

APPENDIX II**Part 1. METHODS OF ANALYSIS AND SAMPLING PLANS FOR ADOPTION AND REVOCATION BY CAC48**

- 1.1 COCOA PRODUCTS AND CHOCOLATE
- 1.2 FISH AND FISHERY PRODUCTS
- 1.3 FOODS FOR SPECIAL DIETARY USES
- 1.4 FRUIT JUICES AND NECTARS
- 1.5 MILK AND MILK PRODUCTS
- 1.6 MISCELLANEOUS PRODUCTS

Part 2. METHODS OF ANALYSIS WHICH REMAINS UNCHANGED IN CXS 234 AS A RESULT OF DECISIONS BY CCMAS44

- 2.1 CEREALS, PULSES AND LEGUMES AND DERIVED PRODUCTS
- 2.2 FRUIT JUICES AND NECTARS
- 2.3 MILK AND MILK PRODUCTS
- 2.4 MISCELLANEOUS PRODUCTS

Part 3. METHODS OF ANALYSIS FOR FURTHER CONSIDERATION

- 3.1 COCOA PRODUCTS AND CHOCOLATE
- 3.2 FOODS FOR SPECIAL DIETARY USES
- 3.3 FRUIT JUICES AND NECTARS

Part 1

METHODS OF ANALYSIS AND SAMPLING FOR ADOPTION AND REVOCATION BY CAC48

Notes:

1. Methods and performance criteria for inclusion and/or amendment in CXS 234-1999: changes indicated in ~~strike through~~, or **bold** and underlined font.
2. Methods for revocation in CXS 234-1999: ~~strike throughs~~ are indicated in **red**.
3. The references to Appendix VIII and Appendix XI in this document relate to the relevant appendices in CXS 234-1999.

1.1 COCOA PRODUCTS AND CHOCOLATE

Cocoa products and chocolate				
Commodity	Provision	Method	Principle	Type
Chocolate and chocolate products	Fat-free cocoa solids	ICA No. 26 / AOAC 977.10 and AOAC 931.05	Oven evaporation and factor <u>Calculation from moisture (Determined as water) and gravimetry</u>	I
Chocolate and chocolate products	Fat-free milk solids <u>(Determined as Milk Protein)</u>	ICA No. 26 and ICA No. 10000 17 and ICA No. 13 or / AOAC 977.10 and AOAC 955.04C and AOAC 939.02	<u>Calculation from moisture content, and</u> Titrimetry, (Kjeldahl digestion); <u>content of extracted and precipitated</u> after extraction of milk proteins.	I
Chocolate and chocolate products	Fat, total <u>on dry basis</u>	ICA No. 26 / AOAC 977.10 and AOAC 963.15	<u>Calculation from moisture (Determined as Water) and</u> Gravimetry (Soxhlet extraction)	I
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Cocoa shell <u>(determined as spiral vessel count)</u>	AOAC 968.10	<u>Microscopy -</u> Spiral vessel count, <u>stone cell count</u>	I
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Cocoa shell <u>(determined as stone cell count)</u>	AOAC 970.23	<u>Microscopy -</u> Spiral vessel count , stone cell count	I

1.2 FISH AND FISHERY PRODUCTS

Fish and fishery products				
Commodity	Provision	Method	Principle	Type
Fish sauce	Amino acid nitrogen	AOAC 920.04 and AOAC 920.03	Determining formaldehyde titration method <u>and</u> Subtracting by ammoniacal nitrogen (magnesium oxide method)	I
<u>Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter</u>	<u>Determination of fish content (declaration) – Nitrogen</u>	<u>ISO 937</u>	<u>Titrimetry (Kjeldahl digestion)</u>	<u>II</u>
<u>Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter</u>	<u>Determination of fish content (declaration) – Moisture</u>	<u>ISO 1442</u>	<u>Gravimetry</u>	<u>I</u>
<u>Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter</u>	<u>Determination of fish content (declaration) – Total fat</u>	<u>ISO 1443</u>	<u>Gravimetry</u>	<u>I</u>
<u>Quick frozen fish sticks (fish fingers), fish portions and fish fillets – breaded or in batter</u>	<u>Determination of fish content (declaration) – Ash</u>	<u>ISO 936</u>	<u>Gravimetry</u>	<u>I</u>
Salted Atlantic herring and salted sprat and sturgeon caviar	Determination of salt content	See Appendix VIII		
Salted fish and dried salted fish of the Gadidae family of fishes	Salt saturation	See equation in footnote^{xii}	Calculation	I

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

~~1. % salt in water = (% salt content / (% salt content + % moisture)) x 100%~~

~~2. % salt saturation = (% salt in water / 26.4 %*) x 100%~~

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

APPENDIX VIII

PREPARATION OF FISH SAMPLES AND DETERMINATION OF SALT AND WATER CONTENT IN FISH AND FISHERY PRODUCTS**PART 1: PREPARATION OF FISH SAMPLES****~~Salted fish and dried salted fish of the Gadidae family of fishes~~**

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

PART 2: DETERMINATION OF SALT CONTENT**~~Salted fish and dried salted fish of the Gadidae family of fishes, salted Atlantic herring and salted sprat, and sturgeon caviar~~****1. Principle**

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

2. Equipment and chemicals

- ~~— Brush~~
- ~~— Sharp knife or saw~~
- ~~— Balance, accurate to ± 0.01 g~~
- ~~— Calibrated volumetric flasks, 250 ml~~
- ~~— Erlenmeyer flasks~~
- ~~— Electric homogenizer~~
- ~~— Magnetic stirrer~~
- ~~— Folded paper filter, quick running~~
- ~~— Pipettes~~
- ~~— Funnel~~
- ~~— Burette~~
- ~~— Potassium hexacyano ferrate (II), $K_4Fe(CN)_6 \cdot 3H_2O$, 15% w/v (aq)~~
- ~~— Zinc sulphate, $ZnSO_4 \cdot 6H_2O$, 30% w/v (aq)~~
- ~~— Sodium hydroxide, NaOH, 0.1 N, 0.41% w/v (aq)~~
- ~~— Silver nitrate, $AgNO_3$, 0.1 N, 1.6987% w/v (aq), standardized~~
- ~~— Potassium chromate, K_2CrO_4 5% w/v (aq)~~
- ~~— Phenolphthalein, 1% in ethanol~~
- ~~— Distilled or deionized water~~

