AOAC 1990. If a cooking evaluation is inconclusive, then one of the cooking methods defined as follows would be used to make the exact determination of moisture content: AOAC Methods "Moisture in Meat" (Method A); 950.46; AOAC 1990.
 - In quick-frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh, the disputed material is sectioned from the block and the gelatinous condition confirmed by cooking as defined in Appendix V or by using procedure in Appendix VII to determine if greater than 86 percent moisture is present in any fillet. If cooking evaluation is inconclusive, then procedure in Appendix VI would be used to make the exact determination of moisture content.
 - In quick-frozen fish fillets and quick-frozen fish sticks (fish fingers), fish portions and fish fillets

 breaded or in batter, the disputed material is sectioned from the product and gelatinous condition confirmed by cooking as defined in Appendix V or AOAC Methods- "Moisture in Meat and Meat Products, Preparation of Sample Procedure"; 983.18 and "Moisture in Meat" (Method A); 950.46 to determine if greater than 86 percent moisture is present in any fillet or product unit. If a cooking evaluation is inconclusive, then AOAC Methods- "Moisture in Meat and Meat Products, Preparation of Sample Procedure"; 983.18 and "Moisture in Meat and Meat Product, Preparation of Sample Procedure"; 983.18 and "Moisture in Meat and Meat Products, Preparation of Sample Procedure"; 983.18 and "Moisture in Meat" (Method A); 950.46 would be used to make the exact determination of moisture content.
- 14. In cases where a final decision regarding the odour/flavour cannot be made in the thawed state **in quick-frozen shrimps or prawns, quick-frozen lobsters**, a small portion of the sample unit (100 g to 200 g) is prepared without delay for cooking and the odour/flavour confirmed by using one of the cooking methods defined in Appendix V.

Crackers from marine and freshwater fish, crustacean and molluscan shellfish and boiled dried salted anchovies

The sample used for sensory evaluation should not be same as that used for other examination.

- 1. For crackers from marine and freshwater fish, crustacean and molluscan shellfish, examine the sample unit for foreign matter, bones and discolouration.
- 2. **For boiled dried salted anchovies,** examine every fish in the sample unit for foreign matter, breakage, pink condition and mould growth.
- 3. For all the mentioned commodities above, assess the odour in the uncooked sample in accordance with the *Guidelines for the sensory evaluation of fish and shellfish in laboratories* (CXG 31-1999).³
- 4. For all the mentioned commodities above, assess the flavour in cooked sample in accordance with CXG 31-1999.
- 5. For crackers from marine and freshwater fish, crustacean and molluscan shellfish, the sample shall be deep fried in fresh cooking oil at 190 °C for 20–60 seconds as appropriate to the thickness of the crackers.
- 6. For boiled dried salted anchovies, the sample shall be cooked prior to assessment according to the cooking instructions on the package. When such instructions are not given, the sample shall be deep fried in fresh cooking oil at 190 °C for 1–2 minutes as appropriate to the size.

Fish sauce

- 1. Complete external packaging unit examination for the presence of any integrity defects, particularly cracks or leakage or loose pieces of the packaging units.
- 2. Examination of the product for translucence and foreign matter.
- 3. Evaluation of odour and taste.

Salted fish and dried salted fish of the Gadidae family of fishes

- 1. Examine every fish in the sample in its entirety.
- 2. Examine the product for the form of presentation.
- 3. Examine the fish for foreign matter, pink conditions, halophilic mould, liver stains, intense bruising, severe burning and texture.
- 4. Assess odour in accordance with CXG 31-1999.³

Salted Atlantic herring and salted sprat

See CXS 244-2004.8

Sturgeon caviar

See CXS 291-2010.9

Live and raw bivalve molluscs

See Appendices III, V and IX, and CXS 292-2008.10

Smoked fish, smoke-flavoured fish and smoke-dried fish

See Appendices III, V and IX.

Live abalone and for raw fresh chilled or frozen abalone for direct consumption or for further processing

See CXS 312-2013.11

APPENDIX V

PART 1: THAWING PROCEDURES IN FISH AND FISHERY PRODUCTS

Quick-frozen shrimps or prawns, quick-frozen lobsters and quick-frozen raw squid

The sample unit is thawed by enclosing it in a film type bag and immersing in water at room temperature (not greater than 35 °C). The complete thawing of the product is determined by gently squeezing the bag occasionally so as not to damage the texture of the sample, until no hard core or ice crystals are left.

Raw bivalve molluscs

For frozen product, the sample unit is thawed by enclosing it in a film type bag and immersing in water at room temperature (not greater than 35 °C). The complete thawing of the product is determined by gently squeezing the bag occasionally so as not to damage the texture of the bivalve molluscs, until no hard core or ice crystals are left.

Smoked fish, smoke-flavoured fish and smoke-dried fish

Temperatures for thawing

Frozen samples of final products shall be thawed at refrigeration temperatures to maintain quality and safety.

Raw fresh chilled or frozen abalone

For frozen product, the sample is thawed by enclosing it in a film type bag allowing it to thaw at room temperature or in a refrigerator (at 2–6 °C). The complete thawing of the product is determined by gently squeezing the bag occasionally so as not to damage the texture of the abalone, until no hard core or ice crystals are left.

Quick-frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh

Air thaw method:

Frozen fish blocks are removed from the packaging. The frozen fish blocks are individually placed into snug fitting impermeable plastic bags or a humidity controlled environment with a relative humidity of at least 80 percent. Remove as much air as possible from the bags and seal. The frozen fish blocks sealed in plastic bags are placed on individual trays and thawed at air temperature of 25 °C (77 °F) or lower. Thawing is completed when the product can be readily separated without tearing. Internal block temperature should not exceed 7 °C (44.6 °F).

Water immersion method:

Frozen fish blocks are removed from the packaging. The frozen fish blocks are sealed in plastic bags. Remove as much air as possible from the bags and seal. The frozen fish blocks are placed into a circulating water bath with temperatures maintained at 21 °C \pm 1.5 °C (70 °F \pm 3 °F). Thawing is completed when the product can be easily separated without tearing. Internal block temperature should not exceed 7 °C (44.6 °F).

PART 2: COOKING PROCEDURES IN FISH AND FISHERY PRODUCTS

Quick-frozen finfish, uneviscerated and eviscerated, quick-frozen shrimps or prawns, quick-frozen lobsters, quick-frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh, quick-frozen fish fillets, quick-frozen raw squid.

The following procedures are based on heating the product to an internal temperature of 65–70 °C. The product must not be overcooked. Cooking times vary according to the size of the product and the temperatures used. The exact times and conditions of cooking for the product should be determined by prior experimentation.

Baking procedure: Wrap the product in aluminium foil and place it evenly on a flat cookie sheet or shallow flat pan.

Steaming procedure: Wrap the product in aluminium foil and place it on a wire rack suspended over boiling water in a covered container.

