CODEX ALIMENTARIUS

INTERNATIONAL FOOD STANDARDS



RECOMMENDED METHODS OF ANALYSIS AND SAMPLING

CXS 234-1999¹

Adopted in 1999.

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 42nd Session of the Codex Alimentarius Commission in 2019.

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

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PART A - METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

| Commodity | Provision | Method | Principle | Туре |
|---|-----------------------------|--|--|------|
| All Foods | | | | |
| All foods | Acesulfame K, Aspartame | EN 12856 | High performance liquid chromatography | II |
| All foods | Cyclamate | EN 12857 | High performance liquid chromatography | II |
| All foods | Cyclamate | NMKL 123 | Spectrophotometry | Ш |
| All foods | Saccharin | EN 12856 | High performance liquid chromatography | III |
| All Foods (see also meat products) | Nitrates and/or Nitrites | EN 12014-1 | Part 1- General considerations | N/A |
| Individual Foods ² | Sulphites | EN 1988-1 AOAC 990.28 | Part 1: Optimized Monier-Williams method | III |
| Individual Foods ³ | Sulphites | EN 1988-2 | Part 2: Enzymatic method | III |
| - Individual Foods | | NMKL 135 | r an 2. Enzymatic method | |
| Cereals, Pulses and Legumes and | Derived Products | | | |
| Certain pulses | Moisture | ISO 665 | Gravimetry | I |
| Degermed maize (corn) meal and maize (corn) grits | Ash | AOAC 923.03 ISO 2171 ICC Method No 104/1 | Gravimetry | I |
| Degermed maize (corn) meal and maize (corn) grits | Fat, crude | AOAC 945.38F; 920.39C | Gravimetry (ether extraction) | I |
| Degermed maize (corn) meal and maize (corn) grits | Moisture | ISO 712 ICC Method No 110/1 | Gravimetry | I |
| Degermed maize (corn) meal and maize (corn) grits | Particle size (granularity) | AOAC 965.22 | Sieving | I |

Hominy, fruit juice, sea food
 Wine, dried apples, lemon juice, potato flakes, sultanas, beer

| Cereals, Pulses and Legumes and I | Derived Products | | | |
|--|---|---|---|-----|
| Degermed maize (corn) meal and maize (corn) grits | Protein | ICC Method No 105/1 | Titrimetry, Kjeldahl digestion | I |
| Durum wheat semolina and durum wheat flour | Ash (semolina) | AOAC 923.03 ISO 2171 | Gravimetry | I |
| Durum wheat semolina and durum wheat flour | Moisture | ISO 712 ICC 110/1 | Gravimetry | I |
| Durum wheat semolina and durum wheat flour | Protein (N x 5.7) | ICC 105/1 | Titrimetry, Kjeldahl digestion | I |
| Instant Noodles | Extraction of oil from instant noodles | described in the standard | Gravimetry | I |
| Instant Noodles | Acid Value | described in the standard | Titrimetry | ı |
| Instant Noodles | Moisture | described in the standard | Gravimetry | ı |
| Maize (corn) | Moisture | ISO 6540 | Gravimetry | ı |
| Peanuts (raw) | Aflatoxins, total | AOAC 991.31 | Immunoaffinity column (Aflatest) | II |
| Peanuts (raw) | Aflatoxins, total | AOAC 993.17 | Thin layer chromatography | Ш |
| Peanuts (intended for further processing) | Aflatoxins, total | AOAC 975.36 | Romer minicolmn | III |
| Peanuts (Cereals, shell-fruits and derived products (including peanuts)) | Sum of aflatoxins B_1 , B_2 , G_1 and G_2 | EN 12955 ISO 16050 | HPLC with post column derivatization and immunoaffinity column clean up | Ш |
| Peanuts (intended for further processing) | Aflatoxins, total | AOAC 979.18 | Holaday-Velasco minicolumn | III |
| Pearl millet flour | Ash | AOAC 923.03 | Gravimetry | 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; 920.39C | Gravimetry (ether extraction) | ı |
| Pearl millet flour | Fibre, crude | ISO 5498: (B.5 Separation) | Gravimetry | ı |
| Pearl millet flour | Moisture | ISO 712: ICC 110/1 | Gravimetry | I |

| Cereals, Pulses and Legume | s and Derived Products | | | |
|----------------------------|--|---|--|----|
| Quinoa | Moisture content | ISO 712 / AACCI 44-15.02 | Gravimetry | I |
| Quinoa | Protein Content (N x 6.25 in dry weight basis) | ISO 1871 | Titrimetry (Kjeldahl) | IV |
| Pearl millet flour | Protein | AOAC 920.87 | Titrimetry, Kjeldahl digestion | I |
| Sorghum flour | Ash | AOAC 923.03 ISO 2171 ICC 104/1 | Gravimetry | 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; 920.39C | Gravimetry (ether extraction) | I |
| Sorghum flour | Fibre, crude | ICC 113 ISO 6541 | Gravimetry | I |
| Sorghum flour | Moisture | ISO 712 ICC 110/1 | Gravimetry | I |
| Sorghum flour | Particle size (granularity) | AOAC 965.22 | Sieving | I |
| Sorghum flour | Protein | ICC 105/1 | Titrimetry, Kjeldahl digestion | I |
| Sorghum flour | Tannins | ISO 9648 | Spectrophotometry | I |
| Sorghum grains | Ash | AOAC 923.03 ISO 2171 ICC 104/1 | Gravimetry | I |
| Sorghum grains | Fat, crude | AOAC 945.38F, 920.39C | Gravimetry (ether extraction) | I |
| Sorghum grains | Moisture | ISO 6540 | Gravimetry | I |
| Sorghum grains | Protein | ICC 105/1 | Titrimetry, Kjeldahl digestion | I |
| Sorghum grains | Tannins | ISO 9648 | Spectrophotometry | I |
| Soy protein products | Ash | AOAC 923.03 ISO 2171: (Method B) | Gravimetry | I |
| Soy protein products | Fat | CAC/RM 55 - Method 1 | Gravimetry (extraction) | I |

| Cereals, Pulses and Legumes and | Derived Products | | | |
|---|-----------------------------|---|---------------------------------|----|
| Soy protein products | Fibre, crude | ISO 5498 | Gravimetry | 1 |
| Soy protein products | Moisture | AOAC 925.09 | Gravimetry (vacuum oven) | I |
| Soy protein products | Protein | AOAC 955.04D (using factor 6.25) | Titrimetry , Kjeldahl digestion | II |
| Vegetable protein products | Ash | AOAC 923.03 ISO 2171 (Method B) | Gravimetry, Direct | I |
| Vegetable protein products | Fat | CAC/RM 55 - Method 1 | Gravimetry (extraction) | I |
| Vegetable protein products | Fibre, crude | AACC 32-17 | Ceramic fiber filteration | I |
| Vegetable protein products | Moisture | AOAC 925.09 | Gravimetry (vacuum oven) | 1 |
| Vegetable protein products | Protein | AOAC 955.04D (using factor 6.25) | Titrimetry, Kjeldahl digestion | II |
| Wheat flour | Ash | AOAC 923.03 ISO 2171 ICC 104/1 | Gravimetry | I |
| Wheat flour | Fat acidity | AOAC 939.05 | Titrimetry | ı |
| Wheat flour | Moisture | ISO 712: ICC 110/1 | Gravimetry | I |
| Wheat flour | Particle size (granularity) | AOAC 965.22 | Sieving | I |
| Wheat flour | Protein | ICC 105/1 | Titrimetry, Kjeldahl digestion | I |
| Wheat protein products including wheat gluten | Protein | Vital wheat gluten and devitalized wheat gluten | Kjeldahl | I |
| | | AOAC 979.09 (wheat protein in grain N x 5.7) | | |
| | | Solubilized wheat protein | Kjeldahl | |
| | | AOAC 920.87 | . 10.000 | |
| | | (wheat protein in flour N x 5.7) | | |
| Wheat protein products including Wheat gluten | Fibre, crude | AOAC 962.09 | Ceramic fiber filteration | I |

| Cereals, Pulses and Legumes and I | | | | |
|---|-----------------------------|--------------------------------------|---|----|
| Wheat protein products including Wheat gluten | Ash | AOAC 923.03 ISO 2171: method B | Gravimetry | I |
| Whole and decorticated pearl millet grains | Ash | AOAC 923.03 | Gravimetry | I |
| Whole and decorticated pearl millet grains | Fat, crude | AOAC 945.38F; 920.39C | Gravimetry (ether extraction) | I |
| Whole and decorticated pearl millet grains | Fibre, crude | ISO 5498 (B.5 Separation) | Gravimetry | I |
| Whole and decorticated pearl millet grains | Moisture | ISO 712 ICC 110/1 | Gravimetry | I |
| Whole and decorticated pearl millet grains | Protein | AOAC 920.87 | Titrimetry, Kjeldahl digestion | I |
| Whole maize (corn) meal | Ash | AOAC 923.03 ISO 2171 ICC 104/1 | Gravimetry | I |
| Whole maize (corn) meal | Fat, crude | AOAC 945.38F; 920.39C | Gravimetry (ether extraction) | I |
| Whole maize (corn) meal | Moisture | ISO 712 ICC 110/1 | Gravimetry | I |
| Whole maize (corn) meal | Particle size (granularity) | AOAC 965.22 | Sieving | I |
| Whole maize (corn) meal | Protein | ICC 105/1 | Titrimetry, Kjeldahl digestion | I |
| Cocoa Products and Chocolate | | | | |
| Chocolate and chocolate products | Cocoa butter | AOAC 963.15 IOCCC 14 | Gravimetry (Soxhlet extraction) | I |
| Chocolate and chocolate products | Fat-free cocoa solids | AOAC 931.05 | Oven evaporation and factor | 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 |

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| Cocoa Products and Chocolate | | | | |
|--|--|--|---|----|
| Chocolate and chocolate products | Milkfat | IOCCC 5 AOAC 945.34; 925.41B; 920.80 | Titrimetry/Distillation | I |
| Chocolate and chocolate products | Moisture | IOCCC 26 or AOAC 977.10 (Karl Fischer method); or AOAC 931.04 or IOCCC 1 | Gravimetry | I |
| Chocolate and chocolate products | Non-cocoa butter vegetable fat | AOCS Ce 10/02 and described in the Standard | Described in the Standard | 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 | ı |
| 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 | | | | |
| Fats and Oils (all) | Arsenic | AOAC 952.13 (Codex general method) | Colorimetry (diethyldithiocarbamate) | 11 |
| Fats and Oils (all) | Arsenic | AOAC 942.17 (Codex general method) | Colorimetry (molybdenum blue) | Ш |
| Fats and Oils (all) | Arsenic | AOAC 986.15 (Codex general method) | Atomic absorption spectrophotometry | Ш |
| Fats and oils | Butylhydroxyanisole, butylhydroxytoluene, tert- butylhydroquinone, & propyl gallate | AOAC 983.15; or AOCS Ce-6-86 | Liquid chromatography | II |
| Fats and Oils (all) | Insoluble impurities | ISO 663 | Gravimetry | I |
| Fats and Oils (all) | Lead | AOAC 994.02 ISO 12193 (Codex general method) or AOCS Ca 18c-91 | Atomic absorption spectrophotometry (direct graphite furnace) | II |

