CODEX ALIMENTARIUS
INTERNATIONAL FOOD STANDARDS

Food and Agriculture Organization of the United Nations
World Health Organization

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RECOMMENDED METHODS OF ANALYSIS AND SAMPLING

CXS 234-1999

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***** The most updated version of the method should be used, in application of ISO/IEC 17025. The present list of methods reflects the amendments adopted by the Forty-sixth Session of the Codex Alimentarius Commission in 2023.

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PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

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Cereals, pulses and legumes and derived products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Certain pulses (soybeans)	Moisture	ISO 665	Gravimetry (oven drying at 103 °C)	I
Certain pulses except soybeans	Moisture	ISO 24557/AACC 44-17.01	Gravimetry (oven drying at 130 °C)	
Degermed maize (corn) meal and maize (corn) grits	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Degermed maize (corn) meal and maize (corn) grits	Fat, crude	AOAC 945.38F and 920.39C and ICC 11/1	Calculation from moisture and Gravimetry (ether extraction)	I
Degermed maize (corn) meal and maize (corn) grits	Moisture	ICC 110/1	Gravimetry (oven drying at 130 °C – 133 °C)	I

[i] Hominy, fruit juice, seafood.
[ii] Wine, dried apples, lemon juice, potato flakes, sultanas, beer.

Cereals, pulses and legumes and derived products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Degermed maize (corn) meal and maize (corn) grits	Particle size (granularity)	AOAC 965.22[iii]	Gravimetry (sieving)	I
Degermed maize (corn) meal and maize (corn) grits	Protein	ICC 105/2 and ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	I
Durum wheat semolina and durum wheat flour	Ash	AOAC 923.03 / ISO 2171 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (incineration at 550 °C)	I
Durum wheat semolina and durum wheat flour	Moisture	ISO 712 / ICC 110/1	Gravimetry (oven drying at 130 °C – 133 °C)	I
Durum wheat semolina and durum wheat flour	Protein	ICC 105/2 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	I
Instant noodles	Extraction of oil from instant noodles	See Appendix I, Part A	Gravimetry	I
Instant noodles	Acid value	See Appendix I, Part B	Titrimetry (ether extraction)	I
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Pearl millet flour	Ash	AOAC 923.03 / ISO 2171 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (incineration at 550 °C)	I
Pearl millet flour	Colour	*Modern Cereal Chemistry*, 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 and 920.39C and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (ether extraction)	I
Pearl millet flour	Fibre, crude	ISO 5498 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (extraction and filtration)	I

[iii] Sieve specifications as in ISO 3310/1.

Cereals, pulses and legumes and derived products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Pearl millet flour	Moisture	ISO 712 / ICC 110/1	Gravimetry (oven drying at 130 ºC–133 ºC)	I
Pearl millet flour	Protein	ISO 20483 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	I
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
Sorghum flour	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Sorghum flour	Colour	*Modern Cereal Chemistry*, 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 and 920.39C and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (ether extraction)	I
Sorghum flour	Fibre, crude	ICC 113 / ISO 6541 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (separation, incineration)	I
Sorghum flour	Moisture	ISO 712 / ICC 110/1	Gravimetry (oven drying at 130 ºC – 133 ºC)	I
Sorghum flour	Particle size (granularity)	AOAC 965.22[iv]	Sieving	I
Sorghum flour	Protein	ICC 105/2 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	I
Sorghum flour	Tannins	ISO 9648 and ISO 712 / ICC 110/1	Calculation from moisture and spectrophotometry	I
Sorghum grains	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I

[iv] Sieve specifications as in ISO 3310/1.

Cereals, pulses and legumes and derived products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Sorghum grains	Fat, crude	AOAC 945.38F and 920.39C and ISO 6540	Calculation from moisture and gravimetry (ether extraction)	I
Sorghum grains	Moisture	ISO 6540	Gravimetry (oven drying at 130 ºC and 133 ºC)	I
Sorghum grains	Protein	ICC 105/2 and ISO 6540	Titrimetry, Kjeldahl digestion	I
Sorghum grains	Tannins	ISO 9648 and ISO 6540	Calculation from moisture and spectrophotometry	I
Soy protein products	Ash	AOAC 923.03 ISO 2171: (Method B)	Gravimetry	I
Soy protein products	Fat	CAC/RM 55 - Method 1	Gravimetry (extraction)	I
Soy protein products	Fibre, crude	ISO 5498 and AOAC 925.09	Calculation from moisture and gravimetry (extraction and filtration)	I
Soy protein products	Moisture	AOAC 925.09	Gravimetry (vacuum oven 98 ºC – 100 ºC)	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 and AOAC 925.09	Calculation from moisture and gravimetry (incineration at 550 ºC)	I
Vegetable protein products	Fat	CAC/RM 55 - Method 1	Gravimetry (extraction)	I
Vegetable protein products	Fibre, crude	AACC 32-10.01 and AOAC 925.09	Calculation from moisture and gravimetry (ceramic fibre filtration)	I
Vegetable protein products	Moisture	AOAC 925.09	Gravimetry (vacuum oven at 98 C – 100 ºC)	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	ISO 7305 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (extraction)	I

Cereals, pulses and legumes and derived products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Wheat flour	Moisture	ISO 712: ICC 110/1	Gravimetry (oven drying at 130 ºC and 133 ºC)	I
Wheat flour	Particle size (granularity)	AOAC 965.22[v]	Gravimery (sieving)	I
Wheat flour	Protein	ICC 105/2 and ISO 712 / ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	I
Wheat protein products including wheat gluten	Crude protein excluding added vitamins, minerals, amino acids and optional ingredients	Vital wheat gluten and devitalized wheat gluten ISO 20483 and AOAC 925.09	Calculation from moisture and titrimetry (Kjeldahl digestion)	I
		Solubilized wheat protein ISO 20483 and AOAC 925.09	Calculation from moisture and titrimetry (Kjeldahl digestion)	I
Wheat protein products including Wheat gluten	Fibre, crude	AOAC 962.09 and AOAC 925.09	Calculation from moisture and gravimetry (ceramic fibre filtration)	I
Wheat protein products including wheat gluten	Moisture	AOAC 925.09	Gravimetry (vacuum oven at 98 ºC – 100 ºC)	I
Wheat protein products including Wheat gluten	Ash	AOAC 923.03 ISO 2171 and AOAC 925.09	Calculation from moisture and gravimetry (incineration at 550 ºC)	I
Whole and decorticated pearl millet grains	Ash	AOAC 923.03 / ISO 2171 and ISO 712/ ICC 110/1	Calculation from moisture and gravimetry (incineration at 550 ºC)	I
Whole and decorticated pearl millet grains	Fat, crude	AOAC 945.38F and 920.39C and ISO 712/ ICC 110/1	Calculation from moisture and gravimetry (ether extraction)	I
Whole and decorticated pearl millet grains	Fibre, crude	ISO 5498 and ISO 712 / ICC 110/1	Calculation from moisture and gravimetry (filtration through filter paper)	I
Whole and decorticated pearl millet grains	Moisture	ISO 712 ICC 110/1	Gravimetry (oven drying 130 ºC – 133 ºC)	I

[v] Sieve specifications as in ISO 3310/1.

Cereals, pulses and legumes and derived products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Whole and decorticated pearl millet grains	Protein	ISO 20483 and ISO 712 / ICC 110/1	Calculation from moisture and 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 945.38Fand 920.39C and ICC 110/1	Calculation from moisture and gravimetry (ether extraction)	I
Whole maize (corn) meal	Moisture	ICC 110/1 ISO 6540	Gravimetry (oven drying 130 ºC – 133 ºC)	I
Whole maize (corn) meal	Particle size (granularity)	AOAC 965.22[vi]	Sieving	I
Whole maize (corn) meal	Protein	ICC 105/2 and ICC 110/1	Calculation from moisture and titrimetry (Kjeldahl digestion)	I

[vi] Sieve specifications as in ISO 3310/1.

Cocoa products and chocolate				
Commodity	***Provision***	***Method***	***Principle***	***Type***
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	I
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
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	I
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Cocoa shell	AOAC 968.10 and 970.23	Spiral vessel count, stone cell count	I
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Fat	AOAC 963.15 or IOCCC 14	Gravimetry (Soxhlet extraction)	I
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	I

Fats and oils and related products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Fats and oils (all)	Arsenic	AOAC 963.21 and AOAC 942.17	Kjeldahl flask digestion and colorimetry (molybdenum blue)	III
Fats and oils (all)	Arsenic	AOAC 963.21 and AOAC 952.13	Kjeldahl flask digestion and colorimetry (diethyldithiorcarbamate)	III
Fats and oils (all)	Arsenic	AOAC 986.15	Atomic absorption spectrophotometry (hydride generation)	II
Fats and oils (all)	Insoluble impurities	ISO 663	Calculation from total insoluble content in *n*-hexane or light petroleum. Gravimetry, drying at 103 ^{0}C	I
Fats and oils (all)	Moisture and volatile matter	ISO 662	Gravimetry, drying at 103 ^{0}C	I
Fats and oils (all)	Soap content	ISO 10539 / AOCS Cc 17-95	Titrimery (colorimetric)	I
Fats and oils	Synthetic phenolic antioxidants	AOCS Ce 6a-2021	Liquid chromatography	II
Fats and oils	Synthetic phenolic antioxidants	AOAC 983.15	Liquid chromatography	III
Fats and oils not covered by individual standards	Acidity: acid value	ISO 660 / AOCS Cd 3d-63	Titrimetry	I
Fats and oils not covered by individual standards	Copper and iron	AOAC 990.05 / ISO 8294 / AOCS Ca 18b-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Fats and oils not covered by individual standards	Peroxide value	AOCS Cd 8b-90 ISO 3960 / NMKL 158	Titrimetry (colorimetric)	I
Fat spreads and blended spreads	Total fat	ISO 17189 | IDF 194	Gravimetry, direct determination of fat using solvent extraction	I
Fish oils	Fatty acid composition	AOCS Ce 2c-66 and AOCS Ce 1i-07 / AOCS Ce 1j-07	Preparation of methyl esters and gas chromatography	III
Fish oils	Fatty acid composition	ISO 12966-2 and ISO 12966-4	Preparation of methyl esters and gas chromatography	III

Fats and oils and related products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Fish oils	Acidity: acid value	AOCS Ca 5a-40 / AOCS Cd 3d-63 / ISO 660 / NMKL 38	Titrimetry	I
Fish oils	Peroxide value	AOCS Cd 8b-90 / ISO 3960 / NMKL 158 / European Pharmacopoeia 2.5.5	Titrimetry (colorimetric)	I
Fish oils	Phospholipids	USP-FCC 12 2S (Krill oil – phospholipids),	Nuclear magnetic resonance spectroscopy	I
Fish oils	P-Anisidine value	European Pharmacopoeia 2.5.36/ AOCS Cd 18-90/ ISO 6885	Spectrophotometry	I
Fish oils	Triglycerides	AOCS Cd 11d-96	Liquid chromatography with evaporative light scattering detection	II
Fish oils	Triglycerides	European Pharmacopoeia 1352	Liquid chromatography with refractive index detection	III
Fish oils	Triglycerides	USP 40 NF37	Liquid chromatography with refractive index detection	III
Fish oils	Vitamin A[vii]	European Pharmacopoeia Monograph on Cod Liver Oil (Type A), Monograph 01/2005:1192, with LC end-point 2.2.29	Liquid chromatography	III
Fish oils	Vitamin A[viii]	EN 12823-1	Liquid chromatography	II
Fish oils	Vitamin D	EN 12821 (Determination of vitamin D by high performance liquid chromatography – Measurement of cholecalciferol (D3) or ergocalciferol (D2))	Liquid chromatography	III
Fish oils	Vitamin D[ix]	NMKL 167 / EN 12821	Liquid chromatography	II

[vii] The respective *Standard for Fish Oils* (CXS 329-2017) states that vitamin A is expressed as 'retinol equivalents' (RE) where RE takes into account the fact that different vitamers of vitamin A differ in activity. ISO/TR 23304:2021 "Food products - Guidance on how to express vitamins and their vitamers".
[viii] See note vii above.
[ix] The provisions account for vitamins D2 and D3.

