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#### **RECOMMENDED METHODS OF ANALYSIS AND SAMPLING**

CXS 234-1999<sup>1</sup>

Adopted in 1999.

<sup>&</sup>lt;sup>1</sup> 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 41st Session of the Codex Alimentarius Commission in 2018.

**Miscellaneous Products** 

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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

Commodity	Provision	Method	Principle	Туре
All Foods				
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	Ш
All foods	Saccharin	EN 12856	High performance liquid chromatography	III
All Foods (see also meat products)	Nitrates and/or Nitrites	EN 12014-1	Part 1- General considerations	N/A
Individual Foods <sup>2</sup>	Sulphites	EN 1988-1 AOAC 990.28	Part 1: Optimized Monier-Williams method	
Individual Foods <sup>3</sup>	Sulphites	EN 1988-2	Part 2: Enzymatic method	111
		NMKL 135	Fait 2. Enzymatic method	
Cereals, Pulses and Legumes and I	Derived Products			
Certain pulses	Moisture	ISO 665	Gravimetry	I
Degermed maize (corn) meal and maize (corn) grits	Ash	AOAC 923.03 ISO 2171 ICC Method No 104/1	Gravimetry	I
Degermed maize (corn) meal and maize (corn) grits	Fat, crude	AOAC 945.38F; 920.39C	Gravimetry (ether extraction)	I
Degermed maize (corn) meal and maize (corn) grits	Moisture	ISO 712 ICC Method No 110/1	Gravimetry	I
Degermed maize (corn) meal and maize (corn) grits	Particle size (granularity)	AOAC 965.22	Sieving	I

<sup>2</sup> Hominy, fruit juice, sea food
<sup>3</sup> Wine, dried apples, lemon juice, potato flakes, sultanas, beer

Cereals, Pulses and Legumes and	Derived Products			
Degermed maize (corn) meal and maize (corn) grits	Protein	ICC Method No 105/1	Titrimetry, Kjeldahl digestion	Ι
Durum wheat semolina and durum wheat flour	Ash (semolina)	AOAC 923.03 ISO 2171	Gravimetry	I
Durum wheat semolina and durum wheat flour	Moisture	ISO 712 ICC 110/1	Gravimetry	Ι
Durum wheat semolina and durum wheat flour	Protein (N x 5.7)	ICC 105/1	Titrimetry, Kjeldahl digestion	Ι
Instant Noodles	Extraction of oil from instant noodles	described in the standard	Gravimetry	I
Instant Noodles	Acid Value	described in the standard	Titrimetry	I
Instant Noodles	Moisture	described in the standard	Gravimetry	Ι
Maize (corn)	Moisture	ISO 6540	Gravimetry	Ι
Peanuts (raw)	Aflatoxins, total	AOAC 991.31	Immunoaffinity column (Aflatest)	П
Peanuts (raw)	Aflatoxins, total	AOAC 993.17	Thin layer chromatography	Ш
Peanuts (intended for further processing)	Aflatoxins, total	AOAC 975.36	Romer minicolmn	
Peanuts (Cereals, shell-fruits and derived products (including peanuts))	Sum of aflatoxins $B_1$ , $B_2$ , $G_1$ and $G_2$	EN 12955 ISO 16050	HPLC with post column derivatization and immunoaffinity column clean up	111
Peanuts (intended for further processing)	Aflatoxins, total	AOAC 979.18	Holaday-Velasco minicolumn	111
Pearl millet flour	Ash	AOAC 923.03	Gravimetry	Ι
Pearl millet flour	Colour	<i>Modern Cereal Chemistry</i> , 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605- 612, Food Trade Press Ltd, London, 1969.	Colorimetry using specific colour grader	IV
Pearl millet flour	Fat, crude	AOAC 945.38F; 920.39C	Gravimetry (ether extraction)	Ι
Pearl millet flour	Fibre, crude	ISO 5498: (B.5 Separation)	Gravimetry	Ι
Pearl millet flour	Moisture	ISO 712: ICC 110/1	Gravimetry	Ι

Cereals, Pulses and Legume	es and Derived Products			
Quinoa	Moisture content	ISO 712 / AACCI 44-15.02	Gravimetry	I
Quinoa	Protein Content (N x 6.25 in dry weight basis)	ISO 1871	Titrimetry (Kjeldahl)	IV
Pearl millet flour	Protein	AOAC 920.87	Titrimetry, Kjeldahl digestion	I
Sorghum flour	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	Ι
Sorghum flour	Colour	<i>Modern Cereal Chemistry</i> , 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605- 612, Food Trade Press Ltd, London, 1969.	Colorimetry using specific colour grader	IV
Sorghum flour	Fat, crude	AOAC 945.38F; 920.39C	Gravimetry (ether extraction)	I
Sorghum flour	Fibre, crude	ICC 113 ISO 6541	Gravimetry	Ι
Sorghum flour	Moisture	ISO 712 ICC 110/1	Gravimetry	I
Sorghum flour	Particle size (granularity)	AOAC 965.22	Sieving	I
Sorghum flour	Protein	ICC 105/1	Titrimetry, Kjeldahl digestion	I
Sorghum flour	Tannins	ISO 9648	Spectrophotometry	I
Sorghum grains	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	Ι
Sorghum grains	Fat, crude	AOAC 945.38F, 920.39C	Gravimetry (ether extraction)	I
Sorghum grains	Moisture	ISO 6540	Gravimetry	I
Sorghum grains	Protein	ICC 105/1	Titrimetry, Kjeldahl digestion	I
Sorghum grains	Tannins	ISO 9648	Spectrophotometry	I
Soy protein products	Ash	AOAC 923.03 ISO 2171: (Method B)	Gravimetry	Ι
Soy protein products	Fat	CAC/RM 55 - Method 1	Gravimetry (extraction)	Ι

Cereals, Pulses and Legumes and	Derived Products			
Soy protein products	Fibre, crude	ISO 5498	Gravimetry	I
Soy protein products	Moisture	AOAC 925.09	Gravimetry (vacuum oven)	I
Soy protein products	Protein	AOAC 955.04D (using factor 6.25)	Titrimetry , Kjeldahl digestion	II
Vegetable protein products	Ash	AOAC 923.03 ISO 2171 (Method B)	Gravimetry, Direct	I
Vegetable protein products	Fat	CAC/RM 55 - Method 1	Gravimetry (extraction)	I
Vegetable protein products	Fibre, crude	AACC 32-17	Ceramic fiber filteration	I
Vegetable protein products	Moisture	AOAC 925.09	Gravimetry (vacuum oven)	I
Vegetable protein products	Protein	AOAC 955.04D (using factor 6.25)	Titrimetry, Kjeldahl digestion	II
Wheat flour	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Wheat flour	Fat acidity	AOAC 939.05	Titrimetry	I
Wheat flour	Moisture	ISO 712: ICC 110/1	Gravimetry	I
Wheat flour	Particle size (granularity)	AOAC 965.22	Sieving	I
Wheat flour	Protein	ICC 105/1	Titrimetry, Kjeldahl digestion	I
Wheat protein products including wheat gluten	Protein	Vital wheat gluten and devitalized wheat gluten AOAC 979.09 (wheat protein in grain N x 5.7)	Kjeldahl	I
		Solubilized wheat protein AOAC 920.87 (wheat protein in flour N x 5.7)	Kjeldahl	I
Wheat protein products including Wheat gluten	Fibre, crude	AOAC 962.09	Ceramic fiber filteration	I

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Cereals, Pulses and Legumes and	Derived Products			
Wheat protein products including Wheat gluten	Ash	AOAC 923.03 ISO 2171: method B	Gravimetry	I
Whole and decorticated pearl millet grains	Ash	AOAC 923.03	Gravimetry	I
Whole and decorticated pearl millet grains	Fat, crude	AOAC 945.38F; 920.39C	Gravimetry (ether extraction)	Ι
Whole and decorticated pearl millet grains	Fibre, crude	ISO 5498 (B.5 Separation)	Gravimetry	Ι
Whole and decorticated pearl millet grains	Moisture	ISO 712 ICC 110/1	Gravimetry	Ι
Whole and decorticated pearl millet grains	Protein	AOAC 920.87	Titrimetry, Kjeldahl digestion	I
Whole maize (corn) meal	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Whole maize (corn) meal	Fat, crude	AOAC 945.38F; 920.39C	Gravimetry (ether extraction)	Ι
Whole maize (corn) meal	Moisture	ISO 712 ICC 110/1	Gravimetry	Ι
Whole maize (corn) meal	Particle size (granularity)	AOAC 965.22	Sieving	Ι
Whole maize (corn) meal	Protein	ICC 105/1	Titrimetry, Kjeldahl digestion	I
Cocoa Products and Chocolate				
Chocolate and chocolate products	Cocoa butter	AOAC 963.15 IOCCC 14	Gravimetry (Soxhlet extraction)	I
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)	I

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Cocoa Products and Chocolate				
Chocolate and chocolate products	Milkfat	IOCCC 5 AOAC 945.34; 925.41B; 920.80	Titrimetry/Distillation	I
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	I
Cocoa powders (cocoa) and dry cocoa-sugar mixtures	Moisture	IOCCC 26 or AOAC 977.10 (Karl Fischer method)	Gravimetry	Ι
Fats and Oils and Related Products				
Fats and Oils (all)	Arsenic	AOAC 952.13 (Codex general method)	Colorimetry (diethyldithiocarbamate)	II
Fats and Oils (all)	Arsenic	AOAC 942.17 (Codex general method)	Colorimetry (molybdenum blue)	
Fats and Oils (all)	Arsenic	AOAC 986.15 (Codex general method)	Atomic absorption spectrophotometry	III
Fats and oils	Butylhydroxyanisole, butylhydroxytoluene, tert- butylhydroquinone, & propyl gallate	AOAC 983.15; or AOCS Ce-6-86	Liquid chromatography	II
Fats and Oils (all)	Insoluble impurities	ISO 663	Gravimetry	I
Fats and Oils (all)	Lead	AOAC 994.02 ISO 12193 (Codex general method) or AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II

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Fats and Oils and Related Products

Fats and Oils (all)	Matter volatile at 105°C	ISO 662	Gravimetry (open-drying)	Ι
Fats and Oils (all)	Soap content	BS 684 Section 2.5; or AOCS Cc 17-95	Gravimetry	I
Fats and oils not covered by individual standards	Acid Value	ISO 660; or AOCS Cd 3d-63	Titrimetry	I
Fats and oils not covered by individual standards	Copper and Iron	AOAC 990.05 ISO 8294 or AOCS Ca 18b-91 (Codex general method)	Atomic absorption Spectrophotometry (direct graphite furnace)	II
Fats and oils not covered by individual standards	Peroxide value	AOCS Cd 8b-90 ISO 3960	Titrimetry using iso-octane	I
Fat spreads and blended spreads	Fat content	ISO 17189   IDF 194	Gravimetry	Ι
Fish oils	Fatty acid composition	ISO 5508	Gas chromatography	Ш
Fish oils	Fatty acid composition	ISO 12966-2	Gas chromatography	111
Fish oils	Fatty acid composition	AOCS Ce 1b-89	GLC	111
Fish oils	Fatty acid composition	AOCS Ce 1-07	Capillary GLC	Ш
Fish oils	Fatty acid composition	AOCS Ce 2b-11	Alkali hydrolysis	111
Fish oils	Fatty acid composition	AOCS Ce 1a-13	Capillary GLC	111
Fish oils	Fatty acid composition	AOCS Ce 2-66	Preparation of methyl esters by fatty acids	111
Fish oils	Acid value	AOCS Ca 5a-40 AOCS CD 3D-63 ISO 3960 NMKL 38	Titration	I
Fish oils	Peroxide value	AOCS Cd 8b-90 ISO 3960 NMKL 158	Titration	I
Fish oils	Peroxide value	European Pharmacopeia 2.5.5 (Part B Iso- octane as solvent)	Titration	Ι