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

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

| Fats and Oils and Related Produ | ucts | | | |
|---------------------------------|--|---|--|-----|
| Named Animal Fats | GLC ranges of fatty acid composition | ISO 5508 and ISO 12966-2 or AOCS Ce 2-66 and Ce 1e-91 or Ce 1f-96 | Gas chromatography of methyl esters | II |
| Named Animal Fats | lodine value (IV) | ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92 | Wijs-Titrimetry | I |
| Named Animal Fats | Peroxide value | AOCS Cd 8b-90 ISO 3960 | Titrimetry using iso-octane | I |
| Named Animal Fats | Relative density | ISO/AOCS method for apparent density to be inserted | Pycnometry | II |
| Named Animal Fats | Refractive index | ISO 6320; or AOCS Cc 7-25 | Refractometry | П |
| Named Animal Fats | Saponification value | ISO 3657; or AOCS Cd 3-25 | Titrimetry | I |
| Named Animal Fats | Unsaponifiable matter | ISO 3596 or ISO 18609; or AOCS Ca 6b-53 | Titrimetry after extraction with diethyl ether | I |
| Named Animal Fats | Titre | ISO 935; or AOCS Cc 12-59 | Thermometry | - 1 |
| Named Vegetable Oils | Acidity: | ISO 660 / AOCS Cd 3d-63 / AOCS Ca 5a- | Titrimetry | I |
| | Acid value | 40 | | |
| 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, with the appropriate conversion factor; or AOCS Cc 10c-95 | Pycnometry | I |
| Named Vegetable Oils | Baudouin test (modified Villavecchia or sesameseed oil test) | AOCS Cb 2-40 | Colour reaction | I |
| Named Vegetable Oils | Carotenoids, total | BS 684 Section 2.20 | Spectrophotometry | П |
| Named Vegetable Oils | Copper and iron | ISO 8294; or AOAC 990.05; or AOCS Ca 18b-91 | AAS | II |
| Named Vegetable Oils | Crismer value | AOCS Cb 4-35 and AOCS Ca 5a-40 | Turbidity | I |
| Named Vegetable Oils | GLC ranges of fatty acid composition | ISO 5508 and ISO 12966-2; or AOCS Ce 2-66 and Ce 162 or Ce 1h-05 | Gas chromatography of methyl esters | II |
| Named Vegetable Oils | Halphen test | AOCS Cb 1-25 | Colorimetry | I |

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| Fats and Oils and Related Products | 3 | | | |
|------------------------------------|-------------------------------------|---|------------------------------|----|
| Named Vegetable Oils | Insoluble impurities | ISO 663 | Gravimetry | I |
| Named Vegetable Oils | lodine value (IV) | Wijs - ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92; or NMKL 39 | Wijs-Titrimetry ⁴ | I |
| Named Vegetable Oils | Lead | AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91 | Atomic Absorption | II |
| Named Vegetable Oils | Moisture & volatile matter at 105°C | ISO 662 | Gravimetry | I |
| Named Vegetable Oils | Peroxide value (PV) | AOCS Cd 8b-90 or ISO 3960 | Titrimetry | I |
| Named Vegetable Oils | Refractive index | ISO 6320 or AOCS Cc 7-25 | Refractometry | II |
| Named Vegetable Oils | Reichert value and Polenske value | AOCS Cd 5-40 | Titrimetry | I |
| Named Vegetable Oils | Relative density | IUPAC 2.101 with the appropriate conversion factor See comment above (Named Animal Fats) ⁵ | Pycnometry | I |
| Named Vegetable Oils | Saponification value (SV) | ISO 3657 or AOCS Cd 3-25 | Titrimetry | I |
| Named Vegetable Oils | Slip point | ISO 6321 for all oils; AOCS Cc 3b-92 for all oils except palm oils; AOCS Cc 3-25 for palm oils only | Open ended capillary tube | I |
| Named Vegetable Oils | Soap content | BS 684 Section 2.5; or AOCS Cc 17-95 | Gravimetry | I |
| Named Vegetable Oils | Sterol content | ISO 12228; or AOCS Ch 6-91 | Gas chromatography | II |
| Named Vegetable Oils | Tocopherol content | ISO 9936 or AOCS Ce 8-89 | HPLC | II |
| Named Vegetable Oils | Unsaponifiable matter | ISO 3596; or ISO 18609; or AOCS Ca 6b-53 | Gravimetry | I |
| Olive Oils and Olive Pomace Oils | Absorbency in ultra-violet | COI/T.20/Doc. No. 19 or ISO 3656 or AOCS Ch 5-91 | Absorption in ultra violet | 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 |

It is possible to calculate the lodine Value from fatty acid composition data obtained by gas chromatography e.g. using AOCS Cd 1b-87
 The method is no longer available.

| Fats and Oils and Related Products | 3 | | | |
|------------------------------------|---|--|---|----|
| 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 | lodine 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 | Lead | AOAC 994.02 or ISO 12193 or AOCS Ca 18c-91 | AAS | II |
| Olive Oils and Olive Pomace Oils | Moisture and volatile matter | ISO 662 | Gravimetry | I |
| 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 | 11 |
| 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 | Ш |
| 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 | | | | |
|---|---|--|--|-----|
| 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 | pΗ | AOAC 981.12 The pH shall be measured in a sample of fish sauce diluted with water to 1:10 using a pH meter. The dilution of fish sauce is necessary because of the high ionic strength in the undiluted sauce. | Electrometry | III |
| Fish Sauce | sodium chloride | AOAC 976.18 | Potentiometry | II |
| Fish Sauce | sodium chloride | AOAC 937.09 | Titrimetry | IV |
| Fish Sauce | histamine | AOAC 977.13 | Fluorimetry | Ш |
| Frozen abalone (covered by glaze) | Net weight | AOAC 963.18 | Gravimetry | |
| Frozen fish and fishery products | Thawing and cooking procedures | Described in the Standards | Thawing and heating | 1 |
| 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 |

| Fish and Fishery Products | | | | |
|---|--|--|--|-----|
| 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 | 1 |
| 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 <i>Gadidae</i> Family | Salt | Described in CXS 167-1989 | Titrimetry (Mohr) Salt determined as chloride expressed as sodium chloride | I |
| Salted Fish and Dried Salted Fish of the <i>Gadidae</i> Family of Fishes | Salt Content Water content | Sampling and method described in the Standard | Gravimetry | I |
| Smoked Fish, Smoke-Flavoured fish and Smoke-dried fish | Water phase salt | AOAC 952.08 AOAC 937.09 Described in standard ⁶ | Calculation | I |
| Smoked Fish, Smoke-Flavoured fish and Smoke-dried fish | Water activity | NMKL 168 ISO 21807 | Electrometry | III |

⁶ % salt × 100 / (%water + %salt)

| Fish and Fishery Products | | | | |
|-------------------------------|------------------------------|---------------------------|--|----|
| Sturgeon Caviar | Salt content | Described in CXS 167-1989 | Titrimetry (Mohr) Salt determined as chloride expressed as sodium chloride | I |
| Live and raw bivalve molluscs | Paralytic shellfish toxicity | AOAC 959.08 | Mouse bioassay | IV |
| Live and raw bivalve molluscs | Paralytic shellfish toxicity | AOAC 2011.27 | Receptor binding assay | IV |

Method Performance Criteria for histamine for fish and fishery products

| 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 1 and may either meet the minimum applicable range, or LOD and LOQ criteria listed.

Table 1. Criteria for determination of Toxin Analogues by Chemical Methods

| Toxin Group | Toxin | Minimum | LOD | LOQ | Precision (RSD _R) | Recovery percent | Applicable methods that meet |
|-------------|-----------|------------------|---------|---------|-------------------------------|------------------|------------------------------|
| | | applicable range | (mg/kg) | (mg/kg) | (%) | | the criteria |
| | | (mg/kg) | | | No more than | | |
| STX Group | Saxitoxin | 0.05 - 0.2 | 0.01 | 0.02 | 44% | 50 – 130 | AOAC 2005.06 |
| | (STX) | | | | | | NMKL 182, EN 14526 |
| | NEO | 0.05 - 0.2 | 0.01 | 0.02 | 44% | 50 – 130 | AOAC 2011.02 |
| | dcSTX | 0.05 - 0.2 | 0.01 | 0.02 | 44% | 50 – 130 | NMKL 197 |
| | GTX1 | 0.05 - 0.2 | 0.01 | 0.02 | 44% | 50 – 130 | |
| | GTX2 | 0.1 - 0.5 | 0.03 | 0.06 | 38% | 50- 130 | |
| | GTX3 | 0.1 – 0.5 | 0.03 | 0.06 | 38% | 50- 130 | |
| | GTX4 | 0.05 - 0.2 | 0.01 | 0.02 | 44% | 50 – 130 | |
| | GTX5 | 0.1 - 0.5 | 0.03 | 0.06 | 38% | 50- 130 | |
| | GTX6 | 0.1 - 0.5 | 0.03 | 0.06 | 38% | 50- 130 | |
| | dcGTX2 | 0.1 - 0.5 | 0.03 | 0.06 | 38% | 50- 130 | |
| | dcGTX3 | 0.1 - 0.5 | 0.03 | 0.06 | 38% | 50- 130 | |
| | C1 | 0.1 - 0.5 | 0.03 | 0.06 | 38% | 50- 130 | |
| | C2 | 0.1 - 0.5 | 0.03 | 0.06 | 38% | 50- 130 | |
| | C3 | 0.5 – 1.5 | 0.1 | 0.2 | 32% | 50- 130 | |
| | C4 | 0.5 – 1.5 | 0.1 | 0.2 | 32% | 50– 130 | |
| OA Group | OA | 0.03 - 0.2 | 0.01 | 0.02 | 44% | 60-115 | See reference below |
| | DTX1 | 0.03 - 0.2 | 0.01 | 0.02 | 44% | 60-115 | |
| | DTX2 | 0.1 – 0.5 | 0.03 | 0.06 | 38% | 60-115 | |
| Domoic Acid | DA | 14 – 26 | 2 | 4 | 20% | 80-110 | |
| AZA Group | AZA1 | 0.03 - 0.2 | 0.01 | 0.02 | 44% | 40 - 120 | See reference below |
| | AZA2 | 0.03 - 0.2 | 0.01 | 0.02 | 44% | 40 - 120 | |
| | AZA3 | 0.03 - 0.2 | 0.01 | 0.02 | 44% | 40 - 120 | |

Reference: http://aesan.msssi.gob.es/en/CRLMB/web/procedimientos_crlmb/crlmb_standard_operating_procedures.shtml Harmonised-SOP-LCMS-OA-Version4.pdf

Total toxicity is estimated as the sum of the molar concentrations of detected analogs multiplied by the relevant specific toxicity equivalency factors (TEFs). Internationally scientifically validated TEFs must be used. The science behind TEFs is developing. Current internationally validated TEF's will be found on the FAO website. Information on TEFs could be incorporated in this standard at a future date.

Methods should be validated and used for the relevant toxin analogues that may contribute to total toxicity. Currently known toxin analogues to consider are listed in Table 1.