Fats and oils and related products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Named animal fats	Acidity: acid value	ISO 660 / AOCS Cd 3d-63	Titrimetry	I
Named animal fats	Fatty acid composition	ISO 12966-2 and ISO 12966-4	Preparation of methyl esters and gas chromatography	III
Named animal fats	Copper and iron	AOAC 990.05 / ISO 8294 / AOCS Ca 18b-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Named animal fats	Iodine value (IV)	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Titrimetry (Wijs)	I
Named animal fats	Peroxide value	AOCS Cd 8b-90 / ISO 3960 / NMKL 158	Titrimetry (colorimetric)	I
Named animal fats	Relative density	ISO 6883, with the appropriate conversion factor / AOCS Cc 10c-95	Pycnometry	I
Named animal fats	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
Named animal fats	Saponification value	ISO 3657 / AOCS Cd 3-25	Titrimetry (colorimetric)	I
Named animal fats	Unsaponifiable matter	ISO 3596 / ISO 18609 / AOCS Ca 6b-53	Gravimetry, drying at 103 ^{0}C and titrimetry (colorimetry)	I
Named animal fats	Titre	ISO 935	Thermometry	I
Named animal fats	Titre	AOCS Cc 12-59[x]	Thermometry	IV
Named vegetable oils	Acidity: Acid value	ISO 660 / AOCS Cd 3d-63 / AOCS Ca 5a-40	Titrimetry	I
Named vegetable oils	Free fatty acids	ISO 660 / AOCS Cd 3d-63 / AOCS Ca 5a-40	Titrimetry	I
Named vegetable oils	Apparent density	ISO 6883 / AOCS Cc 10c-95	Pycnometry	I
Named vegetable oils	Baudouin test (modified Villavecchia or sesame seed oil test)	AOCS Cb 2-40	Colour reaction	I

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

Fats and oils and related products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Named vegetable oils	Carotenoids, total	BS 684-2.20	Spectrophotometry	II
Named vegetable oils	Copper and iron	AOAC 990.05 / ISO 8294 / AOCS Ca 18b-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Named vegetable oils	Crismer value	AOCS Cb 4-35 and AOCS Ca 5a-40	Calculation from individual fatty acid composition (gas chromatography of methyl esters) and turbidity	I
Named vegetable oils	Fatty acid composition	ISO 12966-2 and ISO 12966-4 / AOCS Ce 2-66 and AOCS Ce 1h-05	Gas chromatography of methyl esters	II
Named vegetable oils	Halphen test	AOCS Cb 1-25	Colorimetry	I
Named vegetable oils	Insoluble impurities	ISO 663	Calculation from total insoluble content in *n*-hexane or light petroleum. Gravimetry, drying at 103 ^{0}C	I
Named vegetable oils	Iodine value	ISO 3961 / AOAC 993.20 / AOCS Cd 1d-92 / NMKL 39	Titrimetry (Wijs)	I
Named vegetable oils	Moisture and volatile matter	ISO 662	Gravimetry, drying at 103 ^{0}C	I
Named vegetable oils	Peroxide value (PV)	AOCS Cd 8b-90 / ISO 3960 / NMKL 158	Titrimetry (colorimetric)	I
Named vegetable oils	Refractive index	ISO 6320 / AOCS Cc 7-25	Refractometry	II
Named vegetable oils	Reichert-Meissi value and Polenske value	AOCS Cd 5-40	Calculation from soluble and insoluble volatile fatty acids. Titrimetry (colorimetric)	I
Named vegetable oils	Relative density	ISO 6883 / AOCS Cc 10c-95	Pynometry	I
Named vegetable oils	Saponification value (SV)	ISO 3657 / AOCS Cd 3-25	Titrimetry (colorimetric)	I
Named vegetable oils	Slip point	ISO 6321 / AOCS Cc 3b-92 for all oils, except palm oils or AOCS Cc 3-25 for palm oils only	Open ended capillary tube	I
Named vegetable oils	Sterol composition and total sterols	ISO 12228-1 / AOCS Ch 6-91	Thin-layer chromatography and gas chromatography	II

Fats and oils and related products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Named vegetable oils	Tocopherol content	ISO 9936 / AOCS Ce 8-89	Liquid chromatography with fluorescence detection	II
Named vegetable oils	Unsaponifiable matter	ISO 3596 / AOCS Ca 6b-53	Diethyl ether extraction and gravimetry, drying at 103 ^{0}C and titrimetry (colorimetry) and correction for free fatty acids titrimetry (colorimetry)	I
Named vegetable oils	Unsaponifiable matter	ISO 18609[xi]	Hexane extraction and gravimetry, drying at 103 °C and titrimetry (colorimetry) and correction for free fatty acids titrimetry (colorimetry)[xii]	IV
Olive oils and olive pomace oils	Absorbency in ultraviolet	COI/T.20/Doc. No. 19 or ISO 3656 or AOCS Ch 5-91	Absorption in ultraviolet	II
Olive oils and olive pomace oils	Acidity, free (acid value)	ISO 660 or AOCS Cd 3d-63	Titrimetry	I
Olive oils and olive pomace oils	Alpha-tocopherol	ISO 9936	HPLC	II
Olive oils and olive pomace oils	Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 or AOCS Ce 5b-89	Analysis of triglycerides of HPLC and calculation	I
Olive oils and olive pomace oils	Erythrodiol + uvaol	COI/T.20/Doc.no. 30	Gas chromatography	II
Olive oils and olive pomace oils	Halogenated solvents, traces	COI/T.20/Doc. no. 8	Gas chromatography	II
Olive oils and olive pomace oils	Insoluble impurities in light petroleum	ISO 663	Gravimetry	I
Olive oils and olive pomace oils	Iodine value	ISO 3961 or AOAC 993.20 or AOCS Cd 1d-92 or NMKL 39	Wijs-titrimetry	I
Olive oils and olive pomace oils	Iron and copper	ISO 8294 or AOAC 990.05	AAS	II
Olive oils and olive pomace oils	Moisture and volatile matter	ISO 662	Gravimetry	I

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

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

Fats and oils and related products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Olive oils and olive pomace oils	Organoleptic characteristics	COI/T.20/Doc. no. 15	Panel test	I
Olive oils and olive pomace oils	Peroxide value	ISO 3960 or AOCS Cd 8b-90	Titrimetry	I
Olive oils and olive pomace oils	Relative density	IUPAC 2.101, with the appropriate conversion factor. See comment above	Pycnometry	I
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	I
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	III
Olive oils and olive pomace oils	Trans fatty acids content	COI/T.20/Doc no. 17 or ISO 15304 or AOCS Ch 2a-94	Gas chromatography of methyl esters	II
Olive oils and olive pomace oils	Unsaponifiable matter	ISO 3596 or ISO 18609 or AOCS Ca 6b-53	Gravimetry	I
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				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Fish and fishery products	Histamine	AOAC 977.13	Fluorimetry	II
Fish and fishery products	Mercury	AOAC 977.15	Flameless atomic absorption spectrophotometry	III
Fish and fishery products: canned products	Drained weight	Described in the standard	Weighing	I
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	I
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	pH	AOAC 981.12 The pH shall be measured in a sample of fish sauce diluted with water to 1:10 using a pH metre. The dilution of fish sauce is necessary because of the high ionic strength in the undiluted sauce	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	I

Fish and fishery products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
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
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	I
Quick-frozen fish sticks (fish fingers) and fish portions – breaded or in batter	Net weight	Described in the standard	Weighing	I
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

Fish and fishery products				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Salted fish and dried salted fish of the Gadidae 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[xiii]	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

Table 1. Method performance criteria for histamine for fish and fishery products

Provision	ML (mg/100 g)	Minimum applicable range (mg/100 g)	LOD (mg/100 g)	LOQ (mg/100 g)	RSD_R (%)	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 2 and may either meet the minimum applicable range, or LOD and LOQ criteria listed.

[xiii] % salt × 100/(% water + % salt).

Table 2. Criteria for determination of toxin analogues by chemical methods

Toxin group	Toxin	Minimum applicable range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD_R) (%) No more than	Recovery percent	Applicable methods that meet the criteria
STX group	Saxitoxin (STX)	0.05 – 0.2	0.01	0.02	44%	50 – 130	AOAC 2005.06 NMKL 182, EN 14526 AOAC 2011.02 NMKL 197
	NEO	0.05 – 0.2	0.01	0.02	44%	50 – 130	
	dcSTX	0.05 – 0.2	0.01	0.02	44%	50 – 130	
	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 aid	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	

Total toxicity is estimated as the sum of the molar concentrations of detected analogues multiplied by the relevant specific toxicity equivalency factors (TEFs). Internationally scientifically validated TEFs must be used. The science behind TEFs is developing. Current internationally validated TEFs 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 2.

Where toxin analogues that are not listed in Table 2 are determined the competent authority must assess the contribution of these analogues to total toxicity whilst conducting further investigations.

Table 3. Performance criteria for methods of analysis of methylmercury*

Commodity	Provision	ML (mg/kg)	Min appl. range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (%) Not more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
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	8–110	AOAC 988.11 EN 16801	GC-electron capture GC-ICP/MS

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

Foods for special dietary uses				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Special foods	Ash	AOAC 942.05	Gravimetry	I
Special foods	Calcium	AOAC 984.27	ICP emission spectrometry	III
Special foods	Calories by calculation	Method described in CAC/VOL IX-Ed.1, Part III	Calculation method	III
Special foods	Carbohydrates	Method described in CAC/VOL IX-Ed.1, Part III	Calculation	III
Special foods	Chloride	AOAC 971.27 (Codex general method)	Potentiometry	II
Special foods	Dietary fibre, total	AOAC 985.29	Gravimetry (enzymatic digestion)	I
Special foods	Fat	CAC/RM 55	Gravimetry (extraction)	I

Foods for special dietary uses				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Special foods	Fat in foods not containing starch, meat, or vegetable products	CAC/RM 1, B-2	Gravimetry	I
Special foods	Fill of containers	CAC/RM 46 (See Appendix II)	Weighing	I
Special foods	Folic acid	AOAC 944.12	Microbioassay	II
Special foods	Linoleate (in the form of glycerides)	AOAC 922.06; 969.33; 963.22	Acid hydrolysis, preparation of methyl esters and gas chromatography	II
Special foods	Linoleate (in the form of glycerides)	AOAC 922.06; 979.19	Acid hydrolysis and spectrophotometry	III
Special foods	Loss on drying (milk-based)	AOAC 925.23 ISO 6731 | IDF 21	Gravimetry	I
Special foods	Nicotinamide for foods not based on milk	AOAC 961.14	Colorimetry	II
Special foods	Nicotinamide for milk-based foods	AOAC 944.13	Microbioassay	II
Special foods	Pantothenic acid/enriched foods	AOAC 945.74	Microbioassay	II
Special foods	Pantothenic acid/non-enriched foods	*The Analyst* 89 (1964):1, 3-6, ibid. 232 US Dept Agr., *Agr. Handbook* 97 (1965)	Microbioassay	IV
Special foods	Phosphorous	AOAC 986.24	Colorimetry (molybdovanadate)	II
Special foods	Protein efficiency ratio (PER)	AOAC 960.48	Rat bioassay	I
Special foods	Protein, crude	Method described in CAC/Vol IX-Ed. 1, Part III	Titrimetry, Kjeldahl digestion	I
Special foods	Riboflavin	AOAC 970.65	Fluorometry	II
Special foods	Sodium and potassium	ISO 8070 | IDF 119	Flame atomic absorption spectrometry	II
Special foods	Sodium and potassium	AOAC 984.27	ICP emission spectrometry	III
Special foods	Vitamin A	AOAC 974.29	Colorimetry	IV
Special foods	Vitamin A in foods in which carotenes have been added as a source of vitamin A	AOAC 941.15	Spectrophotometry	III

Foods for special dietary uses				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Special foods	Vitamin B_{12}	AOAC 952.20	Microbioassay	II
Special foods	Vitamin B_6	AOAC 961.15	Microbioassay	II
Special foods	Vitamin C	AOAC 967.22	Microfluorometry	II
Special foods	Vitamin C	AOAC 967.21	Colorimetry (dichloroindophenol)	III
Special foods	Vitamin D (D_3, milk-based infant formula)	AOAC 992.26	Liquid chromatography	II
Special foods	Vitamin E	AOAC 971.30	Colorimetry	IV
Special foods	Vitamin E (milk-based infant formula)	AOAC 992.03	Liquid chromatography	II
Special foods	Sodium and potassium	ISO 8070 | IDF 119	Flame atomic absorption spectrometry	II
Follow-up formula	Dietary fibre, total	AOAC 991.43	Gravimetry (enzymatic digestion)	I
Follow-up formula	Iodine (milk-based formula)	AOAC 992.24	Ion-selective potentiometry	II
Follow-up formula	Pantothenic acid	AOAC 992.07 Measures total pantothenate (free pantothenic acid + CoA- + ACP-bound) and measured as D-pantothenic acid (or calcium D-pantothenate)	Microbioassay	II
Follow-up formula	Vitamin A	AOAC 974.29	Colorimetry	IV
Follow-up formula	Vitamin A (retinol isomers)	AOAC 992.04	HPLC	II
Follow-up formula	Vitamin A (retinol) (above 500 IU/l milk after reconstitution)	AOAC 992.06	HPLC	III
Follow-up formula	Vitamin K	AOAC 2015.09 / ISO 21446	HPLC-FLD	II

Foods for special dietary uses				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Foods with low-sodium content (including salt substitutes)	Iodine	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 *Eur J Gastroenterol Hepatol* 2003; 15: 465-474	Immunoassay	I
Infant formula	Biotin	AOAC 2016.02 / ISO 23305	HPLC-UV	II
Infant formula	Biotin	EN 15607 (d-biotin) (Measures total D-biotin [free + D-biocytin])	HPLC- FLD	III
Infant formula	Calories (by calculation)	Method described in CAC/Vol IX-Ed.1, Part III[xiv]	Calculation	I
Infant formula	Calcium	AOAC 2015.06/ISO 21424 | IDF 243	ICP-MS	II
Infant formula	Calcium	AOAC 2011.14/ISO 15151 | IDF 229	ICP emission spectroscopy	III
Infant formula	Calcium	ISO 8070 | IDF 119	Flame atomic absorption spectrophotometry	III
Infant formula	Calcium	AOAC 985.35	Flame atomic absorption spectroscopy	III

[xiv] Section 9. Calories by calculation – Section 9.2 Conversion factors

(a) protein 4 kcal per g
(b) carbohydrate 4 kcal per g
(c) fat 9 kcal per g
(d) monosaccharides 3.75 kcal per g
(e) specific food ingredients: See “Energy and Protein Requirements” (FAO Nutrition Meeting Report Series No. 52 or WHO Technical Report Series No. 522).
(f) other specific calorie conversion factors may be used where the formulation of the food and the nutrient content are known and where such specific conversion factors are physiologically more meaningful than the factors listed above.