Boil-in-bag procedure: Place the product into a boilable film-type pouch and seal. Immerse the pouch into boiling water and cook.

Microwave procedure: Enclose the product in a container suitable for microwave cooking. If plastic bags are used, check to ensure that no odour is imparted from the plastic bags. Cook according to equipment specifications.

Quick-frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter

The frozen sample shall be cooked prior to sensory assessment according to the cooking instructions on the package. When such instructions are not given, or equipment to cook the sample according to the instructions is not obtainable, the frozen sample shall be cooked according to the applicable method(s) given below:

Use procedure 976.16 of the AOAC. It is based on heating product to an internal temperature of 65–70 °C. Cooking times vary according to size of product and equipment used. If determining cooking time, cook extra samples, using a temperature measuring device to determine internal temperature.

APPENDIX VI

DETERMINATION OF FISH CONTENT (DECLARATION) IN QUICK-FROZEN FISH STICKS (FISH FINGERS), FISH PORTIONS AND FISH FILLETS – BREADED OR IN BATTER

Estimation of fish content

AOAC Method 996.15. (End-product method)

Calculation:

% fish content = (Wd/Wb) X 100 + adjustment factor*

Wd = weight of debattered and/or debreaded test unit

Wb = weight of battered and/or breaded test unit

* Raw breaded frozen coated fish and fishery products: 2.0%

* Batter-dipped frozen coated fish and fishery products: 2.0%

* Precooked frozen coated fish and fishery products: 4.0%

Reference: J. AOAC Int. 80, 1235 (1997)

Other methods

(1) Chemical analysis method (nitrogen factor end-product method)

Appropriate in cases where there is reason to doubt the composition of the fish core (i.e. appears to contain non-fish ingredients). Except for fully cooked products, this method requires confirmation with the AOAC Method 996.15, or with Method #2 (Determination of fish content) in conjunction with investigation at the processing plant when determining product compliance with the labelling provisions in CXS 166-1989.¹² This method should trigger in-factory investigation (e.g. raw ingredient recipe checks) when suspect products are identified.

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

% Fish = $\frac{(\% \text{ total nitrogen - }\% \text{ nonfish flesh nitrogen})}{N \text{ factor}^*} x100$

*appropriate N (nitrogen) factor

The non-fish flesh nitrogen is calculated as follows:

% non-fish flesh nitrogen = % carbohydrate X 0.02

Where the carbohydrate is calculated by difference:

% carbohydrate = 100 - (%water + % fat + % protein + % ash)

References

Determination of nitrogen: ISO 937

Determination of moisture: ISO 1442

Determination of total fat: ISO 1443

Determination of ash: ISO 936

Average nitrogen factors to be used for fish flesh for specific fish species used as raw material for the product can be found at the following website:

http://www.fao.org/in-action/globefish/fishery-information/resource-detail/en/c/338604/

http://www.fao.org/fishery/topic/1514/en

The uncertainty of each nitrogen factor should be taken into account from the statistical data presented with the published nitrogen factor (e.g. 2 standard errors about the mean).

(2) Rapid method used during production

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

% Fish Content =
$$\frac{\text{Weight of ingoing fish}}{\text{Weight of final product}} x100$$

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.

APPENDIX VII

ESTIMATION OF PROPORTION OF FISH FILLETS AND MINCED FISH FLESH

(West European Fish Technologists Association-WEFTA Method)

a) Equipment

Balance, sensitive to 0.1 g

Circular sieve – 200 mm diameter, 2.5 or 2.8 mesh opening (ISO) soft rubber edge (or blunt) spatula, forks, suitable sized plates, water tight plastic bags.

b) Preparation of samples

Fish portions/sticks: Take as many portions as needed to provide a fish core sample of about 200 g (2 kg). If breaded and/or battered first strip coating according to the method described in Appendix VI.

c) Detemination of weights "A" of the frozen fish samples

Weight the single fish portions/decoated fish cores while they are still frozen. Smaller portions are combined to a sample subunits of about 200 g (e.g. 10 fish sticks of about 20 g each). Record the weight "A" n of the subunits. Place the pre-weighed sample subunits into water tight bags.

d) Thawing

Thaw the samples by immersing the bags into a gently agitated water bath of about 20 $^{\circ}$ C, but not more than 35 $^{\circ}$ C.

e) Draining

After thawing has been completed (duration about 20-30 minutes) take each sample unit, one at a time, and drain the exuded fluid (thaw drip) for 2 minutes on a pre-weighed circular sieve incluned at an angle of 17–20 degrees. Remove adhering drip from the bottom of the sieve by use of a paper towel when draining is completed.

f) Determination of weight "B" of the drained fish sample and weight "C" of the thaw drip

Determine the weight of the drained fish sample "B" - sieve plus fish minus sieve weight. The difference of "A" - "B" is the weight of exuded fluid - thaw drip.

g) Separation

Place the drained fish core on a plate and separate the minced flesh from the fillet using a fork to hold the fillet flesh and a soft, rubber edge spatula to scrape off the minced flesh.

APPENDIX VIII

PREPARATION OF FISH SAMPLES AND DETERMINATION OF SALT AND WATER CONTENT IN FISH AND FISHERY PRODUCTS

PART 1: PREPARATION OF FISH SAMPLES

Salted fish and dried salted fish of the Gadidae family of fishes

- 1. Before preparing of a subsample adhering salt crystals should be removed by brushing from the surface of the sample without using water.
- 2. The preparation of fish samples for the determination of salt content, and water content in order to calculate the % salt saturation of the fish should be carried out according to AOAC 937.07. The analysis should be on the edible portion of the fish.
- 3. Determination should be performed at least in duplicate.

PART 2: DETERMINATION OF SALT CONTENT

Salted fish and dried salted fish of the Gadidae family of fishes, salted Atlantic herring and salted sprat, and sturgeon caviar

1. Principle

The salt is extracted by water from the pre-weighed sample. After the precipitation of the proteins, the chloride concentration is determined by titration of an aliquot of the solution with a standardized silver nitrate solution (Mohr method) and calculated as sodium chloride.

2. Equipment and chemicals

- Brush
- Sharp knife or saw
- Balance, accurate to \pm 0.01 g
- Calibrated volumetric flasks, 250 ml
- Erlenmeyer flasks
- Electric homogenizer
- Magnetic stirrer
- Folded paper filter, quick running
- Pipettes
- Funnel
- Burette
- Potassium hexacyano ferrate (II), K4Fe(CN)6·3H2O, 15% w/v (aq)
- Zinc sulphate, ZnSO4·6H2O, 30% w/v (aq)
- Sodium hydroxide, NaOH, 0.1 N, 0.41% w/v (aq)
- Silver nitrate, AgNO3, 0.1 N, 1.6987% w/v (aq), standardized
- Potassium chromate, K2CrO4 5% w/v (aq)
- Phenolphthalein, 1% in ethanol
- Distilled or deionized water

3. Procedure

- (i) Five grams of homogenized subsample is weighted into a 250 ml volumetric flask and vigorously shaken with approximately 100 ml water.
- (ii) Five millilitres of potassium hexacyano-ferrate solution and 5 ml of zinc sulphate solution are added, the flask is shaken.
- (iii) Water is added to the graduation mark.