Fats and Oils and Related Pr	oducts			
Fish oils	Phospholipids	USP-FCC10 2S(Krill oil): Phospholipids, Nuclear Magnetic Resonance, Appendix IIC	NMR Spectroscopy	IV
Fish oils	P-Anisidine value	European Pharmacopoeia 2.5.36/ AOCS Cd 18-90/ ISO 6885	Spectrophotometry	Ι
Fish oils	Triglycerides	USP 40-NF35(Omega-3 Acid Triglycerides): Content of oligomers and partial glyceride	HPLC-RI	
		European Pharmacopoeia 1352 (Omega3 acid triglycerides): Oligomers and partial glycerides	HPLC RI	III
		AOCS Cd 11d-96	HPLC-ELSD	111
Fish oils	Vitamin A	European Parharmcopeia Monograph on Cod Liver Oil (Type A), monograph 01/2005:1192, with LC end-point 2.2.29	LC	III
Fish oils	Vitamin A	EN 12823-1 (Determination of vitamin A by high performance liquid chromatograph – Part 1: Measurement of all-E-retinol and 13- Z-retinol	LC	III
Fish oils	Vitamin D	EN 12821 (Determination of vitamin D by high performance liquid chromatography – Measurement of cholecalciferol (D3) or ergocalciferol (D2))	LC	111
Fish oils	Vitamin D	NMKL 167 (Cholecalciferol (vitamin D3) and Ergocalciferol (vitamin D2). Determination by HPLC in foodstuffs	LC	III
Named Animal Fats	Acidity	ISO 660; or AOCS Cd 3d-63	Titrimetry	I
Named Animal Fats	Copper and Iron	AOAC 990.05 ISO 8294; or AOCS Ca 18b-91 (Codex general method)	Atomic absorption Spectrophotometry (direct graphite furnace)	II
Named Animal Fats	GLC ranges of fatty acid composition	ISO 5508 and ISO 12966-2 or AOCS Ce 2-66 and Ce 1e-91 or Ce 1f-96	Gas chromatography of methyl esters	II

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Fats and Oils and Related Prod	ucts			
Named Animal Fats	lodine value (IV)	ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92	Wijs-Titrimetry	I
Named Animal Fats	Peroxide value	AOCS Cd 8b-90 ISO 3960	Titrimetry using <i>iso</i> -octane	I
Named Animal Fats	Relative density	ISO/AOCS method for apparent density to be inserted	Pycnometry	II
Named Animal Fats	Refractive index	ISO 6320; or AOCS Cc 7-25	Refractometry	II
Named Animal Fats	Saponification value	ISO 3657; or AOCS Cd 3-25	Titrimetry	I
Named Animal Fats	Unsaponifiable matter	ISO 3596 or ISO 18609; or AOCS Ca 6b-53	Titrimetry after extraction with diethyl ether	Ι
Named Animal Fats	Titre	ISO 935; or AOCS Cc 12-59	Thermometry	Ι
Named Vegetable Oils	Acidity	ISO 660; or AOCS Cd 3d-63	Titrimetry	I
Named Vegetable Oils	Apparent density	ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95	Pycnometry	I
Named Vegetable Oils	Baudouin test (modified Villavecchia or sesameseed oil test)	AOCS Cb 2-40	Colour reaction	I
Named Vegetable Oils	Carotenoids, total	BS 684 Section 2.20	Spectrophotometry	П
Named Vegetable Oils	Copper and iron	ISO 8294; or AOAC 990.05; or AOCS Ca 18b-91	AAS	II
Named Vegetable Oils	Crismer value	AOCS Cb 4-35 and AOCS Ca 5a-40	Turbidity	Ι
Named Vegetable Oils	GLC ranges of fatty acid composition	ISO 5508 and ISO 12966-2; or AOCS Ce 2- 66 and Ce 162 or 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	Gravimetry	Ι
Named Vegetable Oils	lodine value (IV)	Wijs - ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92; or NMKL 39	Wijs-Titrimetry <sup>4</sup>	I
Named Vegetable Oils	Lead	AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91	Atomic Absorption	II

<sup>&</sup>lt;sup>4</sup> It is possible to calculate the lodine Value from fatty acid composition data obtained by gas chromatography e.g. using AOCS Cd 1b-87

Fats and Oils and Related Products	6			
Named Vegetable Oils	Moisture & volatile matter at 105°C	ISO 662	Gravimetry	I
Named Vegetable Oils	Peroxide value (PV)	AOCS Cd 8b-90 or ISO 3960	Titrimetry	I
Named Vegetable Oils	Refractive index	ISO 6320 or AOCS Cc 7-25	Refractometry	
Named Vegetable Oils	Reichert value and Polenske value	AOCS Cd 5-40	Titrimetry	I
Named Vegetable Oils	Relative density	IUPAC 2.101 with the appropriate conversion factor See comment above (Named Animal Fats) <sup>5</sup>	Pycnometry	I
Named Vegetable Oils	Saponification value (SV)	ISO 3657 or AOCS Cd 3-25	Titrimetry	Ι
Named Vegetable Oils	Slip point	ISO 6321 for all oils; AOCS Cc 3b-92 for all oils except palm oils; AOCS Cc 3-25 for palm oils only	Open ended capillary tube	I
Named Vegetable Oils	Soap content	BS 684 Section 2.5; or AOCS Cc 17-95	Gravimetry	I
Named Vegetable Oils	Sterol content	ISO 12228; or AOCS Ch 6-91	Gas chromatography	II
Named Vegetable Oils	Tocopherol content	ISO 9936 or AOCS Ce 8-89	HPLC	II
Named Vegetable Oils	Unsaponifiable matter	ISO 3596; or ISO 18609; or AOCS Ca 6b-53	Gravimetry	
Olive Oils and Olive Pomace Oils	Absorbency in ultra-violet	COI/T.20/Doc. No. 19 or ISO 3656 or AOCS Ch 5-91	Absorption in ultra violet	II
Olive Oils and Olive Pomace Oils	Acidity, free (acid value)	ISO 660 or AOCS Cd 3d-63	Titrimetry	Ι
Olive Oils and Olive Pomace Oils	Alpha-tocopherol	ISO 9936	HPLC	
Olive Oils and Olive Pomace Oils	Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 or AOCS Ce 5b-89	Analysis of triglycerides of HPLC and calculation	Ι
Olive Oils and Olive Pomace Oils	Erythrodiol + uvaol	COI/T.20/Doc.no. 30	Gas chromatography	
Olive Oils and Olive Pomace Oils	Halogenated solvents, traces	COI/T.20/Doc. no. 8	Gas chromatography	II

<sup>5</sup> The method is no longer available.

Fats and Oils and Related Products	5			
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 or AOAC 993.20 or AOCS Cd 1d- 92 or NMKL 39	Wijs-Titrimetry	Ι
Olive Oils and Olive Pomace Oils	Iron and copper	ISO 8294 or AOAC 990.05	AAS	
Olive Oils and Olive Pomace Oils	Lead	AOAC 994.02 or ISO 12193 or AOCS Ca 18c-91	AAS	II
Olive Oils and Olive Pomace Oils	Moisture and volatile matter	ISO 662	Gravimetry	Ι
Olive Oils and Olive Pomace Oils	Organoleptic characteristics	COI/T.20/Doc. no. 15	Panel test	Ι
Olive Oils and Olive Pomace Oils	Peroxide value	ISO 3960 or AOCS Cd 8b-90	Titrimetry	Ι
Olive Oils and Olive Pomace Oils	Relative density	IUPAC 2.101, with the appropriate conversion factor. See comment above	Pycnometry	Ι
Olive Oils and Olive Pomace Oils	Refractive index	ISO 6320 or AOCS Cc 7-25	Refractometry	II
Olive Oils and Olive Pomace Oils	Saponification value	ISO 3657 or AOCS Cd 3-25	Titrimetry	
Olive Oils and Olive Pomace Oils	Sterol composition and total sterols	COI/T.20/Doc. no. 30   ISO 12228-2 or AOCS Ch 6-91	Gas chromatography	II
Olive Oils and Olive Pomace Oils	Stigmastadienes	COI/T.20/Doc. no. 11 or ISO 15788-1 or AOCS Cd 26-96	Gas chromatography	II
Olive Oils and Olive Pomace Oils	Stigmastadienes	ISO 15788-2	HPLC	111
Olive Oils and Olive Pomace Oils	Trans fatty acids content	COI/T.20/Doc no. 17 or ISO 15304 or AOCS Ch 2a-94	Gas chromatography of methyl esters	II
Olive Oils and Olive Pomace Oils	Unsaponifiable matter	ISO 3596 or ISO 18609 or AOCS Ca 6b-53	Gravimetry	
Olive Oils and Olive Pomace Oils	Wax content	COI/T.20/Doc. no. 18 or AOCS Ch 8-02	Gas chromatography	II
Fish and Fishery Products				
Fish and fishery products	Histamine	AOAC 977.13	Fluorimetry	П
Fish and fishery products	Mercury	AOAC 977.15	Flameless atomic absorption spectrophotometry	
Fish and fishery products: canned products	Drained weight	Described in the Standard	Weighing	Ι

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Fish and Fishery Products				
Fish and fishery products: canned products	Net weight	Described in the Standard	Weighing	I
Boiled Dried Salted Anchovies	Sodium Chloride (chloride expressed as sodium chloride)	AOAC 937.09	Titrimetry	II
Canned shrimps or prawns	Size, determination of	Described in the Standard	Number per 100 g	Ι
Fish Sauce	total nitrogen	AOAC 940.25	digestion	I
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	AOAC 981.12 The pH shall be measured in a sample of fish sauce diluted with water to 1:10 using a pH meter. The dilution of fish sauce is necessary because of the high ionic strength in the undiluted sauce.	electrometry	III
Fish Sauce	sodium chloride	AOAC 976.18	potentiometry	II
Fish Sauce	sodium chloride	AOAC 937.09	titrimetry	IV
Fish Sauce	histamine	AOAC 977.13	Fluorimetry	II
Frozen abalone (covered by glaze)	Net weight	AOAC 963.18	Gravimetry	I
Frozen fish and fishery products	Thawing and cooking procedures	Described in the Standards	Thawing and heating	Ι
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 blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh	Net content of frozen fish blocks covered by glaze	Described in the Standard	Gravimetry	I
Quick frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh	Sodium chloride	AOAC 971.21 (Codex general method)	Potentiometry	II

Fish and Fishery Products				
Quick frozen fish fillets	Net weight of products covered by glaze	Described in the Standard	Water spraying and sieving	I
Quick Frozen Fish sticks (fish fingers) and fish portions - breaded or in batter	Fish content (declaration)	AOAC 996.15 and calculation (described in the standard)	Gravimetry	Ι
Quick frozen fish sticks (fish fingers) and fish portions - breaded or in batter	Net weight	Described in the Standard	Weighing	Ι
Quick Frozen Fish Sticks (fish fingers) and Fish Portions-Breaded and in Batter (except for certain fish species with soft flesh)	Proportion of fish fillet and minced fish	WEFTA Method (described in the Standard)	Gravimetry	I
Quick frozen fish sticks (fish fingers) and fish portions - breaded or in batter	Sodium chloride	AOAC 971.27 (Codex general method)	Potentiometry	II
Salted Atlantic Herring and Salted Sprat	Water content	AOAC 950.46B	air drying	I
Salted Fish of the Gadidae Family	Salt	Described in CXS 167-1989	Titrimetry (Mohr) Salt determined as chloride expressed as sodium chloride	I
Salted Fish and Dried Salted Fish of the <i>Gadidae</i> Family of Fishes	Salt Content Water content	Sampling and method described in the Standard	Gravimetry	I
Smoked Fish, Smoke-Flavoured fish and Smoke-dried fish	Water phase salt	AOAC 952.08 AOAC 937.09 Described in standard <sup>6</sup>	Calculation	I
Smoked Fish, Smoke-Flavoured fish and Smoke-dried fish	Water activity	NMKL 168   ISO 21807	Electrometry	III
Sturgeon Caviar	Salt content	Described in CXS 167-1989	Titrimetry (Mohr) Salt determined as chloride expressed as sodium chloride	I
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

# Method Performance Criteria for histamine for fish and fishery products

<sup>&</sup>lt;sup>6</sup> % salt × 100 / (%water + %salt)

Provision	ML (mg/100 g)	Minimum applicable range (mg/100 g)	LOD (mg/100 g)	LOQ (mg/100 g)	RSD <sub>R</sub> (%)	Recovery	Applicable methods that meet the criteria	Principle
Histamine	10 (average)	8 – 12	1	2	16.0	90 – 107	AOAC 977.13   NMKL 99, NMKL 196,	Fluorometric HPLC
Histamine	20 (each unit)	16 – 24	2	4	14.4	90 – 107	AOAC 977.13   NMKL 99, NMKL 196,	Fluorometric HPLC

### Determination of Biotoxins in live and raw bivalve molluscs

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

#### Criteria for determination of Toxin Analogues by chemical methods

Methods shall meet the numerical criteria listed in Table 1 and may either meet the minimum applicable range, or LOD and LOQ criteria listed.