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

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 | 80-110 | 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 D | lietary Uses | | | |
|---------------------|--|---|--|-----|
| Special foods | Ash | AOAC 942.05 | Gravimetry | I |
| Special foods | Calcium | AOAC 984.27 | ICP emission spectrometry | Ш |
| Special foods | Calories by calculation | Method described in CAC/VOL IX-Ed.1, Part III | Calculation method | Ш |
| 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 |
| 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 | 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 | Ш |
| 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., <i>Agr. Handbook</i> 97 (1965) | Microbioassay | IV |
| Special foods | Phosphorous | AOAC 986.24 | Colorimetry (molybdovanadate) | II |

| Foods for Special Die | tary Uses | | | |
|-----------------------|--|---|--------------------------------------|-----|
| Special foods | Protein efficiency ratio (PER) | AOAC 960.48 | Rat bioassay | I |
| Special foods | Protein, crude | Method described in CAC/VOL IX-Ed. 1,Part III | Titrimetry, Kjeldahl digestion | I |
| Special foods | Riboflavin | AOAC 970.65 | Fluorometry | 11 |
| 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 | Ш |
| Special foods | Vitamin A | AOAC 974.29 | Colorimetry | IV |
| Special foods | Vitamin A in foods in which carotenes have been added as a source of vitamin A | AOAC 941.15 | Spectrophotometry | III |
| Special foods | Vitamin B ₁₂ | AOAC 952.20 | Microbioassay | II |
| Special foods | Vitamin B ₆ | AOAC 961.15 | Microbioassay | II |
| Special foods | Vitamin C | AOAC 967.22 | Microfluorometry | II |
| Special foods | Vitamin C | AOAC 967.21 | Colorimetry (dichloroindophenol) | III |
| Special foods | Vitamin D | AOAC 936.14 | Rat bioassay | IV |
| 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 | 11 |
| 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 | lodine (milk based formula) | AOAC 992.24 | Ion-selective potentiometry | II |
| Follow-up formula | Pantothenic acid | AOAC 992.07 | Microbioassay | 11 |
| | | Measures total pantothenate (free pantothenic acid + CoA- + ACP-bound) and measured as D-pantothenic acid (or calcium D-pantothenate) | | |

| Foods for Special Dietar | y Uses | | | |
|--|--|--|---|-----|
| 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/I milk after reconstitution) | AOAC 992.06 | HPLC | III |
| Follow-up formula | Vitamin K | AOAC 999.15 | HPLC | II |
| • | | EN 14148 (vitamin K ₁) | with | |
| | | (Measures either aggregated cis + trans K_1 or can measure individual cis and trans forms depending on LC column.) | C30 column to separate the cis- and the trans- K vitamins | |
| Foods with low-sodium content (including salt substitutes) | lodine | AOAC 925.56 | Titrimetry | II |
| Foods with low-sodium content (including salt substitutes) | Silica (colloidal, calcium silicate) | AOAC 950.85N | Gravimetry | IV |
| Gluten-free foods | Gluten | Enzyme-Linked Immunoassay R5 Mendez (ELISA) Method | Immunoassay | I |
| | | Eur J Gastroenterol Hepatol 2003; 15: 465-474 | | |
| Infant formula | Biotin | EN 15607 (d-biotin) | HPLC- fluorescence | III |
| | | (Measures total D-biotin (free + D-biocytin) | | |
| Infant formula | Biotin | AOAC 2016.02 | HPLC-UV | П |
| Infant formula | Calories (by calculation) | Method described in CAC/Vol IX-Ed.1, Part III ⁷ | Calculation | I |

⁷ Section 9. Calories by calculation – Section 9.2 Conversion Factors

(a) protein
(b) carbohydrate
(c) fat
(d) monosaccharides
4 kcal per g
4 kcal per g
9 kcal per g
3.75 kcal per g

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

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

| Foods for Special D | ietary Uses | | | |
|---------------------|--|---------------------------------------|---|-----|
| Infant formula | Calcium | AOAC 2015.06 / ISO 21424 IDF 243 | ICP-MS | II |
| Infant formula | Calcium | ISO 8070 IDF 119 | Flame atomic absorption spectrophotometry | III |
| Infant formula | Calcium | AOAC 985.35 | Flame atomic absorption spectroscopy | III |
| Infant formula | Chloride | AOAC 986.26 | Potentiometry | III |
| Infant formula | Chloride | AOAC 2016.03 / ISO 21422 IDF 242 | Potentiometry | II |
| Infant formula | Choline | AOAC 999.14 | Enzymatic Colorimetric Method with limitations on applicability due to choline and ascorbate concentration. | II |
| 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 | 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 | П |
| Infant formula | Crude protein ⁸ | ISO 8968-1 IDF 20-1 | Titrimetry (Kjeldahl) | 1 |
| Infant formula | Fatty acids (including trans fatty acid) | AOAC 996.06 | Gas chromatography | III |
| Infant formula | Fatty acids (including trans fatty acid) | AOCS Ce 1i-07 | Gas chromatography | Ш |

⁸ Determination of Crude Protein

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

| Foods for Special D | ietary Uses | | | |
|---------------------|--|---|---|-----|
| Infant formula | Folic acid | AOAC 992.05 | Microbioassay | III |
| | | (Measures free folic acid + free, unbound natural folates, aggregated and measured as folic acid) | | |
| | | EN 14131 | | |
| | | (Total folate (free + bound), aggregated and measured as folic acid) | | |
| Infant formula | Folic acid | AOAC 2011.06 | LC-MS/MS | II |
| Infant formula | lodine | AOAC 2012.15 / ISO 20647 IDF 234 | ICP-MS | ll |
| | (for milk-based formula) | | | |
| Infant formula | Iron | AOAC 2015.06 / | ICP-MS | II |
| | | ISO 21424 IDF 243 | | |
| Infant formula | Iron ⁹ | 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 / | ICP-MS | Ш |
| | | ISO 21424 IDF 243 | | |
| Infant formula | Magnesium | ISO 8070 IDF 119 | Flame atomic absorption spectrophotometry | III |
| Infant formula | Magnesium | AOAC 985.35 | Flame atomic absorption spectroscopy | III |
| Infant formula | Manganese | AOAC 2015.06 / | ICP-MS | II |
| | | ISO 21424 243 | | |
| 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 |

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⁹ General Codex methods are also available

CXS 234-1999 24

| Foods for Special D | ietary Uses | | | |
|---------------------|--|---|--|------------------|
| Infant formula | Molybdenum (Section B of CXS 72-1981 only) | AOAC 2006.03 | ICP emission spectroscopy | III |
| | 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 985.34 (niacin (preformed) and nicotinamide) | Microbioassay and turbidimetry | III |
| Infant formula | Niacin | EN 15652 | HPLC | II ¹⁰ |
| | | (Free and bound and phosphorylated forms measured either as aggregate of nicotinic acid + nicotinamide, or as individual forms) | | |
| Infant formula | Pantothenic acid | AOAC 2012.16 ISO 20639 | UHPLC-MS/MS | II |
| Infant formula | Phosphorus | AOAC 2015.06 / | ICP-MS | II |
| | | ISO 21424 IDF 243 | | |
| Infant formula | Phosphorus | AOAC 986.24 | Spectrophotometry (molybdovanadate) | III |
| Infant formula | Riboflavin | AOAC 985.31 ¹¹ | Fluorimetry | Ш |
| Infant formula | Riboflavin | EN 14152 (Measures natural and supplemental forms, free, bound and phosphorylated (FMN and FAD) aggregated and measured as riboflavin.) | HPLC | II |
| 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 |

When published as EN method
 Care should be taken in the application of the method due to spectral interference

| Foods for Special D | ietary Uses | | | |
|---------------------|--|---|--|-----|
| | Selenium | AOAC 2011.19 / ISO 20649 IDF 235 | ICP-MS | II |
| Infant formula | Sodium and potassium | AOAC 2015.06 / ISO 21424 243 | ICP-MS | II |
| Infant formula | Sodium and potassium | ISO 8070 IDF 119 | Flame atomic absorption spectrophotometry | III |
| Infant formula | Thiamine | AOAC 986.27 ¹² | Fluorimetry | III |
| 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 | II |
| Infant formula | Total carbohydrates | AOAC 986.25 AOAC 990.19 or | Determination by difference | I |
| | Moisture/Total Solids | AOAC 990.20 ISO 6731 IDF 21 | Gravimetry | |
| | Ash | AOAC 942.05 | Gravimetry | |
| Infant formula | Total fat | AOAC 989.05 ISO 8381 IDF 123 | Gravimetry (Röse-Gottlieb) | I |
| Infant formula | Total fat for milk-based infant formula (Products not completely soluble in ammonia) | ISO 8262-1 IDF 124-1 | Gravimetry (Weibull-Berntrop) | 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 |

¹² Care should be taken in the application of the method due to spectral interference

| Foods for Special Di | ietary Uses | | | |
|----------------------|--|--|--|------|
| Infant formula | Total phospholipids | AOCS Ja7b-91 | Gas chromatography with suitable extraction and preparation procedures | III |
| Infant formula | Vitamin A | EN 12823-1 (all-trans-retinol and 13-cis-retinol) | HPLC | III |
| | | Vitamin A (both natural + supplemental ester forms) aggregated and quantified as individual retinol isomers (13 - cis and all-trans) | | |
| Infant formula | Vitamin A Palmitate (Retinyl Palmitate), Vitamin A Acetate | AOAC 2012.10 ISO 20633 | HPLC | II |
| | (Retinyl Acetate) | | | |
| Infant formula | Vitamin C | AOAC 2012.22 / ISO/DIS 20635 | HPLC-UV | II |
| Infant formula | Vitamin D | EN 12821 | HPLC-UV | III |
| mant formula | | (D2 and/or D3 measured as single components. Hydroxylated forms not measured.) | | |
| | | NMKL 167 | | |
| Infant formula | Vitamin D | AOAC 995.05 | HPLC-UV | III |
| | | D2 and D3 measured | | |
| Infant formula | Vitamin D | AOAC 2016.05 / ISO 20636 | LC-MS | II |
| Infant formula | Vitamin E | AOAC 992.03 | HPLC | III |
| | | Measures all rac-vitamin E (both natural + supplemental ester forms) aggregated and quantified as α-congeners | | |
| Infant formula | Vitamin E | EN 12822 | HPLC | ll l |
| | | (Measures Vitamin E (both natural + supplemental ester forms) aggregated and quantified as individual tocopherol congeners $(\alpha, \beta, \gamma, \delta)$. | | |
| Infant formula | Vitamin E | AOAC 2012.10 / ISO 20633 | HPLC | II |

| Foods for Special D | ietary Uses | | | |
|---------------------|-------------------------|--|--------------------------------------|-----|
| Infant formula | Vitamin B ₆ | AOAC 985.32 | Microbioassay | III |
| Infant formula | Vitamin B ₆ | EN 14166 | Microbioassay | III |
| | | (Aggregates free and bound pyridoxal, pyridoxine and pyridoxamine and measures as pyridoxine) | | |
| Infant formula | Vitamin B ₆ | AOAC 2004.07 | | II |
| | | EN 14164 | HPLC | |
| | | (Free and bound phosphorylated forms (pyridoxal, pyridoxine and pyridoxamine) converted and measured as pyridoxine) | | |
| Infant formula | Vitamin B ₆ | EN 14663 (includes glycosylated forms) | HPLC | III |
| | | (Free and bound phosphorylated and glycosylated forms measured as the individual forms pyridoxal, pyridoxine and pyridoxamine) | | |
| Infant formula | Vitamin B ₁₂ | AOAC 986.23 | Turbidimetric Method | |
| | | (Measures total vitamin B ₁₂ as cyanocobalamin) | | 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 / | ICP-MS | ll |
| | | ISO 21424 IDF 243 | | •• |
| Infant formula | Zinc | AOAC 985.35 | Flame atomic absorption spectroscopy | III |