- (iv) After shaking again and allowing to stand for precipitation, the flask content is filtered through a folded paper filter.
- (v) An aliquot of the clear filtrate is transferred into an Erlenmeyer flask and two drops of phenolphthalein are added. Sodium hydroxide is added dropwise until the aliquot takes on a faint red colour. The aliquot then diluted with water to approximately 100 ml.
- (vi) After addition of approximately 1 ml potassium chromate solution, the diluted aliquot is titrated under constant stirring, with silver nitrate solution. End-point is indicated by a faint, but distinct, change in colour. This faint reddish-brown colour should persist after brisk shaking.

To recognize the colour change, it is advisable to carry out the titration against a white background.

- (vii) Blank titration of reagents used should be done.
- (viii) End-point determination can also be made by using instruments like potentiometer or colorimeter.

4. Calculation of results

In the equation of the calculation of results the following symbols are used:

A= volume of aliquot (ml)

C= concentration of silver nitrate solution in N

V= volume of silver nitrate solution in ml used to reach end-point and corrected for blank value

W= sample weight (g)

The salt content in the sample is calculated by using the equation:

Salt concentration (%) = (V x C x 58.45 x 250 x 100) / (A x W x 1000)

Results should be reported with one figure after the decimal point.

5. Reference method

As reference method a method should be used which includes the complete ashing of the sample in a muffle furnace at 550 °C before chloride determination according to the method described above (leaving out steps (ii) and (iv)).

6. Comments

By using the given equation all chloride determined is calculated as sodium chloride. However it is impossible to estimate sodium by this methodology, because other chlorides of the alkali and earth alkali elements are present which form the counterparts of chlorides.

The presence of natural halogens other than chloride in fish and salt is negligible.

A step, in which proteins are precipitated (ii), is essential to avoid misleading results.

PART 3: DETERMINATION OF WATER CONTENT

Salted fish and dried salted fish of the Gadidae family of fishes

- i) Determination of % salt saturation as required by the standard, should be in accordance to AOAC 950.46.B (air-drying (a)).
- ii) Determination of water content in the whole fish, when needed in the commercial trade of klippfish and wet salted fish, the method of sampling the fish should be carried out according to the "Determination of water content in whole fish by cross section method" defined in the annex to this appendix.

Salted Atlantic herring and salted sprat

Determination of water content is performed according to AOAC 950.46B (air-drying).

ANNEX TO APPENDIX VIII

DETERMINATION OF WATER CONTENT IN WHOLE FISH BY CROSS SECTION METHOD

1 PRINCIPLE

The fish is cut in sections as described in method. The sections are cut in smaller bits to a collected sample. The water content of the collected sample is determined by drying. Examinations and experience have shown that the water content of this collected sample is closed to the "true" water content of the fish.

2 EQUIPMENT

- Soft brush
- Basins (steel, glass, porcelain)
- Scissors
- Band saw
- Knife
- Weight, 1 g precision
- Oven, 103–105 °C
- Desiccator

3 PREPARATION OF SAMPLE

Salt particles on the surface of the fish are brushed away.

The weight of the fish is determined to 1 g accuracy.

The length of the fish is measured as the distance between the cleft in the tail and a line drawn between the tips of the ear bones.

4 PROCEDURE

- (i) The sampling of the fish is described in the enclosed figure.
 - A) Wet salted fish is sliced in sections by knife
 - B) Salted and dried salted fish is sliced in sections by band saw.
 - 1) A section of 20 mm measured from a line drawn between the ear bones, dotted line on figure, is cut.
 - 2) The next cut is a 40 mm section.
 - 3) A 2 mm section is cut from the front part of the 40 mm section and collected (see Section 7 [Comments]).
 - 4) The next cut is a new cut of a 40 mm section.
 - 5) A 2 mm section is cut from the front part of the 40 mm section and collected.
 - 6) The entire fish is cut in 40 mm sections from which are cut 2 mm sections (see enclosed figure).
 - 7) All sections of 2 mm, marked II, IV, VI, VIII in the figure, even numbers, are collected to a collected sample.
- (ii) The 2 mm sections in the collected sample are cut with scissors in smaller pieces directly in tared basins just after the fish is cut.
- (iii) The basins containing the sample are weighted.
- (iv) The basins containing the samples are put in the oven at 103 °C −105 °C for drying to constant weight (18 hours over night).
- (v) The basins are taken from the oven to a desiccator and cooled.
- (vi) The basins are weighted.

5. CALCULATION OF RESULTS

In the equation of the calculation of results the following symbols are used:

W1 = Weight of fish and basins before drying, g.

W2 = Weight of fish and basins after drying, g.

Ws = Weight of tared basins, g

The water content in the fish is calculated by using the equation:

Water content, $g/100 g = 100^{*}(W1-W2)/(W1-Ws)$

The result is reported to the nearest gram, together with the length and the weight of the analysed fish.

6. CONTROL ANALYSIS OF WHOLE FISH.

The determination of water content in whole fish by cross section method appears to give the closest result compared to water content determined by the drying of the whole fish

7. COMMENTS

Each sampled fish should be packed and sealed in a plastic bag before analysis. The samples should be stored under chilled or refrigerated conditions from the time of sampling to the time of analysis.

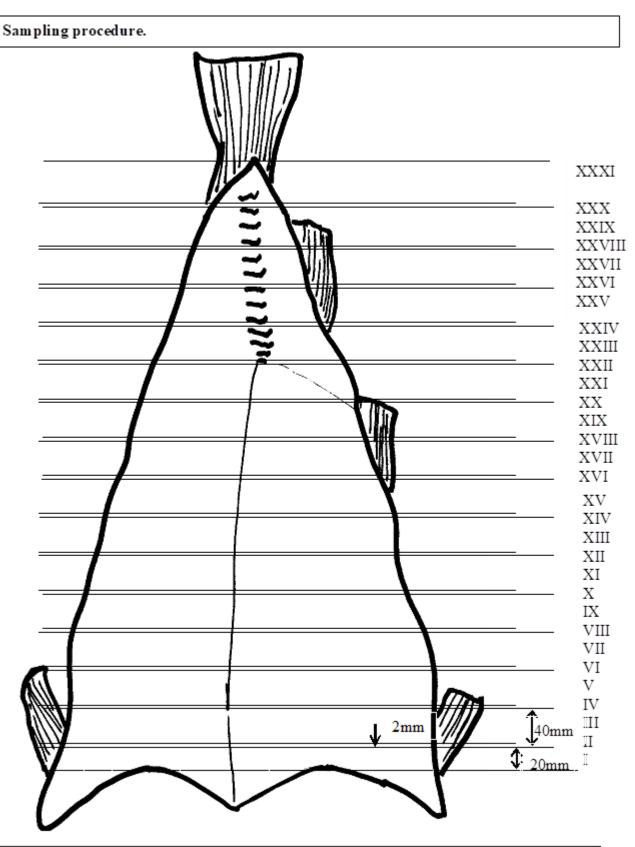
The analysis must be performed as soon as possible after the fish has been sampled.

