

APPENDIX IV

METHODS OF ANALYSIS FOR FURTHER CONSIDERATION (For referral)

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.

Part 1. CEREALS, PULSES AND LEGUMES AND DERIVED PRODUCTS - for referral to CCCPL

Cereals, pulses and legumes and derived products				
Commodity	Provision	Method	Principle	Type
<u>Wheat</u>	<u>Insect bored kernels</u>	<u>ISO 7970</u>	<u>Visual examination and gravimetry</u>	!

Part 2. COCOA PRODUCTS AND CHOCOLATE – for review by the EWG on cocoa products and chocolate workable package

Cocoa products and chocolate				
Commodity	Provision	Method	Principle	Type
Chocolate and chocolate products	Cocoa butter*	AOAC 963.15 / IOCCC <u>ICA No. 14</u>	Gravimetry (Soxhlet extraction)	I
Chocolate and chocolate products	Fat, total <u>Cocoa butter</u> on dry basis*	ICA No. 26 / AOAC 977.10 and AOAC 963.15 <u>/ ICA No. 14</u>	Calculation from moisture (determined as water) <u>water</u> and gravimetry (Soxhlet extraction)	I
Chocolate and chocolate products	Milk_fat	IOCCC <u>ICA No. 5</u>	Titrimetry / Distillation	↓ <u>IV</u>
<u>Chocolate and chocolate products</u>	<u>Milk fat</u>	<u>AOCS Ce 11a-07 / ISO 11053</u>	<u>GC-FID and calculation</u>	!
<u>Chocolate and chocolate products</u>	<u>Milk fat</u>	<u>AOAC 990.27</u>	<u>GC-FID and calculation</u>	!
Chocolate and chocolate products	Milkfat	AOAC 945.34; 925.41B; 920.80	Titrimetry / Distillation	↓
Chocolate and chocolate products	Moisture	IOCCC 26 or AOAC 977.10 (Karl Fischer method); or AOAC 931.04 or IOCCC 1	Gravimetry	↓

Cocoa products and chocolate				
Commodity	Provision	Method	Principle	Type
Chocolate and chocolate products	Non-cocoa butter vegetable fat	AOCS Ce 10-02 and described in the standard	Described in the standard GC-MS	I IV
<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>	I
<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>	I
<u>Chocolate and chocolate products</u>	<u>Determination of centre and coating of filled chocolate</u>	<u>See Appendix **</u>		
Cocoa (cacao) mass or cocoa/ chocolate liquor, and cocoa cake	<u>Cocoa butter Fat</u>	AOAC 963.15 / or ICA No. 10CCC 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 Gravimetry after extraction with diethyl ether	I
<u>Cocoa butter</u>	<u>Unsaponifiable matter**</u>	<u>ISO 18609</u>	<u>Gravimetry after extraction with hexane</u>	IV
Cocoa powders (cocoa) and dry cocoa-sugar mixtures	Moisture <u>(determined as water)</u>	10CCC ICA No. 26 or / AOAC 977.10 (Karl Fischer method)	Gravimetry Titrimetry - Karl Fischer	I II
<u>Cocoa powders (cocoas) and dry mixtures of cocoa and sugars</u>	<u>Determination of content of full-fat cocoa powder, fat-reduced cocoa powder and highly fat-reduced cocoa powder</u>	<u>EU CLEN Method ILIADe 112 and AOAC 963.15 / ICA No. 14</u>	<u>HPLC-UV, Gravimetry (Soxhlet extraction) and calculation</u>	I
<u>Cocoa powders (cocoas) and dry mixtures of cocoa and sugars</u>	<u>Cocoa butter</u>	<u>AOAC 963.15 / ICA No. 14</u>	<u>Gravimetry (Soxhlet extraction)</u>	I

* Applicable for products which do not contain milkfat or other added fats

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

Appendix ** of CXS 234-1999**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.