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

| Standard | Provisions | Method | Principle | Туре |
|---------------|---|---|--|--------|
| General meth | ods that do not measure the lower molecular weight fraction (i.e. monome | eric units<=9) ⁽²⁾ | | • |
| All foods (1) | Method applicable for determining dietary fibres that do not include | AOAC 985.29 | Enzymatic gravimetry | Type I |
| | the lower molecular weight fraction. (4) | AACC Intl 32-05.01 | | |
| All foods (1) | Method applicable for determining dietary fibres that do not include | AOAC 991.43 | Enzymatic gravimetry | Type I |
| | the lower molecular weight fraction and also includes determination for soluble and insoluble dietary fibres (4) | AACC Intl 32-07.01 | | |
| | 101 Soluble and insoluble dietary libres (4) | NMKL 129 | | |
| All foods (1) | Method applicable for determining dietary fibres that do not include the lower molecular weight fraction, in foods and food products containing more than 10% dietary fibres and less than 2% starch (e.g. fruits) (4) | AOAC 993.21 | Gravimetry | Type I |
| All foods (1) | Method applicable for determining dietary fibres that do not include | AOAC 994.13 | Enzymatic GC/ | Type I |
| | the lower molecular weight fraction. Provides sugar residue composition of dietary fibre polysaccharides, as well as content of | AACC Intl 32- 25.01 | colorimetry gravimetry | |
| | Klason lignin (4). | NMKL 162 | | |
| All foods (1) | Insoluble dietary fibres in food and food products (4) | AOAC 991.42 (Specific for insoluble fibre) | Enzymatic gravimetry | Type I |
| | | AACC Intl 32-20.01 | | |
| All foods (1) | Soluble dietary fibres in food and food products (4) | AOAC 993.19 (Specific for soluble fibre) | Enzymatic gravimetry | Type I |
| General meth | nods that measure both the higher (monomeric units > 9) and the lov | wer molecular weight fraction (m | onomeric units <=9) (2) | |
| All foods (1) | Method applicable for determining the content of dietary fibres of | AOAC 2001.03 | Enzymatic gravimetry | Type I |
| | higher and lower molecular weight, in food where resistant starches are not present | AACC Intl 32-41.01 | and Liquid chromatography | |
| All foods (1) | Method applicable for determining the content of dietary fibres of | AOAC 2009.01 | Enzymatic-Gravimetry | Type I |
| | higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches. | AACC Intl 32-45.01 | High Pressure Liquid Chromatography | |
| 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 | Туре |
|---------------|--|--------------------------------|---------------------------|----------|
| General meth | ods that do not measure the lower molecular weight fraction (i.e. mono | omeric units<=9)(2) | | • |
| Methods that | measure individual specific components (monomeric units: the | whole range for each type of o | components is covered)(2) | |
| All foods (1) | (1→3)(1→4) Beta-D-Glucans | AOAC 995.16 | Enzymatic | Type II |
| | | AACC Intl 32-23.01 | | |
| All foods (1) | Fructans (oligofructoses, inulin, hydrolyzed inulin, polyfructoses, | AOAC 997.08 | Enzymatic & HPAEC- | Type II |
| | fructooligosaccharides) | AACC Intl 32-31.01 | PAD | |
| | (applicable to added fructans) | | | |
| All foods (1) | Fructans (oligofructoses, inulin, hydrolyzed inulin, polyfructoses, | AOAC 999.03 | Enzymatic & | Type III |
| | fructooligosaccharides) | AACC Intl 32-32.01 | colorimetric | |
| | (not applicable highly depolymerised fructans) | | | |
| All foods (1) | Polydextrose | AOAC 2000.11 | HPAEC-PAD | Type II |
| | | AACC Intl 32-28.01 | | |
| All foods (1) | Trans-galacto-oligo saccharides | AOAC 2001.02 | HPAEC-PAD | Type II |
| | | AACC Intl 32-33.01 | | |
| All foods (1) | Resistant starch (Recommended for RS3) | AOAC 2002.02 | Enzymatic | Type II |
| | | AACC Intl 32-40.01 | | |

| Other met | thods ⁽²⁾ that have not been subjected to interlaboratory evaluation | on under AOAC international guidelines | 3 | |
|--------------------|---|---|------------------------------|---------|
| Yeast cell wall | Insoluble glucans and mannans of yeast cell wall (for yeast cell wall only) | Eurasyp (European association for specialty yeast product) – LM Bonanno. Biospringer- 2004 – online version: http://www.eurasyp.org/public.techniq ue.home.screen. | Chemical & HPAEC-PAD | Type IV |
| All foods | Fructo-oligosaccharides (monomeric units<5) | Ouarné et al. 1999 in <i>Complex Carbohydrates in Foods</i> . 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 for Nutrition Labelling* (CXG 2-1985).

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

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

| Fruit Juices and Nectars | | | | |
|--------------------------|---|---|---|------|
| Commodity | Provisions | Method | Principle | Туре |
| Fruit Juices and Nectars | Ascorbic acid-L (additives) | IFUMA 17A | HPLC | II |
| Fruit Juices and Nectars | Ascorbic acid-L (additives) | ISO 6557-1 | Fluorescence spectrometry | IV |
| Fruit Juices and Nectars | Ascorbic acid-L (additives) | AOAC 967.21 IFUMA 17 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 ¹³ (additives) | AOAC 986.13 | HPLC | II |
| Fruit Juices and Nectars | Citric acid ⁵ (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 & HIS in apple juice (permitted ingredients) | Determination of HFCS & HIS by Capillary GC method JAOAC 84, 486 (2001) | CAP GC Method | IV |
| Fruit Juices and Nectars | Malic acid (additives) | AOAC 993.05 | Enzymatic determination and HPLC | III |
| Fruit Juices and Nectars | Malic acid-D | EN 12138 IFUMA 64 | Enzymatic determination | II |
| Fruit Juices and Nectars | Malic acid-D in apple juice | AOAC 995.06 | HPLC | II |
| Fruit Juices and Nectars | Malic acid-L | EN 1138 IFUMA 21 | Enzymatic determination | II |
| Fruit Juices and Nectars | Pectin (additives) | IFUMA 26 | Precipitation/photometry | I |
| Fruit Juices and Nectars | Benzoic acid and its salts; sorbic acid and its salts | IFUMA 63 NMKL 124 | HPLC | II |
| Fruit Juices and Nectars | Benzoic acid and its salts | ISO 5518, ISO 6560 | Spectrometry | III |
| Fruit Juices and Nectars | Preservatives in fruit juices (sorbic acid and its salts) | ISO 5519 | Spectrometry | III |

¹³ All juices except citrus based juices

| Fruit Juices and Nectars | | | | |
|--------------------------|---|--|-------------------------------|-----|
| Fruit Juices and Nectars | Quinic, malic & citric acid in cranberry juice cocktail and apple juice (permitted ingredients and additives) | Determination of quinic, malic and citric acid in cranberry juice cocktail and apple juice AOAC 986.13 | HPLC | 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 | Sections 3.2 Quality Criteria and 3.3 Authenticity ¹⁴ | 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 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.

¹⁴ 3.4 Verification of Composition, Quality and Authenticity

| Fruit Juices and Nectars | | | |
|--------------------------|---|---|-----|
| 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 |
| Fruit Juices and Nectars | Determination of carotenoid, total/individual groups EN 12136; IFUMA 59 | Spectrophotometry | I |
| Fruit Juices and Nectars | Determination of centrifugable pulp EN 12134; IFUMA 60 | Centrifugation/% value | I |
| Fruit Juices and Nectars | Determination of chloride (expressed as sodium chloride) EN 12133 IFUMA 37 | Electrochemical titrimetry | III |
| Fruit Juices and Nectars | Determination of chloride in vegetable juice AOAC 971.27 (Codex general method) ISO 3634 | Titration | II |
| Fruit Juices and Nectars | Determination of essential oils (Scott titration AOAC 968.20 - IFUMA 45* | (Scott) distillation, titration | I |
| Fruit Juices and Nectars | Determination of essential oils (in citrus fruit) (volume determination)* ISO 1955 | Distillation and direct reading of the volume determination | I |
| Fruit Juices and Nectars | Determination of fermentability IFUMA 18 | Microbiological method | I |
| Fruit Juices and Nectars | Determination of formol number EN 1133 IFUMA 30 | Potentiometric titration | I |
| Fruit Juices and Nectars | Determination of free amino acids EN 12742 IFUMA 57 | Liquid Chromatography | II |
| Fruit Juices and Nectars | Determination of fumaric acid IFUMA 72 | HPLC | II |

| Fruit Juices and Nectars | | | |
|--------------------------|---|----------------------------------|----------|
| Fruit Juices and Nectars | Determination of glucose fructose and saccharose EN 12630 IFUMA 67 NMKL 148 | HPLC | II |
| Fruit Juices and Nectars | Determination of gluconic acid IFUMA 76 | Enzymatic determination | II |
| 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 | | |
|--------------------------|--|-----|
| Fruit Juices and Nectars | Determination of sodium, potassium, calcium, Atomic Absorption Spectroscopy magnesium in fruit juices EN 1134 IFUMA 33 | 11 |
| Fruit Juices and Nectars | Determination of sorbitol-D Enzymatic determination IFUMA62 | II |
| Fruit Juices and Nectars | Determination of stable carbon isotope ratio in the pulp of fruit juices ENV 13070 Analytica Chimica Acta 340 (1997) | 11 |
| Fruit Juices and Nectars | Determination of stable carbon isotope ratio of Stable isotope mass spectrometry sugars from fruit juices ENV 12140 Analytica Chimica Acta.271 (1993) | II |
| Fruit Juices and Nectars | Determination of stable hydrogen isotope ratio Stable isotope mass spectrometry of water from fruit juices ENV 12142 | II |
| Fruit Juices and Nectars | Determination of stable oxygen isotope ratio in Stable isotope mass spectrometry fruit juice water ENV 12141 | II |
| Fruit Juices and Nectars | Detection of starch Colorimetric AOAC 925.38 IFUMA 73 | I |
| Fruit Juices and Nectars | Determination of sugar beet derived syrups in frozen concentrated orange juice δ ¹⁸ O Measurements in Water AOAC 992.09 Oxygen isotope ratio analysis | I |
| Fruit Juices and Nectars | Determination of titrable acids, total Titrimetry EN 12147 IFUMA 03 ISO 750 | I |
| Fruit Juices and Nectars | Determination of total dry matter (vacuum- Gravimetric determination oven drying at 70°C)* EN 12145 IFUMA 61 | I |
| Fruit Juices and Nectars | Determination of total solids (Microwave oven Gravimetric determination drying)* AOAC 985.26 | I |
| Fruit Juices and Nectars | Determination of Vitamin C (dehydro-ascorbic Microfluorometry acid and ascorbic acid) AOAC 967.22 | III |

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

| Milk and Milk Products | | | | |
|--|--|---|---|-----|
| Milk products | Iron | NMKL 139 AOAC 999.11 | Atomic absorption | ll |
| F | | (Codex general method) | spectrophotometry | |
| Milk products | Iron | NMKL 161 / AOAC 999.10 | Atomic absorption spectrophotometry | III |
| Milk products | Iron | AOAC 984.27 | Inductively Coupled Plasma optical emission spectrophotometry | III |
| Milk products | Iron | ISO 6732 IDF 103 | Photometry (bathophenanthroline) | IV |
| Milk and Milk Products | Melamine | ISO/TS 15495 IDF/RM 230 | LC-MS/MS | IV |
| Milk products (products not completely soluble in ammonia) | Milk fat | ISO 8262-3 IDF 124-3 | Gravimetry (Weibull-Berntrop) | I |
| Blend of evaporated skimmed milk and vegetable fat | Total fat | ISO 1737 IDF 13 | Gravimetry (Röse-Gottlieb) | I |
| Blend of evaporated skimmed milk and vegetable fat | Milk solids-not-fat ¹⁵ (MSNF) | ISO 6731 IDF 21 and ISO 1737 IDF 13 | 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 ¹⁵ | ISO 6731 IDF 21 and ISO 1737 IDF 13 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 ¹⁵ | ISO 6731 IDF 21 and ISO 1737 IDF 13 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 1737 IDF 13 | Gravimetry (Röse-Gottlieb) | I |

_

¹⁵ Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose

| Milk and Milk Products | | | | |
|---|--|---|---|----|
| Reduced fat blend of evaporated skimmed milk and vegetable fat | Milk solids-not-fat (MSNF) ¹⁵ | ISO 6731 IDF 21 and ISO 1737 IDF 13 | 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 ¹⁵ | ISO 6731 IDF 21 and ISO 1737 IDF 13 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 ¹⁵ | ISO 6731 IDF 21 and ISO 1737 IDF 13 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 1736 IDF 9 | Gravimetry (Röse-Gottlieb) | I |
| Blend of skimmed milk and vegetable fat in powdered form | Water ¹⁶ | ISO 5537 IDF 26 | Gravimetry, drying at 87 °C | I |
| Blend of skimmed milk and vegetable fat in powdered form | Milk protein in MSNF ¹⁵ | 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 |
| Blend of skimmed milk and vegetable fat in powdered form | Milk protein in MSNF ¹⁵ | ISO 5537 IDF 26 and ISO 1736 IDF 9 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 | Total fat | ISO 1736 IDF 9 | Gravimetry (Röse-Gottlieb) | I |
| Reduced fat blend of skimmed milk powder and vegetable fat in powdered form | Water ¹⁶ | ISO 5537 IDF 26 | Gravimetry, drying at 87 °C | I |