It might be difficult to cut sections of 2 mm when the fish has a water content above 50 percent, but the section must be close to 2 mm.

To minimize the loss of water from the 2 mm sections it is important to weight the collected sample immediately after the fish is cut in sections.

Determination should be performed at least in duplicate.

FIGURE



All sections labelled by even numbers, II, IV, VI, VIII etc. are collected to constitute one sample.

APPENDIX IX

METHOD OF ANALYSIS OF PATHOGENS IN FISH AND FISHERY PRODUCTS

PART 1: ESCHERICHIA COLI IN LIVE AND RAW BIVALVE MOLLUSCS

The ISO/TS 16649-3 – Horizontal method for the enumeration of beta-glucuronidase-positive *Escherichia coli* – Part 3: Most probable number technique using 5-bromo-4-chloro-3-indolyl beta-D-glucuronide or other validated methods in accordance with the protocol set out in the ISO 16140 or other internationally accepted similar protocol.

PART 2: DETERMINATION OF *LISTERIA MONOCYTOGENES* IN SMOKED FISH, SMOKE-FLAVOURED FISH AND SMOKE-DRIED FISH

The microbiological criteria for products in which growth of *L. monocytogenes* will not occur are based on the use of the ISO 11290-2 method. Other methods that provide equivalent sensitivity, reproducibility, and reliability can be employed if they have been appropriately validated (e.g. based on ISO 16140). The microbiological criteria for products in which growth of *L. monocytogenes* can occur are based on the use of ISO 11290-1 method. Other methods that provide equivalent sensitivity, reproducibility, and reliability can be employed if they have been appropriately can be employed of the use of ISO 11290-1 method. Other methods that provide equivalent sensitivity, reproducibility, and reliability can be employed if they have been appropriately validated (e.g. based on ISO 16140).

PART 3: DETERMINATION OF *CLOSTRIDIUM BOTULINUM IN* SMOKED FISH, SMOKE-FLAVOURED FISH AND SMOKE-DRIED FISH

AOAC 977.26 for the detection of *C. botulinum* and its toxins in foods or other scientifically equivalent validated method. This method is not routinely performed on the product but may be used when there is a suspicion of the presence of toxins.

APPENDIX X

DETERMINATION OF WATER CAPACITY OF CONTAINERS (CAC/RM 46)

1. SCOPE

This method applies to glass containers.

2. DEFINITION

The water capacity of a container is the volume of distilled water at 20 °C which the sealed container will hold when completely filled.

3. PROCEDURE

- **3.1** Select a container which is undamaged in all respects.
- **3.2** Wash, dry and weigh the empty container.
- **3.3** Fill the container with distilled water at 20 °C to the level of the top thereof, and weigh the container thus filled.

4. CALCULATION AND EXPRESSION OF RESULTS

Subtract the weight found in Section 3.2 from the weight found in Section 3.3. The difference shall be considered to be the weight of water required to fill the container. Results are expressed as ml of water.

APPENDIX XI

DETERMINATION OF MOISTURE IN POWDERED MILK, POWDERED CREAM AND BLEND OF SKIMMED MILK POWDER WITH VEGETABLE FAT

TEST MOISTURE METHOD AT NORMAL PRESSURE (102 ± 2)°C IN POWDERED MILK, POWDERED CREAM, AND BLEND OF SKIMMED MILK POWDER WITH VEGETABLE FAT

DESCRIPTION OF THE METHOD: DETERMINATION OF MOISTURE

1. SCOPE

This standard specifies a method for the determination of moisture content for all types of powdered milk, powdered cream and mixtures of powdered skimmed milk with vegetable fat.

2. DEFINITION

The content is the mass loss determined by the procedure specified in this standard. It is expressed in percentage by mass g/100 g.

3. PRINCIPLE

A portion of the sample is dried in an oven set at (102 ± 2) °C until constant weight and weighed to determine the loss of mass.

4. EQUIPMENT

Common laboratory equipment and, in particular, the following.

- **4.1 Analytical balance,** capable of weighing with a precision of 1 mg, with a minimum resolution of 0.1 mg.
- **4.2 Drying oven**, with good ventilation, as far as possible with forced ventilation, capable of being thermostatically maintained at (102 ± 2) °C throughout the workspace, with a temperature controller.
- **4.3 Desiccator**, with freshly dried silica gel with hygrometric indicator or another effective desiccant.
- **4.4** Flat-bottomed dishes, approximately 25 mm deep, approximately 50 mm in diameter, and made of an appropriate material (for example, glass, stainless steel, nickel, or aluminium), fitted with tight-fitting, removable lids easily.

5. SAMPLING

It is important that the laboratory receive a truly representative sample and that it has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this standard. A recommended sampling method is provided in ISO 707 | IDF 50.

6. TEST SAMPLE PREPARATION

Transfer the entire sample to a dry, tightly closed container with a capacity of approximately twice the volume of the sample. Mix thoroughly by turning and shaking the container.

7. PROCEDURE

7.1 Preparation of the dish

- 7.1.1 Heat the uncovered capsule and its lid (4.4) in the oven (4.2) controlled at (102 \pm 2) °C, for 1 h.
- **7.1.2** Transfer the capped dish to the desiccator (4.3), allow it to cool to room temperature in the balance room, and weigh (4.1) to the nearest 0.1 mg.

7.2 Test sample

7.2.1 Place 1 g-1.5 g of the prepared test sample (6) in the dish, cover with the lid and weigh to the nearest 0.1 mg.

7.3 Determination

- **7.3.1** Uncover the capsule and place it together with the lid in the oven (4.2), controlled at (102 \pm 2) °C for 2 hrs.
- **7.3.2** Replace the cap, transfer the capped dish to the desiccator, allow to cool to balance room temperature, and weigh to the nearest 0.1 mg.

- 7.3.3 Uncover the capsule and heat again, along with its lid, on the oven for 1 h. Then repeat operation 7.3.2.
- **7.3.4** Repeat this process until the difference in mass between two successive weightings does not exceed 0.5 mg. Record the lowest mass.