Toxin Group	Toxin	Minimum	LOD	LOQ	Precision (RSD <sub>R</sub> )	Recovery percent	Applicable methods that meet
		applicable range	(mg/kg)	(mg/kg)	(%)		the criteria
		(mg/kg)			No more than		
STX Group	Saxitoxin	0.05 – 0.2	0.01	0.02	44%	50 – 130	AOAC 2005.06
	(STX)						NMKL 182, EN 14526
	NEO	0.05 – 0.2	0.01	0.02	44%	50 – 130	AOAC 2011.02
	dcSTX	0.05 – 0.2	0.01	0.02	44%	50 – 130	NMKL 197
	GTX1	0.05 – 0.2	0.01	0.02	44%	50 – 130	
	GTX2	0.1 – 0.5	0.03	0.06	38%	50– 130	
	GTX3	0.1 – 0.5	0.03	0.06	38%	50– 130	
	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%	60-115	See reference below
	DTX1	0.03 – 0.2	0.01	0.02	44%	60-115	
	DTX2	0.1 – 0.5	0.03	0.06	38%	60-115	
Domoic Acid	DA	14 – 26	2	4	20%	80-110	
AZA Group	AZA1	0.03 – 0.2	0.01	0.02	44%	40 - 120	See reference below
-	AZA2	0.03 – 0.2	0.01	0.02	44%	40 - 120	
	AZA3	0.03 – 0.2	0.01	0.02	44%	40 - 120	]

**Reference:** http://aesan.msssi.gob.es/en/CRLMB/web/procedimientos\_crlmb/crlmb\_standard\_operating\_procedures.shtml Harmonised-SOP-LCMS-OA-Version4.pdf

Total toxicity is estimated as the sum of the molar concentrations of detected analogs 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 TEF's will be found on the FAO website. Information on TEFs could be incorporated in this standard at a future date.

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 1.

Where toxin analogues that are not listed in Table 1 are determined the competent authority must assess the contribution of these analogs to total toxicity whilst conducting further investigations.

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
All Tuna	methylmercury*	1.2	0.64 – 1.8	0.12	0.24	31	80 – 110	EN 16801	GC-ICP/MS
Alfonsino	methylmercury*	1.5	0.82 – 2.2	0.15	0.30	30	80 – 110	AOAC 988.11 EN 16801	GC-electron capture GC-ICP/MS
All Marlin	methylmercury*	1.7	0.95 – 2.5	0.17	0.34	30	80 – 110	AOAC 988.11 EN 16801	GC-electron capture GC-ICP/MS
Shark	methylmercury*	1.6	0.88 – 2.3	0.16	0.32	30	80-110	AOAC 988.11 EN 16801	GC-electron capture GC-ICP/MS

Performance Criteria for methods of analysis of methylmercury\*

\* 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.

Foods for Special Dietary Uses

AOAC 942.05 Special foods Ash Gravimetrv Special foods Calcium AOAC 984.27 ICP emission spectrometry Method described in CAC/VOL IX-Ed.1, Special foods Calories by calculation Calculation method Part III Ш Special foods Carbohydrates Method described in CAC/VOL IX-Ed.1, Calculation Part III Special foods Chloride AOAC 971.27 (Codex general method) Ш Potentiometry Special foods Dietary fibre, total AOAC 985.29 Gravimetry (enzymatic digestion) Fat CAC/RM 55 Special foods Gravimetry (extraction) Special foods Fat in foods not containing starch, meat or CAC/RM 1, B-2 Gravimetry vegetable products Special foods Fill of containers CAC/RM 46 Weighing Special foods Folic acid AOAC 944.12 Microbioassay Ш Acid hydrolysis, preparation of Special foods Linoleate (in the form of glycerides) AOAC 922.06; 969.33; 963.22 Ш methyl esters and gas chromatography Ш 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 Gravimetry -ISO 6731|IDF 21 Special foods Nicotinamide for foods not based on milk AOAC 961.14 Colorimetry Ш Special foods Nicotinamide for milk-based foods AOAC 944.13 Ш Microbioassay AOAC 945.74 Special foods Pantothenic acid/enriched foods Microbioassay Ш Special foods Pantothenic acid/non-enriched foods The Analyst 89 (1964):1, 3-6, ibid. 232 Microbioassay IV US Dept Agr., Agr. Handbook 97 (1965) Special foods Phosphorous AOAC 986.24 Colorimetry Ш (molybdovanadate)

Follow-up formula

Follow-up formula

Follow-up formula

Dietary fibre, total

Pantothenic acid

lodine (milk based formula)

Foods for Special D	ietary Uses			
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	
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	
Special foods	Vitamin B <sub>12</sub>	AOAC 952.20	Microbioassay	II
Special foods	Vitamin B <sub>6</sub>	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	AOAC 936.14	Rat bioassay	IV
Special foods	Vitamin D (D <sub>3</sub> , milk based infant formula)	AOAC 992.26	Liquid chromatography	11
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	II

AOAC 991.43

AOAC 992.24

AOAC 992.07

Measures total pantothenate

calcium D-pantothenate)

(free pantothenic acid + CoA- + ACP-bound) and measured as D-pantothenic acid (or Ш

Ш

spectrometry

Microbioassay

digestion)

Gravimetry (enzymatic

Ion-selective potentiometry

Foods for Special Dietary Uses

	,			
Follow-up formula	Vitamin A	AOAC 974.29	Colorimetry	IV
Follow-up formula	Vitamin A (retinol isomers)	AOAC 992.04	HPLC	П
Follow-up formula	Vitamin A (retinol) (above 500 IU/I milk after reconstitution)	AOAC 992.06	HPLC	III
Follow-up formula	Vitamin K	AOAC 999.15	HPLC	11
·		EN 14148 (vitamin K1)	with	
		(Measures either aggregated cis + trans K <sub>1</sub> or can measure individual cis and trans forms depending on LC column.)	C30 column to separate the cis- and the trans- K vitamins	
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: 465-474		
Infant formula	Biotin	EN 15607 (d-biotin)	HPLC- fluorescence	
		(Measures total D-biotin (free + D-biocytin)		
Infant formula	Biotin	AOAC 2016.02	HPLC-UV	
Infant formula	Calories (by calculation)	Method described in CAC/Vol IX-Ed.1, Part III <sup>7</sup>	Calculation	Ι

<sup>7</sup> Section 9. Calories by calculation – Section 9.2 Conversion Factors

(a) protein 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 maybe 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

<sup>(</sup>b) carbohydrate 4 kcal per g

Foods for Special D	ietary Uses			
Infant formula	Calcium	ISO 8070   IDF 119	Flame atomic absorption spectrophotometry	II
Infant formula	Calcium	AOAC 985.35	Flame atomic absorption spectroscopy	III
Infant formula	Calcium	AOAC 984.27	ICP emission spectroscopy	III
Infant formula	Chloride	AOAC 986.26	Potentiometry	
Infant formula	Chloride	AOAC 2016.03 / ISO 21422   IDF 242	Potentiometry	П
Infant formula	Choline	AOAC 999.14	Enzymatic Colorimetric Method with limitations on applicability due to choline and ascorbate concentration.	II
Infant formula	Copper	AOAC 985.35	Flame atomic absorption spectroscopy	II
Infant formula	Copper	AOAC 984.27	ICP emission spectroscopy	III
Infant formula	Chromium (Section B of CXS 72-1981 only)	EN 14082	Graphite furnace atomic absorption after dry ashing	III
Infant formula	Chromium (Section B of CXS 72-1981 only)	EN 14083	Graphite furnace AAS after pressure digestion	III
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 <sup>8</sup>	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	I
Infant formula	Fatty acids (including trans fatty acid)	AOAC 996.06	Gas chromatography	III
Infant formula	Fatty acids (including trans fatty acid)	AOCS Ce 1i-07	Gas chromatography	III

#### <sup>8</sup> 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

Foods for Special D	ietary Uses			
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	J AOAC Int. 2000:83; 1141-1148	Optical Biosensor	IV
		(Measures free folic acid + proportion of free, natural folate)	Immunoassay	
Infant formula	Folic acid	J Chromatogr. A., 928, 77-90, 2001 (Measures total folates after conversion to, and measurement as 5-Me-H4PteGlu)	HPLC, incorporating immunoaffinity clean-up and conversion to 5- methyltetrahydrofolate	IV
Infant formula	lodine	AOAC 2012.15   ISO 20647   IDF 234	ICP-MS	II
	(for milk-based formula)			
Infant formula	Iron <sup>9</sup>	AOAC 985.35	Flame atomic absorption spectrophotometry	
Infant formula	Iron	AOAC 984.27	ICP emission spectroscopy	111
Infant formula	Iron	AOAC 999.11 NMKL139	AAS after dry ashing	II
Infant formula	Magnesium	ISO 8070   IDF 119	Flame atomic absorption spectrophotometry	II
Infant formula	Magnesium	AOAC 985.35	Flame atomic absorption spectroscopy	III
Infant formula	Magnesium	AOAC 984.27	ICP emission spectroscopy	
Infant formula	Manganese	AOAC 985.35	Flame atomic absorption spectrophotometry	II
Infant formula	Manganese	AOAC 984.27	ICP emission spectroscopy	
Infant formula	Melamine	ISO/TS 15495   IDF/RM 230	LC-MS/MS	IV

<sup>&</sup>lt;sup>9</sup> General Codex methods are also available

Foods for Special Di	ietary Uses			
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	
	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	П
Infant formula	Niacin	AOAC 985.34 (niacin (preformed) and nicotinamide)	Microbioassay and turbidimetry	
Infant formula	Niacin	EN 15652	HPLC	<b>  </b> <sup>10</sup>
		(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 986.24	Spectrophotometry (molybdovanadate)	II
Infant formula	Phosphorus	AOAC 984.27	ICP emission spectroscopy	III
Infant formula	Riboflavin	AOAC 985.3111	Fluorimetry	
Infant formula	Riboflavin	EN 14152 (Measures natural and supplemental forms, free, bound and phosphorylated (FMN and FAD) aggregated and measured as riboflavin.)	HPLC	II
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	