¹⁶ Water content excluding the crystallized water bound to lactose (generally known as "moisture content")

| Milk and Milk Products | | | | |
|--|--|--|---|----|
| Reduced fat blend of skimmed milk powder and vegetable fat in powdered form | Milk protein in MSNF ¹⁵ | 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 ¹⁵ | ISO 5537 IDF 26 and ISO 1736 IDF 9 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 sweetened condensed skimmed milk and vegetable fat | Total fat | ISO 1737 IDF 13 | Gravimetry (Röse-Gottlieb) | I |
| Blend of sweetened condensed skimmed milk and vegetable fat | Sucrose | ISO 2911 IDF 35 | Polarimetry | IV |
| Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only) | Milk solids-not-fat ¹⁵ (MSNF) | ISO 6734 IDF 15 and ISO 1737 IDF 13 and ISO 2911 IDF 35 and | Calculation from total solids content, fat content and sucrose content Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Polarimetry | IV |
| Blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only) | Milk protein in MSNF ¹⁵ | ISO 6734 IDF 15 and ISO 1737 IDF 13 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 ¹⁵ | ISO 6734 IDF 15 and ISO 1737 IDF 13 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 1737 IDF 13 | Gravimetry (Röse-Gottlieb) | I |

| Milk and Milk Products | | | | |
|--|--|--|---|-----|
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only) | Milk solids-not-fat ¹⁵ (MSNF) | ISO 6734 IDF 15 and ISO 1737 IDF 13 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 ¹⁵ | ISO 6734 IDF 15 and ISO 1737 IDF 13 and ISO 2911 IDF 35 and ISO 8968-1 IDF 20-1 | Calculation from total solids content, fat content, sucrose content and protein content Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Polarimetry and Titrimetry (Kjeldahl) | IV |
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (for products sweetened with sucrose only) | Milk protein in MSNF ¹⁵ | ISO 6734 IDF 15 and ISO 1737 IDF 13 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 | Lead | AOAC 972.25 (Codex general method) | Atomic absorption spectrophotometry | IV |
| Butter | Milk solids-not-fat15 (MSNF) | ISO 3727-2 IDF 80-2 | Gravimetry | I |
| Butter | Milkfat | ISO 17189 IDF 194 | Gravimetry | l |
| | | | Direct determination of fat using solvent extraction | |
| Butter | Milk fat purity | ISO 17678 IDF 202 | Calculation from determination of triglycerides by gas chromatography - FID | I |
| Butter | Salt | ISO 1738 IDF 12/ | Titrimetry (Mohr: determination of | III |
| | | AOAC 960.29 | chloride, expressed as sodium chloride) | |
| Butter | Salt | ISO 15648 IDF 179 | Potentiometry (determination of chloride, expressed as sodium chloride) | II |

| Milk and Milk Products | | | | |
|--------------------------|--|--|--|-----|
| Butter | Water ¹⁶ | ISO 37271-1 IDF 80-1 | Gravimetry | I |
| Cheese | Milkfat | ISO 1735 IDF 5 | Gravimetry (Schmid-Bondzynski- Ratslaff) | I |
| Cheese | Moisture | ISO 5534 IDF 4 | Gravimetry, drying at 102 °C | I |
| Cheese (and cheese rind) | Natamycin | ISO 9233-1 IDF 140-1 | Molecular absorption spectrophotometry | 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) | |
| Cheeses, individual | Dry matter (Total solids)15 | ISO 5534 IDF 4 | Gravimetry, drying at 102°C | I |
| Cheeses, individual | Milkfat in dry matter ISO 5534 IDF 4 Calculation from dry matter content ISO 1735 IDF 5 and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzyr | | 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 (FDM) | ISO 5534 IDF 4 ISO 1735 IDF 5 | Calculation from dry matter content and fat content Gravimetry, drying at 102°C and | |
| | | | Gravimetry (Schmid-Bondzynski- Ratzlaff) | |
| Cottage cheese | Fat-free dry matter | ISO 5534 IDF 4 and ISO 1735 IDF 5 | Calculation from dry matter content and fat content Gravimetry, drying at 102 °C Gravimetry (Schmid-Bondzynski- Ratzlaff) | I |

| Milk and Milk Products | | | | |
|--|--|--|--|---|
| 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) | l |
| Cottage cheese (for samples containing lactose up to 5%) | Milkfat in dry matter | ISO 5534 IDF 4 and ISO 1735 IDF 5 | Calculation from dry matter content and fat content Gravimetry, drying at 102 °C and Gravimetry (Schmid-Bondzynski-Ratzlaff) | |
| Cottage cheese (for samples containing lactorse up to 5%) | Milkfat | ISO 1735 IDF 5 | Gravimetry (Schmid-Bondzynski- Ratzlaff) | 1 |
| Cottage cheese (for samples containing lactorse 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) | ı |
| Cream | Milkfat | ISO 2450 IDF 16 | Gravimetry (Röse-Gottlieb) | I |
| Cream | Solids ¹⁵ | ISO 6731 IDF 21 | Gravimetry (drying at 102°C) | I |
| Creams Lowered in Milkfat Content | Milkfat | ISO 2450 IDF 16 / AOAC 995.19 | Gravimetry (Röse-Gottlieb) | ı |
| Creams, Whipped Creams and Fermented Creams | Milk solids-not-fat (MSNF) ¹⁵ | 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 |
| Cream cheese | Moisture on fat free basis | ISO 5534 IDF 4 ISO 1735 IDF 5 | Calculation from fat content and moisture content Gravimetry drying at 102°C (forced air oven) | I |
| Dairy fat spreads | Milk fat purity | ISO 17678 IDF 202 | Gravimetry (Schmid-Bondzynski- Ratzlaff) Calculation from determination of triglycerides by gas chromatography - FID | I |

| Milk and Milk Products | | | | |
|---|--|---|---|----|
| Dairy fat spreads | Total fat | ISO 17189 IDF 194 | Gravimetry Direct determination of fat using solvent extraction | |
| Dairy permeate powders | Milkfat | ISO 1736 IDF 9 | Gravimetry (Röse-Gottlieb) | I |
| Dairy permeate powders | Nitrogen | ISO 8968-1 IDF 20-1 | Titrimetry (Kjeldahl) | I |
| Dairy permeate powders | Moisture ¹⁷ | ISO 5537 IDF 26 | Gravimetry (drying at 87°C) | ı |
| Dairy permeate powders | Ash | NMKL 173 | Gravimetry (ashing at 550 °C) | IV |
| Edible casein products | Acids, free | ISO 5547 IDF 91 | Titrimetry (aqueous extract) | IV |
| Edible casein products (caseins obtained by rennet precipitation and of caseinates, with the exception of ammonium caseinate) | Ash (including P ₂ O ₅) | ISO 5545 IDF 90 | Gravimetry (ashing at 825 °C) | I |
| Edible casein products (acid caseins, of ammonium caseinates, of their mixtures with rennet casein and with caseinates, and of caseins of unknown type) | Ash (including P ₂ O ₅) | ISO 5544 IDF 89 | Gravimetry (ashing at 825 °C) | I |
| Edible casein products | Copper | AOAC 2015.06 / | ICP-MS | П |
| | | ISO 21424 IDF 243 | | |
| Edible casein products | Copper | AOAC 985.35 | Atomic absorption spectrophotometry | Ш |
| Edible casein products | Copper | ISO 5738 IDF 76 | Colorimetry (diethyldiethiocarbamate) | Ш |
| Edible casein products | Lactose | ISO 5548 IDF 106 | Photometry (phenol and H ₂ SO ₄) | IV |
| Edible casein products Lead | | NMKL 139 (Codex general method) AOAC 999.11 | Atomic absorption spectrophotometry | IV |
| Edible casein products | Lead | NMKL 161 / | Atomic absorption spectrophotometry | IV |
| | | AOAC 999.10 | asserption open aprilately | |
| Edible casein products | Lead | ISO/TS 6733 IDF/RM 133 | Spectrophotometry (1,5-diphenylthiocarbazone) | IV |

¹⁷ Moisture content excluding the water of crystallization of lactose

| Milk and Milk Products | | | | |
|------------------------|--|------------------------------|---|----|
| Edible casein products | Milkfat | ISO 5543 IDF 127 | Gravimetry (Schmid-Bondzynski- Ratslaff) | I |
| Edible casein products | рН | ISO 5546 IDF 115 | Electrometry | IV |
| Edible casein products | Milk Protein (total N x 6.38 in dry matter) | ISO 5550 IDF 78 and | Calculation from dry matter content and protein content | I |
| | , | ISO 8968-1 IDF 20-1 | Gravimetry, drying at 102°C and | |
| | | | Titrimetry (Kjeldahl) | |
| Edible casein products | Sediment (scorched particles) | ISO 5739 IDF 107 | Visual comparison with standard disks, after filtration | IV |
| Edible casein products | Water ¹⁶ | ISO 5550 IDF 78 | Gravimetry (drying at 102 °C) | I |
| Emmental | Calcium ISO 8070 IDF 1 >= 800mg/100g | | Flame atomic absorption | |
| 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 1737 IDF 13 | Gravimetry (Röse-Gottlieb) | I |
| Evaporated milks | ISO 8968-1 IDF 20-1 Gravimetry (Röse-Gottlieb) | | 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 ¹⁵ | 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) ¹⁵ | 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 | I |
| Fermented milks | Lactobacillus acidophilus | ISO 20128 IDF 192 | Colony count at 37 °C | I |
| | • | | | |

| Milk and Milk Products | | | | | |
|---|---|--|--|----------|--|
| Fermented milks - Yoghurt and yoghurt products | Quantification of Lactobacillus delbrueckii subsp bulgaricus & Streptococcus thermophilus | ISO 7889 IDF 117 | Colony count at 37°C | | |
| Fermented milks - Yoghurt and yoghurt products | Identification of Lactobacillus delbrueckii subsp bulgaricus & Streptococcus thermophilus | ISO 9232 IDF 146 | Test for strain identification | I | |
| Fermented milks | Microorganisms constituting the starter culture | ISO 27205 IDF 149 (Annex A) | Colony count at 25 °C, 30 °C, 37 °C and 45 °C according to the starter organism in question | IV | |
| Fermented milks | Milkfat | ISO 1211 IDF 1 | Gravimetry (Röse-Gottlieb) | I | |
| Fermented milks Milk powders and cream powders | Milk Protein Acidity, titratable | ISO 8968-1 IDF 20-1 ISO 6091 IDF 86 | Titrimetry (Kjeldahl) Titrimetry, titration to pH 8.4 | I | |
| Milk powders and cream powders | Milkfat | ISO 1736 IDF 9 | Gravimetry (Röse-Gottlieb) | <u> </u> | |
| 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 disks, after filtration | IV | |
| Milk powders and cream powders | Solubility Index | ISO 8156 IDF 129 | Centrifugation | | |
| Milk powders and cream powders | Water ¹⁶ | ISO 5537 IDF 26 | Gravimetry (drying at 87°C) | ı | |
| Milk fat Products | Copper | ISO 5738 IDF 76 | Photometry, diethyldithiocarbamate | Ш | |
| Milk fat Products | Copper | AOAC 960.40 | Photometry, diethyldithiocarbamate | 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 | |
| Milkfat products (anhydrous milkfat) | Peroxide value | AOAC 965.33 | Titrimetry | I | |
| Milk fat products | Water ¹⁶ | ISO 5536 IDF 23 | Titrimetry (Karl Fischer) | II | |
| Mozzarella | Milkfat in dry matter – with high moisture | ISO 5534 IDF 4 and ISO 1735 IDF 5 | Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski- Ratzlaff) | I | |