8. CALCULATION AND EXPRESSION OF RESULTS

8.1 Calculation

The moisture content in the sample, expressed in g/100 g, is equal to:

moisture = $(m_1 - m_2) \times 100$

where,

m $_{\rm 0}\, is$ the mass, in grams, of the dish and lid (Section 7.1.2)

m 1 is the mass, in grams, of the dish, lid and test sample before drying (Section 7.2.1)

m 2 is the mass, in grams, of the dish, lid and test sample after drying (Section 7.3.4)

8.2 Expression of test results

Express the sample results to two decimal places.

STANDARD PROCEDURE FOR TOUGH STRING TEST OF CANNED AND QUICK-FROZEN GREEN AND WAX BEANS (CAC/RM 39-1970)

1. DEFINITION

A tough string is a string that will support the weight of 250 g for 5 seconds or longer when tested in accordance with the procedure described below.

2. PRINCIPLE

Strings are removed from individual pods, fastened through a clamp assembly weighing 250 g, and hung so that the string supports the entire weight. If the string supports the weight for 5 seconds or more, it is considered a tough string.

3. APPARATUS

3.1 Weighted clamp

Use battery clamp (with teeth filed off or turned back), spring operated clothes pin or binder clip which presents a flat clamping surface. Attach weight so that entire assembly of weight and clamp weighs 250 g. See Figure 1. A bag containing lead pellets is convenient as a weight.

4. PROCEDURE

- **4.1** From the drained product select a representative sample of not less than 285 g. Record the weight of this test sample.
- **4.2** Break the individual bean units and set aside those that show evidence of tough strings. Remove the strings from the pods and retain the pod material for weighing.
- **4.3** Fasten the clamp assembly to one end of the string. Grasp the other end of the string with the fingers (a cloth may be used to aid in holding the string) and lift gently.
- **4.4** If the string supports the 250 g assembly for at least five seconds consider the bean unit as containing tough string. If the string breaks in less than five seconds, retest the broken parts that are 13 mm or longer to determine if such portions are tough.
- **4.5** Weigh the bean units which contain tough strings.

5. CALCULATION AND EXPRESSION OF RESULTS

% m/m pods containing tough strings = $\frac{\text{pods containing tough strings (g)}}{\text{test sample (g)}} \times 100$

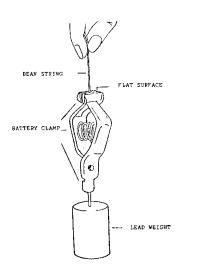


Figure 1: Tough string tester for green or wax beans

METHOD FOR DISTINGUISHING TYPE OF PEAS (CAC/RM 48-1972)

1. **DEFINITION**

This method is based on differentiation between starch granules of the wrinkled-seeded types and starch granules of the smooth-seeded types.

2. REAGENTS AND MATERIALS

- **2.1** Compound microscope:
 - 100 to 250 magnification
 - phase contrast
- 2.2 Microscope slide and cover glass.
- 2.3 Spatula.
- **2.4** Ethanol 95% (v/v).
- 2.5 Glycerine.

3. PROCEDURE

3.1 Preparing mount

- 3.1.1 Remove a small portion of the endosperm and place on glass slide;
- 3.1.2 Using a spatula grind the material with 95% (v/v) ethanol;
- **3.1.3** Add a drop of glycerine, place cover glass on material and examine under microscope.

3.2 Identification

- **3.2.1** Starch granules of the wrinkled-seeded types (garden peas, sweet) show up as clear cut, well defined, generally spherical particles.
- **3.2.2** Starch granules of the smooth-seeded types (round, early, Continental) show up as an amorphous mass with no well-defined geometric shape.

APPENDIX XIV

DETERMINATION OF INTERNAL DEFECTS: DATES

Examine each date carefully for internal defects using a strong light. If the dates are pitted, open up the flesh so that the internal cavity can be viewed. If the dates are unpitted, slit the date open so as to expose the pit, remove the pit and examine the pit cavity.

APPENDIX XV

EXTRANEOUS VEGETABLE MATTER IN DESICCATED COCONUT

The determination is carried out by spreading 100 g of the sample in a thin layer against a white background and counting the extraneous material with the naked eye.

APPENDIX XVI

DETERMINATION OF BROKEN, SLABS, DIRTY, MOULDY, DAMAGED AND IMMATURE FRUITS: **DRIED APRICOTS**

Examine the fruits (sample size: 1 kg) visually and weigh the defective items. Calculate the percentage of defects:

 $\frac{\text{Weight of defective unites}}{\text{Total weight of sample}} x100 = \% \text{ defective}$

DETERMINATION OF VOLUME OF FILL (BY DISPLACEMENT): PICKLED CUCUMBERS

METHOD 1

- (1) This method may be used for all sub-types of pickles. Use a 4 to 8 litre size can with an overflow spout constructed from 0.5 cm to 1 cm inside diameter metal tubing (see Figure 1). The tubing is soldered to an opening in the side of the can 2 cm to 3 cm from the bottom and is bent upward parallel to sides. The tube is bent over and slightly downward from the can at the upper end to form a spout about 4 cm below the top of the can. The lower tip end of the spout is lower than the inside lower curve of the spout (point A). The upper tip end of the spout is slightly shorter than the lower tip end of the spout. A brace near the top of the can holds the tubing firmly in place. A woven wire basket made from screen wire with about eight meshes to the inch with a handle is used for lowering the pickle ingredient into the overflow can.
- (2) Place overflow can on a level table so that overflow will discharge into a sink. Fill the overflow can with water at room temperature (approximately 20 °C or 68 °F). Place the empty basket into the filled overflow can.
- (3) When overflow ceases, place a beaker or graduated cylinder under the spout.
- (4) Remove basket and place drained pickle ingredient (at room temperature) in basket and lower slowly into the overflow can. When overflow ceases, measure the volume of the fluid overflow. The percent volume of pickle ingredient (volume occupied) is calculated as follows:

- x100 = percent volume of pickle ingredient

Figure 1 Point "A" 4 cm 1 brace 0.5 to 1 cm inside diameter 2.5 cm 2 Overflow can

Overflow Volume

Total capacity (volume) of container (see Method E)

METHOD 2

- (1) Use water to partially fill a graduated cylinder (or other technical measuring device) large enough so that the pickle ingredient from one container may be completely submerged.
- (2) Prior to adding the pickle ingredient, record the volume of water in the partially filled cylinder.
- (3) Add all the drained pickle ingredient from one container so that it is entirely submerged.
- (4) Measure the volume of liquid and pickle ingredient after submersion of pickle ingredient.
- (5) Subtract the value in (2) from the value in (4) to obtain the pickle ingredient displacement.
- (6) To determine Volume fill, calculate:

Pickle Ingredient Displacement

 $\frac{1}{Total Capacity (volume) of Container (see Method E)} x100 = percent volume of pickle ingredient$

METHOD 3

- (1) Remove and collect the packing medium from the container for other quality determinations.
- (2) With the pickle ingredient in the container fill it to capacity with water.
- (3) Drain, collect and measure the water.