3. Procedure

- ~~(i) Five grams of homogenized subsample is weighted into a 250 ml volumetric flask and vigorously shaken with approximately 100 ml water.~~
- ~~(ii) Five millilitres of potassium hexacyano-ferrate solution and 5 ml of zinc sulphate solution are added, the flask is shaken.~~
- ~~(iii) Water is added to the graduation mark.~~
- ~~(iv) After shaking again and allowing to stand for precipitation, the flask content is filtered through~~

~~a folded paper filter.~~

~~(v) An aliquot of the clear filtrate is transferred into an Erlenmeyer flask and two drops of phenolphthalein are added. Sodium hydroxide is added dropwise until the aliquot takes on a faint red colour. The aliquot then diluted with water to approximately 100 ml.~~

~~(vi) After addition of approximately 1 ml potassium chromate solution, the diluted aliquot is titrated under constant stirring, with silver nitrate solution. End point is indicated by a faint, but distinct, change in colour. This faint reddish-brown colour should persist after brisk shaking.~~

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

~~(vii) Blank titration of reagents used should be done.~~

~~(viii) End-point determination can also be made by using instruments like potentiometer or colorimeter.~~

4. Calculation of results

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

~~A= volume of aliquot (ml)~~

~~C= concentration of silver nitrate solution in N~~

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

~~W= sample weight (g)~~

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

$$\text{Salt concentration (\%)} = (V \times C \times 58.45 \times 250 \times 100) / (A \times W \times 1000)$$

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

5. Reference method

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

6. Comments

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

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

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

PART 3: DETERMINATION OF WATER CONTENT

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

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

Salted Atlantic herring and salted sprat

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

Table 1. Method performance criteria for sodium chloride and for salt determined as chloride expressed as sodium chloride

Note: The columns “Examples of applicable methods that meet the criteria” and “Principle” are not for adoption or revocation by CAC.

Commodity	Provision	ML (%)	Min. appl. range (%)	LOD (%)	LOQ (%)	Precision (RSD _R) (%) no more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
Boiled dried salted anchovies	Sodium chloride and salt determined as chloride expressed as sodium chloride	15 (NaCl) 9.1 (Cl ⁻)	13.8–16.2 8.3–9.9	1.5 0.91	3.0 1.8	5.3 5.7	98–102 98–102	NMKL 178 AOAC 971.27 AOAC 937.09	Potentiometric titration Potentiometric titration Titration
Fish sauce	<u>Sodium chloride and Salt</u> determined as chloride expressed as sodium chloride	20 (NaCl) Minimum limit <u>From 20 (NaCl)</u> <u>From 12 (Cl⁻)</u>	18 - <u>22</u> 11 - <u>13</u>	2.0 1.2	4.0 2.4	5.1 5.5	98–102 98–102	NMKL 178 AOAC 971.27 AOAC 976.18 AOAC 937.09	Potentiometric titration <u>Titrimetry (Potentiometric)</u> Potentiometric titration <u>Titrimetry (Potentiometric)</u> Titration <u>Titrimetry</u> Potentiometric titration <u>Titrimetry (Potentiometric)</u>
<u>Salted Atlantic herring and salted sprat</u>	<u>Sodium chloride and Salt</u> determined as <u>Chloride expressed as Sodium chloride</u>	<u>From 1 to 20 (NaCl)</u> <u>From 0.6 to 12 (Cl⁻)</u>	<u>0.9 – 22</u> <u>0.5 - 13</u>	<u>0.1</u> <u>0.06</u>	<u>0.2</u> <u>0.12</u>	<u>8.0</u> <u>8.6</u>	<u>97-103</u>	<u>NMKL 178</u> <u>AOAC 971.27</u> <u>AOAC 976.18</u> <u>AOAC 937.09</u>	<u>Titrimetry (Potentiometric)</u> <u>Titrimetry (Potentiometric)</u> <u>Titrimetry</u> <u>Titrimetry (Potentiometric)</u>

Commodity	Provision	ML (%)	Min. appl. range (%)	LOD (%)	LOQ (%)	Precision (RSD _R) (%) no more than	Recovery (%)	Examples of applicable methods that meet the criteria	Principle
<u>Salted Fish and dried salted fish of Gadidae family of fishes</u>	<u>Sodium chloride and Salt determined as Chloride expressed as Sodium chloride</u>	<u>From 12 (NaCl)</u>	<u>11 – 13</u>	<u>1.2</u>	<u>2.4</u>	<u>5.5</u>	<u>98-102</u>	<u>NMKL 178</u>	<u>Titrimetry (Potentiometric)</u>
		<u>From 7.3 (Cl⁻)</u>	<u>6.8 – 8.1</u>	<u>0.8</u>	<u>1.5</u>	<u>5.9</u>		<u>AOAC 971.27</u>	<u>Titrimetry (Potentiometric)</u>
								<u>AOAC 976.18</u> <u>AOAC 937.09</u>	<u>Titrimetry</u> <u>Titrimetry (Potentiometric)</u>
<u>Sturgeon Caviar</u>	<u>Sodium chloride and Salt determined as Chloride expressed as Sodium chloride</u>	<u>From 3 to 5 (NaCl)</u>	<u>2.7 -5.5</u>	<u>0.3</u>	<u>0.6</u>	<u>6.8</u>	<u>97-103</u>	<u>NMKL 178</u>	<u>Titrimetry (Potentiometric)</u>
								<u>AOAC 971.27</u>	<u>Titrimetry (Potentiometric)</u>
		<u>From 1.8 to 3.0 (Cl⁻)</u>	<u>1.7 – 3.4</u>	<u>0.2</u>	<u>0.4</u>	<u>7.3</u>		<u>AOAC 976.18</u> <u>AOAC 937.09</u>	<u>Titrimetry</u> <u>Titrimetry (Potentiometric)</u>