| Milk and Milk Products | | | | |
|--|---|--|---|----------|
| Mozzarella | Milkfat in dry matter – with low moisture | ISO 5534 IDF 4 and ISO 1735 IDF 5 | Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski- Ratzlaff) | I |
| Sweetened condensed milk | Milkfat | ISO 1737 IDF 13 | Gravimetry (Röse-Gottlieb) | I |
| Sweetened Condensed Milks (for products sweetened with sucrose only) | Milk Protein in MNSF ¹⁵ | ISO 6734 IDF 15 and ISO 1737 IDF 13 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 ¹⁵ | ISO 6734 IDF 15 | Gravimetry, drying at 102 °C | I |
| Whey cheeses by coagulation | Milkfat | ISO 1735 IDF 5 | Gravimetry (Schmid-Bondzynski- Ratzlaff) | I |
| Whey cheeses by coagulation | Milkfat in dry matter | ISO 1735 IDF 5and ISO 5534 IDF 4 | Calculation from fat content and dry matter content | I |
| | | | Gravimetry (Schmid-Bondzynski-Ratzlaff) Gravimetry, drying at 102°C | |
| Whey cheeses by concentration | Milkfat | ISO 1854 IDF 59 | Gravimetry, drying at 102 C Gravimetry (Röse Gottlieb) | |
| <u> </u> | | • | , | <u>!</u> |
| Whey cheeses by concentration | Milkfat in dry matter | ISO 1854 IDF 59 and ISO 2920 IDF 58 | Calculation from fat content and dry matter content | ı |
| | | 13O 2920 IDF 36 | Gravimetry (Röse Gottlieb) | |
| | | | Gravimetry, drying at 88 C | |
| Whey powders | Ash | ISO 5545 IDF 90 | Gravimetry (ashing at 825°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 1736 IDF 9 | Gravimetry (Röse-Gottlieb) | I |
| Whey powders Milk protein (total N x 6.38) | | ISO 8968-1 IDF 20-1 | Titrimetry (Kjeldahl) | |
| Whey powders | Moisture, "Free" | ISO 2920 IDF 58 | Gravimetry (drying at 88°C ±2°C) | IV |

| Milk and Milk Products | | | | |
|------------------------|---------------------|---|-------------------------------------|-----|
| Whey powders | Water ¹⁶ | ISO 5537 IDF 26 | Gravimetry (drying at 87°C) | I |
| Natural Mineral Waters | | | | |
| 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 | Sodium | Examination of Water Pollution Control. WHO Pergamon Press (1982) Vol.2, pp. 151-152 | | III |
| Natural mineral waters | Sulphates | ISO 9280 | Gravimetry | Ш |
| Natural mineral waters | Sulphide | Handb. Spurenanal. 1974 | | IV |
| | | | | |

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 ¹⁸ ICP-MS ¹⁸ |
| Cadmium | 0.003 | 0.0017 | 0.0006 | 0.0012 | 44 | 80-110 | ISO 11885 ISO 17294-2 ISO 15586 ISO 5961 (Section 3) EPA 200.8 | ICP-OES ICP-MS GF-AAS AAS ICP-MS |
| Chromium | 0.05 | 0.028 | 0.01 | 0.02 | 44 | 90-107 | ISO 11885 ISO 17294-2 ISO 15586ISO 18412 (Cr VI) ISO 23913 (Cr VI) ISO 9174 (Section 4) EPA 200.8 | ICP-OES ICP-MS GF-AAS Photometric CIA, spectrophotometry AAS ICP-MS |
| Copper | 1 | 0.52 | 0.1 | 0.2 | 32 | 97-103 | ISO 11885 ISO 17294-2 ISO 15586 ISO 8288 EPA 200.8 | ICP-OES ICP-MS GF-AAS Flame-AAS ICP-MS |
| Cyanide | 0.07 | 0.039 | 0.014 | 0.028 | 44 | 90-107 | ISO 14403ISO 6703-1 | CFA Photometric, trimetric |
| Fluoride | 1.0 | 0.52 | 0.1 | 0.2 | 32 | 97-103 | ISO 10304-1 ISO 10359-1 (dissolved fluoride) ISO 10359-2 (inorganic bound) | LC of ions Electrochemical probe Digestion, distillation |

¹⁸ Total Boron is determined

| Provision | ML | Min. applicable | LOD | LOQ | Precision RSDR (%) | Recovery | Suggested methods | Principle |
|-----------|--------|-----------------|--------|--------|--------------------|----------|----------------------|----------------------------------|
| | (mg/L) | range (mg/L) | (mg/L) | (mg/L) | No more than | (%) | meeting the criteria | - |
| Lead | 0.01 | 0.0056 | 0.002 | 0.004 | 44 | 90-107 | ISO 17294-2 | ICP-MS |
| | | | | | | | ISO 15586 | GF-AAS |
| | | | | | | | EPA 200.8 | ICP-MS |
| Manganese | 0.4 | 0.18 | 0.04 | 0.08 | 37 | 95-105 | ISO 11885I | ICP-OES |
| | | | | | | | SO 17294-2 | ICP-MS |
| | | | | | | | ISO 15586 | GF-AAS |
| | | | | | | | EPA 200.8 | ICP-MS |
| Mercury | 0.001 | 0.00056 | 0.0002 | 0.0004 | 44 | 80-110 | EN 1483 | AAS |
| | | | | | | | ISO 17852 | Enrichment by amalgamation (III) |
| | | | | | | | ISO 5666 | AFS |
| | | | | | | | ISO 16590 | AAS after tin(II) chloride |
| | | | | | | | EPA 200.8 | reduction |
| | | | | | | | | Enrichment by amalgamation (III) |
| | | | | | | | | ICP-MS |
| Nickel | 0.02 | 0.011 | 0.004 | 0.008 | 44 | 90-107 | ISO 17294-2 | ICP-MS |
| | | | | | | | ISO 15586 | GF-AAS |
| | | | | | | | EPA 200.8 | ICP-MS |
| Nitrate | 50 | 37 | 5 | 10 | 18 | 98-102 | ISO 10304-1 | LC of ions |
| | | | | | | | ISO 13395 | CFA, FIA, Spectrophotometry |
| | | | | | | | ISO 7890-3 | Spectrophotometry |
| Nitrite | 0.1 | 0.03 | 0.01 | 0.02 | 44 | 95-105 | ISO 10304-1 | LC of ions UV |
| | | | | | | | ISO 13395 | CFA, FIA, Spectrophotometry |
| | | | | | | | ISO 6777 | Spectrophotometry |
| Selenium | 0.01 | 0.0056 | 0.002 | 0.004 | 44 | 90-107 | ISO 17294-2 | ICP-MS |
| | | | | | | | ISO 15586 | GF-AAS |
| | | | | | | | ISO 9965 | AAS (Hydride) |
| | | | | | | | EPA 200.8 | ICP-MS |

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 | | <10 | 80-110 | ISO 17993 | HPLC FD |
| | | 0.04 μg/L | | <18 | 80-110 | ISO 7981-1 | TLC |
| | | 0.005 µg/L | | <19 | 80-100 | ISO 7981-2 | HPLC |

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

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

| Provision Proper fill (in lieu of drained weight) Types of peas, distinguishing Syrup | Method CAC/RM 45 CAC/RM 48 | Principle Pouring and measuring | Type I |
|--|--|--|--|
| weight) Types of peas, distinguishing | | Pouring and measuring | I |
| 71 1 7 0 0 | CAC/RM 48 | | |
| Syrup | | Visual inspection | I |
| · · | AOAC 932.14C | Brix spindle method | I |
| Washed drained weight | CAC/RM 44 | Sieving | ı |
| Mineral impurities | ISO 762 | Gravimetry | I |
| Drained weight | AOAC 968.30 ISO:2173 | Gravimetry | I |
| Soluble solids | AOAC 932.14C | Refractometry | ı |
| Calcium | AOAC 968.31 | Complexometric titrimetry | П |
| Mineral impurities | AOAC 971.33 | Gravimetry | I |
| Calcium | NMKL 153 | Atomic Absorption Spectrophotometry | II |
| Calcium | AOAC 968.31 | Complexometry Titrimetry | III |
| Mineral impurities (sand) | AOAC 971.33 | Gravimetry | I |
| 0.1: | | 0 1 | |
| | | <u> </u> | II |
| Identification of defects | Described in the Standard | Visual inspection | <u> </u> |
| Moisture | AOAC 934.06 | Gravimetry (vacuum oven) | I |
| Total acidity of the extracted oil | ISO 660 or AOCS Cd 3d-63 | Titrimetry | I |
| Ash | AOAC 950.49 | Gravimetry | I |
| Extraneous vegetable matter | Described in the Standard | Counting extraneous material with the naked eye | IV |
| Moisture | AOAC 925.40 | Gravimery (loss on drying) | ı |
| | Mineral impurities Drained weight Soluble solids Calcium Mineral impurities Calcium Calcium Mineral impurities (sand) Calcium Identification of defects Moisture Total acidity of the extracted oil Ash Extraneous vegetable matter | Washed drained weight Mineral impurities ISO 762 Drained weight AOAC 968.30 ISO:2173 Soluble solids AOAC 932.14C Calcium AOAC 968.31 Mineral impurities AOAC 971.33 Calcium AOAC 968.31 Mineral impurities (sand) AOAC 968.31 Mineral impurities (sand) AOAC 971.33 ISO 762 Calcium AOAC 968.31 Identification of defects Described in the Standard Moisture AOAC 934.06 Total acidity of the extracted oil Ash AOAC 950.49 Extraneous vegetable matter Described in the Standard | Washed drained weight CAC/RM 44 Sieving Mineral impurities ISO 762 Gravimetry Drained weight AOAC 968.30 ISO:2173 Gravimetry Soluble solids AOAC 932.14C Refractometry Calcium AOAC 968.31 Complexometric titrimetry Mineral impurities AOAC 971.33 Gravimetry Calcium NMKL 153 Atomic Absorption Spectrophotometry Calcium AOAC 968.31 Complexometry Titrimetry Mineral impurities (sand) AOAC 971.33 Gravimetry Calcium AOAC 968.31 Complexometry Titrimetry Mineral impurities (sand) AOAC 971.33 Gravimetry ISO 762 Calcium AOAC 968.31 Complexometric titrimetry Identification of defects Described in the Standard Visual inspection Moisture AOAC 934.06 Gravimetry (vacuum oven) Total acidity of the extracted oil ASh AOAC 950.49 Gravimetry Extraneous vegetable matter Described in the Standard Counting extraneous material with the naked eye |

| Processed Fruits and Vegetables | | | | |
|------------------------------------|-------------------------------|------------------------------------|---|------|
| Commodity | Provision | Method | Principle | Туре |
| Desiccated coconut | Oil content | AOAC 948.22 | Gravimetry | I |
| Dried apricots | Identification of defects | Described in the Standard | Visual inspection (weighing) | I |
| Dried apricots | Moisture | AOAC 934.06 | Gravimetry (vacuum oven) | I |
| Dried apricots | Sulphur dioxide | AOAC 963.20 | Colorimetry | II |
| Jams (fruit preserves) and jellies | Fill of Containers | CAC/RM 46 | Weighing | I |
| Jams (fruit preserves) and jellies | Soluble solids | ISO 2173 AOAC 932.12 | Refractometry | I |
| Mango chutney | Ash insoluble in HCI | 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 | AOAC 971.33 | Gravimetry | I |
| Pickled cucumbers | Salt in brine | AOAC 971.27 (Codex general method) | Potentiometry | II |
| Pickled cucumbers | Volume fill by displacement | Described in the Standard | Displacement | I |
| Preserved tomatoes | Calcium | AOAC 968.31 | Complexometric titrimetry | III |
| Preserved tomatoes | Calcium | NMKL 153 | Atomic Absorption Spectrophotometry | II |
| Preserved tomatoes | Minimum 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 2631 | Enzymatic determination | II |
| Processed tomato concentrates | Mineral impurities (sand) | AOAC 971.33 | 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 Fruits and Vegetables | | | | |
|--|-----------------------|---|--------------------------------------|------|
| Commodity | Provision | Method | Principle | Туре |
| 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 | CAC/RM 51 | Ashing | I |
| Raisins | Mineral oil | CAC/RM 52 | Extraction and separation on alumina | II |
| Raisins | Moisture | AOAC 972.20 | Electrical conductance | I |
| Raisins | Sorbitol | AOAC 973.28 | Gas chromatography | II |
| Raisins | Sulphur dioxide | AOAC 963.20 | Colorimetry | II |
| Table olives | Drained weight | AOAC 968.30 (Codex general method for processed fruits and vegetables) | Sieving Gravimetry | I |
| Table olives | Fill of containers | CAC/RM 46* (for glass containers) (Codex general method for processed fruits and vegetables) and ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables) | Weighing | I |
| Table olives | pH of brine | NMKL 179 (Codex general method for processed fruits and vegetables) | Potentiometry | II |
| | | AOAC 981.12 (Codex general method for processed fruits and vegetables) ISO 1842 | _ | III |
| Table olives | Salt in brine | AOAC 971.27 NMKL 178 (Codex general method) | Potentiometry | II |
| Table olives | Lead | AOAC 999.11 NMKL 139 (Codex general method) | AAS (Flame absorption) | II |
| Table olives | Tin | NMKL 190 EN 15764 | AAS | 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 3.2 from the weight found in 3.3. The difference shall be considered to be the weight of water required to fill the container. Results are expressed as mL of water.