To determine 'volume fill', calculate:

percent volume of pickle ingredient = $\frac{V_1 - V_2}{V_1}$

Where,

V1=Total capacity (volume) of container; and V2=Volume of drained water from (3) above

APPENDIX XVIII

STANDARD PROCEDURE FOR THAWING OF QUICK-FROZEN FRUITS AND VEGETABLES

1. SCOPE

This thawing procedure is for the purposes of analysis and assessing the organoleptic the characteristics and is generally applicable to all quick-frozen fruits and vegetables.

2. FIELD OF APPLICATION

- **2.1** Most on quick-frozen fruits and many vegetables can be examined on the basis of their organoleptic characteristics in a thawed condition. Where a vegetable requires cooking prior to organoleptic testing the prescribed procedure for the cooking of quick-frozen vegetables is to be followed (Appendix XIX, CAC/RM 33-1970).
- **2.2** Where a particular quick-frozen fruit or vegetable requires special treatment not fully covered by this general procedure for examination, the treatment outlined in the appropriate Codex commodity standard should be followed.

3. DEFINITIONS

- **3.1** Thawing of quick-frozen fruits and vegetables for the purpose of this examination procedure, means subjecting the product to controlled conditions of temperature until the product is sufficiently free from ice crystals so that the individual units can be readily separated and handled.
- **3.2** Air thawing means thawing of the product in unopened container by exposure to air of an ambient temperature in free or forced ventilation.
- **3.3** Water thawing by indirect contact, means thawing of the product in a tightly sealed container by immersion in water, stationary or flowing, at a temperature not exceeding 30 °C.
- **3.4** Water thawing by direct contact, means thawing of the unpacked product by immersion in water at a temperature not exceeding 30 °C. (This method is applicable only to some vegetables).

4. PRINCIPLE OF METHODS

By rapidly thawing quick-frozen products under controlled conditions, the quality factors of the original product retained by the quick-freezing process are preserved to a high degree.

For the purpose of this examination procedure there are two general methods for thawing quick-frozen fruits and vegetables: air thawing and water thawing, Water thawing is faster and in some instances more desirable than air thawing, some quick-frozen commodities, especially those where the product consists of small individual units surrounded, by air, thaw much faster than others, Through experience the analyst will learn to judge the best procedure and time requirement for adequate thawing for each commodity.

5. APPARATUS

- **5.1** Electric fan (optional), for forced ventilation air thawing.
- **5.2** Water bath with thermostat and circulation pump, for indirect or direct water thawing.
- **5.3** Plastic bags or other suitable watertight and closable container, for samples to be subjected to water thawing.
- **5.4** Clamps or weights, to prevent agitation of package in water bath during thawing.
- **5.5** Screen, to remove excess water after water thawing by direct contact.
- 5.6 Tray, on which the product is placed after removal of excess water when thawed by direct contact with water.

6. SAMPLES

The entire package or sample unit is used intact, except that in the case of bulk or industrial size containers a representative sample of 1-2 kg is adequate for testing and organoleptic examination.

7. PROCEDURE

For the rapid thawing of quick-frozen products contained in consumer-size packages, bulk or industrial packages and subsamples of these in suitable containers, one of the following methods should be used:

7.1 Air thawing

Thaw in unopened containers at ambient temperature. To hasten the thawing process forced air ventilation may be applied and the packages may be separated from each other.

7.2 Water thawing by indirect contact

Products packed in tightly sealed containers may be thawed by immersion of the container in water at a temperature not exceeding 30 °C, e.g. a water bath with thermostat and circulation pump.

7.3 Water thawing b direct contact (applicable only to some vegetables)

The vegetable is removed from the pack and thawed by immersion in water at a temperature not exceeding 30 °C. As soon as the product is thawed sufficiently to permit easy separation of the individual units, it is drained on a suitable screen to remove excess water and placed on a tray for final air thawing and examination.

8. NOTES ON PROCEDURE

8.1 Selection of thawing method

- **8.1.1** Certain quick-frozen vegetables should not be subjected to water thawing by direct contact in order to prevent leaching of soluble solids or product material.
- **8.1.2** If there is an indication of off flavours or off odours in the quick-frozen product when the packages are opened, water thawing by direct contact is not to be used as a preparatory step to cooking, as the off flavour or off odour may be partially removed during such thawing. Such suspect samples are to be placed in a cooking receptacle while still frozen.

8.2 Prevention of damage

Extreme care should be taken during the thawing process in order that the product is not damaged or exposed to abuse that will alter or degrade the true characteristics of the product. Quick-frozen fruits are more susceptible to abuse during thawing than quick-frozen vegetables. Some fruits, especially light-coloured fruits, oxidize quite readily and should be examined for colour before thawing is completed. Also, some fruits show a breakdown in texture or "bleed" when thawed more than necessary. Consequently, rapid thawing under controlled conditions is most desirable in preparing the product for laboratory examination.

9. TEST REPORT

The identity of the sample and the thawing procedure used should be recorded.

10. ADDITIONAL NOTES

- **10.1** Quick-frozen corn (maize) or products containing corn should always be air thawed or water thawed by indirect contact to avoid leaching of soluble solids or product material.
- **10.2** Quick-frozen peaches and apricots (light-coloured fruits) and red cherries oxidize quite readily and should be examined while some ice crystals remain in the product.

STANDARD PROCEDURE FOR COOKING OF QUICK-FROZEN VEGETABLES (CAC/RM 33-1970)

1. SCOPE

This cooking procedure is for the purposes of analysis and assessing the organoleptic characteristics and is generally applicable to all quick-frozen vegetables.

2. FIELD OF APPLICATION

- **2.1** The cooking procedure described below applies to those quick-frozen vegetables which are normally cooked prior to consumption for the proper evaluation of such organoleptic quality factors as texture, tenderness, maturity or flavour.
- **2.2** Where a particular quick-frozen vegetable requires a special cooking procedure not fully covered by this general procedure for examination, the method outlined in the appropriate Codex commodity standard shall be followed.

3. DEFINITION

Cooking of vegetables, for the purpose of this examination procedure, means to prepare, food for the table by subjecting quick-frozen vegetables to an appropriate standard (cooking) procedure by partial or whole immersion of the product in boiling water for a specified time.

4. PRINCIPLE OF METHOD

By heating the quick-frozen vegetable, through partial or whole immersion in water at boiling temperature for such a period of time as to undergo specific changes of conditions.

5. APPARATUS

- **5.1** Two-litre saucepan with cover.
- **5.2** Hot plate or gas fire.
- **5.3** Tray on which product is placed after cooking for cooling and presentation.
- **5.4** Graduated cylinder or similar measuring device for water.