1.3 FOODS FOR SPECIAL DIETARY USES

Foods for special dietary uses				
Commodity	Provision	Method	Principle	Type
<u>Follow-up formula</u>	<u>Vitamin A palmitate (retinyl palmitate), Vitamin A acetate (retinyl acetate)</u>	<u>AOAC 2012.10 / ISO 20633</u>	<u>HPLC-UV</u>	<u>II</u>
Follow-up formula	Vitamin A (retinol isomers)	AOAC 992.04	HPLC- <u>UV</u>	II <u>III</u>
Follow-up formula	Vitamin A (retinol) (above 500 IU/l milk after reconstitution)	AOAC 992.06	HPLC- <u>UV</u>	II <u>III</u>
Follow-up formula	Vitamin A	AOAC 974.29	Colorimetry	IV
<u>Follow-up formula</u>	<u>Vitamin E</u>	<u>AOAC 2012.10 / ISO 20633</u>	<u>HPLC-UV</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Vitamin D</u>	<u>AOAC 2016.05 / ISO 20636</u>	<u>UHPLC-MS/MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Thiamine</u>	<u>AOAC 2015.14 / ISO 21470</u>	<u>Enzymatic digestion and UHPLC-MS/MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Riboflavin</u>	<u>AOAC 2015.14 / ISO 21470</u>	<u>Enzymatic digestion and UHPLC-MS/MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Niacin</u>	<u>AOAC 2015.14 / ISO 21470</u>	<u>Enzymatic digestion and UHPLC-MS/MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Vitamin B₆</u>	<u>AOAC 2015.14 / ISO 21470</u>	<u>Enzymatic digestion and UHPLC-MS/MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Vitamin B₁₂</u>	<u>AOAC 2011.10 / ISO 20634</u>	<u>HPLC-VIS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Vitamin B₁₂</u>	<u>AOAC 2014.02</u>	<u>HPLC-UV</u>	<u>III</u>

Foods for special dietary uses				
Commodity	Provision	Method	Principle	Type
<u>Follow-up formula</u>	<u>Pantothenic acid</u>	<u>AOAC 2012.16 / ISO 20639</u>	<u>UHPLC-MS/MS</u>	<u>II</u>
Follow-up formula	Pantothenic acid	AOAC 992.07 Measures total pantothenate (free pantothenic acid + CoA- + ACP-bound) and measured as D-pantothenic acid (or calcium D-pantothenate)	Microbioassay	II <u>III</u>
<u>Follow-up formula</u>	<u>Folic Acid</u>	<u>AOAC 2011.06 / ISO 20631</u>	<u>HPLC-MS/MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Vitamin C</u>	<u>AOAC 2012.22 / ISO 20635</u>	<u>UHPLC-UV</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Biotin</u>	<u>AOAC 2016.02 / ISO 23305</u>	<u>HPLC-UV</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Iron</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP-MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Iron</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP-OES</u>	<u>III</u>
<u>Follow-up formula</u>	<u>Calcium</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP-MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Calcium</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP-OES</u>	<u>III</u>
<u>Follow-up formula</u>	<u>Phosphorus</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP-MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Phosphorous</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP-OES</u>	<u>III</u>
<u>Follow-up formula</u>	<u>Magnesium</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP-MS</u>	<u>II</u>

Foods for special dietary uses				
Commodity	Provision	Method	Principle	Type
<u>Follow-up formula</u>	<u>Magnesium</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP-OES</u>	<u>III</u>
<u>Follow-up formula</u>	<u>Sodium</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP-MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Sodium</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP-OES</u>	<u>III</u>
<u>Follow-up formula</u>	<u>Chloride</u>	<u>AOAC 2016.03 / ISO 21422 IDF 242</u>	<u>Potentiometry</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Potassium</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP-MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Potassium</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP-OES</u>	<u>III</u>
<u>Follow-up formula</u>	<u>Manganese</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP-MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Manganese</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP-OES</u>	<u>III</u>
<u>Follow-up formula</u>	<u>Iodine</u>	<u>AOAC 2012.15 / ISO 20647 IDF 234</u>	<u>ICP-MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Iodine (milk-based formula)</u>	<u>AOAC 992.24</u>	<u>Ion-selective potentiometry</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Selenium</u>	<u>AOAC 2011.19 / ISO 20649 IDF 235</u>	<u>ICP-MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Copper</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP-MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Copper</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP-OES</u>	<u>III</u>
<u>Follow-up formula</u>	<u>Zinc</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP-MS</u>	<u>II</u>

Foods for special dietary uses				
Commodity	Provision	Method	Principle	Type
<u>Follow-up formula</u>	<u>Zinc</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP-OES</u>	<u>III</u>
<u>Follow-up formula</u>	<u>Total nucleotides</u>	<u>AOAC 2011.20 / ISO 20638</u>	<u>LC SPE -HPLC-UV</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Choline</u>	<u>AOAC 2015.10 / ISO 21468</u>	<u>UHPLC-MS/MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Myo-inositol</u>	<u>AOAC 2011.18 / ISO 20637</u>	<u>HPLC-PAD</u>	<u>II</u>
<u>Follow-up formula</u>	<u>L-carnitine</u>	<u>AOAC 2015.10 / ISO 21468</u>	<u>UHPLC-MS/MS</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Total amino acids (excluding taurine and tryptophan) for use according to section 3.1.3 (a) notes 2) and 3) of CXS 156-1987</u>	<u>AOAC 2018.06 / ISO 4214 IDF 254 / AACC 07-50.01</u>	<u>UHPLC-UV</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Tryptophan</u>	<u>AOAC 2017.03</u> <u>For use according to Section 3.1.3 (a) notes 2 and 3 of CXS 72-1981</u>	<u>HPLC-FLD</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Total fatty acids</u>	<u>AOAC 2012.13 / ISO 16958 IDF 231</u>	<u>GC-FID</u>	<u>II</u>
<u>Follow-up formula</u>	<u>Total fatty acids</u>	<u>AOAC 996.06</u>	<u>GC</u>	<u>III</u>
<u>Follow-up formula</u>	<u>Crude protein</u>	<u>ISO 8968-1 IDF 20-1</u>	<u>Titrimetry (Kjeldahl digestion)</u>	<u>I</u>
Infant formula	Folic acid	AOAC 2011.06	<u>UHPLC-MS/MS</u>	<u>II</u>
<u>Infant formula</u>	<u>Folic acid</u>	<u>ISO 20631</u>	<u>UHPLC-MS/MS</u>	<u>II</u>

Table 2. Methods of analysis for dietary fibre: *Guidelines for use of nutrition and health claims* (CXG 23-1997): Table of conditions for claims

Standard	Provisions	Method	Principle	Type
General methods that measure both the higher (monomeric units > 9) and the lower molecular weight fraction (monomeric units ≤9) ⁽²⁾				
<u>All foods (1)</u>	<u>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</u>	<u>AOAC 2022.01/ AACC 32-61.01/ ICC Standard No. 191**</u>	<u>Enzymatic-gravimetry and HPLC</u>	<u>Type I</u>
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

** Isolated, purified, and/or synthetic fibres captured by AOAC 2022.01/ICC Standard 191/AACC 32-61.01 that do not meet the Codex definition of dietary fibre in the *Guidelines on nutrition labelling* (CXG 2-1985) should be subtracted from the final measurement, where deemed appropriate by competent authorities.