Foods for special dietary uses				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Infant formula	Carnitine	AOAC 2015.10/ISO 21468	UHPLC-MS/MS	II
Infant formula	Chloride	AOAC 986.26	Potentiometry	III
Infant formula	Chloride	AOAC 2016.03/ISO 21422 | IDF 242	Potentiometry	II
Infant formula	Choline	AOAC 2015.10/ISO 21468	UHPLC-MS/MS	II
Infant formula	Choline	AOAC 999.14	Enzymatic colorimetric method with limitations on applicability due to choline and ascorbate concentration.	III
Infant formula	Copper	AOAC 2015.06/ISO 21424 | IDF 243	ICP-MS	II
Infant formula	Copper	AOAC 985.35	Flame atomic absorption spectroscopy	III
Infant formula	Copper	AOAC 2011/14/ISO 15151 | IDF 229	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	III
Infant formula	Chromium (Section B of CXS 72-1981 only)	AOAC 2011.19/ISO 20649 |IDF 235	ICP-MS	II
Infant formula	Crude protein[xv]	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Infant formula	Fatty acids (including trans fatty acid)	AOAC 996.06	Gas chromatography	III

[xv] **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 dietary uses				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Infant formula	Fatty acids (including trans fatty acid)	AOCS Ce 1i-07	Gas chromatography	III
Infant formula	Folic acid	AOAC 992.05 (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)	Microbioassay	III
Infant formula	Folic acid	AOAC 2011.06	LC-MS/MS	II
Infant formula	Iodine (for milk-based formula)	AOAC 2012.15 / ISO 20647 | IDF 234	ICP-MS	II
Infant formula	Iron	AOAC 2015.06 / ISO 21424 | IDF 243	ICP-MS	II
Infant formula	Iron	AOAC 2011.14 / ISO 15151 | IDF 229	ICP emission spectroscopy	III
Infant formula	Iron[xvi]	AOAC 985.35	Flame atomic absorption spectrophotometry	III
Infant formula	Iron	AOAC 999.11 | NMKL139	AAS after dry ashing	II
Infant formula	Magnesium	AOAC 2015.06 / ISO 21424 | IDF 243	ICP-MS	II
Infant formula	Magnesium	AOAC 2011.14 / ISO 15151 | IDF 229	ICP emission spectroscopy	III
Infant formula	Magnesium	ISO 8070 | IDF 119	Flame atomic absorption spectrophotometry	III
Infant formula	Magnesium	AOAC 985.35	Flame atomic absorption spectroscopy	III

[xvi] General Codex methods are also available.

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

[xvii] When published as EN method.

Foods for special dietary uses				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Infant formula	Phosphorus	AOAC 986.24	Spectrophotometry (molybdovanadate)	III
Infant formula	Riboflavin	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II
Infant formula	Riboflavin	AOAC 985.31[xviii]	Fluorimetry	III
Infant formula	Riboflavin	EN 14152 (Measures natural and supplemental forms, free, bound and phosphorylated (FMN and FAD) aggregated and measured as riboflavin.)	HPLC	III
Infant formula	Selenium	AOAC 996.16 or AOAC 996.17	Continuous hydride generation flame atomic absorption spectrometry (HGAAS)	III
Infant formula	Selenium	EN 14627	Hydride generation atomic absorption spectrometry (HGAAS)	III
Infant formula	Selenium	AOAC 2006.03	ICP emission spectroscopy	III
	Selenium	AOAC 2011.19 / ISO 20649 | IDF 235	ICP-MS	II
Infant formula	Sodium and potassium	AOAC 2015.06 / ISO 21424 | 243	ICP-MS	II
Infant formula	Sodium and potassium	AOAC 2011.14 / ISO 15151 | IDF 229	ICP emission spectroscopy	III
Infant formula	Sodium and potassium	ISO 8070 | IDF 119	Flame atomic absorption spectrophotometry	III
Infant formula	Thiamine	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II
Infant formula	Thiamine	AOAC 986.27[xix]	Fluorimetry	III

[xviii] Care should be taken in the application of the method due to spectral interference.
[xix] See note xviii above.

Foods for special dietary uses				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Infant formula	Thiamine	EN 14122 (Measures all vitamin B_1 forms (natural and added free, bound and phosphorylated) following extraction and conversion to thiamine)	HPLC with pre-or post-column derivatization to thiochrom	III
Infant formula	Total amino acids (excluding taurine and tryptophan) for use according to Section 3.1.3 (a) notes 2) and 3) of CXS 72-1981	AOAC 2018.06 / ISO 4214 | IDF 254 / AACC 07-50.01	UHPLC-UV	II
Infant formula	Total carbohydrates	AOAC 986.25 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 23318 | IDF 249	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)	I
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
Infant formula	Total phospholipids	AOCS Ja7b-91	Gas chromatography with suitable extraction and preparation procedures	III

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

Foods for special dietary uses

Commodity	*Provision*	*Method*	*Principle*	*Type*
Infant formula	Vitamin E	EN 12822 (Measures vitamin E (both natural + supplemental ester forms) aggregated and quantified as individual tocopherol congeners (α, β, γ, δ)	HPLC	II
Infant formula	Vitamin E	AOAC 2012.10 / ISO 20633	HPLC	II
Infant formula	Vitamin B_6	AOAC 2015.14 / ISO 21470	Enzymatic digestion and UHPLC-MS/MS	II
Infant formula	Vitamin B_6	AOAC 985.32	Microbioassay	III
Infant formula	Vitamin B_6	EN 14166 (Aggregates free and bound pyridoxal, pyridoxine and pyridoxamine and measures as pyridoxine)	Microbioassay	III
Infant formula	Vitamin B_6	AOAC 2004.07 EN 14164 (Free and bound phosphorylated forms (pyridoxal, pyridoxine and pyridoxamine) converted and measured as pyridoxine)	HPLC	III
Infant formula	Vitamin B_{12}	AOAC 986.23 (Measures total vitamin B_{12} as cyanocobalamin)	Turbidimetric method	III
Infant formula	Vitamin B_{12}	AOAC 2011.10 / ISO 20634	HPLC	II
Infant formula	Vitamin K	AOAC 2015.09 / ISO 21446	HPLC-FLD	II
Infant formula	Zinc	AOAC 2015.06 / ISO 21424 | IDF 243	ICP-MS	II
Infant formula	Zinc	AOAC 2011.14 / ISO 15151 | IDF 229	ICP emission spectroscopy	III
Infant formula	Zinc	AOAC 985.35	Flame atomic absorption spectroscopy	III

Table 4. Methods of analysis for dietary fibre: *Guidelines for Use of Nutrition and Health Claims* (CXG 23-1997): Table of conditions for claims

Standard	Provisions	Method	Principle	Type
General methods that do not measure the lower molecular weight fraction (i.e. monomeric units < = 9)[2]				
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	Type I
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	Type I
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	Type I
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 methods that measure both the higher (monomeric units > 9) and the lower molecular weight fraction (monomeric units <=9) [2]				
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	Type I
All foods (1)	Method applicable for determining the content of dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starch	ICC Standard No. 185 / AOAC 2017.16 / AACC 32-60-01	Enzymatic gravimetry High pressure liquid chromatography	Type I
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	Type I

Standard	Provisions	Method	Principle	Type
General methods that do not measure the lower molecular weight fraction (i.e. monomeric units < = 9)[2]				
Methods that measure individual specific components (monomeric units: the whole range for each type of components is covered)[2]				
All foods (1)	(1→3)(1→4) *Beta*-D-Glucans	AOAC 995.16 AACC Intl 32-23.01	Enzymatic	Type II
All foods (1)	Fructans (oligofructoses, inulin, hydrolysed 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, hydrolysed inulin, polyfructoses, fructooligosaccharides) (not applicable highly depolymerized 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 methods[2] that have not been subjected to interlaboratory evaluation under AOAC international guidelines				
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.technique.home.screen	Chemical & HPAEC-PAD	Type IV
All foods	Fructo-oligosaccharides (monomeric units < 5)	Ouarné *et al.* 1999 in *Complex Carbohydrates in Foods*. Edited by S. Sungsoo, L. Prosky & M. Dreher. Marcel Dekker Inc, New York	HPAEC-PAD	Type 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	Type IV

(1) Users should consult the description of each method for the food matrices that were the subject of interlaboratory study in the Official Methods of Analysis of AOAC International.

(2) Two issues are left for national authorities: to include monomeric units 3-9 and which isolated or synthetic compounds have physiological benefit. (Refer to the *Guidelines on Nutrition Labelling* (CXG 2-1985).

(3) Quantitation lost for resistant starch. Refer to specific methods.

(4) Quantitation lost for inulin, resistant starch, polydextrose and resistant maltodextrins. Refer to specific methods.

Fruit juices and nectars

Commodity	***Provisions***	***Method***	***Principle***	***Type***
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[xx] (additives)	AOAC 986.13	HPLC	II

[xx] All juices except citrus based juices.

Fruit juices and nectars				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Fruit juices and nectars	Citric acid[xxi] (additives)	EN 1137 IFUMA 22	Enzymatic determination	III
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 and HIS in apple juice (permitted ingredients)	Determination of HFCS and 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	III

[xxi] All juices except citrus based juices.

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Fruit juices and nectars				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Fruit juices and nectars	Preservatives in fruit juices (sorbic acid and its salts)	ISO 5519	Spectrometry	III
Fruit juices and nectars	Quinic, malic and citric acid in cranberry juice cocktail and apple juice (permitted ingredients and additives)	Determination of quinic, malic, and citric acid in cranberry juice cocktail and apple juice AOAC 986.13	HPLC	III
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	III
Fruit juices and nectars	Sucrose (permitted ingredients)	EN 12630 IFUMA 67 NMKL 148	HPLC	II
Fruit juices and nectars	Sulphur dioxide (additives)	Optimized Monier-Williams AOAC 990.28 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	III
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				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005[xxii]	Determination of acetic acid EN 12632; IFUMA 66	Enzymatic determination	II
Fruit juices and nectars		Determination of alcohol (ethanol) IFUMA 52	Enzymatic determination	II
Fruit juices and nectars		Detection of anthocyanins IFUMA 71	HPLC	I
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
Fruit juices and nectars		Determination of benzoic acid as a marker in orange juice AOAC 994.11	HPLC	III
Fruit juices and nectars		Determination of C^{13}/C^{12} 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

[xxii] **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				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
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[xxiii]	(Scott) distillation, titration	I
Fruit juices and nectars		Determination of essential oils (in citrus fruit) (volume determination)[xxiv] ISO 1955	Distillation and direct reading of the volume determination	I
Fruit juices and nectars		Determination of fermentability IFUMA 18	Microbiological method	I
Fruit juices and nectars		Determination of formol number EN 1133 IFUMA 30	Potentiometric titration	I
Fruit juices and nectars		Determination of free amino acids EN 12742 IFUMA 57	Liquid chromatography	II
Fruit juices and nectars		Determination of fumaric acid IFUMA 72	HPLC	II
Fruit juices and nectars		Determination of glucose fructose and saccharose EN 12630 IFUMA 67 NMKL 148	HPLC	II
Fruit juices and nectars		Determination of gluconic acid IFUMA 76	Enzymatic determination	II

[xxiii] *Because there is no numerical value in the standard duplicate Type I methods have been included which may lead to different results.*
[xxiv] See note xxiii above.