Part 3. CODEX COMMITTEE ON FATS AND OILS – for referral to CCFO

Fats and oils				
Commodity	Provision	Method	Principle	Type
<u>Microbial omega-3 oils</u>	<u>Moisture</u>	<u>ISO 8534</u>	<u>Titrimetry (Karl Fischer)</u>	<u>II</u>
<u>Microbial omega-3 oils</u>	<u>Moisture</u>	<u>AOCS Ca 2e-84</u>	<u>Titrimetry (Karl Fischer)</u>	<u>III</u>
<u>Microbial omega-3 oils</u>	<u>Moisture and volatile matter at 103 °C</u>	<u>ISO 662</u>	<u>Gravimetry</u>	<u>I</u>
<u>Microbial omega-3 oils</u>	<u>Moisture and volatile matter at 130 °C</u>	<u>AOCS Ca 2c-25</u>	<u>Gravimetry</u>	<u>I</u>

Part 4. CODEX COMMITTEE ON SPICES AND CULINARY HERBS – for referral to CCSCH

Spices and culinary herbs				
Commodity	Provision	Method	Principle	Type
<u>Large cardamom</u>	<u>Mammalian and/or other excreta</u>	<u>AOAC 993.27</u>	<u>Colorimetry</u>	<u>III</u>
<u>Dried or dehydrated coriander</u>	<u>Mammalian and/or other excreta</u>	<u>AOAC 993.27</u>	<u>Colorimetry</u>	
<u>Dried or dehydrated coriander</u>	<u>Mammalian and/or other excreta</u>	<u>ISO 927</u>	<u>Visual examination (gravimetry)</u>	

Part 5. FRUIT JUICES AND NECTARS – for review by the EWG on fruit juices and nectars workable package

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Pectin (additives)	IFUMA 26	Precipitation / photometry	I
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 ENV 12142	Stable isotope mass spectrometry	II
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) (Quality / Authenticity)</u>	Determination of vitamin C (dehydro-ascorbic acid and ascorbic acid) AOAC 967.22	Microfluorometry	III
Fruit juices and nectars	Ascorbic acid-L <u>(additives)</u>	IFU 17a	HPLC- <u>UV</u>	II
Fruit juices and nectars	Ascorbic acid-L <u>(additives)</u>	AOAC 967.21 / ISO 6557-2	<u>Titrimetry</u> (Indophenol method)	III
Fruit juices and nectars	Ascorbic acid-L <u>(additives)</u>	IFU 17b	<u>Potentiometric titrimetry</u> (iodine) method	III
<u>Fruit juices and nectars</u>	<u>Determination of glucose, fructose, sucrose and sorbitol</u> <u>(additive / authenticity)</u>	<u>IFU 67</u>	<u>HPLC-RI</u>	<u>II</u>

^{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
<u>Fruit juices and nectars</u>	<u>Determination of glucose, fructose and sucrose (additive / authenticity)</u>	<u>NMKL 148</u>	<u>HPLC-RI</u>	<u>III</u>
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**	Determination of glucose fructose and saccharose EN 12630 IFUMA 67 NMKL 148	HPLC	II
Fruit juices and nectars	Quinic, malic and citric acid in cranberry juice cocktail and apple juice (permitted ingredients and additives) <u>(Quality / Additive / Authenticity)</u>	Determination of quinic, malic and citric acid in cranberry juice cocktail and apple juice AOAC 986.13	HPLC- <u>UV</u>	III
Fruit juices and nectars	Sucrose (permitted ingredients) <u>(Additive / Authenticity)</u>	EN 12630 IFUMA 67 <u>NMKL 148</u>	HPLC- <u>RI</u>	II
<u>Fruit juices and nectars</u>	<u>Sucrose (Additive / Authenticity)</u>	<u>NMKL 148</u>	<u>HPLC-RI</u>	<u>III</u>
Fruit juices and nectars	Tartaric acid in grape juice (additives) <u>(Quality/Additive/Authenticity)</u>	EN 12137 IFUMA 65	HPLC- <u>UV</u>	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Fermentability (Quality/Authenticity)</u>	Determination of fermentability IFUMA 18	Microbiological method	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** <u>Anthocyanins (Quality/Authenticity)</u>	Detection of anthocyanins IFUMA 71	HPLC- <u>UV</u>	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Beet sugar in fruit juices (Authenticity)</u>	Detection of beet sugar in fruit juices AOAC 995.17	<u>Magnetic Resonance spectrometry (D-NMR)</u> Deuterium NMR	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>C¹³/C¹² ratio of ethanol derived from fruit juices (Authenticity)</u>	Determination of C¹³/C¹² ratio of ethanol derived from fruit juices JAOAC 79, No. 1, 1996, 62-72	Stable isotope mass spectrometry <u>IRMS</u>	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Carbon stable isotope ratio of apple juice (Authenticity)</u>	Determination of carbon stable isotope ratio of apple juice AOAC 981.09 - JAOAC 64, 85 (1981)	Stable isotope mass spectrometry <u>IRMS</u>	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Carbon stable isotope ratio of orange juice (Authenticity)</u>	Determination of carbon stable isotope ratio of orange juice AOAC 982.21	Stable isotope mass spectrometry <u>IRMS</u>	II