1.4 FRUIT JUICES AND NECTARS

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Ascorbic acid-L (additives)	IFUM A 17 Aa	HPLC	II
Fruit juices and nectars	Ascorbic acid-L (additives)	AOAC 967.21 IFUMA 17 ISO 6557-2	Indophenol method	III
Fruit juices and nectars	Ascorbic acid-L (additives)	IFUM A 17 b	Indophenol <u>Iodine</u> method	III
Fruit juices and nectars	Ascorbic acid-L (additives)	ISO 6557-1	Fluorescence spectrometry <u>(reference method)</u>	IV
Fruit juices and nectars	Carbon dioxide (additives and processing aids)	IFUMA 42	Titrimetry (back-titration after precipitation)	IV
Fruit juices and nectars	Citric acid ^{xviii} (additives)	AOAC 986.13	HPLC	II
Fruit juices and nectars	<u>High Fructose Corn Syrup HFCS</u> and <u>Hydrolyzed Inulin Syrup HIS</u> in apple juice (permitted ingredients)	Determination of HFCS and HIS by Capillary GC method JAOAC 84, 486 (2001) / <u>IFU recommendation No. 4</u>	CAP GC method	IV
Fruit juices and nectars	Malic acid-L	EN-1138	Enzymatic determination	II
Fruit juices and nectars	Malic acid-L	IFUM A 21	Enzymatic determination	II
Fruit juices and nectars	Saccharin	NMKL 122	Liquid chromatography <u>HPLC</u>	II
Fruit juices and nectars	Soluble solids	AOAC 983.17 / EN 12143 / IFUM A 8 / ISO 2173	Indirect by refractometry	I

^{xviii} All juices except citrus based juices.

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Sucrose (permitted ingredients)	EN 12146 / IFUM A 56	Enzymatic determination	III
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005 <u>Phosphorous / phosphate</u>	Determination of phosphorus/phosphate EN 1136 / IFUM A No 50	Photometric determination	II

1.5 MILK AND MILK PRODUCTS

Milk and milk products				
Commodity	Provision	Method	Principle	Type
<u>Butter</u>	<u>Salt (Determined as chloride expressed as NaCl)</u>	<u>AOAC 2016.03 / ISO 21422 IDF 242</u>	<u>Potentiometry</u>	<u>III</u>
Butter	Salt (Determined as chloride expressed as NaCl)	ISO 15648 IDF 179	Potentiometry (determination of chloride, expressed as sodium chloride)	II
<u>Cheese</u>	<u>Sodium Chloride (Determined as chloride, expressed as NaCl)</u>	<u>AOAC 2016.03 / ISO 21422 IDF 242</u>	<u>Potentiometry</u>	<u>III</u>
Cheese	Sodium chloride (Determined as chloride expressed as NaCl)	ISO 5943 IDF 88	Potentiometry (determination of chloride, expressed as sodium chloride)	II
<u>Whey powders</u>	<u>Water^{xlii} (moisture)^{***}</u>	<u>Described in Appendix XI</u>	<u>Gravimetry (drying at 102°C)</u>	<u>IV</u>

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

^{***} Due to accessibility to equipment and calibration of the method ISO 5537 | IDF 26, the method as described in Appendix XI is listed as Type IV. In a dispute situation, the Type I method shall be used. This 102°C method is less precise, and results may not be consistent with results obtained with ISO 5537 | IDF 26, in particular for powders with high natural lactose such as whey powders.

APPENDIX XI

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

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

DESCRIPTION OF THE METHOD: DETERMINATION OF MOISTURE

1. SCOPE

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

Table 3: Numeric performance criteria for methods of analysis for copper and iron in milk fat products

(Note: the numeric performance criteria are not for adoption or revocation. The only changes in underlined and/or ~~strike through~~ font are amendments / removal of example methods / principles)

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

1.6 MISCELLANEOUS PRODUCTS

Miscellaneous products				
Commodity	Provision	Method	Principle	Type
<u>Dried meat</u>	<u>Chloride as sodium chloride</u>	<u>AOAC 935.47 and AOAC 937.09B</u>	<u>Titrimetry (Volhard method)</u>	<u>III</u>
Food-grade <u>Food grade</u> salt	Iodine	WHO/UNICEF/ICCIDD method ^{xlv} Only applicable to a product which has been fortified with iodate	Titrimetry using sodium thiosulphate	IV
<u>Food grade salt</u>	<u>Sodium chloride</u>	<u>See Appendix * Part A</u>	<u>Calculation</u>	<u>I</u>

PART B – METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

Commodity categories	Method of sampling	Notes
Miscellaneous products		
<u>Food grade salt</u>	<u>See Appendix * Part B</u>	

^{xlv} 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~~ <https://www.who.int/publications/i/item/9789241595827>

APPENDIX ***DETERMINATION OF SODIUM CHLORIDE AND RELATED
SAMPLING METHOD FOR FOOD GRADE SALT****Part A. DETERMINATION OF SODIUM CHLORIDE CONTENT**

This method allows the calculation of sodium chloride content, as provided for in CXS 150 Section 3.1, on the basis of the results of the determinations of sulphate, calcium and magnesium, potassium and loss on drying. Convert sulphate to CaSO_4 and unused calcium to CaCl_2 , unless sulphate in sample exceeds the amount necessary to combine with calcium, in which case convert calcium to CaSO_4 and unused sulphate first to MgSO_4 and any remaining sulphate to Na_2SO_4 . Convert unused magnesium to MgCl_2 . Convert potassium to KCl. Convert unused halogens to NaCl. Report the NaCl content on a dry matter basis, multiplying the percentage NaCl by $100/100-P$, where P is the percentage loss on drying.