<sup>10</sup> When published as EN method
<sup>11</sup> Care should be taken in the application of the method due to spectral interference

Foods for Special D	ietary Uses			
	Selenium	AOAC 2011.19   ISO 20649   IDF 235	ICP-MS	II
Infant formula	Sodium and potassium	AOAC 984.27	ICP emission spectrometry	III
Infant formula	Sodium and potassium	ISO 8070   IDF 119	Flame atomic absorption spectrophotometry	II
Infant formula	Thiamine	AOAC 986.27 <sup>12</sup>	Fluorimetry	III
Infant formula	Thiamine	EN 14122 (Measures all vitamin B <sub>1</sub> forms (natural and added free, bound and phosphorylated) following extraction and conversion to thiamine)	HPLC with pre-or post column derivatization to thiochrom	II
Infant formula	Total carbohydrates	AOAC 986.25 AOAC 990.19 or	Determination by difference	I
	Moisture/Total Solids	AOAC 990.20 ISO 6731 IDF 21	Gravimetry	
	Ash	AOAC 942.05	Gravimetry	
Infant formula	Total fat	AOAC 989.05 ISO 8381 IDF 123	Gravimetry (Röse-Gottlieb)	I
Infant formula	Total fat for milk-based infant formula (Products not completely soluble in ammonia)	ISO 8262-1  IDF 124-1	Gravimetry (Weibull-Berntrop)	Ι
Infant formula	Total fatty acids	AOAC 996.06	Gas Chromatography	III
Infant formula	Total fatty acids			
		AOAC 2012.13   ISO 16958   IDF231	Gas Chromatography	II
Infant formula	Total nucleotides	AOAC 2011.20 ISO 20638	LC	II

<sup>&</sup>lt;sup>12</sup> Care should be taken in the application of the method due to spectral interference

Foods for Special Dietary Uses

Infant formula	Total phospholipids	AOCS Ja7b-91	Gas chromatography	
			with suitable extraction and preparation procedures	
Infant formula	Vitamin A	EN 12823-1 (all-trans-retinol and 13-cis- retinol)	HPLC	III
		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 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
Infant formula	Vitamin E	AOAC 992.03	HPLC	
		Measures all rac-vitamin E (both natural + supplemental ester forms) aggregated and quantified as α-congeners		
Infant formula	Vitamin E	EN 12822	HPLC	II
		(Measures Vitamin E (both natural + supplemental ester forms) aggregated and quantified as individual tocopherol congeners $(\alpha, \beta, \gamma, \delta)$ .		

Foods for Special D	ietary Uses			
Infant formula	Vitamin E	AOAC 2012.10   ISO 20633	HPLC	
Infant formula	Vitamin B <sub>6</sub>	AOAC 985.32	Microbioassay	
Infant formula	Vitamin B <sub>6</sub>	EN 14166	Microbioassay	
		(Aggregates free and bound pyridoxal, pyridoxine and pyridoxamine and measures as pyridoxine)		
Infant formula	Vitamin B <sub>6</sub>	AOAC 2004.07		II
		EN 14164	HPLC	
		(Free and bound phosphorylated forms (pyridoxal, pyridoxine and pyridoxamine) converted and measured as pyridoxine)		
Infant formula	Vitamin B <sub>6</sub>	EN 14663 (includes glycosylated forms)	HPLC	
		(Free and bound phosphorylated and glycosylated forms measured as the individual forms pyridoxal, pyridoxine and pyridoxamine)		
Infant formula	Vitamin B <sub>12</sub>	AOAC 986.23	Turbidimetric Method	
		(Measures total vitamin B <sub>12</sub> as cyanocobalamin)		111
Infant formula	Vitamin B <sub>12</sub>	AOAC 2011.10   ISO 20634	HPLC	II
Infant formula	Zinc	AOAC 985.35	Flame atomic absorption spectroscopy	II
Infant formula	Zinc	AOAC 984.27	ICP emission spectroscopy	III

#### Methods of analysis for dietary fibre: Guidelines for Use of Nutrition and Health Claims: Table of Conditions for Claims

Standard	Provisions	Method	Principle	Туре
General meth	ods that do not measure the lower molecular weight fraction (i.e. mo	onomeric units<=9) <sup>(2)</sup>		
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction. (4)	AOAC 985.29 AACC Intl 32-05.01	Enzymatic gravimetry	Type I
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction and also includes determination for soluble and insoluble dietary fibres (4)	AOAC 991.43 AACC Intl 32-07.01 NMKL 129	Enzymatic gravimetry	Туре І
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 the lower molecular weight fraction. Provides sugar residue composition of dietary fibre polysaccharides, as well as content of Klason lignin (4).	AOAC 994.13 AACC Intl 32- 25.01 NMKL 162	Enzymatic GC/ colorimetry gravimetry	Туре І
All foods (1)	Insoluble dietary fibres in food and food products (4)	AOAC 991.42 (Specific for insoluble fibre) AACC Intl 32-20.01	Enzymatic gravimetry	Type I
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 low	ver molecular weight fraction (r	nonomeric units <=9) <sup>(2)</sup>	
All foods (1)	Method applicable for determining the content of dietary fibres of higher and lower molecular weight, in food where resistant starches are not present	AOAC 2001.03 AACC Intl 32-41.01	Enzymatic gravimetry and Liquid chromatography	Туре І
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 starches.	AOAC 2009.01 AACC Intl 32-45.01	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
Methods that	measure individual specific components (monomeric units: the	whole range for each type of c	omponents is covered) <sup>(2)</sup>	•
All foods (1)	$(1\rightarrow 3)(1\rightarrow 4)$ Beta-D-Glucans	AOAC 995.16 AACC Intl 32-23.01	Enzymatic	Type II
All foods (1)	Fructans (oligofructoses, inulin, hydrolyzed inulin, polyfructoses, fructooligosaccharides) (applicable to added fructans)	AOAC 997.08 AACC Intl 32-31.01	Enzymatic & HPAEC- PAD	Type II
All foods (1)	Fructans (oligofructoses, inulin, hydrolyzed inulin, polyfructoses, fructooligosaccharides) (not applicable highly depolymerised fructans)	AOAC 999.03 AACC Intl 32-32.01	Enzymatic & colorimetric	Type III
All foods (1)	Polydextrose	AOAC 2000.11 AACC Intl 32-28.01	HPAEC-PAD	Type II
All foods (1)	Trans-galacto-oligo saccharides	AOAC 2001.02 AACC Intl 32-33.01	HPAEC-PAD	Type II
All foods (1)	Resistant starch (Recommended for RS3)	AOAC 2002.02 AACC Intl 32-40.01	Enzymatic	Type II

Other me	thods <sup>(2)</sup> that have not been subjected to interlaboratory evaluati	on under AOAC international guidelines	S	
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 et al. 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 – Analyst 119, 1497-1509	Gas-Liquid Chromatography	Туре IV

<sup>(1)</sup> 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.

<sup>(2)</sup> 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 for Nutrition Labelling* (CXG 2-1985).

<sup>(3)</sup> Quantitation lost for resistant starch. Refer to specific methods.

<sup>(4)</sup> 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 ISO 6557-2	Indophenol method	III
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 <sup>13</sup> (additives)	AOAC 986.13	HPLC	II
Fruit Juices and Nectars	Citric acid <sup>5</sup> (additives)	EN 1137 IFUMA 22	Enzymatic determination	
Fruit Juices and Nectars	Glucose and fructose (permitted ingredients)	EN 12630 IFUMA 67 NMKL 148	HPLC	III
Fruit Juices and Nectars	Glucose-D and fructose-D (permitted ingredients)	EN 1140 IFUMA 55	Enzymatic determination	II
Fruit Juices and Nectars	HFCS & HIS in apple juice (permitted ingredients)	Determination of HFCS & HIS by Capillary GC method JAOAC 84, 486 (2001)	CAP GC Method	IV
Fruit Juices and Nectars	Malic acid (additives)	AOAC 993.05	Enzymatic determination and HPLC	III
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 IFUMA 21	Enzymatic determination	II
Fruit Juices and Nectars	Pectin (additives)	IFUMA 26	Precipitation/photometry	I
Fruit Juices and Nectars	Benzoic acid and its salts; sorbic acid and its salts	IFUMA 63 NMKL 124	HPLC	II
Fruit Juices and Nectars	Benzoic acid and its salts	ISO 5518, ISO 6560	Spectrometry	
Fruit Juices and Nectars	Preservatives in fruit juices (sorbic acid and its salts)	ISO 5519	Spectrometry	

<sup>13</sup> All juices except citrus based juices

Fruit Juices and Nectars				
Fruit Juices and Nectars	Quinic, malic & 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 ISO 2173	Indirect by refractometry	I
Fruit Juices and Nectars	Sucrose (permitted ingredients)	EN 12146 IFUMA 56	Enzymatic determination	
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 IFUMA 7A NMKL 132	Titrimetry after distillation	II
Fruit Juices and Nectars	Sulphur dioxide (additives)	NMKL 135	Enzymatic determination	III
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
Fruit Juices and Nectars	Sections 3.2 Quality Criteria and 3.3 Authenticity <sup>14</sup>	Determination of acetic acid EN 12632; IFUMA 66	Enzymatic determination	11
Fruit Juices and Nectars		Determination of alcohol (ethanol) IFUMA 52	Enzymatic determination	II
Fruit Juices and Nectars		Detection of anthocyanins IFUMA 71	HPLC	Ι
Fruit Juices and Nectars		Determination of ash in fruit products AOAC 940.26; EN 1135; IFUMA 9	Gravimetry	I
Fruit Juices and Nectars		Detection of beet sugar in fruit juices AOAC 995.17	Deuterium NMR	II

#### <sup>14</sup> 3.4 Verification of Composition, Quality and Authenticity

Fruit juices and nectars should be subject to testing for authenticity, composition, and quality where applicable and where required. The analytical methods used should be those found in Section 9, Methods of Analysis and Sampling.