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** <u>Carotenoid, total/individual groups (Authenticity)</u>	Determination of carotenoid, total/individual groups EN 12136; IFUMA 59	Spectrophotometry	I
Fruit juices and nectars	<u>Cellobiose (Quality/Authenticity)</u>	IFUMA 4	Capillary gas chromatography <u>Cap-GC-FID</u>	IV
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Formol number (Quality/Authenticity)</u>	Determination of formol number EN 1133 IFUMA 30	Potentiometric titration	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Free amino acids (Quality/Authenticity)</u>	Determination of free amino acids EN 12742 IFUMA 57	Liquid Chromatography	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Hesperidin and naringin (Quality/Authenticity)</u>	Determination of hesperidin and naringin EN 12148 IFUMA 58	HPLC	II
Fruit juices and nectars	High Fructose Corn Syrup and Hydrolysed Inulin Syrup in apple juice (permitted ingredients) <u>(Additive / Authenticity)</u>	Determination of HFCS and HIS by Capillary GC method JAOAC 84, 486 (2001) / IFU recommendation No. 4	<u>CAP-GC-FID</u>	IV

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>Naringin and neohesperidin in orange juice (Quality/Authenticity)</u>	Determination of naringin and neohesperidin in orange juice AOAC 999.05	HPLC- <u>UV</u>	III
Fruit juices and nectars	Phosphorus/phosphate <u>(Quality/Additive/Authenticity)</u>	EN 1136 / IFU 50	Photometric determination <u>Photometry</u>	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Proline by photometry – non-specific determination (Quality/Authenticity)</u>	Determination of proline by photometry – non-specific determination EN 1144 IFUMA 49	Photometry	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Sodium, potassium, calcium, magnesium in fruit juices (Quality/Authenticity)</u>	Determination of sodium, potassium, calcium, magnesium in fruit juices EN 1134 IFUMA 33	Atomic absorption spectroscopy <u>AAS</u>	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Stable carbon isotope ratio in the pulp of fruit juices (Authenticity)</u>	Determination of stable carbon isotope ratio in the pulp of fruit juices ENV 13070 Analytica Chimica Acta 340 (1997) / <u>IFU 88</u>	Stable isotope mass spectrometry <u>IRMS</u>	II

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** <u>Stable carbon isotope ratio of sugars from fruit juices (Authenticity)</u>	Determination of stable carbon isotope ratio of sugars from fruit juices ENV 12140 Analytica Chimica Acta 271 (1993) / <u>IFU 88</u>	Stable isotope mass spectrometry <u>IRMS</u>	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005**	Determination of stable oxygen isotope ratio in fruit juice water ENV 12141	Stable isotope mass spectrometry	II
<u>Fruit juices and nectars</u>	<u>Stable oxygen isotope ratio in fruit juice water (Authenticity)</u>	<u>IFU 89</u>	<u>IRMS</u>	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Sugar beet derived syrups in frozen concentrated orange juice $\delta^{18}\text{O}$ measurements in water (Authenticity)</u>	Determination of sugar beet derived syrups in frozen concentrated orange juice $\delta^{18}\text{O}$ Measurements in water AOAC 992.09	Oxygen isotope ratio analysis <u>IRMS</u>	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Benzoic acid as a marker in orange juice for pulp wash (Quality/Authenticity)</u>	Determination of benzoic acid as a marker in orange juice AOAC 994.11	HPLC	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>Chloride (expressed as sodium chloride) (Authenticity)</u>	Determination of chloride (expressed as sodium chloride) EN 12133 IFUMA 37	Electrochemical titrimetry	III
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Fumaric acid (Quality/Authenticity)</u>	Determination of fumaric acid IFUMA 72	HPLC	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Essential oils (Scott titration) (Quality/Authenticity)</u>	Determination of essential oils (Scott titration) AOAC 968.20 / IFUMA45 ^{xxi}	(Scott) Distillation, / titration	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx}	Determination of pH-value NMKL 179	Potentiometry	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>pH-value (Quality)</u>	Determination of pH-value EN 1132 IFUMA 11 / <u>NMKL 174</u> / ISO 1842	Potentiometry	IV II
Fruit juices and nectars	Soluble solids <u>(Quality)</u>	AOAC 983.17 / EN 12143 / IFU 8 / ISO 2173	Indirect by refractometry	I