Part B. SAMPLING: METHOD FOR THE SAMPLING OF FOOD GRADE SALT FOR THE DETERMINATION OF SODIUM CHLORIDE**1. SCOPE**

This method specifies the sampling procedure to be applied when determining the main component in order to assess the food grade quality of sodium chloride (salt) as provided for in the Codex Standard for Food Grade Salt, Section 3: "Essential Composition and Quality Factors".

The criterion to be used for acceptance or rejection of a lot or consignment on the basis of this sample is also provided.

2. FIELD OF APPLICATION

This method is applicable to the sampling of any type of salt intended for use as food, either prepacked or in bulk.

3. PRINCIPLE

This method represents a variables sampling procedure for mean quality: blended bulk sample analysis.

A blended bulk sample is produced in such a way that it is representative of the lot or consignment. It is composed of a proportion of items drawn from the lot or consignment to be analyzed.

Acceptance criterion is on the basis that the mean value obtained from analyses of those blended bulk samples must comply with the provision in the Standard.

4. DEFINITIONS

The terms used in this sampling method refer to those in the "General Guidelines on Sampling" (CXG 50-2004) unless stated otherwise.

5. EQUIPMENT

The sampling equipment used should be adapted to the nature of the tests to be carried out (for example: sampling by borer, sampling equipment made of chemically inert material, etc.). The containers used for collecting the samples should be made of a chemically inert material and should be air-tight.

6. PROCEDURE**6.1 PREPACKED SALT**

Sampling may be carried out by "random sampling" or by "systematic sampling". The choice of the method to be used depends on the nature of the lot (for example: if the packages are marked with successive numbers, systematic sampling may be suitable).

6.1.1 Random sampling

Draw the n items from the lot in such a way that each item in the lot has the same chance of being selected.

6.1.2 Systematic sampling

If the N units in the lot have been classified and can be numbered from 1 to N, the 1-in-k systematic sampling of n items can be obtained as follows:

- a) Determine the k value as $k = N/n$. (If k is not an integer, then round to the nearest integer).

- b) From the first k items in the lot take one at random and then take every kth item thereafter.

6.2 SALT IN BULK

Here, the lot is fictitiously divided into items (strata); a lot with a total mass of m kg is considered to be composed of m/100 items. In this case, it is necessary to draw up a "stratified sampling" plan appropriate to the lot dimension. The samples are selected from all the strata in proportion to the stratum sizes.

Note: Stratified sampling of a population which can be divided into different subpopulations (called strata) is carried out in such a way that specified proportions of the sample are drawn from the different strata.

6.3 CONSTITUTION OF THE SAMPLE

6.3.1 The size and the number of the items forming the sample depend on the type of salt and the lot magnitude. The minimum size to be taken into account should be in accordance with one of the following specifications according to the circumstances:

- 250 g of salt in bulk or prepacked in more than 1 kg packages;
- one package for prepacked salt in 500 g or 1 kg packages.

The appropriate number of samples to be drawn from the lot, shall be determined in accordance with "General Guidelines on Sampling" (CXG 50-2004).

6.3.2 Combine and mix well the different items drawn from the lot. This blended bulk sample constitutes the laboratory sample. More than one laboratory sample may be composed in such a manner.

7. ACCEPTANCE CRITERION

7.1 Determine the NaCl content (%) of at least two test portions of the laboratory sample.

7.2 Calculate the average of the results obtained for the n test portions of the laboratory sample using:

$$\bar{x} = \frac{\sum x}{n} (n \geq 2)$$

7.3 In accordance with the provision for the relevant NaCl content (%), a lot or a consignment shall be considered acceptable if the following condition is verified:

$\bar{x} \geq$ minimum level specified.

8. SAMPLING REPORT

The sampling report should contain the following information:

- a) type and origin of the salt;
- b) alterations of state of the salt (e.g. presence of foreign matter);
- c) date of sampling;
- d) lot or consignment number;
- e) method of packing;
- f) total mass of lot or consignment
- g) number, unit mass of packages and whether the mass is given net or gross;
- h) number of items sampled;
- i) number, nature and initial position of sampled items;
- j) number, composition and mass of the bulk sample(s) and the method used to obtain and conserve it (them);
- k) names and signatures of the people who carried out the sampling.

Part 2

METHODS OF ANALYSIS WHICH REMAINS UNCHANGED IN CXS 234 AS A RESULT OF DECISIONS BY CCMAS44

2.1 CEREALS, PULSES AND LEGUMES AND DERIVED PRODUCTS

Cereals, pulses and legumes and derived products				
Commodity	Provision	Method	Principle	Type
Quinoa	Protein	ISO 1871	Titrimetry (Kjeldahl digestion)	IV

2.2 FRUIT JUICES AND NECTARS

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Malic acid (additives)	AOAC 993.05	Enzymatic determination and HPLC	III
Fruit juices and nectars	Preservatives in fruit juices (sorbic acid and its salts)	ISO 5519	Spectrometry	III

2.3 MILK AND MILK PRODUCTS

Milk and milk products				
Commodity	Provision	Method	Principle	Type
Whey powders	Water ^{xlii} (moisture)	ISO 5537 IDF 26	Gravimetry (drying at 87°C)	I

2.4 MISCELLANEOUS PRODUCTS

Miscellaneous products				
Commodity	Provision	Method	Principle	Type
Dried meat	Chloride as sodium chloride (≥ 1.0%)	ISO 1841-1	Titrimetry (Volhard method)	III
Dried meat	Chloride as sodium chloride (≥ 0.25%)	ISO 1841-2	Titrimetry (potentiometry)	II

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

Part 3

METHODS OF ANALYSIS FOR FURTHER CONSIDERATION

3.1 COCOA PRODUCTS AND CHOCOLATE (For further consideration by the EWG on cocoa products and chocolate)

Note: Text indicated in ~~strike through~~, or **bold** and underlined font indicate changes and/or additions discussed in relation to the method of analysis as it currently appears in CXS 234-1999.