Fruit juices and nectars				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Fruit juices and nectars		Determination of glycerol IFUMA 77	Enzymatic determination	II
Fruit juices and nectars		Determination of hesperidin and naringin EN 12148 IFUMA 58	HPLC	II
Fruit juices and nectars		Determination of hydroxymethylfurfural IFUMA 69	HPLC	II
Fruit juices and nectars		Determination of hydroxymethylfurfural ISO 7466	Spectrometry	III
Fruit juices and nectars		Determination of isocitric acid-D IFUMA 54	Enzymatic determination	II
Fruit juices and nectars		Determination of Lactic acid- D and L EN 12631 IFUMA 53	Enzymatic determination	II
Fruit juices and nectars		Determination of L-malic/total malic acid ratio in apple juice AOAC 993.05	Enzymatic determination and HPLC	II
Fruit juices and nectars		Determination of naringin and neohesperidin in orange juice AOAC 999.05	HPLC	III
Fruit juices and nectars		Determination of pH value NMKL 179 EN 1132 IFUMA 11 ISO 1842	Potentiometry	 II IV
Fruit juices and nectars		Determination of phosphorus/phosphate EN 1136 IFUMA No 50	Photometric determination	II
Fruit juices and nectars		Determination of proline by photometry – non-specific determination EN 1141 IFUMA 49	Photometry	I
Fruit juices and nectars		Determination of relative density EN 1131 (1993); IFUMA 01 & IFU Method No General sheet (1971)	Pycnometry	II
Fruit juices and nectars		Determination of relative density IFUMA 01A	Densitometry	III
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				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
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	I
Fruit juices and nectars		Determination of titrable acids, total EN 12147 IFUMA 03 ISO 750	Titrimetry	I
Fruit juices and nectars		Determination of total dry matter (vacuum oven drying at 70 °C)[xxv] EN 12145 IFUMA 61	Gravimetric determination	I
Fruit juices and nectars		Determination of total solids (microwave oven drying)[xxvi] AOAC 985.26	Gravimetric determination	I
Fruit juices and nectars		Determination of vitamin C (dehydro-ascorbic acid and ascorbic acid) AOAC 967.22	Microfluorometry	III

[xxv] *Because there is no numerical value in the standard duplicate Type I methods have been included which may lead to different results.*
[xxvi] See note xxv above.

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

[xxvii] Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.
[xxviii] See note xxvii above.
[xxix] See note xxvii above.

Milk and milk products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat (MSNF)[xxx]	ISO 6731 | IDF 21 and ISO 23318 | IDF 249	Calculation from total solids content and fat content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb)	I
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF[xxxi]	ISO 6731 | IDF 21 and ISO 23318 | IDF 249 and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF[xxxii]	ISO 6731 | IDF 21 and ISO 23318 | IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Total fat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	I
Blend of skimmed milk and vegetable fat in powdered form	Water[xxxiii] (moisture)	Described in Appendix III[xxxiv]	Gravimetry, drying at 102 °C	IV
Blend of skimmed milk and vegetable fat in powdered form	Water[xxxv] (moisture)	ISO 5537 | IDF 26	Gravimetry, drying at 87 °C	I
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF[xxxvi]	ISO 5537 | IDF 26 and ISO 23318 | IDF 249 and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 87 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV

[xxx] Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.
[xxxi] See note xxx above.
[xxxii] See note xxx above.
[xxxiii] Water content excluding the crystallized water bound to lactose (generally known as moisture content).
[xxxiv] Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method described in Appendix III is listed as Type IV.
[xxxv] See note xxxiii above.
[xxxvi] See note xxx above.

Milk and milk products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF[xxxvii]	Described in Appendix III[xxxviii] and ISO 23318 | IDF 249 and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF[xxxix]	ISO 5537 | IDF 26 and and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 87 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF[xl]	Described in Appendix III[xli] and ISO 23318 |IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Total fat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water[xlii] (moisture)	ISO 5537 | IDF 26	Gravimetry, drying at 87 °C	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water[xliii] (moisture)	Described in Appendix III[xliv]	Gravimetry, drying at 102 °C	IV

[xxxvii] Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.
[xxxviii] Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method as described in Appendix III is listed as Type IV.
[xxxix] See note xxxvii above.
[xl] See note xxxvii above.
[xli] See note xxxviii above.
[xlii] Water content excluding the crystallized water bound to lactose (generally known as moisture content).
[xliii] See note xlii above.
[xliv] See note xxxviii above.

Milk and milk products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF[xlv]	ISO 5537 | IDF 26 and ISO 1736 | IDF 9 and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 87 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF[xlvi]	ISO 5537 | IDF 26 and ISO 23318 | IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 87 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF[xlvii]	Described in Appendix III[xlviii] and ISO 23318 |IDF 249 and AOAC 991.20	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	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 (for products sweetened with sucrose only)	Milk solids-not-fat (MSNF)[xlix]	ISO 6734 | IDF 15 and ISO 23318 | IDF 249 and ISO 2911 | IDF 35	Calculation from total solids content, fat content and sucrose content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry	IV

[xlv] Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.
[xlvi] See note xlv above.
[xlvii] See note xlv above.
[xlviii] Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method as described in Appendix III is listed as Type IV.
[xlix] See note xlv above.

Milk and milk products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF[l]	ISO 6734 | IDF 15 and ISO 23318 | IDF 249 and ISO 2911 | IDF 35 and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content, sucrose content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry and titrimetry (Kjeldahl)	IV
Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF[li]	ISO 6734 | IDF 15 and ISO 23318 | IDF 249 and ISO 2911 | IDF 35 and AOAC 991.20	Calculation from total solids content, fat content, sucrose content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry and titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk solids-not-fat (MSNF)[lii]	ISO 6734 | IDF 15 and ISO 23318 | IDF 249 and ISO 2911 | IDF 35	Calculation from total solids content, fat content and sucrose content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF[liii]	ISO 6734 | IDF 15 and ISO 23318 | IDF 249 and ISO 2911 | IDF 35 and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content, sucrose content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry and titrimetry (Kjeldahl)	IV

[l] Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.
[li] See note l above.
[lii] See note l above.
[liii] See note l above.

Milk and milk products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only)	Milk protein in MSNF[liv]	ISO 6734 | IDF 15 and ISO 23318 | IDF 249 and ISO 2911 | IDF 35 and AOAC 991.20	Calculation from total solids content, fat content, sucrose content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and polarimetry and titrimetry (Kjeldahl)	IV
Butter	Milk solids-not-fat (MSNF)[lv]	ISO 3727-2 | IDF 80-2	Gravimetry	I
Butter	Milkfat (total fat)	ISO 17189 | IDF 194	Gravimetry (direct determination of fat using solvent extraction)	I
Butter	Milk fat purity	ISO 17678 | IDF 202	Calculation from determination of triglycerides by gas chromatography - FID	I
Butter	Salt	ISO 1738 | IDF 12/ AOAC 960.29	Titrimetry (Mohr: determination of chloride, expressed as sodium chloride)	III
Butter	Salt	ISO 15648 | IDF 179	Potentiometry (determination of chloride, expressed as sodium chloride)	II
Butter	Water[lvi]	ISO 3727-1 | IDF 80-1	Gravimetry	I
Cheese	Milkfat	ISO 23319 | IDF 250	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cheese	Moisture	ISO 5534 | IDF 4	Gravimetry, drying at 102 °C	I

[liv] Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.
[lv] See note liv above.
[lvi] Water content excluding the crystallized water bound to lactose (generally known as moisture content).

Milk and milk products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Cheese (and cheese rind)	Natamycin	ISO 9233-1 | IDF 140-1	Molecular absorption spectrophotometry	III
Cheese (and cheese rind)	Natamycin	ISO 9233-2 | IDF 140-2	HPLC-UV	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)[lvii]	ISO 5534 | IDF 4	Gravimetry, drying at 102 °C	I
Cheeses, individual	Milkfat in dry matter	ISO 5534 | IDF 4 ISO 23319 | IDF 250	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cheeses in brine	Milkfat in dry matter	ISO 5534 | IDF 4 ISO 23319 | IDF 250	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cottage cheese	Fat-free dry matter	ISO 5534 | IDF 4 and ISO 23319 | IDF 250	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Schmid-Bondzynski-Ratzlaff)	I

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

Milk and milk products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Cottage cheese (for samples containing lactose over 5% or with non-dairy ingredients)	Milkfat in dry matter	ISO 5534 | IDF 4 and ISO 8262-3 | IDF 124-3	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Weibull-Berntrop)	I
Cottage cheese (for samples containing lactose up to 5%)	Milkfat in dry matter	ISO 5534 | IDF 4 and ISO 23319 | IDF 250	Calculation from dry matter content and fat content, gravimetry, drying at 102 °C and gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cottage cheese (for samples containing lactose up to 5%)	Milkfat	ISO 23319 | IDF 250	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cottage cheese (for samples containing lactose over 5% or with non-dairy ingredients)	Milkfat	ISO 8262-3 | IDF 124-3	Gravimetry (Weibull-Berntrop)	I
Cheese, unripened, including fresh cheese	Milk protein	ISO 8968-1 | IDF 20-1	Titrimetry, Kjeldahl	I
Cream and prepared creams	Milk protein	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Cream	Milkfat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	I
Cream	Solids[lviii]	ISO 6731 | IDF 21	Gravimetry (drying at 102 °C)	I
Creams lowered in milkfat content	Milkfat	ISO 23318 | IDF 249 / AOAC 995.19	Gravimetry (Röse-Gottlieb)	I
Creams, whipped creams and fermented creams	Milk solids-not-fat (MSNF)[lix]	ISO 3727-2 | IDF 80-2	Gravimetry	I
Cream cheese	Dry matter	ISO 5534 | IDF 4	Gravimetry drying at 102 °C (forced air oven)	I

[lviii] Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.
[lix] See note lviii above.

Milk and milk products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Cream cheese	Moisture on fat-free basis	ISO 5534 | IDF 4 ISO 23319 | IDF 250	Calculation from fat content and moisture content, gravimetry drying at 102 °C (forced air oven), gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Dairy fat spreads	Milk fat purity	ISO 17678 | IDF 202	Calculation from determination of triglycerides by gas chromatography - FID	I
Dairy fat spreads	Milkfat (total fat)	ISO 17189 | IDF 194	Gravimetry Gravimetry (direct determination of fat using solvent extraction)	I
Dairy permeate powders	Lactose	ISO 22662 | IDF 198	High performance liquid chromatography	II
Dairy permeate powders	Milkfat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	I
Dairy permeate powders	Nitrogen	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Dairy permeate powders	Moisture[lx]	ISO 5537 | IDF 26	Gravimetry (drying at 87 °C)	I
Dairy permeate powders	Ash	NMKL 173 / AOAC 930.30	Gravimetry (ashing at 550 °C)	I
Edible casein products	Free acidity	ISO 5547 | IDF 91	Titrimetry (aqueous extract)	I
Edible casein products (caseins obtained by rennet precipitation and of caseinates, with the exception of ammonium caseinate)	Ash (including P_2O_5)	ISO 5545 | IDF 90	Gravimetry (ashing at 825 °C)	I

[lx] Moisture content excluding the water of crystallization of lactose.

Milk and milk products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Edible casein products (acid caseins, of ammonium caseinates, of their mixtures with rennet casein and with caseinates, and of caseins of unknown type)	Ash (including P_2O_5)	ISO 5544 | IDF 89	Gravimetry (ashing at 825 °C)	I
Edible casein products	Lactose	ISO 5548 | IDF 106	Photometry (phenol and H_2SO_4)	IV
Edible casein products	Milkfat (total fat)	ISO 23319 | IDF 250	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Edible casein products	pH	ISO 5546 | IDF 115	Electrometry	II
Edible casein products	Milk protein (total N x 6.38 in dry matter)	ISO 5550 | IDF 78 and ISO 8968-1 | IDF 20-1	Calculation from dry matter content and protein content Gravimetry, drying at 102 °C and titrimetry (Kjeldahl)	I
Edible casein products	Sediment (scorched particles)	ISO 5739 | IDF 107	Visual comparison with standard discs, after filtration	IV
Edible casein products	Water[lxi]	ISO 5550 | IDF 78	Gravimetry (drying at 102 °C)	I
Emmental	Calcium > = 800 mg/100 g	ISO 8070 | IDF 119	Flame atomic absorption	III
Emmental	Calcium > = 800 mg/100 g	AOAC 2015.06 / ISO 21424 |IDF 243	ICP mass spectrometry	II
Emmental	Calcium > = 800 mg/100 g	AOAC 2011.14 / ISO 15151 | IDF 229	ICP emission spectroscopy	III
Emmental	Propionic acid	ISO/TS 19046-1I IDF/RM 233-1	Gas Chromatography -FID	IV
Emmental	Propionic acid	ISO/TS 19046-2I IDF/RM 233-2	Ion exchange chromatography - UV	IV
Evaporated milks	Milkfat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	I

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

Milk and milk products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Evaporated milks	Milk protein in MSNF[lxii]	ISO 6731 | IDF 21 and ISO 23318 | IDF 249 and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content and protein content, gravimetry, drying at 102 °C and gravimetry (Röse-Gottlieb) and titrimetry (Kjeldahl)	I
Evaporated milks	Milk solids[lxiii] **¡Error! Marcador no definido.**	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)[lxiv]	ISO 13580 | IDF 151	Gravimetry, drying at 102 °C	I
Fermented milks	Total acidity expressed as percentage of lactic acid	ISO/TS 11869 | IDF/RM 150	Potentiometry, titration to pH 8.30	IV
Fermented milks	*Lactobacillus acidophilus*	ISO 20128 | IDF 192	Colony count at 37 °C	I
Fermented milks - Yoghurt and yoghurt products	Quantification of *Lactobacillus delbrueckii* subsp *bulgaricus* and *Streptococcus thermophilus*	ISO 7889 | IDF 117	Colony count at 37 °C	I
Fermented milks - Yoghurt and yoghurt products	Identification of *Lactobacillus delbrueckii* subsp *bulgaricus* and *Streptococcus thermophilus*	ISO 9232 | IDF 146	Test for strain identification	I
Fermented milks	Sum of microorganisms constituting the starter culture (bacteria in fermented milk deriving (or originating) from starter culture)	ISO 27205 | IDF 149 (Annex A)	Colony count at 25 °C, 30 °C, 37 °C and 45 °C according to the starter organism in question	I
Fermented milks	Milkfat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	I

[lxii] Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.
[lxiii] See note lxii above.
[lxiv] See note lxii above.