^{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** Starch (Quality)	Detection of starch AOAC 925.38 / IFUMA 73	Colorimetric	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** Titrateable acids, total (Quality/Authenticity)	Determination of titrable acids, total EN 12147 IFUMA 03 ISO 750	Titrimetry	I
Fruit juices and nectars	Benzoic acid and its salts; sorbic acid and its salts (Additive)	IFUMA 63 / NMKL 124	HPLC- UV	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** Ash in fruit products (Quality/Authenticity)	Determination of ash in fruit products AOAC 940.26; / EN 1135 ; IFUMA 9	Gravimetry	I
Fruit juices and nectars	Sulphur dioxide (additives)	Optimized Monier Williams AOAC 990.28 / IFUMA 7A NMKL 132	Titrimetry (after distillation)	II
<u>Fruit juices and nectars</u>	<u>Sulphur dioxide (additives)</u>	<u>NMKL 132</u>	<u>Spectrophotometric (after distillation)</u>	<u>III</u>
Fruit juices and nectars	Ascorbic acid-L	ISO 6557-2	<u>Titrimetry</u> (Indophenol method)	III
Fruit juices and nectars	Ascorbic acid-L	ISO 6557-1	Fluorescence spectroscopy	IV
Fruit juices and nectars	Malic acid (additives)	AOAC 993.05	HPLC and Enzymatic determination and HPLC	III

Fruit juices and nectars				
Commodity	Provision	Method	Principle	Type
Fruit juices and nectars	Malic acid-D	IFUMA 64	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005^{xx} <u>Isocitric acid-D (Quality criteria / Authenticity)</u>	Determination of isocitric acid-D IFUMA 54	Enzymatic determination	II
Fruit juices and nectars	Citric acid ^{xix} (additives / <u>authenticity</u>)	IFUMA 22	Enzymatic determination	III
Fruit juices and nectars	Glucose-D and fructose-D (permitted ingredients) <u>(Additive / Authenticity)</u>	IFUMA 55	Enzymatic determination	II
Fruit juices and nectars	Malic acid-L (<u>Additive / Authenticity</u>)	IFU 21	Enzymatic determination	II
Fruit juices and nectars	Sucrose (<u>Additive / Authenticity</u>)	IFU 56	Enzymatic determination	III
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 in apple juice (Quality/Authenticity)</u>	Determination of L-malic/total malic acid ratio in apple juice AOAC 993.05	Enzymatic determination and HPLC	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.

^{xix} All juices except citrus based juices.

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** <u>Sorbitol-D (Quality / Authenticity)</u>	Determination of sorbitol-D IFUMA_62	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Acetic acid (Quality / Authenticity)</u>	Determination of acetic acid IFUMA 66	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Alcohol (ethanol) (Quality)</u>	Determination of alcohol (ethanol) IFUMA 52	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Gluconic acid (Quality)</u>	Determination of gluconic acid IFUMA 76	Enzymatic determination	II
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Glycerol (Quality)</u>	Determination of glycerol IFUMA-77	Enzymatic determination	II

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** <u>Lactic acid- D and L (Quality)</u>	Determination of Lactic acid- D and L IFUMA-53	Enzymatic determination	II
Fruit juices and nectars	Sulphur dioxide (additives)	NMKL 135	Enzymatic determination	III
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>pH-value (Quality)</u>	Determination of pH value ISO 1842	Potentiometry	IV II
Fruit juices and nectars	Soluble solids <u>(Quality)</u>	ISO 2173	Indirect by refractometry	I
Fruit juices and nectars	Sections 3.2 Quality criteria and 3.3 Authenticity of CXS 247-2005** <u>Titrateable acids, total (Quality/Authenticity)</u>	Determination of titrable acids, total ISO 750	Titrimetry	I

*ACA = anhydrous citric acid

Part 6. SUGARS AND HONEY – for review by the EWG on sugars and honey workable package**Part 6.1 Methods for consideration for the development of method performance criteria**