6. SAMPLES

Generally, a separate set of samples for cooking purposes only need not be taken. Ordinarily part of the contents of a larger retail size package or part of a sample of a bulk container, used for testing other product characteristics can be used for the cooking procedure. Care should be taken, however, that the portion used for cooking is not treated differently from the normal procedure, e.g. thawed prior to cooking whereas the product would usually be put in boiling water while still in the frozen state.

DETERMINATION OF THE ALCOHOL-INSOLUBLE SOLIDS CONTENT OF QUICK-FROZEN PEAS

1. PRINCIPLE OF THE METHOD

The alcohol-insoluble solids in peas consist mainly of insoluble carbohydrates (starch) and protein. A weighed quantity of the sample is boiled with slightly diluted alcohol. The solids are washed with alcohol until the filtrate is clear. The alcohol-insoluble solids are dried and weighed. The percentage by mass present is used as a guide to maturity.

2. REAGENTS

2.1 Ethanol (95 percent) or denaturated ethanol.

Ethanol denaturated with 5 percent v/v methanol.

2.2 Diluted ethanol or diluted denaturated ethanol 80 percent v/v.

Dilute 8 parts by volume of reagent under Section 2.1 to 9.5 parts by volume with H₂O.

3. APPARATUS

- **3.1** Analytical balance.
- **3.2** Beaker, 600 ml, if sample is boiled or 250 ml (standard taper ground-glass joint) flask with reflux condenser if refluxed.
- 3.3 Buchner funnel.
- 3.4 Drying dish with lid, flat bottomed.
- **3.5** Hot plates or boiling water bath for refluxing or boiling.
- 3.6 Clamps or weights to prevent agitation of package in water bath during thawing.
- **3.7** Desiccator with active desiccant.
- **3.8** Drying oven, well-ventilated and thermostatically controlled and adjusted to operate at 100 ± 2 °C.
- **3.9** Filter paper, Whatman No. 1 or equivalent.
- 3.10 Macerator or blender.
- **3.11** Plastics bag of sufficient capacity to hold the entire sample for thawing.
- **3.12** "Policemen" on glass rods, bent so as to facilitate cleaning flask or beaker.
- 3.13 Water bath, with continuous flow at room temperature or regulated at room temperature for thawing.

4. PREPARATION OF TEST SAMPLE

Place frozen peas or frozen peas with sauce in plastic bag and tie off. Immerse sample in water bath with continuous flow at room temperature or regulated at room temperature. Avoid agitation of package during thawing by using clamps or weights if necessary. When completely thawed, remove package from bath. Blot off adhering water from the plastic bag. Transfer the peas from container to a sieve, the meshes of which are made by so weaving wire as to from square openings of 2.8 mm by 2.8 mm. If sauce is present, wash with gentle spray of water at room temperature until the sauce is removed. Without shifting the peas, incline the sieve as to facilitate drainage, and drain for two minutes. Wipe the bottom of the sieve. Weight 250 g peas into blender, add 250 ml distilled water and macerate to a smooth paste. If there is less than 250 g sample, use the entire sample of peas with an equivalent quantity by mass of distilled water and macerate to a smooth paste.

5. PROCEDURE

- **5.1** Dry a filter paper in flat bottomed dish, lid off, for two hours at 100 ± 2 °C. Cover dish, cool in a desiccator and weigh accurately. (The filter paper should be larger than the base of the funnel and folded at the circumference to facilitate subsequent removal without loss of solids).
- **5.2** Weight 20 g \pm 0.01 g paste into a 250 ml ground-joint flask, add 120 ml denaturated ethanol or ethanol, and swirl to mix. Reflux on a steam or water bath for 30 minutes.

If boiling rather than refluxing is preferred, weight 40 g \pm 0.01 g paste into a 600 ml beaker. Add 240 ml denaturated ethanol or ethanol, stir and cover beaker. Bring solution in the beaker to a boil and simmer slowly for 30 minutes on a hot plate.

Immediately filter with suction on a Buchner funnel through the dried and weighed filter paper. Decant most of the supernatant liquid through the filter paper. Wash the solids in the flask or beaker without delay, with small portions of 80 percent denaturated ethanol or 80 percent ethanol until the washings are colourless, allow solids to become dry during the washing. Transfer solids to the filter paper, spreading the solids evenly.

5.3 Remove the filter paper containing the residue from the funnel, transfer to the dish used in preparing the filter paper and dry uncovered in an air over for two hours at 100 ± 2 °C. Cover the dish, cool in a desiccator and weigh accurately. The weight of the dry residue is the difference between the weight under Section 5.1 and this final weight.

6. CALCULATION AND EXPRESSION OF RESULTS

Calculate the alcohol-insoluble solids content of the sample by means of the following formula:

6.1 If 20 g sample is refluxed:

Alcohol-insoluble solids content (% m/m) = 10 M

Where:

 \underline{M} = the mass in g of dry residue (see Section 5.3)

6.2 If 40 g sample is refluxed:

Alcohol-insoluble solids content (% m/m) = 5 M

Where:

 \underline{M} = the mass in g of dry residue (see Section 5.3)

7. REPEATABILITY OF RESULTS

The difference between results of duplicate determination (results obtained simultaneously or in rapid succession by the same analyst) should not exceed 0.6 g alcohol-insoluble solids for 100 g of the product.

8. EXPRESSION OF RESULTS

Results are expressed as g alcohol-insoluble solids per 100 g of the product (% m/m).

APPENDIX XXI

DETERMINATION OF SALT-FREE DRY MATTER (QUICK-FROZEN SPINACH)

PROCEDURE

- 1. Determine the total dry matter of the product by drying over sand for 4 hours at 105 °C.
- 2. From the value obtained in (1) deduct the amount of salt (NaCl) determine by either (a) electrometric titration using a pH metre with a silver electrode; or (b) direct titration with AgNO₃. Express the result, after deducting salt from total dry matter, as salt-free dry matter.

DETERMINATION OF PEROXIDE VALUE IN COOKED RICE WRAPPED IN PLANT LEAVES: EXTRACTION OF OILS FROM THE PRODUCT

Apparatus

- (a) Rotary evaporator
- (b) Water bath

Extraction

Remove the product package and plant leaves etc. take out the edible part of the representative sample; crush it and put it in a homogenizer or glass mortar and grind it continuously to make the sample fully mashed and mixed well and then put it in the wide-mouth bottle and add 2 to 3 times the sample volume of petroleum ether (boiling range: 30 °C–60 °C). After fully mixing, stopper the bottle and leave for more than 12 hours. Filter all the solution with a funnel filled with anhydrous sodium sulphate into a round-bottom flask. Rinse the residue in the wide-mouth bottle with petroleum ether. Repeat the filtration once with a new anhydrous sodium sulphate funnel, if the filtrate is not clear enough. Evaporate the petroleum ether in the round-bottom flask under reduced pressure on a rotary evaporator at below 40 °C, and the residue is the test sample. A sufficient number of representative samples should be selected to ensure that not less than 8 grams of the test sample can be obtained. The test sample should be tested as soon as possible.