Milk and milk products

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

[lxv] Water content excluding the crystallized water bound to lactose (generally known as moisture content).
[lxvi] See note lxv above.
[lxvii] Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method described in Appendix III is listed as Type IV.
[lxviii] See note lxv above.

Milk and milk products

Commodity	*Provisions*	*Method*	*Principle*	*Type*
Sweetened condensed milk	Milkfat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	I
Sweetened condensed milks (for products sweetened with sucrose only)	Milk protein in MSNF[lxix]	ISO 6734 | IDF 15 and ISO 23318 | IDF 249 and ISO 2911 | IDF 35 and ISO 8968-1 | IDF 20-1	Calculation from total solids content, fat content, sucrose and protein content, gravimetry, drying at 102 °C and polarimetry, gravimetry (Röse-Gottlieb), titrimetry (Kjeldahl)	I
Sweetened condensed milks	Solids[lxx]	ISO 6734 | IDF 15	Gravimetry, drying at 102 °C	I
Whey cheeses by coagulation	Milkfat	ISO 23319 | IDF 250	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Whey cheeses by coagulation	Milkfat in dry matter	ISO 23319 | IDF 250 and ISO 5534 | IDF 4	Calculation from fat content and dry matter content, gravimetry (Schmid-Bondzynski-Ratzlaff), gravimetry, drying at 102 °C	I
Whey cheeses by concentration (carbohydrate contents below 5%)	Milkfat (total fat)	ISO 23318 | IDF 59249	Gravimetry (Röse-Gottlieb)	I

[lxix] Milk total solids and milk solids-not-fat (MSNF) content include water of crystallization of lactose.
[lxx] See note lxix above.

Milk and milk products

Commodity	*Provisions*	*Method*	*Principle*	*Type*
Whey cheeses by concentration (does not dissolve completely in the ammonia, contains fats and fatty acid (FFA) in significant quantities or carbohydrate content > 5%)	Milkfat (total fat)	ISO 8262-3 |IDF 124-3	Gravimetry (Weibull-Berntrop)	I
Whey cheeses by concentration (for carbohydrate content under 5%)	Milkfat in dry matter (total fat in dry matter)	ISO 23318 | IDF 249 and ISO 2920 | IDF 58	Calculation from fat content and dry matter content, gravimetry (Röse-Gottlieb) gravimetry, drying at 88 °C	I
Whey cheeses by concentration (does no dissolve completely in the ammonia, contains FFA in significant quantities, or carbohydrate content >5%)	Milkfat in dry matter (total fat in dry matter)	ISO 8262-3 | IDF 124-3 and ISO 2920 | IDF 58	Calculation from fat content and dry matter contents, gravimetry (Weibull-Berntrop) gravimetry, drying at 88 °C	I
Whey powders	Ash	ISO 5545 | IDF 90	Gravimetry (ashing at 825 M °C)	IV
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	Milkfat	ISO 23318 | IDF 249	Gravimetry (Röse-Gottlieb)	I
Whey powders	Milk protein (total N x 6.38)	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Whey powders	Water[lxxi] (moisture)	ISO 5537 | IDF 26	Gravimetry (drying at 87 °C)	I

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

Table 5. Numeric performance criteria for methods of analysis for copper and iron in milk fat products

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

Table 6. Numeric performance criteria for copper and iron in edible casein products

Commodity	Provision	ML (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	RSDR (%)	Recovery	Minimum applicable range Minimum	Minimum applicable range Maximum	Examples of applicable methods that meet the criteria	Principle
Edible casein products	Copper	5	0.50	1.0	25.1	80–110%	3.1	6.9	AOAC 2015.06 / ISO 21424 | IDF 243 AOAC 2011.14 / ISO 15151 | IDF 229	ICP mass spectrometry ICP emission spectroscopy
									AOAC 985.35	Atomic absorption spectrophotometry
									ISO 5738 | IDF 76	Colorimetry (diethyldiethiocarbamate)
Edible casein products	Iron	20	2.0	4.0	20.4	80–110%	13,9	26.1	AOAC 2015.06 / ISO 21424 | IDF 243 AOAC 2011.14 / ISO 15151 | IDF 229	ICP mass spectrometry ICP emission spectroscopy
	Iron (in roller dried caseinates)	50	5.0	10.0	17.8	90–107%	36.7	63,3	AOAC 2015.06 / ISO 21424 | IDF 243 AOAC 2011.14 / ISO 15151 | IDF 229	ICP mass spectrometry ICP emission spectroscopy

Table 7. Numeric performance criteria for lead in butter, edible casein and whey powders

Commodity	Provision	ML (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	RSDR (%)	Recovery	Minimum applicable range Minimum	Minimum applicable range Maximum	Examples of applicable methods that meet the criteria	Principle
Butter, edible casein products and whey powders (secondary milk products)	Lead	0.02	0.004	0.008	≤ 44	60-115%	0.011	0.029	-	-

Natural mineral waters				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Natural mineral waters	Calcium	ISO 7980	Atomic absorption spectrophotometry	III
Natural mineral waters	Chloride	*Examination of Water Pollution Control.* WHO Pergamon Press (1982) Vol. 2, pp. 205-208		II
Natural mineral waters	Chloride	AOAC 973.51	Titrimetry (mercuric nitrate)	III
Natural mineral waters	Chloride	ISO 9297	Titrimetry	III
Natural mineral waters	Iron, dissolved	ISO 6332	Spectrophotometry	II
Natural mineral waters	Magnesium	ISO 6059	Titrimetry	II
Natural mineral waters	Magnesium	ISO 7980	Atomic absorption spectrophotometry	III
Natural mineral waters	Phenols	ISO 6439	Spectrophotometry	I
Natural mineral waters	Potassium	*Examination of Water Pollution Control.* WHO Pergamon Press (1982) Vol. 2, pp. 142-145		II
Natural mineral waters	Sodium	*Examination of Water Pollution Control.* WHO Pergamon Press (1982) Vol. 2 pp. 148-151		II

Natural mineral waters				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Natural mineral waters	Sodium	*Examination of Water Pollution Control.* WHO Pergamon Press (1982) Vol.2, pp. 151-152		III
Natural mineral waters	Sulphates	ISO 9280	Gravimetry	III
Natural mineral waters	Sulphide	*Handb. Spurenanal.* 1974		IV

Table 8a. Criteria applicable to health-related substances in the *Standard for Natural Mineral Waters* (CXS 108-1981)

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[lxxii] ICP-MS[lxxiii]
Cadmium	0.003	0.0017 – 0.0043	0.0006	0.0012	44	40 - 120	ISO 11885 ISO 17294-2 ISO 15586 ISO 5961 (Section 3)	ICP-OES ICP-MS GF-AAS AAS

[lxxii] Total Boron is determined.
[lxxiii] See note lxxii above.

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
							EPA 200.8	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 14403 ISO 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
Lead	0.01	0.006 – 0.014	0.002	0.004	44	60-115	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	AAS Enrichment by amalgamation (III) AFS

Provision	ML (mg/L)	Min. applicable range (mg/L)	LOD (mg/L)	LOQ (mg/L)	Precision RSDR (%) No more than	Recovery (%)	Suggested methods meeting the criteria	Principle
							ISO 16590 EPA 200.8	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
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
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

Table 8b. 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
PCB	-	>15 ng/L		< 20	70-130	AOAC 990.06	GC ECD
Pesticide (organochlorine)	-	> 15 ng/L		< 20	70-130	AOAC 990.06	GC ECD
PAH	-	0.005 µg/L 0.04 µg/L 0.005 µg/L		< 10 < 18 < 19	80-110 80-110 80-100	ISO 17993 ISO 7981-1 ISO 7981-2	HPLC FD TLC HPLC

Processed fruits and vegetables				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Processed fruits and vegetables (also includes jams, jellies, marmalades, pickled cucumbers, mango chutney, coconut milk and coconut cream)	Benzoic acid	NMKL 124	Liquid chromatography (UV)	II
Processed fruits and vegetables (jams, jellies, marmalades, pickled cucumbers, mango chutney, coconut milk and coconut cream)	Benzoic acid	AOAC 983.16	Gas chromatography	III
Processed fruits and vegetables (canned strawberries, pickled cucumbers, preserved tomatoes, canned citrus fruits, certain canned vegetables)	Calcium	AOAC 968.31	Complexometry/Titrimetry	II
Processed fruits and vegetables	Drained weight	AOAC 968.30 (Codex general method)	Gravimetry (sieving)	I
Processed fruits and vegetables	Fill of glass containers	ISO 8106	Gravimetry	I

Processed fruits and vegetables				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Processed fruits and vegetables	Fill of metal containers	ISO 90-1	Gravimetry	I
Processed fruits and vegetables	Lead	AOAC 972.25 (Codex general method)	AAS (flame absorption)	III
Processed fruits and vegetables	Packing medium canned berry fruits (raspberry, strawberry)	AOAC 932.12 ISO 2173	Refractometry	I
Processed fruits and vegetables (pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney, and aqueous coconut products)	pH	ISO 1842	Potentiometry	IV
Canned bamboo shoots	pH	AOAC 981.12	Potentiometry	IV
Processed fruits and vegetables (pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney and aqueous coconut products)	pH	AOAC 981.12	Potentiometry	III

Processed fruits and vegetables				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Processed fruits and vegetables (pickled cucumbers, table olives, processed tomato concentrates, preserved tomatoes, mango chutney, and aqueous coconut products)	pH	NMKL 179	Potentiometry	II
Processed fruits and vegetables (pickled cucumbers, processed tomato concentrates, preserved tomatoes, canned applesauce, jams, jellies and marmalades, mango chutney, and certain canned fruit)	Soluble solids (packing medium)	ISO 2173	Refractometry	I
Processed fruits and vegetables (jams, jellies, marmalades, pickled cucumbers)	Sorbates	AOAC 983.16	Gas chromatography (Flame ionization)	III
Processed fruits and vegetables (jams, jellies, marmalades, pickled cucumbers)	Sorbates	NMKL 124	Liquid chromatography (UV)	II
Processed fruits and vegetables	Tin	AOAC 980.19 (Codex general method)	Atomic absorption spectrophotometry (flame)	II

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

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

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

Processed fruits and vegetables				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Preserved tomatoes	Drained weight	AOAC 968.30	Gravimetry (sieving) Note: Use a No. 14 screen instead of '7/16' or No. 8	I
Preserved tomatoes	Mould count	AOAC 965.41	Howard mould count	I
Processed tomato concentrates	Lactic acid	EN 12631	Spectrometry (enzymatic determination)	II
Processed tomato concentrates	Mineral impurities (sand)	ISO 762	Gravimetry	IV
Processed tomato concentrates	Mould count	AOAC 965.41	Howard mould count	I
Processed tomato concentrates	Natural tomato soluble solids	AOAC 970.59	Refractometry	I
Processed tomato concentrates	Sodium chloride	AOAC 971.27 (Codex general method)	Potentiometry	II
Processed tomato concentrates	Tomato soluble solids	AOAC 970.59	Refractometry	I
Raisins	Mineral impurities	ISO 762	Ashing	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 (flame ionization)	II
Raisins	Sulphur dioxide	AOAC 963.20	Colorimetry	II
Table olives	Drained weight	AOAC 968.30 (Codex general method for processed fruits and vegetables)	Gravimetry (sieving)	I
Table olives	Fill of glass containers	ISO 8106	Gravimetry	I

Processed fruits and vegetables				
Commodity	***Provision***	***Method***	***Principle***	***Type***
Table olives	Fill of metal containers	ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)	Gravimetry	I
Table olives	pH of brine	NMKL 179 (Codex general method for processed fruits and vegetables)	Potentiometry	II
Table olives	pH of brine	AOAC 981.12 (Codex general method for processed fruits and vegetables)	Potentiometry	III
Table olives	pH of brine	ISO 1842	Potentiometry	IV
Table olives	Salt in brine	AOAC 971.27 | NMKL 178 (Codex general method)	Potentiometry	II
Table olives	Tin	NMKL 190 | EN 15764	Atomic absorption spectrophotometry (flame)	II