Sugars and honey				
Commodity	Provision	Method	Principle	Type
<u>Honey</u>	<u>Hydroxymethylfurfural</u>	<u>AOAC 980.23</u>	<u>Spectrophotometry-UV</u>	<u>III</u>
<u>Honey</u>	<u>Hydroxymethylfurfural</u>	<u>IHC 5</u>	<u>HPLC-UV</u>	<u>II</u>
Sugars (fructose)	D-Fructose	ISO 10504	Liquid chromatography (refractive index detection) <u>HPLC-RI</u>	II
Sugars (fructose)	D-Glucose	ISO 10504	Liquid chromatography (refractive index detection) <u>HPLC-RI</u>	II
<u>Sugars (plantation or mill white sugar)</u>	<u>Sulfites (expressed as sulphur dioxide)</u> <u>> 50 mg/kg ML</u>	<u>AOAC 962.16 (for > 50 mg/kg ML)</u>	<u>Titrimetry Modified Monier – Williams</u>	<u>III</u>
<u>Sugars (plantation or mill white sugar)</u>	<u>Sulfites (expressed as sulphur dioxide)</u>	<u>AOAC 990.28</u>	<u>Titrimetry Modified Monier – Williams</u>	<u>III</u>
<u>Sugars (plantation or mill white sugar)</u>	<u>Sulfites (expressed as sulphur dioxide)</u>	<u>ICUMSA GS 2-33</u>	<u>Colorimetry</u>	<u>IV</u>
<u>Sugars (all)</u>	<u>Sulfites (expressed as sulphur dioxide)</u>	<u>US FDA Method C-004.04</u>	<u>LC-MS/MS</u>	<u>IV</u>
Sugars (plantation or mill white sugar, <u>powdered sugar and powdered dextrose, raw cane sugar, soft white sugar and soft brown sugar, white sugar</u>)	<u>Sulphur dioxide</u> <u>Sulfites (expressed as sulphur dioxide)</u>	ICUMSA GS 2/3-35 <u>2-35</u> NMKL 135 EN 1988-2	Enzymatic <u>spectrophotometry-UV</u>	II

Sugars and honey				
Commodity	Provision	Method	Principle	Type
Sugars (dextrose anhydrous and dextrose monohydrate, fructose, glucose syrup and dried glucose syrup)	Sulphur dioxide Sulfites (expressed as sulphur dioxide)	ISO 5379	Acidimetry and nephelometry	IV
Sugars (fructose)	Sulphur dioxide Sulfites (expressed as sulphur dioxide)	ISO 5379	Acidimetry and nephelometry	IV
Sugars (glucose syrup and dried glucose syrup)	Sulphur dioxide Sulfites (expressed as sulphur dioxide)	ISO 5379	Acidimetry and nephelometry	IV

Method performance criteria for powdered sugar and powdered dextrose

<u>Commodity</u>	<u>Provision</u>	<u>ML (%)</u>	<u>Method performance criteria</u>					<u>Example of methods that meet the criteria</u>	<u>Principle</u>
			<u>Minimal applicable range (%)</u>	<u>Limit of detection (LOD) (%)</u>	<u>Limit of quantification (LOQ) (%)</u>	<u>Precision (RSD_R) (%) no more than</u>	<u>Recovery (%)</u>		
Powdered sugar and powdered dextrose	Dextrose anhydrous (as D-glucose)	99.5	93.5 - 105.5	9.95	19.9	4			
Powdered sugar and powdered dextrose	Dextrose monohydrate (as D-glucose)	99.5	93.5 - 105.5	9.95	19.9	4			
Powdered sugar and powdered dextrose	Glucose syrup	20	18.5 - 21.5	2	4	5			
Powdered sugar and powdered dextrose	Fructose (laevulose)	98	92.1 - 103.9	9.8	19.6	4			
Powdered sugar and powdered dextrose	Fructose (laevulose)	0.5	0.43 - 0.57	0.05	0.1	9			

Method performance criteria for honey

<u>Commodity</u>	<u>Provision</u>	<u>ML</u> <u>(mg/kg)</u>	<u>Method performance criteria</u>					<u>Example of methods that meet the criteria</u>	<u>Principle</u>
			<u>Minimal applicable range</u> <u>(mg/kg)</u>	<u>Limit of detection (LOD)</u> <u>(mg/kg)</u>	<u>Limit of quantification (LOQ)</u> <u>(mg/kg)</u>	<u>Precision (RSD_R) (%)</u> <u>no more than</u>	<u>Recovery (%)</u>		
Honey	Hydroxymethylfurfural content	40	29.0 – 51.0	4.0	8.0	18			
Honey (declared origin from countries or regions with tropical ambient temperatures, and blends of these honeys)	Hydroxymethylfurfural content	80	60.1 – 99.9	8.0	16.0	17			