PART A – IDENTIFICATION OF SCOPOLETIN IN FERMENTED NONI FRUIT JUICE

1. PREPARATION OF SAMPLES

Noni fruit juice is filtered through a 0.45 μ m membrane filter and then purified by solid-phase extraction (SPE) with Waters OASIS® HLB 6cc 200 mg extraction cartridges (or similar solid-phase extraction cartridge), after first equilibrating with methanol (5 mL) followed by deionized water (5 mL). The filtered juice samples (3 mL) are then loaded onto the equilibrated cartridge and washed with 5 percent methanol (MeOH) in deionized water (5 mL). The cartridges are allowed to dry under flow of air for 5 mins and then, eluted with MeOH (3 mL). The MeOH eluate is retained for thin layer chromatography (TLC) analysis. The SPE flow rates of equilibration, wash and elution solvents through the cartridge is approximately 1 drop per second.

2. PREPARATION OF REFERENCE STANDARD

- **2.1** A reference standard is prepared by dissolving 0.1 mg scopoletin in 1 millilitre of methanol.
- **2.2** Alternately, certified *Morinda citrifolia* reference plant material may be prepared in the same manner as the samples to be analysed. The certified *Morinda citrifolia* reference material should be from the same part of the plant as the samples to be analysed.

3. IDENTIFICATION

3.1 Thin layer chromatography

Spot 5 microlitres of sample solutions and reference standard solution on a silica gel 60 F254 TLC plate. After spotting the plates are dried at 110 °C for 15 minutes in a drying oven. Develop the plate with a mobile phase of dichloromethane: methanol (19:1, v/v). View bright fluorescent blue colours on developed plate under UV lamp, 365 nm. Identify scopoletin in samples by comparing Rf values and colours to the standard.

PART B- IDENTIFICATION OF DEACETYLASPERULOSIDIC ACID IN FERMENTED NONI FRUIT JUICE

1. PREPARATION OF SAMPLES

Noni fruit juice is filtered through a 0.45 µm membrane filter and diluted 1:1 with MeOH.

2. PREPARATION OF REFERENCE STANDARD

- **2.1** A reference standard is prepared by dissolving 1 mg deacetylasperulosidic acid in 1 millilitre of methanol.
- **2.2** Alternately, certified *Morinda citrifolia* reference plant material may be prepared in the same manner as the samples to be analysed. The certified *Morinda citrifolia* reference material should be from the same part of the plant as the samples to be analysed.

3. PREPARATION OF p-ANISALDEHYDE SOLUTION

Anisaldehyde solution was prepared by dissolving 2 g of p-anisaldehyde in 96 mL of ethanol with stirring. The solution was then acidified through dropwise addition of concentrated sulfuric acid (4 mL).

4. IDENTIFICATION

4.1 THIN LAYER CHROMATOGRAPHY

Spot 5 microlitres of sample solutions and reference standard solution on a silica gel 60 F254 TLC plate, previously dried at 110 °C for 15 minutes in a drying oven. After spotting samples are again dried at 110 °C or through application of heat via a heat gun for a period of 8-10 seconds. The TLC plates are developed with a mobile phase of dichloromethane: methanol: water (13:6:1, v/v/v). Upon completion of elution, the plate is air dried and developed by spraying with 2% anisaldehyde/4% sulfuric acid in ethanol (EtOH) solution and then heat in oven at 110 °C for 1–5 minutes to reveal and maximize the blue colour. Identify deacetylasperulosidic in samples by comparing spot Rf values and colour with reference standard solution on same TLC plate.

APPENDIX XXIV

METHOD FOR THE EXTRACTION OF OIL (LAVER PRODUCTS)

Weigh 50 g of test sample into a 1 000 ml Erlenmeyer flask.

Add 500 ml of petroleum ether to the flask followed by replacing air in the flask by N_2 gas

Put a stopper on the flask and let it stand for 2 hours.

Decant the extracted solution (A) through a filter paper, on which Na₂SO₄ is mounted to remove moisture, on a funnel into a 1 000 ml round flask-flat bottom.

Add an additional 250 ml of petroleum ether to the residue in the Erlenmeyer flask and decant the extracted solution (B) into the round flask-flat bottom again as done previously.

Evaporate the whole extracted solution (mixture of solutions A and B) on the rotary evaporator in vacuum less than 40 °C.

NOTES

¹ FAO and WHO. 1981. *Standard for natural mineral waters*. Codex Alimentarius Standard, No. CXS 108-1981. Codex Alimentarius Commission. Rome.

² FAO and WHO. 1991. *Standard for Canned Shrimps or Prawns.* Codex Alimentarius Standard, No. CXS 37-1991. Codex Alimentarius Commission. Rome.

³ FAO and WHO. 1999. *Guidelines for the sensory evaluation of fish and shellfish in laboratories*. Codex Alimentarius Guideline, No. CXG 31-1999. Codex Alimentarius Commission. Rome.

⁴ FAO and WHO. 1981. *Standard for quick-frozen shrimps or prawns*. Codex Alimentarius Standard, No. CXS 92-1981. Codex Alimentarius Commission. Rome.

⁵ FAO and WHO. 1981. *Standard for quick-frozen lobsters.* Codex Alimentarius Standard, No. CXS 95-1981. Codex Alimentarius Commission. Rome.

⁶ FAO and WHO. 1995. *Standard for quick-frozen raw squid*. Codex Alimentarius Standard, No. CXS 191-1995. Codex Alimentarius Commission. Rome.

⁷ FAO and WHO. 2014. *Standard for fresh and quick-frozen raw scallop products*. Codex Alimentarius Standard, No. CXS 315-2014. Codex Alimentarius Commission. Rome.

⁸ FAO and WHO. 2024. *Standard for salted Atlantic herring and salted sprat.* Codex Alimentarius Standard, No. CXS 244-2004. Codex Alimentarius Commission. Rome.

⁹ FAO and WHO. 2010. *Standard for sturgeon caviar*. Codex Alimentarius Standard, No. CXS 291-2010. Codex Alimentarius Commission. Rome.

¹⁰ FAO and WHO. 2008. *Standard for live and raw bivalve molluscs*. Codex Alimentarius Standard, No. CXS 292-2008. Codex Alimentarius Commission. Rome.

¹¹ FAO and WHO. 2013. Standard for live abalone and for raw fresh chilled or frozen abalone for direct consumption or for further processing. Codex Alimentarius Standard, No. CXS 312-2013. Codex Alimentarius Commission. Rome.
¹² FAO and WHO. 1989. Standard for guick-frozen fish sticks (fish fingers), fish portions and fish fillets - breaded or in

batter. Codex Alimentarius Standard, No. CXS 166-1989. Codex Alimentarius Commission. Rome.