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			
Fruit Juices and Nectars	Determination of benzoic acid as a marker in orange juice AOAC 994.11	HPLC	
Fruit Juices and Nectars	Determination of C <sup>13</sup> /C <sup>12</sup> ratio of ethanol derived from fruit juices JAOAC 79, No. 1, 1996, 62-72	Stable isotope mass spectrometry	II
Fruit Juices and Nectars	Determination of carbon stable isotope ratio of apple juice AOAC 981.09 - JAOAC 64, 85 (1981)	Stable isotope mass spectrometry	II
Fruit Juices and Nectars	Determination of carbon stable isotope ratio of orange juice AOAC 982.21	Stable isotope mass spectrometry	II
Fruit Juices and Nectars	Determination of carotenoid, total/individual groups EN 12136; IFUMA 59	Spectrophotometry	I
Fruit Juices and Nectars	Determination of centrifugable pulp EN 12134; IFUMA 60	Centrifugation/% value	I
Fruit Juices and Nectars	Determination of chloride (expressed as sodium chloride) EN 12133 IFUMA 37	Electrochemical titrimetry	III
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*	(Scott) distillation, titration	I
Fruit Juices and Nectars	Determination of essential oils (in citrus fruit) (volume determination)* 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		
Fruit Juices and Nectars	Determination of glucose fructose and HPLC saccharose EN 12630 IFUMA 67 NMKL 148	II
Fruit Juices and Nectars	Determination of gluconic acid Enzymatic determination IFUMA 76	II
Fruit Juices and Nectars	Determination of glycerol Enzymatic determination IFUMA 77	II
Fruit Juices and Nectars	Determination of hesperidin and naringin HPLC EN 12148 IFUMA 58	II
Fruit Juices and Nectars	Determination of hydroxymethylfurfural HPLC IFUMA 69	II
Fruit Juices and Nectars	Determination of hydroxymethylfurfural Spectrometry ISO 7466	
Fruit Juices and Nectars	Determination of isocitric acid-D Enzymatic determination IFUMA 54	II
Fruit Juices and Nectars	Determination of Lactic acid- D and L Enzymatic determination EN 12631 IFUMA 53	II
Fruit Juices and Nectars	Determination of L-malic/total malic acid ratio Enzymatic determination and HPLC in apple juice AOAC 993.05	II
Fruit Juices and Nectars	Determination of naringin and neohesperidin in HPLC orange juice AOAC 999.05	111
Fruit Juices and Nectars	Determination of pH-value Potentiometry NMKL 179 EN 1132 IFUMA 11 ISO 1842	II IV
Fruit Juices and Nectars	Determination of phosphorus/phosphate Photometric determination EN 1136 IFUMA No 50	II
Fruit Juices and Nectars	Determination of proline by photometry – non- Photometry specific determination EN 1141 IFUMA 49	I
Fruit Juices and Nectars	Determination of relative density Pycnometry EN 1131 (1993); IFUMA 01 & IFU Method No General sheet (1971)	II
Fruit Juices and Nectars	Determination of Relative density Densitometry IFUMA 01A	

Fruit Juices and Nectars			
Fruit Juices and Nectars	Determination of sodium, potassium, calcium, magnesium in fruit juices EN 1134 IFUMA 33	Atomic Absorption Spectroscopy	II
Fruit Juices and Nectars	Determination of sorbitol-D IFUMA62	Enzymatic determination	II
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	I
Fruit Juices and Nectars	Determination of sugar beet derived syrups in frozen concentrated orange juice $\delta^{18}$ O Measurements in Water AOAC 992.09	Oxygen isotope ratio analysis	Ι
Fruit Juices and Nectars	Determination of titrable acids, total EN 12147 IFUMA 03 ISO 750	Titrimetry	Ι
Fruit Juices and Nectars	Determination of total dry matter (vacuum- oven drying at 70°C)* EN 12145 IFUMA 61	Gravimetric determination	I
Fruit Juices and Nectars	Determination of total solids (Microwave oven drying)* AOAC 985.26	Gravimetric determination	Ι
Fruit Juices and Nectars	Determination of Vitamin C (dehydro-ascorbic acid and ascorbic acid) AOAC 967.22	Microfluorometry	

\* Because there is no numerical value in the Standard duplicate Type I methods have been included which may lead to different results.

Milk and Milk Products				
Milk products	Iron	NMKL 139 AOAC 999.11 (Codex general method)	Atomic absorption spectrophotometry	II
Milk products	Iron	NMKL 161 / AOAC 999.10	Atomic absorption spectrophotometry	
Milk products	Iron	AOAC 984.27	Inductively Coupled Plasma optical emission spectrophotometry	III
Milk products	Iron	ISO 6732   IDF 103	Photometry (bathophenanthroline)	IV
Milk and Milk Products	Melamine	ISO/TS 15495   IDF/RM 230	LC-MS/MS	IV
Milk products (products not completely soluble in ammonia)	Milk fat	ISO 8262-3   IDF 124-3	Gravimetry (Weibull-Berntrop)	Ι
Blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 1737   IDF 13	Gravimetry (Röse-Gottlieb)	Ι
Blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat <sup>15</sup> (MSNF)	ISO 6731   IDF 21 and ISO 1737  IDF 13	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)	Ι
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF <sup>15</sup>	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	IV
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF <sup>15</sup>	AOAC 991/20	Titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 1737  IDF 13	Gravimetry (Röse-Gottlieb)	Ι
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat (MSNF) <sup>15</sup>	ISO 6731   IDF 21 and ISO 1737   IDF 13	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)	Ι
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF <sup>15</sup>	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF <sup>15</sup>	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Total fat	ISO 1736   IDF 9	Gravimetry (Röse-Gottlieb)	

<sup>&</sup>lt;sup>15</sup> Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose

Milk and Milk Products				
Blend of skimmed milk and vegetable fat in powdered form	Water <sup>16</sup>	ISO 5537   IDF 26	Gravimetry, drying at 87 °C	I
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF <sup>15</sup>	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF <sup>15</sup>	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Total fat	ISO 1736   IDF 9	Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water <sup>16</sup>	ISO 5537   IDF 26	Gravimetry, drying at 87 °C	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF <sup>15</sup>	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF <sup>15</sup>	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 1737   IDF 13	Gravimetry (Röse-Gottlieb)	I
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	Milk solids-not-fat <sup>15</sup> (MSNF)	ISO 6734   IDF 15	Calculation from total solids content, fat content and sugar content	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF <sup>15</sup>	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF <sup>15</sup>	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 1737   IDF 13	Gravimetry (Röse-Gottlieb)	Ι
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk solids-not-fat <sup>15</sup> (MSNF)	ISO 6734   IDF 15	Calculation from total solids content and sugar content	IV

<sup>&</sup>lt;sup>16</sup> Water content excluding the crystallized water bound to lactose (generally known as "moisture content")

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Milk and Milk Products				
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF <sup>15</sup>	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF <sup>15</sup>	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Butter	Copper	ISO 5738   IDF 76 AOAC 960.40	Photometry, diethyldithiocarbamate	II
Butter	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Butter	Milk solids-not-fat <sup>15</sup> (MSNF)	ISO 3727-2   IDF 80-2	Gravimetry	I
Butter	Milkfat	ISO 17189   IDF 194	Gravimetry Direct determination of fat using solvent extraction	Ι
Butter	Milk fat purity	ISO 17678   IDF 202	Calculation from determination of triglycerides by gas chromatography	I
Butter	Salt	ISO 1738   IDF 12/ AOAC 960.29	Titrimetry (Mohr: determination of chloride, expressed as sodium chloride)	111
Butter	Salt	ISO 15648   IDF 179	Potentiometry (determination of chloride, expressed as sodium chloride)	II
Butter	Vegetable fat (sterols)	ISO 12078   IDF 159	Gas chromatography	П
Butter	Vegetable fat (sterols)	ISO 18252   IDF 200	Gas chromatography	111
Butter	Water <sup>16</sup>	ISO 37271-1   IDF 80-1	Gravimetry	I
Cheese	Citric acid	ISO/TS 2963   IDF/RM 34	Enzymatic method	IV
Cheese	Citric acid	AOAC 976.15	Photometry	П
Cheese	Milkfat	ISO 1735   IDF 5	Gravimetry (Schmid-Bondzynski- Ratslaff)	Ι
Cheese	Moisture	ISO 5534   IDF 4	Gravimetry, drying at 102 °C	I

Milk and Milk Products				
Cheese (and cheese rind)	Natamycin	ISO 9233-1   IDF 140-1	Molecular absorption spectrophotometry	
		ISO 9233-2   IDF 140-2	HPLC	II
Cheese	Propionic acid	ISO/TS 19046-1I 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)	II
Cheeses, individual	Dry matter (Total solids) <sup>15</sup>	ISO 5534   IDF 4	Gravimetry, drying at 102°C	Ι
Cheeses, individual	Milk fat in dry matter	ISO 1735   IDF 5	Gravimetry (Schmid-Bondzynski- Ratzlaff)	I
Cheeses in brine	Milk fat in dry matter (FDM)	ISO 1735   IDF 5	Gravimetry (Schmid-Bondzynski- Ratzlaff)	Ι
Cottage cheese	Fat-free dry matter	ISO 5534   IDF 4 and ISO 1735   IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102 °C Gravimetry (Schmid-Bondzynski- Ratzlaff)	I
Cottage cheese	Milk fat	ISO 1735   IDF 5	Gravimetry (Schmid-Bondzynski- Ratzlaff) (for samples containing lactose up to 5%)	I
		ISO 8262-3   IDF 124-3	Gravimetry (Weibull-Berntrop) (for samples containing lactose over 5%)	I
Cottage cheese	Milk fat in dry matter	ISO 8262-3   IDF 124-3	Gravimetry (Weibull-Berntrop)	
Cheese, Unripened Including Fresh Cheese	Milk Protein	ISO 8968-1   IDF 20-1	Titrimetry, Kjeldahl	I
Cream and Prepared Creams	Milk protein	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	I
Cream	Milkfat	ISO 2450   IDF 16	Gravimetry (Röse-Gottlieb)	Ι
Cream	Solids <sup>15</sup>	ISO 6731   IDF 21	Gravimetry (drying at 102°C)	I
Creams Lowered in Milkfat Content	Milkfat	ISO 2450   IDF 16 / AOAC 995.19	Gravimetry (Röse-Gottlieb)	
Creams, Whipped Creams and Fermented Creams	Milk solids-not-fat (MSNF) <sup>15</sup>	ISO 3727-2   IDF 80-2 AOAC 920.116	Gravimetry	Ι

Milk and Milk Products

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Cream cheese	Dry matter	ISO 5534   IDF 4	Gravimetry drying at 102 °C (forced air oven)	I
Cream cheese	Moisture on fat free basis	ISO 5534   IDF 4	Calculation from fat content and moisture content	I
		ISO 1735   IDF 5	Gravimetry drying at 102°C (forced air oven) Gravimetry (Schmid-Bondzynski-	
Dairy fat spreads	Milk fat purity	ISO 17678   IDF 202	Ratzlaff) Calculation from determination of triglycerides by gas chromatography	I
Dairy fat spreads	Total fat ISO 17189   IDF 194 Gravimetry Direct determination of fat us			Ι
Dairy fat spreads	Vegetable fat (sterols)	ISO 12078   IDF 159	Gas chromatography	
Dairy fat spreads	Vegetable fat (sterols)	ISO 18252   IDF 200	Gas chromatography	
Dairy permeate powders	Milkfat	ISO 1736   IDF 9	Gravimetry (Röse-Gottlieb)	I
Dairy permeate powders	Nitrogen	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	Ι
Dairy permeate powders	Moisture <sup>17</sup>	ISO 5537   IDF 26	Gravimetry (drying at 87°C)	Ι
Dairy permeate powders	Ash	NMKL 173	Gravimetry (ashing at 550 °C)	IV
Edible casein products	Acids, free	ISO 5547   IDF 91	Titrimetry (aqueous extract)	IV
Edible casein products	Ash (including $P_2O_5$ )	ISO 5545   IDF 90 or <sup>18</sup> ISO 5544   IDF 89	Gravimetry (ashing at 825 °C)	I
Edible casein products	Copper	AOAC 985.35	Atomic absorption spectrophotometry	II
Edible casein products	Copper	ISO 5738   IDF 76	Colorimetry (diethyldiethiocarbamate)	Ш
Edible casein products	Lactose	ISO 5548   IDF 106	Photometry (phenol and H <sub>2</sub> SO <sub>4</sub> )	IV

 <sup>&</sup>lt;sup>17</sup> Moisture content excluding the water of crystallization of lactose
<sup>18</sup> Refer to scope of methods