Part 6.2 Other methods returned for further consideration

Sugars and honey				
<u>Commodity</u>	<u>Provision</u>	<u>Method</u>	<u>Principle</u>	<u>Type</u>
<u>Honey</u>	<u>Diastase activity</u>	<u>AOAC 958.09 / IHC 6.1</u>	<u>Enzymatic spectrophotometry-visible</u>	<u>I</u>
<u>Honey</u>	<u>Sugars added (authenticity)</u>	<u>EN 17958**</u> <u>** For authenticity ranges, refer to: Apidologie 2008, 39 (5), 574-587</u>	<u>HPLC-IRMS</u>	<u>III</u>
<u>Honey</u>	<u>Sugars profile (glucose, fructose, sucrose)</u>	<u>AOAC 977.20</u>	<u>HPLC-RI</u>	<u>IV</u>
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
<u>Honey excluding manuka honey</u>	<u>Sugars added: detection of C₄ sugar</u>	<u>AOAC 998.12</u>	<u>IRMS</u>	<u>II</u>

Sugars and honey				
Commodity	Provision	Method	Principle	Type
Sugars (dextrose anhydrous and dextrose monohydrate)	D-Glucose	ISO 5377	Titrimetry (<u>Lane & Eynon</u>)	I
Sugars (fructose, <u>powdered sugar</u> , <u>white sugar</u> , <u>plantation or mill white sugar</u>)	Conductivity ash	ICUMSA GS 2/3-17 <u>2-17</u>	Conductimetry	I
Sugars (powdered sugar)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	I
Sugars (white sugar)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	I
Sugars (plantation or mill white sugar, <u>soft white sugar and soft brown sugar</u>)	Conductivity ash	ICUMSA GS 1/3/4/7/8-13 <u>1-13</u>	Conductimetry	I
Sugars (soft white sugar and soft brown sugar)	Conductivity ash	ICUMSA GS 1/3/4/7/8-13	Conductimetry	I
Sugars (lactose)	Loss on drying	USP General Chapter 731	Gravimetry (drying at 120 °C for 16h)	I
Sugars (plantation or mill white sugar)	Invert sugar (<u>as reducing sugars</u>)	ICUMSA GS 1/3/7-3 <u>1-3</u>	Titrimetry (Lane & Eynon)	I <u>IV</u>
<u>Sugars (plantation or mill white sugar)</u>	<u>Invert sugar (as reducing sugars)</u>	<u>ICUMSA GS 1-5</u>	<u>Titrimetry – Luff Schoorl</u>	<u>IV</u>
<u>Sugars (plantation or mill white sugar)</u>	<u>Invert sugar</u>	<u>FCC 14th Ed Sucrose monograph, for Organic Impurities - Invert Sugar</u>	<u>HPLC - PAD</u>	<u>II</u>
Sugars (<u>white sugar</u> , powdered sugar)	Invert sugar (<u>as reducing sugars</u>)	ICUMSA GS 2/3-5 <u>2-5</u> after filtration if necessary to remove any anticaking agents	Titrimetry - <u>Knight & Allen</u>	I

Sugars and honey				
Commodity	Provision	Method	Principle	Type
Sugars (white sugar)	Invert sugar	ICUMSA GS 2/3-5	Titrimetry	I
Sugars (powdered sugar)	Invert sugar	ICUMSA GS 2-4 after filtration if necessary to remove any anticaking agents	Enzymatic spectrophotometry-UV	IV
Sugars (soft white sugar and soft brown sugar)	Invert sugar (<u>as reducing sugars</u>)	ICUMSA GS 4/3-3 <u>4-3</u> (applicable at levels >10% m/m)	Titrimetry (Lane & Eynon)	I
Sugars (soft white sugar and soft brown sugar)	Invert sugar (<u>as reducing sugars</u>)	ICUMSA GS 1/3/7-3 <u>1-3</u> (applicable at levels <10% m/m)	Titrimetry (Lane & Eynon)	I <u>IV</u>