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

| Processed Meat and Poultry Products | s and Soups and Broths | | | |
|--|--|------------------------------------|---|-----|
| Meat Products | Nitrates and/or Nitrites | EN 12014-3 | Spectrometric determination of nitrate and nitrite content of meat products after enzymatic reduction of nitrate to nitrite | 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) | Ш |
| Processed meat and poultry products | Nitrites | ISO 2918 | Colorimetry | IV |
| Processed meat and poultry products | Tin | AOAC 985.16 (Codex general method) | Atomic absorption spectrophotometry | II |
| Processed meat and poultry products | Nitrogen/protein | ISO 937 | Titrimetry | Ш |
| 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 | Ш |
| Bouillons and Consommés (soups and broths) | Sodium chloride | AIIBP Method No 2/4 | Potentiometric titration (chloride expressed as sodium chloride) | Ш |
| Canned corned beef | Lead | AOAC 972.25 (Codex general method) | Atomic absorption spectrophotometry | Ш |
| Canned corned beef | Nitrites, potassium and/or sodium salt | AOAC 973.31 (Codex general method) | Colorimetry | П |
| Canned corned beef | Nitrites, potassium and/or sodium salt | ISO 2918 | Colorimetry | IV |

| Processed Meat and Poultry Proc | ducts and Soups and Broth | s | | |
|---------------------------------|---|------------------------------------|-------------------------------------|----|
| Canned corned beef | Tin (Products in tinplate and other containers) | AOAC 985.16 (Codex general method) | Atomic absorption spectrophotometry | II |
| Cooked cured chopped meat | Fat | ISO 1443 | Gravimetry (extraction) | I |
| Cooked cured chopped meat | Lead | AOAC 972.25 (Codex general method) | Atomic absorption spectrophotometry | II |
| Cooked cured chopped meat | Nitrites | AOAC 973.31 (Codex general method) | Colorimetry | П |
| 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 |
| 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 |

| Processed Meat and Poultry Produc | ts and Soups and Broths | S | | |
|--|--|------------------------------------|-------------------------------------|----|
| 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 | 5 | | | |
| Quick frozen fruits and vegetables(non-glazed) | Net weight | AOAC 963.26 | Weighing | I |
| Quick frozen fruits and vegetables | Thawing procedure | see Appendix I | Thawing | I |
| Quick frozen fruits and vegetables: Berries, leek and carrot | Mineral impurities | AOAC 971.33 | Gravimetry | I |
| Quick frozen fruits and vegetables: Berries, Whole kernel corn and Corn-on-the-cob | Soluble solids, total | AOAC 932.12 | Refractometry | I |
| Quick frozen fruits and vegetables: Peaches and berries | Drained fruit/drained berries | AOAC 953.15 | Draining | I |
| Quick frozen fruits and vegetables: Vegetables | Cooking procedure | see Appendix II | Cooking | I |
| Quick frozen French fried potatoes | Moisture | AOAC 984.25 | Gravimetry (convection oven) | I |
| Quick frozen green and wax beans | Tough strings | see Appendix III | Stretching | I |

| Quick Frozen Fruits and Vegeta | bles | | | | |
|--------------------------------|----------------------------------|---|--------------------------------|---|----|
| Quick frozen peas | Solids, alcohol insoluble | see Appendix IV | Gravimetry | I | |
| Quick frozen spinach | Dry matter, Sodium chloride-free | See Appendix V | Weighing | I | |
| Spices and Culinary Herbs | | | | | |
| Cumin | Moisture | ISO 939 | Distillation | | I |
| Cumin | Total ash | ISO 928 | Gravimetry | | I |
| Cumin | Acid-insoluble ash | ISO 930 | Gravimetry | | I |
| Cumin | Volatile oils | ISO 6571 | Distillation / Volumetric | | I |
| Cumin | Extraneous vegetable matte | er ISO 927 | Visual examination/ Gravimetry | | I |
| Cumin | Foreign matter | ISO 927 | Visual examination/Gravimetry | | I |
| Cumin | Insect damage | Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA) | Visual examination | | IV |
| | | http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32 | | | |
| Cumin | Mammalian excreta | Macroanalytical procedure manual USFDA technical bulletin V.39 B(for whole) | Visual examination | | IV |
| Cumin | Mammalian excreta | AOAC 993.27 (for ground) | Enzymatic Detection method | | IV |
| | | | | | |

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| metric I |
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| tion /Gravimetry I |
| ion IV |
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| ction method IV |
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| Spices and Culinary Herbs | | | | |
|-------------------------------|-----------------------------|---|---------------------------------|----|
| Thyme | Mould damage | Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA) | Visual examination | IV |
| | | http://www.fda.gov/Food/FoodSc ienceResearch/LaboratoryMetho ds/ucm084394.htm#v-32 | | |
| Black and white pepper | Bulk density | ISO 959-1 Annex B (black) | Gravimetry | IV |
| | | ISO 959-2 Annex A (white) | | |
| Black pepper | Light berries | ISO 959-1 Annex A (black) | Flotation | IV |
| Black, white and green pepper | Extraneous vegetable matter | ISO 927 | Visual examination / Gravimetry | 1 |
| Black, white and green pepper | Foreign matter | ISO 927 | Visual examination / Gravimetry | 1 |
| Black, white and green pepper | Black berries | Physical separation and weighing | Visual examination | IV |
| | | ISO 959-2 | | |
| Black, white and green pepper | Broken berries | Physical separation and weighing | Visual examination | IV |
| | | ISO 959-2 | | |
| Black, white and green pepper | Mouldy berries | Macroanalytical procedure manual USFDA technical bulletin V.39 B | Visual examination | IV |
| Black, white and green pepper | Insect damage | Macroanalytical procedure manual USFDA technical bulletin V.39 B | Visual examination | IV |
| Black, white and green pepper | Pinheads or broken berries | Physical separation and weighing ISO 959-1 | Visual examination | IV |

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| Spices and Culinary Herbs | | | | |
|-------------------------------|----------------------------|---|--|----|
| Black, white and green pepper | Mammalian excreta | Macroanalytical procedure manual USFDA technical bulletin V.39 B (For Pepper Whole) | Visual examination(For whole pepper) | IV |
| Black, white and green pepper | Mammalian excreta | AOAC 993.27 (for ground pepper) | Enzymatic Detection method (For ground pepper) | I |
| Black, white and green pepper | Moisture content | ISO 939 | Distillation | 1 |
| Black, white and green pepper | Total ash | ISO 928 | Gravimetry | ļ |
| Black, white and green pepper | Non-volatile ether extract | ISO 1108 | Soxhlet extration | ļ |
| Black, white and green pepper | Volatile oils | ISO 6571 | Distillation | I |
| Black, white and green pepper | Piperine content | ISO 5564 | Spectrophotometry | I |
| Black, white and green pepper | Acid-Insoluble ash | ISO 930 | Gravimetry | I |
| Black, white and green pepper | Crude Fibre | ISO 5498 | Gravimetry | 1 |

| Sugars and Honey | | | | |
|------------------|--------------------|--|---------------|----|
| Honey | Acidity | MAFF Validated Method V19 | Titrimetry | |
| | | J. Assoc. Public Analysts (1992) 28 (4) 171-175 | | |
| Honey | diastase activity | IHC Method for Determination of Diastase activity with Phadebas, 2009 except that the incubation time should be increased from 15 to 30 minutes. | | IV |
| Honey | Moisture | AOAC 969.38B or MAFF Validated Method V21 | Refractometry | l |
| Honey | Sample preparation | AOAC 920.180 | - | - |

| Sugars and Honey | | | | |
|--|---|--|--|----|
| Honey | Solids, water-insoluble | MAFF Validated Method V22 J. Assoc. Public Analysts (1992) 28(4) 189-193 | Gravimetry | I |
| Honey | Sugars added (for sugar profile) | AOAC 998.18 | Carbon isotope ratio mass spectrometry | I |
| Honey | Sugars added: detection of corn and cane sugar products | AOAC 978.17 | Carbon isotope ratio mass spectrometry | 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 (dextrose anhydrous and dextrose monohydrate, dried glucose syrup, glucose syrup, powdered dextrose, lactose) | Sulphated ash | ISO 5809 | Single sulphonation | I |
| Sugars (dextrose anhydrous and dextrose monohydrate) | Sulphur dioxide | ISO 5379 | Acidimetry and nephelometry | IV |
| Sugars (fructose) | рН | ICUMSA GS 1/2/3/4/7/8-23 | Potentiometry | 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 and Honey | | | | |
|--|--------------------|---|---------------------------------------|----|
| Sugars (glucose syrup and dried glucose syrup) | Sulphur dioxide | ISO 5379 | Acidimetry and nephelometry | IV |
| Sugars (lactose) | Lactose, anhydrous | ICUMSA GS 4/3-3 | Titrimetry | Ш |
| Sugars (lactose) | Loss on drying | USP General Chapter 731 | Gravimetry (Drying at 120°C for 16 h) | I |
| Sugars (lactose) | рН | ICUMSA GS 1/2/3/4/7/8-23 | Potentiometry | I |
| Sugars (plantation and mill white sugar) | Colour | ICUMSA GS9/1/2/3-8 | Photometry | I |
| Sugars (plantation or mill white sugar) | Conductivity ash | ICUMSA GS 1/3/4/7/8-13 | Conductimetry | I |
| 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 and Honey | | | | |
|--|---------------------------|--|---------------------------|----------|
| Sugars (soft white sugar and soft brown sugar) | Conductivity ash | ICUMSA GS 1/3/4/7/8-13 | Conductimetry | ļ |
| Sugars (soft white sugar and soft brown sugar) | Invert sugar | ICUMSA GS 4/3-3 (applicable at levels >10% m/m) | Titrimetry (Lane & 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 & Eynon) | I |
| Sugars (soft white sugar and soft brown sugar) | Loss on drying | ICUMSA GS 2/1/3-15 | Gravimetry | I |
| 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 | П |
| Sugars (white sugar | Sulphur dioxide | ICUMSA GS 2/3-35 NMKL 135 EN 1988-2 | Enzymatic method | II |
| Miscellaneous Products | | | | |
| Chili sauce | рН | NMKL 179 (Codex general method) | Potentiometry | II |
| Chili sauce | рН | AOAC 981.12 (Codex general method) | Potentiometry | III |
| Chili sauce | Fill of containers | CAC/RM 46 (Codex general method) | Weighing | I |
| Date Paste | Moisture | AOAC 934.06 | Gravimetry | <u> </u> |
| Date Paste | Mineral impurities | ISO 762 | Gravimetry | I |

| Miscellaneous Products | | | | |
|-------------------------|-----------------------|--|--------------------------------------|-----|
| Date Paste | Ash | AOAC 940.26 | Gravimetry | I |
| Date Paste | Acid Soluble Ash | AOAC 900.02D | Gravimetry, Calculation | I |
| Edible cassava flour | Fibre, crude | ISO 5498 (B.5 separation) | Gravimetry | I |
| Edible cassava flour | Granularity | ISO 2591-1 | Sieving | I |
| Edible cassava flour | Moisture | ISO 712 | Gravimetry | I |
| 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 ¹⁹ | 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 | Ш |
| Food grade salt | Cadmium | EuSalt/AS 014 | Atomic absorption spectrophotometry | IV |
| Food grade salt | Calcium and magnesium | ISO 2482 | Complexometric titrimetry | Ш |
| 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 |
| Food grade salt | lodine | EuSalt/AS 002 | Titrimetry using sodium thiosulphate | II |
| Food grade salt | lodine | EuSalt/AS 019 | ICP-OES | Ш |

¹⁹ 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 $^{\circ}$ C) NH₃-free H₂O and then stir the mixture for 60 min. Next, decant the mixture through a quantitative filter and collect the filtrate in a 100 ml volumetric flask.