Milk and Milk Products

Edible casein products	Lead	NMKL 139 (Codex general method) AOAC 999.11	Atomic absorption spectrophotometry	II
Edible casein products	Lead	NMKL 161 / AOAC 999.10	Atomic absorption spectrophotometry	
Edible casein products	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	
Edible casein products	Lead	AOAC 982.23 (Codex general method)	Anodic stripping voltanmetry	
Edible casein products	Lead	ISO/TS 6733   IDF/RM 133	Spectrophotometry (1,5- diphenylthiocarbazone)	IV
Edible casein products	Milkfat	ISO 5543   IDF 127	Gravimetry (Schmid-Bondzynski- Ratslaff)	Ι
Edible casein products	рН	ISO 5546   IDF 115	Electrometry	IV
Edible casein products	Milk Protein (total N x 6.38 in dry matter)	ISO 8968-1   IDF 20-1	Titrimetry, Kjeldahl	Ι
Edible casein products	Sediment (scorched particles)	ISO 5739   IDF 107	Visual comparison with standard disks, after filtration	IV
Edible casein products	Water <sup>16</sup>	ISO 5550   IDF 78	Gravimetry (drying at 102 °C)	I
Emmental	Calcium >= 800mg/100g	ISO 8070   IDF 119	Flame atomic absorption	IV
Emmental	Propionic acid	ISO/TS 19046-11 IDF/RM 233-1	Gas Chromatography -FID	IV
Emmental	Propionic acid	ISO/TS 19046-21 IDF/RM 233-2	Ion exchange chromatography - UV	IV
Evaporated milks	Milk fat	ISO 1737   IDF 13	Gravimetry (Röse-Gottlieb)	Ι
Evaporated milks	Milk Protein in MSNF <sup>15</sup>	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	Ι
Evaporated milks	Solids, total <sup>15</sup>	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) <sup>15</sup>	ISO 13580   IDF 151	Gravimetry (drying at 102 °C)	Ι
	,	·		

# Milk and Milk Products

Fermented milks	Total acidity expressed as percentage of lactic acid	ISO/TS 11869   IDF/RM 150	Potentiometry, titration to pH 8.30	I
Fermented milks	Lactobacillus acidophilus	ISO 20128   IDF 192	Colony count at 37 °C	I
Fermented milks - Yoghurt and yoghurt products	Lactobacillus delbrueckii subsp bulgaricus & Streptococcus thermophilus	ISO 7889   IDF 117	Colony count at 37°C	Ι
Fermented milks - Yoghurt and yoghurt products	Lactobacillus delbrueckii subsp bulgaricus & Streptococcus thermophilus	ISO 9232   IDF 146	Test for strain identification	I
Fermented milks	Microorganisms constituting the 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	IV
Fermented milks	Milk fat	ISO 1211   IDF 1 / AOAC 989.05	Gravimetry (Röse-Gottlieb)	I
Fermented milks Milk powders and cream powders	Milk Protein Acidity, titratable	ISO 8968-1   IDF 20-1 ISO 6091   IDF 86	Titrimetry (Kjeldahl) Titrimetry, titration to pH 8.4	
Milk powders and cream powders	Milk fat	ISO 1736   IDF 9	Gravimetry (Röse-Gottlieb)	İ
Milk powders and cream powders	Milk Protein	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	
Milk powders and cream powders	Scorched particles	ISO 5739   IDF 107	Visual comparison with standard disks, after filtration	IV
Milk powders and cream powders	Solubility Index	ISO 8156   IDF 129	Centrifugation	
Milk powders and cream powders	Water <sup>16</sup>	ISO 5537   IDF 26	Gravimetry (drying at 87°C)	
Milk fat Products	Copper	ISO 5738   IDF 76 AOAC 960.40	Photometry, diethyldithiocarbamate	II
Milk fat products	Fatty acids, free (expressed as oleic acid)	ISO 1740   IDF 6	Titrimetry	I
Milk fat products	Milk fat purity	ISO 17678   IDF 202	Calculation from determination of triglycerides by gas chromatography	I
Milk fat Products (anhydrous milkfat)	Peroxide value (expressed as meq. of oxygen/kg fat)	ISO 3976   IDF 74	Photometry	I
Milkfat products (anhydrous milkfat)	Peroxide value	AOAC 965.33	Titrimetry	I
Milk fat products	Vegetable fat (sterols)	ISO 12078   IDF 159	Gas chromatography	
		ISO 18252   IDF 200	Gas chromatography	
Milk fat products	Water <sup>16</sup>	ISO 5536   IDF 23	Titrimetry (Karl Fischer)	II

Milk and Milk Products				
Mozzarella	Milkfat in dry matter – with high moisture	ISO 1735   IDF 5	Gravimetry after solvent extraction	I
Mozzarella	Milkfat in dry matter – with low moisture	ISO 1735   IDF 5	Gravimetry after solvent extraction	I
Sweetened condensed milk	Milkfat	ISO 1737   IDF 13	Gravimetry (Röse-Gottlieb)	Ι
Sweetened Condensed Milks	Milk Protein in MNSF <sup>15</sup>	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	I
Sweetened Condensed Milks	Solids <sup>15</sup>	ISO 6734   IDF 15	Gravimetry, drying at 102 °C	I
Whey cheeses by coagulation	Milk fat	ISO 1735   IDF 5	Gravimetry (Schmid-Bondzynski- Ratzlaff)	I
Whey cheeses by coagulation	Milk fat in dry matter	ISO 1735   IDF 5and	Calculation from fat content and dry	I
		ISO 5534   IDF 4	matter content	
			Gravimetry (Schmid-Bondzynski-Ratzlaff	
			Gravimetry, drying at 102°C	
Whey cheeses by concentration	Milk fat	ISO 1854   IDF 59	Gravimetry (Röse Gottlieb)	Ι
Whey cheeses by concentration	Milk fat in dry matter	ISO 1854   IDF 59 and	Calculation from fat content and dry	I
	-	ISO 2920   IDF 58	matter content	
			Gravimetry (Röse Gottlieb)	
			Gravimetry, drying at 88 C	
Whey powders	Ash	ISO 5545   IDF 90	Gravimetry (ashing at 825°C)	IV
Whey powders	Copper	AOAC 985.35	Atomic absorption spectrophotometry	П
Whey powders	Copper	ISO 5738   IDF 76	Photometry (diethyldithiocarbamate)	
Whey Powders	Lactose	ISO 5765-1/2   IDF 79-1/2	Enzymatic method: Part 1 - Glucose moiety or Part 2 - Galactose moiety	II
Whey powders	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	П
Whey powders	Milkfat	ISO 1736   IDF 9	Gravimetry (Röse-Gottlieb)	I
Whey powders	Milk protein (total N x 6.38)	ISO 8968-1   IDF 20-1	Titrimetry (Kjeldahl)	I
Whey powders	Moisture, "Free"	ISO 2920   IDF 58	Gravimetry (drying at 88°C ±2°C)	IV
Whey powders	Water <sup>16</sup>	ISO 5537   IDF 26	Gravimetry (drying at 87°C)	

Natural Mineral Waters

ISO 7980 Atomic absorption Natural mineral waters Calcium Ш spectrophotometry Examination of Water Pollution Control. Ш Natural mineral waters Chloride WHO Pergamon Press (1982) Vol. 2, pp. 205-208 Titrimetry (Mercuric nitrate) Ш Chloride AOAC 973.51 Natural mineral waters Natural mineral waters Chloride ISO 9297 Titrimetry Ш ISO 6332 Spectrophotometry Natural mineral waters Iron, dissolved Ш Natural mineral waters Magnesium ISO 6059 Titrimetry 11 Magnesium Natural mineral waters ISO 7980 Atomic absorption spectrophotometry ISO 6439 Spectrophotometry Natural mineral waters Phenols Examination of Water Pollution Control. Ш Natural mineral waters Potassium WHO Pergamon Press (1982) Vol.2, pp. 142-145 Examination of Water Pollution Control. Ш Natural mineral waters Sodium WHO Pergamon Press (1982) Vol.2 pp. 148-151 Examination of Water Pollution Control. Natural mineral waters Sodium Ш WHO Pergamon Press (1982) Vol.2, pp. 151-152 Sulphates ISO 9280 Ш Natural mineral waters Gravimetry Natural mineral waters Sulphide Handb. Spurenanal. 1974 IV

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
Antimony	0.005	0.0028	0.001	0.002	44	80-110	ISO 17294-2 ISO 15586 EPA 200.8	ICP-MS GF-AAS ICP-MS
Arsenic	0.01	0.0056	0.002	0.004	44	90-107	ISO 17294-2 ISO 15586 ISO 11969 EPA 200.8	ICP-MS GF-AAS AAS (Hydride) ICP-MS
Barium	0.7	0.35	0.07	0.14	34	95-105	ISO 11885 ISO 17294-2 EPA 200.8	ICP-OES ICP-MS ICP-MS
Borate	5	3.1	0.5	1	25	97-103	ISO 9390 ISO 11885 ISO 17294-2	Spectrophotometry ICP-OES <sup>19</sup> ICP-MS <sup>19</sup>
Cadmium	0.003	0.0017	0.0006	0.0012	44	80-110	ISO 11885 ISO 17294-2 ISO 15586 ISO 5961 (Section 3) EPA 200.8	ICP-OES ICP-MS GF-AAS AAS ICP-MS
Chromium	0.05	0.028	0.01	0.02	44	90-107	ISO 11885 ISO 17294-2 ISO 15586ISO 18412 (Cr VI) ISO 23913 (Cr VI) ISO 9174 (Section 4) EPA 200.8	ICP-OES ICP-MS GF-AAS Photometric CIA, spectrophotometry AAS ICP-MS
Copper	1	0.52	0.1	0.2	32	97-103	ISO 11885 ISO 17294-2 ISO 15586 ISO 8288 EPA 200.8	ICP-OES ICP-MS GF-AAS Flame-AAS ICP-MS
Cyanide	0.07	0.039	0.014	0.028	44	90-107	ISO 14403ISO 6703-1	CFA Photometric, trimetric
Fluoride	1.0	0.52	0.1	0.2	32	97-103	ISO 10304-1 ISO 10359-1 (dissolved fluoride) ISO 10359-2 (inorganic bound)	LC of ions Electrochemical probe Digestion, distillation

## Criteria applicable to health-related substances in the *Standard for Natural Mineral Waters* (CXS 108-1981)

<sup>19</sup> 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
Lead	0.01	0.0056	0.002	0.004	44	90-107	ISO 17294-2 ISO 15586 EPA 200.8	ICP-MS GF-AAS ICP-MS
Manganese	0.4	0.18	0.04	0.08	37	95-105	ISO 11885I SO 17294-2 ISO 15586 EPA 200.8	ICP-OES ICP-MS GF-AAS ICP-MS
Mercury	0.001	0.00056	0.0002	0.0004	44	80-110	EN 1483 ISO 17852 ISO 5666 ISO 16590 EPA 200.8	AAS Enrichment by amalgamation (III) AFS AAS after tin(II) chloride reduction Enrichment by amalgamation (III) ICP-MS
Nickel	0.02	0.011	0.004	0.008	44	90-107	ISO 17294-2 ISO 15586 EPA 200.8	ICP-MS GF-AAS ICP-MS
Nitrate	50	37	5	10	18	98-102	ISO 10304-1 ISO 13395 ISO 7890-3	LC of ions CFA, FIA, Spectrophotometry Spectrophotometry
Nitrite	0.1	0.03	0.01	0.02	44	95-105	ISO 10304-1 ISO 13395 ISO 6777	LC of ions UV CFA, FIA, Spectrophotometry Spectrophotometry
Selenium	0.01	0.0056	0.002	0.004	44	90-107	ISO 17294-2 ISO 15586 ISO 9965 EPA 200.8	ICP-MS GF-AAS AAS (Hydride) ICP-MS