Processed meat and poultry products and soups and broths				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
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	III
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)	II
Processed meat and poultry products	Nitrates	ISO 3091	Colorimetry (cadmium reduction)	II
Processed meat and poultry products	Nitrites	ISO 2918	Colorimetry	IV
Processed meat and poultry products	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Processed meat and poultry products	Nitrogen/protein	ISO 937	Titrimetry	II
Bouillons and consommés (soups and broths)	Amino nitrogen	AIIBP Method No 2/7	Volumetry (modified Van Slyke)	II
Bouillons and consommés (soups and broths)	Creatinine	AIIBP Method No 2/5	HPLC	II
Bouillons and consommés (soups and broths)	Nitrogen, total	AOAC 928.08	Kjeldahl	II
Bouillons and consommés (soups and broths)	Sodium chloride	AIIBP Method No 2/4	Potentiometric titration (chloride expressed as sodium chloride)	II
Canned corned beef	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Canned corned beef	Nitrites, potassium and/or sodium salt	AOAC 973.31 (Codex general method)	Colorimetry	II

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

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

Quick-frozen fruits and vegetables				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Quick-frozen fruits and vegetables(non-glazed)	Net weight	AOAC 963.26	Weighing	I
Quick-frozen fruits and vegetables	Thawing procedure	See Appendix X	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				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
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 XI	Cooking	I
Quick-frozen French-fried potatoes	Moisture	AOAC 984.25	Gravimetry (convection oven)	I
Quick-frozen green and wax beans	Tough strings	See Appendix IV	Stretching	I
Quick-frozen peas	Solids, alcohol insoluble	See Appendix XII	Gravimetry	I
Quick-frozen spinach	Dry matter, Sodium chloride-free	See Appendix XIII	Weighing	I

Spices and culinary herbs				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Cumin	Moisture	ISO 939	Distillation	I
Cumin	Total ash	ISO 928	Gravimetry	I
Cumin	Acid-insoluble ash	ISO 930	Gravimetry	I
Cumin	Volatile oils	ISO 6571	Distillation/Volumetric	I
Cumin	Extraneous vegetable matter	ISO 927	Visual examination/Gravimetry	I
Cumin	Foreign matter	ISO 927	Visual examination/Gravimetry	I

Spices and culinary herbs				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Cumin	Insect damage	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual, FDA) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV
Cumin	Mammalian excreta	Macroanalytical Procedure Manual USFDA technical bulletin V.39 B (for whole)	Visual examination	IV
Cumin	Mammalian excreta	AOAC 993.27 (for ground)	Enzymatic detection method	IV
Cumin	Mould damage	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual, FDA) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV
Dried oregano	Moisture	ISO 939	Distillation	I
Dried oregano	Total ash (dry weight basis)	ISO 939 and ISO 928	Calculation from moisture and ash Distillation and gravimetry	I
Dried oregano	Acid-insoluble ash (dry weight basis)	ISO 939 and ISO 930	Calculation from moisture and ash Distillation and gravimetry	I
Dried oregano	Volatile oils (dry weight basis)	ISO 939 and ISO 6571	Calculation from moisture and volatile oils distillation and distillation	I
Dried oregano	Extraneous matter	ISO 927	Visual examination followed by gravimetry	I
Dried oregano	Foreign matter	ISO 927	Visual examination followed by gravimetry	I

Spices and culinary herbs				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Dried oregano	Mammalian excreta other excreta	Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (for whole) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavours-and-crude-drugs#v32	Visual examination	IV
Dried oregano	Whole dead insect	ISO 927	Visual examination	IV
Dried oregano	Whole dead insect	MPM V-8 Spices, Condiments, Flavours and Crude Drugs A. General methods for spices, herbs and botanicals (V 32) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavours-and-crude-drugs#v32	Visual examination	IV
Dried oregano	Mould visible	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) https://www.fda.gov/food/laboratory-methods-food/mpm-v-8-spices-condiments-flavours-and-crude-drugs#v32	Visual examination	IV
Dried oregano	Insect damage	ISO 927	Visual examination	I
Thyme	Moisture	ISO 939	Distillation	I
Thyme	Total ash	ISO 928	Gravimetry	I
Thyme	Acid-insoluble ash	ISO 930	Gravimetry	I
Thyme	Volatile oils	ISO 6571	Distillation/Volumetric	I
Thyme	Extraneous vegetable matter	ISO 927	Visual examination/Gravimetry	I

Spices and culinary herbs				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Thyme	Foreign matter	ISO 927	Visual examination/Gravimetry	I
Thyme	Insect damage	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual, FDA) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV
Thyme	Mammalian excreta	Macroanalytical procedure manual USFDA technical bulletin V.39 B (for whole)	Visual examination	IV
Thyme	Mammalian excreta	AOAC 993.27 (for ground)	Enzymatic detection method	IV
Thyme	Mould damage	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual, FDA) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV
Black and white pepper	Bulk density	ISO 959-1 Annex B (black) ISO 959-2 Annex A (white)	Gravimetry	IV
Black pepper	Light berries	ISO 959-1 Annex A (black)	Flotation	IV
Black, white and green pepper	Extraneous vegetable matter	ISO 927	Visual examination/Gravimetry	I
Black, white and green pepper	Foreign matter	ISO 927	Visual examination/Gravimetry	I

Spices and culinary herbs				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
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
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	I
Black, white and green pepper	Non-volatile ether extract	ISO 1108	Soxhlet extraction	I
Black, white and green pepper	Volatile oils	ISO 6571	Distillation	I
Black, white and green pepper	Piperine content	ISO 5564	Spectrophotometry	I

Spices and culinary herbs				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Black, white and green pepper	Acid-insoluble ash	ISO 930	Gravimetry	I
Black, white and green pepper	Crude fibre	ISO 5498	Gravimetry	I

Sugars and honey				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Honey	Acidity	MAFF Validated Method V19 *J. Assoc. Public Analysts* (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	-	-
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	I
Sugars (dextrose anhydrous and dextrose monohydrate)	D-Glucose	ISO 5377	Titrimetry	I
Sugars (dextrose anhydrous and dextrose monohydrate)	Solids, total	ISO 1741	Gravimetry (vacuum oven)	I

Sugars and honey				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
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)	pH	ICUMSA GS 1/2/3/4/7/8-23	Potentiometry	I
Sugars (fructose)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	I
Sugars (fructose)	D-Fructose	ISO 10504	Liquid chromatography (refractive index detection)	II
Sugars (fructose)	D-Glucose	ISO 10504	Liquid chromatography (refractive index detection)	II
Sugars (fructose)	Loss on drying	ISO 1742	Gravimetry	I
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)	I
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	II
Sugars (lactose)	Loss on drying	USP General Chapter 731	Gravimetry (drying at 120 °C for 16 h)	I
Sugars (lactose)	pH	ICUMSA GS 1/2/3/4/7/8-23	Potentiometry	I
Sugars (plantation and mill white sugar)	Colour	ICUMSA GS 9/1/2/3-8	Photometry	I
Sugars (plantation or mill white sugar)	Conductivity ash	ICUMSA GS 1/3/4/7/8-13	Conductimetry	I

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

Sugars and honey				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Sugars (soft white sugar and soft brown sugar)	Sucrose plus invert sugar	ICUMSA GS 4/3-7	Titrimetry	I
Sugars (soft brown sugar)	Sulphated ash	ICUMSA GS 1/3/4/7/8-11	Gravimetry	I
Sugars (soft white sugar and soft brown sugar)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II
Sugars (soft white sugar)	Colour	ICUMSA GS 2/3-9	Photometry	I
Sugars (white sugar)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	I
Sugars (white sugar)	Invert sugar	ICUMSA GS 2/3-5	Titrimetry	I
Sugars (white sugar)	Loss on drying	ICUMSA GS 2/1/3-15	Gravimetry	I
Sugars (white sugar)	Polarization	ICUMSA GS 2/3-1	Polarimetry	II
Sugars (white sugar)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II

Miscellaneous products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Chilli sauce	pH	NMKL 179 (Codex general method) / AOAC 981.12	Potentiometry	II
Chilli sauce	pH	AOAC 981.12 (Codex general method)	Potentiometry	III
Chilli sauce	Fill of containers	CAC/RM 46 (see Appendix II) (Codex general method) (for glass container)	Gravimetry	I
Cooked rice wrapped in plant leaves	Peroxide value	ISO 3960 / AOCS Cd 8b-90 Extraction of oils from product (see Appendix XIV)	Titrimetry	IV
Date paste	Moisture	AOAC 934.06	Gravimetry	I
Date paste	Mineral impurities	ISO 762	Gravimetry	I
Date paste	Ash	AOAC 940.26	Gravimetry	I

Miscellaneous products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Date paste	Acid soluble ash	AOAC 900.02D	Gravimetry, calculation	I
Dried fruits	Identification of defects	Described in the standard	Visual examination	I
Dried fruits (except prunes and raisins)	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	I
Dried meat	Chloride as sodium chloride (≥ 1.0%)	ISO 1841-1	Titrimetry (Volhard method)	III
Dried meat	Chloride as sodium chloride (≥ 0.25%)	ISO 1841-2	Titrimetry (potentiometry)	II
Dried meat	Ash	ISO 936	Gravimetry	I
Dried meat	Water activity	ISO 18787	Electrometry	II
Dried meat	Moisture content	AOAC 950.46B	Gravimetry	I
Dried meat	Protein* (*nitrogen-to-protein conversion factor = 6.25)	ISO 937	Calculation and titrimetry	I
Dried meat	Total fat	ISO 1443	Gravimetry	I
Edible cassava flour	Ash	ISO 2171 and ISO 712	Calculation from moisture and gravimetry (incineration at 550 ^{o}C)	I
Edible cassava flour	Fibre, crude	ISO 5498	Gravimetry	I
Edible cassava flour	Granularity	ISO 2591-1	Sieving	I
Edible cassava flour	Moisture	ISO 712	Gravimetry (oven drying at 98 – 100 ^{o}C)	I
Fermented noni fruit juice	Brix value (soluble solids)	AOAC 983.17 / EN 12143 / IFUMA 8 / ISO 2173	Refractometry	IV
Fermented noni fruit juice	Ethanol	AOAC 2017.07	Enzymatic determination	IV
Fermented noni fruit juice	Ethanol	IFUMA 52	Enzymatic determination	IV

Miscellaneous products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Fermented noni fruit juice	Ethanol	AOAC 2016.12	Headspace GC-FID	IV
Fermented noni fruit juice	Identification of scopoletin	Method described in Appendix XV, Part A	Solid phase extraction and thin layer chromatography	IV
Fermented noni fruit juice	Identification of deacetylasperulosidic acid	Method described in Appendix XV, Part B	Thin layer chromatography	IV
Fermented noni fruit juice	pH value	NMKL 179 / AOAC 981.12	Potentiometry	II
Fermented soybean paste	Total nitrogen	AOAC 984.13	Kjeldahl	I
Fermented soybean paste	Amino nitrogen	AOAC 920.154 on the conditions specified in the standard[lxxiv]	Volumetry	I
Fermented soybean paste	Moisture	AOAC 934.01 (≤ 70°C, ≤ 50 mm Hg)	Gravimetry	I
Food grade salt	Arsenic	EuSalt/AS 015	ICP-OES	IV
Food grade salt	Cadmium	EuSalt/AS 015	ICP-OES	III
Food grade salt	Cadmium	EuSalt/AS 014	Atomic absorption spectrophotometry	IV
Food grade salt	Calcium and magnesium	ISO 2482	Complexometric titrimetry	II
Food grade salt	Calcium and magnesium	EuSalt/AS 009	Flame atomic absorption spectrometry	III
Food grade salt	Calcium and magnesium	EuSalt/AS 015	ICP-OES	III
Food grade salt	Copper	EuSalt/AS 015	ICP-OES	III
Food grade salt	Insoluble matter	ISO 2479	Gravimetry	II

[lxxiv] **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_3-free H_2O 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				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Food grade salt	Iodine	EuSalt/AS 002	Titrimetry using sodium thiosulphate	II
Food grade salt	Iodine	EuSalt/AS 019	ICP-OES	III
Food grade salt	Iodine	WHO/UNICEF/ICCIDD method[lxxv] Only applicable to a product which has been fortified with iodate	Titrimetry using sodium thiosulphate	IV
Food grade salt	Loss on drying	ISO 2483	Gravimetry (drying at 110°C)	I
Food grade salt	Mercury	EuSalt/AS 012	Cold vapour atomic absorption spectrophotometry	IV
Food grade salt	Potassium	EuSalt/AS 008	Flame atomic absorption spectrophotometry	II
Food grade salt	Potassium	EuSalt/AS 015	ICP-OES	III
Food grade salt	Sodium chloride	Described in the standard	Calculation	I
Food grade salt	Sulphate	ISO 2480	Gravimetry	II
Food grade salt	Sulphate	EuSalt/AS 015	ICP-OES	III
Food grade salt	Sulphate	EuSalt/AS 018	Ion chromatography	III
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	I