Cocoa products and chocolate				
Commodity	Provision	Method	Principle	Type
Chocolate and chocolate products	Cocoa butter <u>(determined as fat)</u>	<u>ICA No. 26 / AOAC 977.10 and</u> AOAC 963.15 / ICA IOCCC 14	<u>Calculation from moisture (Determined as Water) and</u> Gravimetry (Soxhlet extraction)	I
Chocolate and chocolate products	Milk_fat	IOCCC ICA No. 5 <u>AOAC 945.34; 925.41B; 920.80</u>	Titrimetry/Distillation	↓ <u>IV</u>
Chocolate and chocolate products	Moisture	IOCCC 26 or AOAC 977.10 (Karl Fischer method); or AOAC 931.04 or IOCCC ICA No. 1	Gravimetry - <u>drying at 100-102°C</u>	↓ <u>IV</u>
<u>Chocolate and chocolate products</u>	<u>Moisture (Determined as Water)</u>	<u>ICA No. 26 / AOAC 977.10</u>	<u>Titrimetry - Karl Fischer</u>	<u>II</u>
Chocolate and chocolate products	Non-cocoa butter vegetable fat	AOCS Ce 10/02 and described in the standard	Described in the standard <u>GC-MS</u>	↓ <u>IV</u>
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	Fat	<u>ICA No. 26 / AOAC 977.10 and</u> AOAC 963.15 / or IOCCC ICA No. 14	<u>Calculation from moisture (Determined as Water) and</u> Gravimetry (Soxhlet extraction)	I
Cocoa butter	Free fatty acids	ISO 660 or / AOCS Cd 3d-63	Titrimetry	I
Cocoa butter	Unsaponifiable matter	ISO 3596 or / ISO 18609 or / AOCS Ca 6b-53	Titrimetry after extraction with diethyl ether	I
Cocoa powders (cocoa) and dry cocoa-sugar mixtures	Moisture <u>(Determined as Water)</u>	IOCCC ICA No. 26 or / AOAC 977.10 (Karl Fischer method)	Gravimetry <u>Titrimetry - Karl Fischer</u>	↓ <u>II</u>

Cocoa products and chocolate				
Commodity	Provision	Method	Principle	Type
<u>Chocolate and chocolate products</u>	<u>Cocoa butter equivalents in cocoa butter and plain chocolate</u>	<u>ISO 23275-1 and ISO 23275-2 / AOCS Ce 11-05</u>	<u>GC-FID</u>	!
<u>Chocolate and chocolate products</u>	<u>Cocoa Butter Equivalents in Milk Chocolate</u>	<u>ISO 11053 / AOCS Ce 11a-07</u>	<u>GC-FID</u>	!
<u>Chocolate and chocolate products</u>	<u>Determination of centre and coating of filled chocolate</u>	<u>See Appendix **</u>		
<u>Cocoa powders (cocoas) and dry mixtures of cocoa and sugars</u>	<u>Determination of full-fat cocoa powder, fat-reduced cocoa powder and highly fat-reduced cocoa powder</u>	<u>AOAC 977.04 or IOCCC 26 (1988)-Karl Fisher Method</u>		
<u>Cocoa powders (cocoas) and dry mixtures of cocoa and sugars</u>	<u>Determination of cocoa butter</u>	<u>To be developed</u>		

APPENDIX **: DETERMINATION OF CENTRE AND COATING OF FILLED CHOCOLATE IN CHOCOLATE AND CHOCOLATE PRODUCTS

All methods approved for the chocolate type used for the coating and those approved for the type of centre concerned.

3.2 FOODS FOR SPECIAL DIETARY USES (For CCNFSDU's consideration)

Foods for special dietary uses				
Commodity	Provision	Method	Principle	Type
Follow-up formula	Vitamin A	EN 12823-1 (all-trans-retinol and 13-cis-retinol) Vitamin A (both natural + supplemental ester forms) aggregated and quantified as individual retinol isomers (13-cis and all-trans)	HPLC-UV or FL	III
Follow-up formula	Vitamin E	EN 12822 (Measures vitamin E (both natural + supplemental ester forms) aggregated and quantified as individual tocopherol congeners (α , β , γ , δ))	HPLC-UV or FL	III
Follow-up formula	Vitamin E	AOAC 992.03 Measures all rac-vitamin E (both natural + supplemental ester forms) aggregated and quantified as α -congeners	HPLC-UV	III
Follow-up formula	Vitamin D	EN 12821 / NMKL 167 (D2 and/or D3 measured as single components. Hydroxylated forms not measured)	HPLC-UV	III
Follow-up formula	Vitamin D	AOAC 995.05 D2 and D3 measured	HPLC-UV	III
Follow-up formula	Thiamine	AOAC 986.27****	Fluorimetry	III
Follow-up formula	Thiamine	EN 14122 (Measures all vitamin B1 forms (natural and added free, bound and phosphorylated) following extraction and conversion to thiamine)	HPLC-FL (with pre-or post-column derivatization to thiochrome)	III
Follow-up formula	Riboflavin	EN 14152	HPLC-FL	III

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

Foods for special dietary uses				
Commodity	Provision	Method	Principle	Type
		(Measures natural and supplemental forms, free, bound and phosphorylated (FMN and FAD) aggregated and measured as riboflavin)		
Follow-up formula	Riboflavin	AOAC 985.31****	Fluorimetry	III
Follow-up formula	Niacin	EN 15652 (Free and bound and phosphorylated forms measured either as aggregate of nicotinic acid + nicotinamide, or as individual forms)	HPLC-FL (with post-column photochemical derivatization)	III
Follow-up formula	Niacin	AOAC 985.34 (niacin (preformed) and nicotinamide)	Microbioassay and turbidimetry	III
Follow-up formula	Vitamin B ₆	EN 14166 (Aggregates free and bound pyridoxal, pyridoxine and pyridoxamine and measures as pyridoxine)	Microbioassay	III
Follow-up formula	Vitamin B ₆	AOAC 985.32	Microbioassay	III
Follow-up formula	Vitamin B ₆	AOAC 2004.07 / EN 14164 (Free and bound phosphorylated forms (pyridoxal, pyridoxine and pyridoxamine) converted and measured as pyridoxine)	HPLC-FL	III
Follow-up formula	Vitamin B ₁₂	AOAC 986.23 (Measures total vitamin B12 as cyanocobalamin)	Turbidimetry	III
Follow-up formula	Folic acid	EN 14131 (Total folate (free + bound), aggregated and measured as folic acid)	Microbioassay	III
Follow-up formula	Folic acid	AOAC 992.05 (Measures free folic acid + free, unbound natural folates, aggregated, and measured as folic acid)	Microbioassay	III