# 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
РАН	-	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	Benzoic acid	NMKL 124	Liquid Chromatography	П
Processed fruits and vegetables	Benzoic acid	NMKL 103; or AOAC 983.16	Gas Chromatography	111
Processed fruits and vegetables	Calcium	AOAC 968.31	Complexometry/ Titrimetry	II
Processed fruits and vegetables	Drained Weight	AOAC 968.30 (Codex General Method)	Sieving Gravimetry	Ι
Processed fruits and vegetables	Fill of containers	CAC/RM 46 (reference to "metal containers" deleted and refer to ISO 90-1 for determination of water capacity in metal containers)	Weighing	I
Processed fruits and vegetables	Lead	AOAC 972.25 (Codex general method)	AAS (Flame absorption)	111
Processed fruits and vegetables	Packing medium Canned berry fruits (raspberry, strawberry)	AOAC 932.12 ISO 2173	Refractometry	I
Processed fruits and Vegetables (except canned bamboo shoots, pH determined by AOAC 981.12)	рН	ISO 1842	Potentiometry	IV

Processed Fruits and Vegetables

Commodity	Provision	Method	Principle	Туре
Processed fruits and vegetables	рН	AOAC 981.12	Potentiometry	
Processed fruits and vegetables	рН	NMKL 179	Potentiometry	II
Processed fruits and vegetables	Soluble solids	ISO 2173 AOAC 932.12	Refractometry	I
Processed fruits and vegetables	Sorbates	NMKL 103 / AOAC 983.16	Gas Chromatography	III
Processed fruits and vegetables	Sorbates	NMKL 124	Liquid Chromatography	II
Processed fruits and vegetables	Tin	AOAC 980.19 (Codex general method)	AAS	II
Processed fruits and vegetables	Total solids	AOAC 920.151	Gravimetry	Ι
Aqueous Coconut Products	Total Fats	ISO 1211   IDF 1	Gravimetry (Röse-Gottlieb)	I
Aqueous Coconut Products	Total solids	ISO 6731   IDF 21	Gravimetry	I
Aqueous Coconut Products	Non-fat solids	ISO 1211   IDF 1ISO 6731   IDF 21	Calculation: Gravimetry (Röse-Gottlieb) Gravimetry	I
Aqueous Coconut Products	Moisture	ISO 6731   IDF 21	Calculation: Gravimetry	Ι
Canned Apple Sauce	Fill of containers	CAC/RM 46 <sup>*</sup> (for glass containers) (Codex general method for processed fruits and vegetables) and ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)	Weighing	Ι
Canned Apple Sauce	Soluble solids	AOAC 932.12 ISO 2173 (Codex general method for processed fruits and vegetables)	Refractometry	I
Canned green beans and wax beans	Tough strings	CAC/RM 39	Stretching	I

Processed Fruits and Vegetables

Commodity	Provision	Method	Principle	Туре
Canned green peas	Proper fill (in lieu of drained weight)	CAC/RM 45	Pouring and measuring	Ι
Canned green peas	Types of peas, distinguishing	CAC/RM 48	Visual inspection	Ι
Canned mangoes	Syrup	AOAC 932.14C	Brix spindle method	Ι
Canned mushrooms	Washed drained weight	CAC/RM 44	Sieving	I
Canned palmito	Mineral impurities	ISO 762	Gravimetry	Ι
Canned Stone Fruits	Drained weight	AOAC 968.30 ISO:2173	Gravimetry	Ι
Canned Stone Fruits	Soluble solids	AOAC 932.14C	Refractometry	I
Canned strawberries	Calcium	AOAC 968.31	Complexometric titrimetry	II
Canned strawberries	Mineral impurities	AOAC 971.33	Gravimetry	Ι
Certain canned citrus fruits	Calcium	NMKL 153	Atomic Absorption Spectrophotometry	11
Certain canned citrus fruits	Calcium	AOAC 968.31	Complexometry Titrimetry	III
Certain Canned Vegetables (palmito)	Mineral impurities (sand)	AOAC 971.33	Gravimetry	Ι
Citrus marmalade	Calcium	ISO 762 AOAC 968.31	Complexometric titrimetry	
Dates	Identification of defects	Described in the Standard	Visual inspection	
Dates	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	
Desiccated coconut	Total acidity of the extracted oil	ISO 660 or AOCS Cd 3d-63	Titrimetry	
Desiccated coconut	Ash	AOAC 950.49	Gravimetry	I
Desiccated coconut	Extraneous vegetable matter	Described in the Standard	Counting extraneous material with the naked eye	IV
Desiccated coconut	Moisture	AOAC 925.40	Gravimery (loss on drying)	Ι

**Processed Fruits and Vegetables** 

Processed tomato concentrates

#### Principle Provision Method Commoditv Туре Desiccated coconut Oil content AOAC 948.22 Gravimetry Visual inspection (weighing) Identification of defects Described in the Standard Dried apricots AOAC 934.06 Dried apricots Moisture Gravimetry (vacuum oven) Т Dried apricots Sulphur dioxide AOAC 963.20 Colorimetry Ш CAC/RM 46 Jams (fruit preserves) and jellies Fill of Containers Weighing Jams (fruit preserves) and jellies **ISO 2173** Refractometry Soluble solids AOAC 932.12 Mango chutney Ash insoluble in HCI ISO 763 Gravimetry Pickled cucumbers Acidity, total AOAC 942.15 Titrimetry AOAC 968.30 **Pickled cucumbers** Drained weight Gravimetry **Pickled cucumbers** Mineral impurities AOAC 971.33 Gravimetry AOAC 971.27 (Codex general method) Pickled cucumbers Salt in brine Potentiometry Ш **Pickled cucumbers** Volume fill by displacement Described in the Standard Displacement Т Preserved tomatoes Calcium AOAC 968.31 Complexometric titrimetry Ш Atomic Absorption Preserved tomatoes Calcium **NMKL 153** Ш Spectrophotometry Minimum Drained Weight Gravimetry (sieving) AOAC 968.30 Preserved tomatoes note: Use a No. 14 screen instead of '7/16' or No. 8 AOAC 965.41 Preserved tomatoes Mould count Howard mould count Т Enzymatic Ш Processed tomato concentrates Lactic acid EN 2631 determination Processed tomato concentrates Mineral impurities (sand) AOAC 971.33 IV Gravimetry Mould count AOAC 965.41 Howard mould count Processed tomato concentrates

AOAC 970.59

Refractometry

Natural tomato soluble solids

Т

Processed Fruits and Vegetables				
Commodity	Provision	Method	Principle	Туре
Processed tomato concentrates	Sodium chloride	AOAC 971.27 (Codex general method)	Potentiometry	11
Processed tomato concentrates	Tomato soluble solids	AOAC 970.59	Refractometry	I
Raisins	Mineral impurities	CAC/RM 51	Ashing	I
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	11
Raisins	Sulphur dioxide	AOAC 963.20	Colorimetry	II
Table olives	Drained weight	AOAC 968.30 (Codex general method for processed fruits and vegetables)	Sieving Gravimetry	I
Table olives	Fill of containers	CAC/RM 46 <sup>*</sup> (for glass containers) (Codex general method for processed fruits and vegetables) and ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)	Weighing	I
Table olives	pH of brine	NMKL 179 (Codex general method for processed fruits and vegetables)	Potentiometry	II
		AOAC 981.12 (Codex general method for processed fruits and vegetables) ISO 1842	-	
Table olives	Salt in brine	AOAC 971.27   NMKL 178 (Codex general method)	Potentiometry	IV II
Table olives	Lead	AOAC 999.11   NMKL 139 (Codex general method)	AAS (Flame absorption)	II
Table olives	Tin	NMKL 190   EN 15764	AAS	

# \* 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 3.2 from the weight found in 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.

Products	Provisions	Method	Principle	Туре
Aqueous coconut products	Total Fats	ISO 1211   IDF 1	Gravimetry (Röse- Gottlieb)	I
Aqueous coconut products	Totals Solids	ISO 6731   IDF 21	Gravimetry	I
Aqueous coconut products	Non-fat solids	ISO 1211   IDF 1 ISO 6731   IDF 21	Calculation: Gravimetry (Röse- Gottlieb) Gravimetry	Ι
Aqueous coconut products	Moisture	ISO 6731   IDF 21	Gravimetry	I

# Processed Meat and Poultry Products and Soups and Broths

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	
Meat Products	Nitrates and/or Nitrites	EN 12014-4 NMKL 165	Ion-exchange chromatographic method	III
Processed meat and poultry products	Fat	ISO 1443	Gravimetry	I
Processed meat and poultry products	Lead	AOAC 934.07	Colorimetry (dithizone)	П
Processed meat and poultry products	Nitrates	ISO 3091	Colorimetry (cadmium reduction)	Ш
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	П
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
Canned corned beef	Nitrites, potassium and/or sodium salt	ISO 2918	Colorimetry	IV

Processed Meat and Poultry Prod	lucts and Soups and Broth	s		
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	11
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	I
Cooked cured pork shoulder	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured pork shoulder	Nitrites	AOAC 973.31 (Codex general method)	Colorimetry	
Cooked cured pork shoulder	Nitrites	ISO 2918	Colorimetry	IV
Cooked cured pork shoulder	Protein	ISO 937	Titrimetry, Kjeldahl digestion	11

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Processed Meat and Poultry Produc	cts and Soups and Broth	\$		
Cooked cured pork shoulder	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Luncheon meat	Fat	ISO 1443	Gravimetry (extraction)	Ι
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	11
Quick Frozen Fruits and Vegetable	s			
Quick frozen fruits and vegetables(non-glazed)	Net weight	AOAC 963.26	Weighing	Ι
Quick frozen fruits and vegetables	Thawing procedure	see Appendix I	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
Quick frozen fruits and vegetables: Peaches and berries	Drained fruit/drained berries	AOAC 953.15	Draining	I
Quick frozen fruits and vegetables: Vegetables	Cooking procedure	see Appendix II	Cooking	Ι
Quick frozen French fried potatoes	Moisture	AOAC 984.25	Gravimetry (convection oven)	Ι
Quick frozen green and wax beans	Tough strings	see Appendix III	Stretching	Ι

Quick Frozen Fruits and Veget	tables			
Quick frozen peas	Solids, alcohol insoluble	see Appendix IV	Gravimetry	Ι
Quick frozen spinach	Dry matter, Sodium chloride-free	See Appendix V	Weighing	ļ
Spices and Culinary Herbs				
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	Ι
Cumin	Extraneous vegetable matte	er ISO 927	Visual examination/ Gravimetry	I
Cumin	Foreign matter	ISO 927	Visual examination/Gravimetry	Ι
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

Spices and Culinary Herb	DS			
Cumin	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		
Thyme	Moisture	ISO 939	Distillation	Ι
Thyme	Total ash	ISO 928	Gravimetry	I
Thyme	Acid-insoluble ash	ISO 930	Gravimetry	Ι
Thyme	Volatile oils	ISO 6571	Distillation/Volumetric	Ι
Thyme	Extraneous vegetable matter	ISO 927	Visual examination /Gravimetry	Ι
Thyme	Foreign matter	ISO 927	Visual examination /Gravimetry	Ι
Thyme	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		
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

Spices and Culinary Herbs				
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		
Black and white pepper	Bulk density	ISO 959-1 Annex B (black)	Gravimetry	IV
		ISO 959-2 Annex A (white)		
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
Black, white and green pepper	Black berries	Physical separation and weighing ISO 959-2	Visual examination	IV
Black, white and green pepper	Broken berries	Physical separation and weighing ISO 959-2	Visual examination	IV
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

Spices and Culinary Herbs				-
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 content	ISO 939	Distillation	I
Black, white and green pepper	Total ash	ISO 928	Gravimetry	Ι
Black, white and green pepper	Non-volatile ether extract	ISO 1108	Soxhlet extration	Ι
Black, white and green pepper	Volatile oils	ISO 6571	Distillation	Ι
Black, white and green pepper	Piperine content	ISO 5564	Spectrophotometry	Ι
Black, white and green pepper	Acid-Insoluble ash	ISO 930	Gravimetry	Ι
Black, white and green pepper	Crude Fibre	ISO 5498	Gravimetry	Ι
Sugars and Honey				
Honey	Acidity	MAFF Validated Method V19 <i>J. Assoc. Public Analyst</i> s (1992) 28 (4) 171-175	Titrimetry	I
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	-	-