[lxxv] 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

Commodity	*Provisions*	*Method*	*Principle*	*Type*
Gari	Ash	ISO 2171 and ISO 712	Calculation from moisture and Gravimetry (incineration at 550 °C)	I
Gari	Fibre, crude	ISO 5498 and ISO 712	Gravimetry (separation)	I
Gari	Granularity	ISO 2591-1	Sieving	I
Gari	Moisture	ISO 712	Gravimetry (oven drying at 130 – 133 °C)	I
Gari	Total acidity	ISO 7305 and ISO 712	Titrimetry (ethanol extraction)	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	I
Ginseng products	Solids	AOAC 925.45 B (dried ginseng) calculated by subtracting the content of water from 100% Quantity of sample: 2 g	Calculation	I
Ginseng products	Ash	AOAC 923.03 AACC Intl 08-01.01	Gravimetry	I
Ginseng products	Water-insoluble solids	Described in the standard (Annex I)	Gravimetry	I
Ginseng products	Water-saturated n-butanol extracts	Described in the standard (Annex II)	Gravimetry	I
Ginseng products	Identification of ginsenosides Rb1 and Rf	Described in the standard (Annex III)	TLC or HPLC	IV
Gochujang	Capsaicin	Journal of AOAC International Vol. 91 No. 2, 2008, pp 387-391	HPLC-Fluorescence	IV

Miscellaneous products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Gochujang	Capsaicin	Journal of AOAC International Vol. 91 No. 2, 2008, pp 387-391	Gas chromatography-FID	IV
Gochujang	Crude protein	AOAC 984.13 (Nitrogen conversion factor: 6.25)	Titrimetry, Kjeldahl	I
Gochujang	Moisture	AOAC 945.43	Gravimetry	I
Guideline level for acrylonitrile	Acrylonitrile	AOAC 985.13	Gas chromatography	II
Guideline levels for vinyl chloride monomer	Vinyl chloride monomer	ISO 6401	Gas chromatography	II
Guideline levels for vinyl chloride monomer	Vinyl chloride monomer	Commission Directive 81/432/EEC O.J. No. L.167, p. 6, 24.6.81	Gas chromatography ("headspace")	III
Guidelines for nutrition labelling	Polyunsaturated fatty acids	AOCS Ce 1h-05[lxxvi]	Gas-liquid chromatography	II
Guidelines for nutrition labelling	Saturated fat	AOAC 996.06; or AOCS Ce 1h-05	Gas-liquid chromatography	II
Guidelines for nutrition labelling	Saturated fatty acids	AOCS Ce 1h-05	Gas-liquid chromatography	II
Harissa	Acidity	ISO 750	Titrimetry	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

[lxxvi] Can also be used to measure trans unsaturated fatty acids.

Miscellaneous products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
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[lxxvii]	Titrimetry	IV
Humus with tehena	Salt content	AOAC 971.27 NMKL 178	Potentiometry	II
Humus with tehena	Total acidity	AOAC 925.53	Titrimetry	I

[lxxvii] http://www.starch.dk/isi/methods/28luff.htm

Miscellaneous products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Kava products for use as a beverage when mixed with water	Moisture	AOAC 925.45	Gravimetry	I
Mixed zaatar	Sodium chloride (dry weight basis)	ISO 939 and AOAC 971.27	Calculation by moisture and ash Distillation and titrimetry	I
Mixed zaatar	Moisture	ISO 939	Distillation	I
Mixed zaatar	Acid-insoluble ash (dry weight basis)	ISO 939 and AOAC 941.12 (corrected for moisture by ISO 930)	Calculation by moisture and ash Distillation and gravimetry, Furnace, 550 ^{0}C	I
Mixed zaatar	Extraneous matter	ISO 927	Visual examination Gravimetry	I
Mixed zaatar	Foreign matter	ISO 927	Visual examination Gravimetry	I
Mixed zaatar	Insects/Insect fragments	ISO 927	Visual examination	IV
Mixed zaatar	Insects/Insect fragments	AOAC 969.44	Visual examination	IV
Mixed zaatar	Insects/Insect fragments	AOAC 975.49	Visual examination	IV
Mixed zaatar	Mould damage	Method V-8 Spices, Condiments, Flavours and Crude Drugs (Macroanalytical Procedure Manual, FDA, Technical Bulletin Number 5)	Visual examination	IV

Miscellaneous products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Mixed zaatar	Mammalian excreta	Macroanalytical Procedure Manual, USFDA, Technical Bulletin V.39 B (for whole)	Visual examination	IV
Mixed zaatar	Mammalian excreta	AOAC 993.27 (for ground)	Enzymatic detection method	IV
Non-fermented soybean products	Moisture content	AOAC 925.09 | AACCI 44-40.01	Gravimetry (vacuum oven)	I
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

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

Miscellaneous products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
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
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	I
Laver products	Moisture content	AOAC 925.45B	Gravimetry, drying at atmospheric pressure	IV
Laver products	Acidity: acid value for the extracted oil	See Appendix XVI and ISO 660 | AOCS Cd 3d-63	Extraction of oil Titrimetry	I
Laver products	Moisture content	AOAC 925.45	Gravimetry, drying at atmospheric pressure	I
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	I
Unrefined shea butter	Relative density	AOCS Cc 10c-95/ ISO 6883	Pycnometry	I

Miscellaneous products				
Commodity	***Provisions***	***Method***	***Principle***	***Type***
Unrefined shea butter	Saponification value	ISO 3657 AOCS Cd 3d-25	Titrimetry	I
Unrefined shea butter	Iodine 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
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

Table 9. Numeric performance criteria for lead and cadmium in foods

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Infant formula, formula for special medical purposes intended for infants and follow-up formula	lead	0.01	0.006 - 0.014	0.002	0.004	44	60-115%		
Milk	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%		
Secondary milk products	lead	0.02	0.011 - 0.029	0.004	0.008	44	60-115%		
Fruit juices, except juices exclusively from berries and other small fruits	lead	0.03	0.017 - 0.043	0.006	0.012	44	60-115%		
Fat spreads and blended spreads	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%		
Grape juice	lead	0.04	0.022 - 0.058	0.008	0.016	44	60-115%		
Canned chestnuts and canned chestnuts puree	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Fruit juices obtained exclusively from berries and other small fruits, except grape juice	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Fruiting vegetables, except fungi and mushrooms	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Preserved tomatoes	lead	0.05	0.028 - 0.072	0.010	0.020	44	60-115%		
Edible fats and oils	lead	0.08	0.045 - 0.115	0.016	0.032	44	60-115%		
Berries and other small fruits, except cranberry, currant, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Brassica vegetables, except kale and leafy Brassica vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Bulb vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Canned fruits	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Canned vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Fruits, except cranberry, currants, and elderberry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Legume vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Meat and fat of poultry	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Meat of cattle, pigs and sheep	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pickled cucumbers (cucumber pickles)	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Poultry, edible offal of	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pulses	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Root and tuber vegetables	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Wine from grapes harvested after July 2019	lead	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Fortified/liqueur wine from grapes harvested after 2019	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%		
Pig, edible offal of	lead	0.15	0.05 - 0.25	0.015	0.03	43	80-110%		
Cattle, edible offal of	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Cereal grains, except buckwheat, cañihua and quinoa	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Cranberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Currants	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Elderberry	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Wine (wine and fortified/ liqueur wine) made from grapes harvested before July 2019	lead	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Fish	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Fresh farmed mushrooms (common mushrooms [*Agaricus bisporous*], shiitake mushrooms (*Lentinula edodes*), and oyster mushrooms [*Pleurotus ostreatus*])	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Leafy vegetables, except spinach	lead	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Jams, jellies, and marmalades	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Mango chutney	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Table olives	lead	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Salt, food grade	lead	1	0.5 - 1.5	0.1	0.2	32	80-110%		
Brassica vegetables, except Brassica leafy vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Bulb vegetables	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Fruiting vegetables, except tomatoes and edible fungi	cadmium	0.05	0.03 - 0.07	0.01	0.02	44	60-115%		
Cereal grains, except buckwheat, cañihua, quinoa, wheat and rice	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Legume vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Pulses, except soya bean (dry)	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Root and tuber vegetables, except celeriac	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		
Stalk and stem vegetables	cadmium	0.1	0.03 - 0.17	0.01	0.02	44	80-110%		

Commodity	Provision	ML (mg/kg)	Method performance criteria						
			Minimum applicable range (mg/kg)	Limit of detection (LOD) (mg/kg)	Limit of quantification (LOQ) (mg/kg)	Precision (RSDR) (%) No more than	Recovery (%)	Example of applicable methods that meet the criteria	Principle
Leafy vegetables	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Wheat (common wheat, durum wheat, spelt and emmer)	cadmium	0.2	0.08 - 0.32	0.02	0.04	41	80-110%		
Chocolate containing or declaring < 30% total cocoa solids on a dry matter basis	cadmium	0.3	0.13 - 0.47	0.03	0.06	38	80-110%		
Rice, polished	cadmium	0.4	0.18 - 0.62	0.04	0.08	37	80-110%		
Salt, food grade	cadmium	0.5	0.23 - 0.77	0.05	0.10	36	80-110%		
Chocolate containing or declaring ≥ 30% to < 50% total cocoa solids on a dry matter basis	cadmium	0.7	0.35 - 1.05	0.07	0.14	34	80-110%		
Chocolate containing or declaring ≥ 50% to < 70% total cocoa solids on a dry matter basis, including sweet chocolate, gianduja chocolate, semi – bitter table chocolate, vermicelli chocolate/	cadmium	0.8	0.40 - 1.20	0.08	0.16	33	80-110%		

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

PART B – METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

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

Appendix I

PART A – EXTRACTION OF OIL FROM INSTANT NOODLES

1. Extraction of oil from instant noodles

1.1 Apparatus

(a) Rotary evaporator

(b) Water bath

1.2 Preparation of test sample

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

1.3 Extraction

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

PART B – DETERMINATION OF ACID VALUE

2. Determination of acid value

2.1 Definition and principle

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

2.2 Apparatus

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

2.3 Reagents

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

Standardization:

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

Calculation:

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

(b) Alcohol-ether mixture: equal volumes ethanol (99.5%) and ether.

(c) Phenolphthalein solution: 1% in alcohol.

2.4 Titration

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

2.5 Calculation

Calculate using following equation:

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

2.5.1 *Definition and principle*

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

2.5.2 *Apparatus*

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

2.5.3 *Reagents*

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

Standardization:

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

Calculation:

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

(e) Alcohol-ether mixture: equal volumes ethanol (99.5%) and ether.

(f) Phenolphthalein solution: 1% in alcohol.

2.5.4 *Titration*

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

2.5.5 *Calculation*

Calculate using following equation:

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

PART C - DETERMINATION OF MOISTURE

3. Determination of moisture

3.1 Apparatus

(a) Aluminum dish: diameter ≥ 55 mm, height ≥ 15 mm, and with inverted tight-fitting lid.

(b) Air-oven: with control accuracy ±1 °C.

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

3.2 Preparation of test sample

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

3.3 Determination

3.3.1 *Fried noodles*

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

3.3.2 *Non-fried noodles*

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

3.4 Calculation

Calculate using the following equation:

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

Appendix II

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

1. SCOPE

This method applies to glass containers.

2. DEFINITION

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

3. PROCEDURE

3.1 Select a container which is undamaged in all respects.

3.2 Wash, dry and weigh the empty container.

3.3 Fill the container with distilled water at 20 °C to the level of the top thereof, and weigh the container thus filled.

4. CALCULATION AND EXPRESSION OF RESULTS

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

Appendix III

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

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

DESCRIPTION OF THE METHOD: DETERMINATION OF MOISTURE

1. SCOPE

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

2. DEFINITION

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

3. PRINCIPLE

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

4. EQUIPMENT

Common laboratory equipment and, in particular, the following.

4.1 **Analytical balance,** capable of weighing with a precision of 1 mg, with a minimum resolution of 0.1 mg.

4.2 **Drying oven,** with good ventilation, as far as possible with forced ventilation, capable of being thermostatically maintained at (102 ± 2) °C throughout the workspace, with a temperature controller.

4.3 **Desiccator,** with freshly dried silica gel with hygrometric indicator or another effective desiccant.

4.4 **Flat-bottomed dishes,** approximately 25 mm deep, approximately 50 mm in diameter, and made of an appropriate material (for example, glass, stainless steel, nickel, or aluminium), fitted with tight-fitting, removable lids easily.