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

| Miscellaneous Products | | | | |
|------------------------|--------------------|--|---|-----|
| Food grade salt | lodine | WHO/UNICEF/ICCIDD method ²⁰ Only applicable to a product which has been fortified with iodate | Titrimetry using sodium thiosulphate | IV |
| Food grade salt | Lead | EuSalt/AS 015 | ICP-OES | III |
| Food grade salt | Lead | EuSalt/AS 013 | Atomic absorption spectrophotometry | IV |
| Food grade salt | Loss on drying | ISO 2483 | Gravimetry (drying at 110°C) | 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 | Potentiometry | П |
| | | NMKL 178 | | |
| Foul medames | Drained weight | AOAC 968.30 | Sieving | I |
| Gari | Ash | ISO 2171 | Gravimetry | i |
| Gari | Fibre, crude | ISO 5498 (B.5 separation) | Gravimetry | i |
| Gari | Granularity | ISO 2591-1 | Sieving | I |

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

| Gari | Moisture | ICC 109/1 ISO 712 | Gravimetry | I |
|-----------------------------------|---|--|--------------------|----|
| Ginseng Products | Moisture | AOAC 925.45 B (Dried ginseng) Quantity of sample: 2 g | Gravimetry | I |
| Ginseng Products | Moisture | AOAC 925.45 D (Ginseng extract) Quantity of sample: 1.5 g (mixing with 20 g of sea sand) | Gravimetry | l |
| 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 | ı |
| Ginseng Products | Water-saturated n-butanol extracts | described in the Standard (Annex II) | Gravimetry | ı |
| Ginseng Products | Identification of ginsenosides Rb1 and Rf | described in the Standard (Annex III) | TLC or HPLC | IV |
| Gochujang | Capsaicin | AOAC 995.03 | HPLC | II |
| Gochujang | | described in the Standard (Annex D) | Gas chromatography | IV |
| Gochujang | Crude protein | AOAC 984.13 (Nitrogen conversion factor: 6.25) | Kjeldahl | I |
| Gochujang | Moisture | AOAC 934.01 (≤ 70°C, ≤ 50 mm Hg) | Gravimetry | I |
| Guideline level for acrylonitrile | Acrylonitrile | AOAC 985.13 | Gas chromatography | II |

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| Miscellaneous Products | | | | |
|---|------------------------------|---|-----------------------------------|-----|
| Guideline levels for vinyl chloride monomer | Vinyl chloride monomer | ISO 6401 | Gas chromatography | II |
| Guideline levels for vinyl chloride monomer | Vinyl chloride monomer | Commission Directive 81/432/EEC O.J. No. L.167, p. 6, 24.6.81 | Gas chromatography ("head-space") | III |
| Guidelines for nutrition labelling | Polyunsaturated fatty acids | AOCS Ce 1h-05 ²¹ | 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 |
| 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 ²² | Titrimetry | IV |
| Humus with tehena | Salt content | AOAC 971.27 NMKL 178 | Potentiometry | II |

 $^{^{21}}$ Can also be used to measure $\it trans$ unsaturated fatty acids 22 http://www.starch.dk/isi/methods/28luff.htm

| Miscellaneous Products | | | | |
|--------------------------------|-----------------------------------|---|--------------------------------|---|
| Humus with tehena | Total acidity | AOAC 925.53 | Titrimetry | I |
| Non-fermented soybean products | Moisture content | AOAC 925.09 AACCI 44- 40.01 | Gravimetry (vacuum oven) | 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 |
| 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 |
| Tehena | Sesame oil | AOCS Cb 2-40 (Baudouin Test) | Colour reaction | I |
| Tempe | Moisture content | AOAC 925.09 AACCI 44- 40.01 | Gravimetry (vacuum oven) | I |

| Miscellaneous Products | | | | |
|------------------------|--|--|--|----|
| Tempe | Protein content | NMKL 6 or AOAC 988.05 or AACCI 46-16.01 (Nitrogen factor 5.71) | Titrimetry, Kjeldahl digestion | I |
| Tempe | Lipid Content | AOAC 963.15 | Gravimetry (Soxhlet Extraction) | I |
| Tempe | Crude fibre | ISO 5498 or AOAC 962.09 or AACCI 32-10.01 | Gravimetry | I |
| Laver products | Moisture Content | AOAC 925.45B | Gravimetry, drying at atmospheric pressure | IV |
| Unrefined shea butter | Moisture content | ISO 662 | Gravimetry | ı |
| Unrefined shea butter | Free fatty acid content acid value and acidity | ISO 660 | Titrimetry | I |
| | | AOCS Cd 3d-63 | | |
| Unrefined shea butter | Relative density | AOCS Cc 10c-95/ | Pycnometry | I |
| | | ISO 6883 | | |
| Unrefined shea butter | Saponification value | ISO 3657 | Titrimetry | I |
| | | AOCS Cd 3d-25 | | |
| Unrefined shea butter | lodine value | AOAC 993.20/ | Wijs Titrimetry | I |
| | | ISO 3961/ | | |
| | | AOCS Cd 1d-92/ | | |
| | | NMKL 39 | | |
| Unrefined shea butter | Peroxide value | AOCS Cd 8b-90/ | Titrimetry | I |
| | | ISO 3960/ | · | |
| | | NMKL 158 | | |
| Unrefined shea butter | Unsaponifiable matter | ISO 3596/ | Gravimetry | I |

| Miscellaneous Products | | | | |
|------------------------|------------------------------|---------------|---------------------------|---|
| | | AOCS Ca 6a-40 | | |
| Unrefined shea butter | Insoluble impurities content | ISO 663/ | Gravimetry | I |
| | | AOCS Ca 3a-46 | | |
| Unrefined shea butter | Melting point | ISO 6321 | Open ended capillary tube | I |
| | | AOCS Cc 3b-92 | | |

PART B - METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

| Commodity Categories | Method of Sampling | Notes | |
|--|---------------------------|---|--|
| Cereals, Pulses and Legumes and Derived Products | | | |
| Wheat protein products including wheat gluten | ISO 13690 | | |
| Fats and Oils | | | |
| Olive Oils and Olive-Pomace Oils | ISO 661 and ISO 5555. | | |
| Fish oils | ISO 5555 | | |
| Milk and Milk Products | | | |
| Milk products | ISO 707 IDF 50 | General instructions for obtaining a sample from a bulk | |
| Milk products | ISO 5538 IDF 113 | Inspection by attributes | |
| Milk products | ISO 3951-1 | Inspection by variables | |
| Processed Fruits and Vegetables | | | |
| Desiccated coconut | Described in the Standard | | |
| Certain canned vegetables, jams and jellies | Described in the Standard | | |
| Chili sauce | Described in the Standard | | |
| Table Olives | Described in the Standard | | |

Appendix I

STANDARD PROCEDURE FOR THAWING OF QUICKEN FROZEN FRUITS AND VEGETABLES

1. SCOPE

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

2. FIELD OF APPLICATION

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

3. DEFINITIONS

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

4. PRINCIPLE OF METHODS

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

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

5. APPARATUS

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

6. SAMPLES

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

7. PROCEDURE

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

7.1 Air thawing

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

7.2 Water thawing by indirect contact

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

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

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

8. NOTES ON PROCEDURE

8.1 Selection of thawing method

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

8.2 Prevention of damage

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

9. TEST REPORT

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

10. ADDITIONAL NOTES

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

Appendix II

STANDARD PROCEDURE FOR COOKING OF QUICK FROZEN VEGETABLES

1. SCOPE

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

2. FIELD OF APPLICATION

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

3. **DEFINITION**

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

4. PRINCIPLE OF METHOD

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

5. APPARATUS

- 5.1 Two-litre sauce pan with cover:
- 5.2 Hot plate or gasfire;
- 5.3 Tray on which product is placed after cooking for cooling and presentation;
- 5.4 Graduated cylinder or similar measuring device for water.

6. SAMPLES

Generally a separate set of samples for cooking purposes only need not be taken. Ordinarily part of the, contents of a larger retail size package or part of a sample of a bulk container, used, for testing other product characteristics can be used for the cooking procedure. Care should be taken, however, that the portion used for cooking is not treated differently from the normal procedure, e.g. thawed prior to cooking whereas the product would usually be put in boiling water while stil1 in the frozen state.

Appendix III

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

1. **DEFINITION**

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

2. PRINCIPLE

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

3. APPARATUS

3.1 Weighted clamp

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

4. PROCEDURE

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

5. CALCULATION AND EXPRESSION OF RESULTS

| | | pods containing tough strings (g) | | |
|-------------------------------------|---|-----------------------------------|-----|-----|
| % m/m pods containing tough strings | = | | _ x | 100 |
| | | test sample (g) | | |

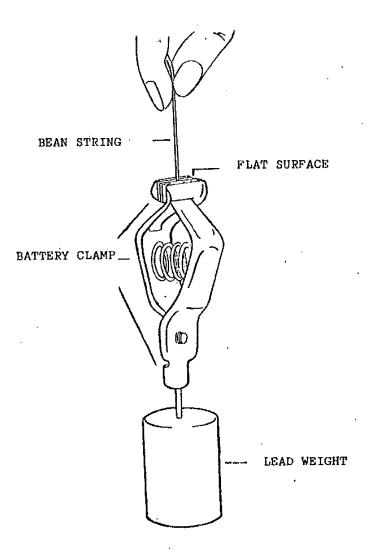


Figure 1 - Tough String Tester for Green or Wax Beans

Appendix IV

DETERMINATION OF THE ALCOHOL-INSOLUBLE SOLIDS CONTENT OFQUICK FROZEN PEAS

1. PRINCIPLE OF THE METHOD

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

2. REAGENTS

2.1 Ethanol (95%) or denaturated ethanol

Ethanol denaturated with 5% v/v methanol.

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

Dilute 8 parts by volume of reagent under 8.2.1 to 9.5 parts by volume with H₂O.

3. APPARATUS

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

4. PREPARATION OF TEST SAMPLE

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

5. PROCEDURE

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

5.2 Weight 20 g \pm 0.01 g paste into a 250 ml ground-joint flask, add 120 ml denaturated ethanol or ethanol, and swirl to mix. Reflux on a steam or water bath for 30 minutes.

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

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

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

6. CALCULATION AND EXPRESSION OF RESULTS

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

6.1 If 20 g sample is refluxed:

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

Where:

M = the mass in g of dry residue (see Section 5.3)

6.2 If 40 g sample is refluxed:

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

Where:

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

7. REPEATIBILITY OF RESULTS

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

8. EXPRESSION OF RESULTS

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

Appendix V

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

PROCEDURE

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