Sugars and Honey				
Honey	Solids, water-insoluble	MAFF Validated Method V22 J. Assoc. Public Analysts (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	Ι
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 (dextrose anhydrous and dextrose monohydrate, dried glucose syrup, glucose syrup, powdered dextrose, lactose)	Sulphated ash	ISO 5809	Single sulphonation	I
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	Ι
Sugars (fructose)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	Ι
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	
Sugars (fructose)	Sulphur dioxide	ISO 5379	Acidimetry and nephelometry	IV
Sugars (glucose syrup and dried glucose syrup)	Reducing sugar	ISO 5377	Titrimetry	I
Sugars (glucose syrup and dried glucose syrup)	Solids, total	ISO 1742	Gravimetry (vacuum oven)	Ι

Sugars and Honey				
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	Ш
Sugars (lactose)	Loss on drying	USP General Chapter 731	Gravimetry (Drying at 120°C for 16 h)	I
Sugars (lactose)	рН	ICUMSA GS 1/2/3/4/7/8-23	Potentiometry	I
Sugars (plantation and mill white sugar)	Colour	ICUMSA GS9/1/2/3-8	Photometry	I
Sugars (plantation or mill white sugar)	Conductivity ash	ICUMSA GS 1/3/4/7/8-13	Conductimetry	Ι
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	Ι
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 and Honey				
Sugars (soft white sugar and soft brown sugar)	Conductivity ash	ICUMSA GS 1/3/4/7/8-13	Conductimetry	I
Sugars (soft white sugar and soft brown sugar)	Invert sugar	ICUMSA GS 4/3-3 (applicable at levels >10% m/m)	Titrimetry (Lane & 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 & Eynon)	Ι
Sugars (soft white sugar and soft brown sugar)	Loss on drying	ICUMSA GS 2/1/3-15	Gravimetry	Ι
Sugars (soft white sugar and soft brown sugar)	Sucrose plus invert sugar	ICUMSA GS 4/3-7	Titrimetry	Ι
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	Ι
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				
Chili sauce	рН	NMKL 179 (Codex general method)	Potentiometry	II
Chili sauce	рН	AOAC 981.12 (Codex general method)	Potentiometry	
Chili sauce	Fill of containers	CAC/RM 46 (Codex general method)	Weighing	I
Date Paste	Moisture	AOAC 934.06	Gravimetry	
Date Paste	Mineral impurities	ISO 762	Gravimetry	I

Miscellaneous Products				
Date Paste	Ash	AOAC 940.26	Gravimetry	I
Date Paste	Acid Soluble Ash	AOAC 900.02D	Gravimetry, Calculation	I
Edible cassava flour	Fibre, crude	ISO 5498 (B.5 separation)	Gravimetry	Ι
Edible cassava flour	Granularity	ISO 2591-1	Sieving	Ι
Edible cassava flour	Moisture	ISO 712	Gravimetry	Ι
Fermented Soybean Paste	Total Nitrogen	AOAC 984.13	Kjeldahl	Ι
Fermented Soybean Paste	Amino Nitrogen	AOAC 920.154 on the conditions specified in the standard <sup>20</sup>	Volumetry	I
Fermented Soybean Paste	Moisture	AOAC 934.01 (≤70°C, ≤ 50 mm Hg)	Gravimetry	ļ
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	П
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	П
Food grade salt	lodine	EuSalt/AS 002	Titrimetry using sodium thiosulphate	II
Food grade salt	lodine	EuSalt/AS 019	ICP-OES	

# <sup>20</sup> 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) NH<sub>3</sub>-free H<sub>2</sub>O 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.

Endpoint - A pH meter shall be used to determine the endpoint instead of optical verification of colours

**Miscellaneous Products** 

Miscellancous i roudets				
Food grade salt	lodine	WHO/UNICEF/ICCIDD method <sup>21</sup> Only applicable to a product which has been fortified with iodate	Titrimetry using sodium thiosulphate	IV
Food grade salt	Lead	EuSalt/AS 015	ICP-OES	III
Food grade salt	Lead	EuSalt/AS 013	Atomic absorption spectrophotometry	IV
Food grade salt	Loss on drying	ISO 2483	Gravimetry (drying at 110°C)	Ι
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	Ι
Food grade salt	Sulphate	ISO 2480	Gravimetry	II
Food grade salt	Sulphate	EuSalt/AS 015	ICP-OES	
Food grade salt	Sulphate	EuSalt/AS 018	lon chromatography	
Foul medames	Sample Preparation	AOAC 945.68		_
Foul medames	Salt content	AOAC 971.27 NMKL 178	Potentiometry	II
Foul medames	Drained weight	AOAC 968.30	Sieving	Ι
Gari	Ash	ISO 2171	Gravimetry	Ι
Gari	Fibre, crude	ISO 5498 (B.5 separation)	Gravimetry	Ι
Gari	Granularity	ISO 2591-1	Sieving	Ι

<sup>&</sup>lt;sup>21</sup> 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

Gari	Moisture	ICC 109/1 ISO 712	Gravimetry	I	
Ginseng Products	Moisture	AOAC 925.45 B (Dried ginseng) Quantity of sample: 2 g	Gravimetry	I	
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 Gravimetry AACC Intl 08-01.01		I	
Ginseng Products	Water-insoluble Solids	described in the Standard Gravimetry (Annex I)		I	
Ginseng Products	Water-saturated n-butanol extracts	described in the Standard Gravimetry (Annex II)		I	
Ginseng Products	Identification of ginsenosides Rb1 and Rf	described in the Standard TLC or HPLC (Annex III)		IV	
Gochujang	Capsaicin	AOAC 995.03	HPLC	II	
Gochujang		described in the Standard (Annex D)	Gas chromatography	IV	
Gochujang	Crude protein	AOAC 984.13 (Nitrogen conversion factor: 6.25)	Kjeldahl	I	
Gochujang	Moisture	AOAC 934.01 (≤ 70°C, ≤ 50 Gravimetry mm Hg)		I	
Guideline level for acrylonitrile	Acrylonitrile	AOAC 985.13	Gas chromatography	II	

Miscellaneous Products				
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 ("head-space")	II
Guidelines for nutrition labelling	Polyunsaturated fatty acids	AOCS Ce 1h-05 <sup>22</sup>	Gas liquid chromatography	II
Guidelines for nutrition labelling	Saturated fat	AOAC 996.06; or AOCS Ce 1h-05	Gas liquid chromatography	11
Guidelines for nutrition labelling	Saturated fatty acids	AOCS Ce 1h-05	Gas liquid chromatography	II
Harissa	Acidity	ISO 750	titrimetry	I
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
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 23	Titrimetry	IV
Humus with tehena	Salt content	AOAC 971.27 NMKL 178	Potentiometry	II

<sup>22</sup> Can also be used to measure *trans* unsaturated fatty acids
<sup>23</sup> http://www.starch.dk/isi/methods/28luff.htm

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Miscellaneous Products				
Humus with tehena	Total acidity	AOAC 925.53	Titrimetry	I
Non-fermented soybean products	Moisture content	AOAC 925.09   AACCI 44- 40.01	Gravimetry (vacuum oven)	Ι
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	I
Sago Flour	Crude Fibre	ISO 6541	Gravimetry	I
Sago Flour	Starch	AOAC 920.44	Gravimetry	I
Tehena	Moisture Content	ISO 934	Gravimetry	Ι
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
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)	I

Miscellaneous Products				
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
Unrefined shea butter	Moisture content	ISO 662	Gravimetry	I
Unrefined shea butter	Free fatty acid content acid value and acidity	ISO 660 AOCS Cd 3d-63	Titrimetry	Ι
Unrefined shea butter	Relative density	AOCS Cc 10c-95/ ISO 6883	Pycnometry	Ι
Unrefined shea butter	Saponification value	ISO 3657 AOCS Cd 3d-25	Titrimetry	Ι
Unrefined shea butter	lodine value	AOAC 993.20/ ISO 3961/ AOCS Cd 1d-92/ NMKL 39	Wijs Titrimetry	I
Unrefined shea butter	Peroxide value	AOCS Cd 8b-90/ ISO 3960/ NMKL 158	Titrimetry	I
Unrefined shea butter	Unsaponifiable matter	ISO 3596/ AOCS Ca 6a-40	Gravimetry	I

Miscellaneous Products				
Unrefined shea butter	Insoluble impurities content	ISO 663/ AOCS Ca 3a-46	Gravimetry	I
Unrefined shea butter	Melting point	ISO 6321 AOCS Cc 3b-92	Open ended capillary tube	I

# 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	
Chili sauce	Described in the Standard	
Table Olives	Described in the Standard	

# STANDARD PROCEDURE FOR THAWING OF QUICKEN 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 (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 30oC.
- 3.4 Water thawing by direct contact, means thawing of the unpacked product by immersion in water at a temperature not exceeding 30oC. (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 sub-samples 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

#### 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 sauce pan with cover:
- 5.2 Hot plate or gasfire;
- 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 stil1 in the frozen state.

# STANDARD PROCEDURE FOR TOUGH STRING TEST OF QUICK FROZEN GREEN AND WAX BEANS

# 1. **DEFINITION**

A tough string is a string that will support the weight of 250 g for five 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 five 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

pods containing tough strings (g)

% m/m pods containing tough strings

test sample (g)

100

х

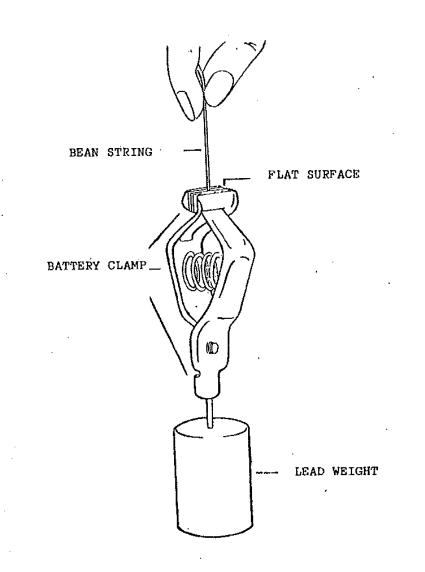


Figure 1 - Tough String Tester for Green or Wax Beans

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#### Appendix IV

# DETERMINATION OF THE ALCOHOL-INSOLUBLE SOLIDS CONTENT OFQUICK 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%) or denaturated ethanol

Ethanol denaturated with 5% v/v methanol.

2.2 Diluted ethanol or diluted denaturated ethanol 80% v/v

Dilute 8 parts by volume of reagent under 8.2.1 to 9.5 parts by volume with  $H_2O$ .

#### 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  $100 \pm 2^{\circ}C$ ;
- 3.9 Filter paper, Whatman No. 1 or equivalent;
- 3.10 Macerator or blendor;
- 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 two minutes. Wipe the bottom of the sieve. Weight 250 g peas into blendor, 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 2 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 ± 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% denaturated ethanol or 80% 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 unconvered in an air over for 2 hours at 100 ± 2°C. Cover the dish, cool in a desiccator, and weight 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:

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Alcohol-insoluble solids content (% m/m) = 10 \underline{M}
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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  $\underline{M}$ 

Where:

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

# 7. REPEATIBILITY 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 V

# 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 meter wil a silver electrode; or (b) direct titration with AgNO3 Express the result, after deducting salt from total dry matter, as "salt-free dry matter.