5. SAMPLING

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

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

6. TEST SAMPLE PREPARATION

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

7. PROCEDURE

7.1 Preparation of the dish

7.1.1 Heat the uncovered capsule and its lid (4.4) in the oven (4.2) controlled at (102 ± 2) °C, for 1 h.

7.1.2 Transfer the capped dish to the desiccator (4.3), allow it to cool to room temperature in the balance room, and weigh (4.1) to the nearest 0.1 mg.

7.2 Test sample

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

7.3 Determination

7.3.1 Uncover the capsule and place it together with the lid in the oven (4.2), controlled at (102 ± 2) °C for 2 hrs.

7.3.2 Replace the cap, transfer the capped dish to the desiccator, allow to cool to balance room temperature, and weigh to the nearest 0.1 mg.

7.3.3 Uncover the capsule and heat again, along with its lid, on the oven for 1 h. Then repeat operation 7.3.2.

7.3.4 Repeat this process until the difference in mass between two successive weightings does not exceed 0.5 mg. Record the lowest mass.

8. CALCULATION AND EXPRESSION OF RESULTS

8.1 Calculation

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

$$\text{moisture} = \frac{(m_1 - m_2) \times 100}{(m_1 - m_0)}$$

where,

m_0 is the mass, in grams, of the dish and lid (Section 7.1.2)

m_1 is the mass, in grams, of the dish, lid and test sample before drying (Section 7.2.1)

m_2 is the mass, in grams, of the dish, lid and test sample after drying (Section 7.3.4)

8.2 Expression of test results

Express the sample results to two decimal places.

Appendix IV

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

1. DEFINITION

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

2. PRINCIPLE

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

3. APPARATUS

3.1 Weighted clamp

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

4. PROCEDURE

4.1 From the drained product select a representative sample of not less than 285 g. Record the weight of this test sample.

4.2 Break the individual bean units and set aside those that show evidence of tough strings. Remove the strings from the pods and retain the pod material for weighing.

4.3 Fasten the clamp assembly to one end of the string. Grasp the other end of the string with the fingers (a cloth may be used to aid in holding the string) and lift gently.

4.4 If the string supports the 250 g assembly for at least five seconds consider the bean unit as containing tough string. If the string breaks in less than five seconds, retest the broken parts that are 13 mm or longer to determine if such portions are tough.

4.5 Weigh the bean units which contain tough strings.

5. CALCULATION AND EXPRESSION OF RESULTS

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

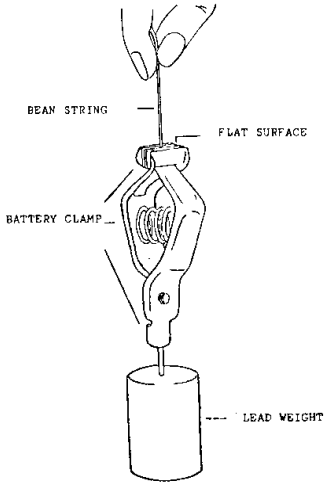


Figure 1 - Tough string tester for green or wax beans

Appendix V

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

1. DEFINITION

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

2. REAGENTS AND MATERIALS

2.1 Compound microscope: -

- 100 to 250 magnification.
- Phase contrast.

2.2 Microscope slide and cover glass.

2.3 Spatula.

2.4 Ethanol - 95% (v/v).

2.5 Glycerine.

3. PROCEDURE

3.1 Preparing mount

3.1.1 Remove a small portion of the endosperm and place on glass slide;

3.1.2 Using a spatula grind the material with 95% (v/v) ethanol;

3.1.3 Add a drop of glycerine, place cover glass on material and examine under microscope.

3.2 Identification

3.2.1 Starch granules of the wrinkled-seeded types (garden peas, sweet) show up as clear cut, well defined, generally spherical particles.

3.2.2 Starch granules of the smooth-seeded types (round, early, Continental) show up as an amorphous mass with no well-defined geometric shape.

Appendix VI

DETERMINATION OF INTERNAL DEFECTS: DATES

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

Appendix VII

EXTRANEOUS VEGETABLE MATTER IN DESICCATED COCONUT

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

Appendix VIII

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

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

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

Appendix IX

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

METHOD 1

(1) This method may be used for all sub-types of pickles. Use a 4 to 8 litre size can with an overflow spout constructed from 0.5 cm to 1 cm inside diameter metal tubing (see Figure 1). The tubing is soldered to an opening in the side of the can 2 cm to 3 cm from the bottom and is bent upward parallel to sides. The tube is bent over and slightly downward from the can at the upper end to form a spout about 4 cm below the top of the can. The lower tip end of the spout is lower than the inside lower curve of the spout (point A). The upper tip end of the spout is higher than the inside lower curve of the spout (point A). The upper tip end of the spout is slightly shorter than the lower tip end of the spout. A brace near the top of the can holds the tubing firmly in place. A woven wire basket made from screen wire with about eight meshes to the inch with a handle is used for lowering the pickle ingredient into the overflow can.

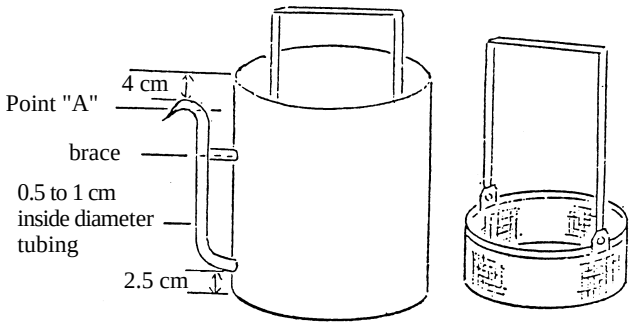
(2) Place overflow can on a level table so that overflow will discharge into a sink. Fill the overflow can with water at room temperature (approximately 20 °C or 68 °F). Place the empty basket into the filled overflow can.

(3) When overflow ceases, place a beaker or graduated cylinder under the spout.

(4) Remove basket and place drained pickle ingredient (at room temperature) in basket and lower slowly into the overflow can. When overflow ceases, measure the volume of the fluid overflow. The percent volume of pickle ingredient (volume occupied) is calculated as follows:

$$\frac{\textit{Overflow Volume}}{\textit{Total capacity (volume) of container (see Method E)}} \textit{ x100 = percent volume of pickle ingredient}$$

Figure 1



Overflow can

METHOD 2

(1) Use water to partially fill a graduated cylinder (or other technical measuring device) large enough so that the pickle ingredient from one container may be completely submerged.

(2) Prior to adding the pickle ingredient, record the volume of water in the partially filled cylinder.

(3) Add all the drained pickle ingredient from one container so that it is entirely submerged.

(4) Measure the volume of liquid and pickle ingredient after submersion of pickle ingredient.

(5) Subtract the value in (2) from the value in (4) to obtain the pickle ingredient displacement.

(6) To determine Volume Fill, calculate:

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

METHOD 3

(1) Remove and collect the packing medium from the container for other quality determinations.

(2) With the pickle ingredient in the container fill it to capacity with water.

(3) Drain, collect and measure the water.

To determine 'volume fill', calculate:

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

Where,

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

Appendix X

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 (Appendix XI, CAC/RM 33-1970).

2.2 Where a particular quick-frozen fruit or vegetable requires special treatment not fully covered by this general procedure for examination, the treatment outlined in the appropriate Codex commodity standard should be followed.

3. DEFINITIONS

3.1 Thawing of quick-frozen fruits and vegetables for the purpose of this examination procedure, means subjecting the product to controlled conditions of temperature until the product is sufficiently free from ice crystals so that the individual units can be readily separated and handled.

3.2 Air thawing means thawing of the product in unopened container by exposure to air of an ambient temperature in free or forced ventilation.

3.3 Water thawing by indirect contact, means thawing of the product in a tightly sealed container by immersion in water, stationary or flowing, at a temperature not exceeding 30 ^{o}C.

3.4 Water thawing by direct contact, means thawing of the unpacked product by immersion in water at a temperature not exceeding 30 ^{o}C. (This method is applicable only to some vegetables).

4. PRINCIPLE OF METHODS

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

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

5. APPARATUS

5.1 Electric fan (optional), for forced ventilation air thawing.

5.2 Water bath with thermostat and circulation pump, for indirect or direct water thawing.

5.3 Plastic bags or other suitable watertight and closable container, for samples to be subjected to water thawing.

5.4 Clamps or weights, to prevent agitation of package in water bath during thawing.

5.5 Screen, to remove excess water after water thawing by direct contact.

5.6 Tray, on which the product is placed after removal of excess water when thawed by direct contact with water.

6. SAMPLES

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

7. PROCEDURE

For the rapid thawing of quick-frozen products contained in consumer-size packages, bulk or industrial packages and 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.

Appendix XI

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

1. SCOPE

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

2. FIELD OF APPLICATION

2.1 The cooking procedure described below applies to those quick-frozen vegetables which are normally cooked prior to consumption for the proper evaluation of such organoleptic quality factors as texture, tenderness, maturity or flavour.

2.2 Where a particular quick-frozen vegetable requires a special cooking procedure not fully covered by this general procedure for examination, the method outlined in the appropriate Codex commodity standard shall be followed.

3. DEFINITION

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

4. PRINCIPLE OF METHOD

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

5. APPARATUS

5.1 Two-litre saucepan with cover.

5.2 Hot plate or gas fire.

5.3 Tray on which product is placed after cooking for cooling and presentation.

5.4 Graduated cylinder or similar measuring device for water.

6. SAMPLES

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

Appendix XII

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

1. PRINCIPLE OF THE METHOD

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

2. REAGENTS

2.1 Ethanol (95 percent) or denaturated ethanol.

Ethanol denaturated with 5 percent v/v methanol.

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

Dilute 8 parts by volume of reagent under Section 2.1 to 9.5 parts by volume with 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 ± 2 °C.

3.9 Filter paper, Whatman No. 1 or equivalent.

3.10 Macerator or blender.

3.11 Plastics bag of sufficient capacity to hold the entire sample for thawing.

3.12 “Policemen” on glass rods, bent so as to facilitate cleaning flask or beaker.

3.13 Water bath, with continuous flow at room temperature or regulated at room temperature for thawing.

4. PREPARATION OF TEST SAMPLE

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

5. PROCEDURE

5.1 Dry a filter paper in flat bottomed dish, lid off, for two hours at 100 ± 2 °C. Cover dish, cool in a desiccator, and weigh accurately. (The filter paper should be larger than the base of the funnel and folded at the circumference to facilitate subsequent removal without loss of solids).

5.2 Weight 20 g ± 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 ± 0.01 g paste into a 600 ml beaker. Add 240 ml denaturated ethanol or ethanol, stir, and cover beaker. Bring solution in the beaker to a boil and simmer slowly for 30 minutes on a hot plate.

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

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

6. CALCULATION AND EXPRESSION OF RESULTS

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

6.1 If 20 g sample is refluxed:

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

Where:

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

6.2 If 40 g sample is refluxed:

Alcohol-insoluble solids content (% m/m) = $5\,\underline{M}$

Where:

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

7. REPEATABILITY OF RESULTS

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

8. EXPRESSION OF RESULTS

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

Appendix XIII

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

PROCEDURE

1. Determine the total dry matter of the product by drying over sand for 4 hours at 105 °C.

2. From the value obtained in (1) deduct the amount of salt (NaCl) determine by either (a) electrometric titration using a pH metre with a silver electrode; or (b) direct titration with $AgNO_3$. Express the result, after deducting salt from total dry matter, as salt-free dry matter.

Appendix XIV

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

Apparatus

(a) Rotary evaporator

(b) Water bath

Extraction

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

Appendix XV

PART A - IDENTIFICATION OF SCOPOLETIN IN FERMENTED NONI FRUIT JUICE

1. PREPARATION OF SAMPLES

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

2. PREPARATION OF REFERENCE STANDARD

2.1 A reference standard is prepared by dissolving 0.1 mg scopoletin in 1 millilitre of methanol.

2.2 Alternately, certified *Morinda citrifolia* reference plant material may be prepared in the same manner as the samples to be analyzed. The certified *Morinda citrifolia* reference material should be from the same part of the plant as the samples to be analyzed.

3. IDENTIFICATION

3.1 Thin layer chromatography

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

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

1. PREPARATION OF SAMPLES

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

2. PREPARATION OF REFERENCE STANDARD

2.1 A reference standard is prepared by dissolving 1 mg deacetylasperulosidic acid in 1 millilitre of methanol.

2.2 Alternately, certified *Morinda citrifolia* reference plant material may be prepared in the same manner as the samples to be analysed. The certified *Morinda citrifolia* reference material should be from the same part of the plant as the samples to be analysed.

3. PREPARATION OF p-ANISALDEHYDE SOLUTION

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

4. IDENTIFICATION

4.1 THIN LAYER CHROMATOGRAPHY

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

Appendix XVI

METHOD FOR THE EXTRACTION OF OIL (LAVER PRODUCTS)

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

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

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

Decant the extracted solution (A) through a filter paper, on which Na_2SO_4 is mounted to remove moisture, on a funnel into a 1 000 ml round flask-flat bottom.

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

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