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

Foods for special dietary uses				
Commodity	Provision	Method	Principle	Type
Follow-up formula	Biotin	EN 15607 (d-biotin) (Measures total D-biotin [free + D-biocytn])	HPLC- FLD (post-column derivatization)	III
Follow-up formula	Iron	AOAC 985.35	FAAS	III
Follow-up formula	Iron	AOAC 999.11 NMKL 139	FAAS	III
Follow-up formula	Calcium	ISO 8070 IDF 119	FAAS	III
Follow-up formula	Calcium	AOAC 985.35	FAAS	III
Follow-up formula	Phosphorous	AOAC 986.24	Spectrophotometry	III
Follow-up formula	Magnesium	ISO 8070 IDF 119	FAAS	III
Follow-up formula	Magnesium	AOAC 985.35	FAAS	III
Follow-up formula	Sodium	ISO 8070 IDF 119	FAAS	III
Follow-up formula	Chloride	AOAC 986.26	Potentiometry	III
Follow-up formula	Potassium	ISO 8070 IDF 119	FAAS	III
Follow-up formula	Manganese	AOAC 985.35	FAAS	III
Follow-up formula	Selenium	AOAC 2006.03	ICP-OES	III
Follow-up formula	Selenium	EN 14627	HGAAS	III
Follow-up formula	Selenium	AOAC 996.16	Fluorimetry	III

Foods for special dietary uses				
Commodity	Provision	Method	Principle	Type
Follow-up formula	Selenium	AOAC 996.17	HGAAS	III
Follow-up formula	Copper	AOAC 985.35	FAAS	III
Follow-up formula	Zinc	AOAC 985.35	FAAS	III
Follow-up formula	Choline	AOAC 999.14	Enzymatic colorimetric method with limitations on applicability due to choline and ascorbate concentration	III

3.3 FRUIT JUICES AND NECTARS (For further consideration by the Expert Group)

Note: Text indicated in ~~strike through~~, or **bold** and underlined font indicate changes and/or additions discussed in relation to the method of analysis as it currently appears in CXS 234-1999.

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Ascorbic acid-L (additives)	ISO 6557-2: <u>1995</u>	Indophenol method A) <u>Titrimetry</u> B) <u>(for strongly coloured)</u> <u>Spectrometry</u>	III <u>IV</u>
Fruit juices and nectars	Citric acid ^{xviii} (additives)	AOAC 986.13 <u>(1996)</u>	HPLC	II
Fruit <u>Apple</u> juices and nectars	Malic acid-D in apple juice	AOAC 995.06 <u>(1998)</u>	HPLC	II
Fruit juices and nectars	Quinic, malic and citric acid in cranberry juice cocktail and apple juice (permitted ingredients and additives)	Determination of quinic, malic and citric acid in cranberry juice cocktail and apple juice AOAC 986.13 <u>(1986)</u>	HPLC	III
Fruit juices and nectars	Sucrose (permitted ingredients)	EN 12630 IFUMA 67 <u>(2005)</u> / NMKL 148 <u>(1993)</u>	HPLC	II
Fruit juices and nectars	Cellobiose	IFUMA <u>Recommendation No. 4 October 2000</u>	Capillary gas chromatography <u>GC</u>	IV
Fruit juices and nectars	Citric acid ^{xxix} (additives)	EN 1137 IFUMA 22 <u>(2005)</u>	Enzymatic determination	III
Fruit juices and nectars	Glucose-D and fructose-D (permitted ingredients)	EN 1140 IFUMA 55 <u>(2005)</u>	Enzymatic determination	II
Fruit juices and nectars	Malic acid-D	EN 12138 IFUMA 64 <u>(2005)</u>	Enzymatic determination	II
Fruit juices and nectars	Pectin (additives)	IFUMA 26 <u>(2012)</u>	Precipitation/photometry	I

^{xviii} All juices except citrus based juices.

^{xxix} All juices except citrus based juices.

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Benzoic acid and its salts; sorbic acid and its salts	IFUMA 63 (2005) NMKL 124 (1997)	HPLC	II
Fruit juices and nectars	Benzoic acid and its salts	ISO 5518:2011, ISO 6560:1983	Spectrometry	III
Fruit juices and nectars	Sulphur dioxide (additives)	Optimized Monier-Williams AOAC 990.28 (2005) IFUMA 7A (2018) NMKL 132 (1989)	Titrimetry after distillation	II
Fruit juices and nectars	Sulphur dioxide (additives)	NMKL 135 (1990)	Enzymatic determination	III
Fruit juices and nectars	Sulphur dioxide (additives)	ISO 5522:1995, ISO 5523:1995	Titrimetry after distillation	III
Fruit juices and nectars	Tartaric acid in grape juice (additives)	EN 12137 IFUMA 65 (2005)	HPLC	II
Fruit juices and nectars	Total nitrogen	EN 12135 IFUMA 28 (2005)	Digestion/titration	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Acetic acid (acetate)</u>	Determination of acetic acid <u>EN 12632; IFUMA 66 (2019)</u>	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Alcohol (ethanol)</u>	Determination of alcohol (ethanol) <u>IFUMA 52 (2005)</u>	Enzymatic determination	II

^{xx} 3.4 Verification of composition, quality and authenticity

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

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

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Anthocyanins</u>	Detection of anthocyanins IFUMA 71 (2023)	HPLC	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Ash</u>	Determination of ash in fruit products AOAC 940.26 (1940): 525°C; EN 1435; IFUMA 9 (2005): 500-550°C	Gravimetry	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Beet sugar</u>	Detection of beet sugar in fruit juices AOAC 995.17 (1998)	Deuterium <u>SNIF-NMR</u>	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Benzoic acid</u>	Determination of benzoic acid as a marker in orange juice AOAC 994.11 (1964)	HPLC	III
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>C¹³/C¹² ratio of ethanol derived from fruit juices</u>	Determination of C¹³/C¹² ratio of ethanol derived from fruit juices JAOAC 79, No. 1, 1996, 62-72	Stable isotope mass spectrometry	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Carbon stable isotope ratio</u>	Determination of carbon stable isotope ratio of apple juice AOAC 981.09 (1997)– JAOAC 64, 85 (1981)	Stable isotope mass spectrometry	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Carbon stable isotope ratio</u>	Determination of carbon stable isotope ratio of orange juice AOAC 982.21 (1997)	Stable isotope mass spectrometry	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Carotenoid, total/individual groups</u>	Determination of carotenoid, total/individual groups EN 12136; IFUMA 59 (2008)	Spectrophotometry	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Centrifugable pulp</u>	Determination of centrifugable pulp EN 12134; IFUMA 60 (2005)	Centrifugation/% value	I

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Chloride (expressed as sodium chloride)</u>	Determination of chloride (expressed as sodium chloride) EN 12133 IFUMA 37 (2005)	Electrochemical titrimetry	III
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Chloride</u>	Determination of chloride in vegetable juice AOAC 971.27 (1996) (Codex general method) ISO 3634:1995	Titration	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Essential oils</u>	Determination of essential oils (Scott titration) AOAC 968.20 (1969) – IFUMA 45 (2005) ^{xxi}	(Scott) distillation, titration	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Essential oils in citrus fruit</u>	Determination of essential oils (in citrus fruit) (volume determination) Error! Bookmark not defined. ISO 1955:1995	Distillation and direct reading of the volume determination	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Fermentability</u>	Determination of fermentability IFUMA 18 (1998)	Microbiological method	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Formol number</u>	Determination of formol number EN 1133 IFUMA 30 (2005)	Potentiometric titration	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Free amino acids</u>	Determination of free amino acids EN 12742 IFUMA 57 (2005)	Liquid chromatography LC	II

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

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Fumaric acid</u>	Determination of fumaric acid IFUMA 72 (1998)	HPLC	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Glucose, fructose, saccharose</u>	Determination of glucose fructose and saccharose EN 12630 IFUMA 67 (2005) NMKL 148 (1993)	HPLC	II or III
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Gluconic acid</u>	Determination of gluconic acid IFUMA 76 (2006)	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Glycerol</u>	Determination of glycerol IFUMA 77 (2005)	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Hesperidin and naringin</u>	Determination of hesperidin and naringin EN 12148 IFUMA 58 (2005)	HPLC	II
Fruit Apple juices and nectars	<u>High Fructose Corn Syrup and Hydrolyzed Inulin Syrup</u> HFCS and HIS in apple juice (permitted ingredients)	Determination of HFCS and HIS by Capillary GC method JAOAC 84, 486 (2001)	CAP GC method	IV
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Hydroxymethylfurfural</u>	Determination of hydroxymethylfurfural IFUMA 69 (2005)	HPLC	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Hydroxymethylfurfural</u>	Determination of hydroxymethylfurfural ISO 7466:1986	Spectrometry	III
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Isocitric acid-D</u>	Determination of isocitric acid-D IFUMA 54 (2005)	Enzymatic determination	II

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Isocitric acid-D	EN 1139 (1999)	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Lactic acid- D and L</u>	Determination of Lactic acid- D and L <u>EN 12631</u> <u>IFUMA 53 (2005)</u>	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>L-malic/total malic acid ratio – to detected added D-malic acid</u>	Determination of L-malic/total malic acid ratio in apple juice <u>AOAC 993.05 (1997)</u>	Enzymatic determination and HPLC	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Naringin and Neohesperidin</u>	Determination of naringin and neohesperidin in orange juice <u>AOAC 999.05 (2002)</u>	HPLC	III
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>pH - value</u>	Determination of pH value <u>NMKL 179 (2005)</u>	Potentiometry	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>pH - value</u>	EN 1132 <u>IFUMA 11 (2015)</u> <u>ISO 1842:1995</u>	Potentiometry	IV
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Proline</u>	Determination of proline by photometry – non-specific determination <u>EN 1141 IFUMA 49 (2005)</u>	Photometry	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Relative density</u>	Determination of relative density <u>EN 1131 (1993); IFUMA 01 (2005) & IFU</u> <u>Method No General sheet (1971)</u>	Pycnometry	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Relative density</u>	Determination of relative density <u>IFUMA 01A (2005)</u>	Densitometry	III

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Sodium, potassium, calcium, magnesium</u>	Determination of sodium, potassium, calcium, magnesium in fruit juices <u>EN 1134-IFUMA 33 (2005)</u>	Atomic absorption spectroscopy AAS	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Sorbitol-D</u>	Determination of sorbitol-D <u>IFUMA 62 (2005)</u>	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Stable carbon isotope ratio</u>	Determination of stable carbon isotope ratio in the pulp of fruit juices <u>ENV 13070</u> Analytica Chimica Acta 340 (1997)	Stable isotope mass spectrometry	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Stable carbon isotope ratio of sugars from fruit juices</u>	Determination of stable carbon isotope ratio of sugars from fruit juices <u>ENV 12140</u> Analytica Chimica Acta 271 (1993)	Stable isotope mass spectrometry	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx}	Determination of stable hydrogen isotope ratio of water from fruit juices <u>ENV 12142</u>	Stable isotope mass spectrometry	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx}	Determination of stable oxygen isotope ratio in fruit juice water <u>ENV 12141</u>	Stable isotope mass spectrometry	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Starch</u>	Detection of starch AOAC 925.38 (1925) <u>IFUMA 73 (2000)</u>	Colorimetric	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Sugar beet derived syrups in frozen concentrated orange juice</u>	Determination of sugar beet derived syrups in frozen concentrated orange juice <u>$\delta^{18}\text{O}$ Measurements in water</u> AOAC 992.09 (1997)	Oxygen isotope ratio analysis (<u>$\delta^{18}\text{O}$ in water</u>)	I

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Titrateable acids</u>	Determination of titrable acids, total EN 12147 IFUMA 03 (2017) ISO 750: 1998	Titrimetry	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Total dry matter at 70°C</u>	Determination of total dry matter (vacuum oven drying at 70 °C)^{xxii} EN 12145 IFUMA 61 (2005)	Gravimetry ie determination	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Total solids (Microwave oven drying)</u>	Determination of total solids (microwave oven drying) ^{Error! Bookmark not defined.} AOAC 985.26 (2001)	Gravimetry ie determination	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Vitamin C (dehydro-ascorbic acid and ascorbic acid)</u>	Determination of vitamin C (dehydro-ascorbic acid and ascorbic acid) AOAC 967.22 (1968)	Microfluorometry	III

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