

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
United Nations



World Health
Organization

Viale delle Terme di Caracalla, 00153 Rome, Italy - Tel: (+39) 06 57051 - E-mail: codex@fao.org - www.codexalimentarius.org

Agenda Item 3

MAS/39 CRD/4

ORIGINAL LANGUAGE ONLY

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS SAMPLING

Thirty-ninth Session
Budapest, Hungary, 7 – 11 May 2018

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS AND SAMPLING PLANS IN CODEX STANDARDS

(Comments submitted by Switzerland and IDF)

SWITZERLAND

Switzerland would like to support the proposals made by the CCNFSDU39 and major standard developing organizations to endorse several new methods for the provisions cited above in infant formula.

A short summary of the reasons why this endorsement should be done can be found in the table below.

Provision	Situation Now	Proposal and motivation for change
Biotin	EN 15607 Foodstuffs - Determination of d-biotin by HPLC – Type II	Proposal Endorse AOAC 2016.02 ISO DIS 23305 as Type II and reclassify EN 15607 as Type III. This new method has extensive Single Laboratory (SLV) and MLT study data. The MLT study utilized 14 carefully selected infant, child and adult nutritional formulas covering a broad matrix range. 12 laboratories covering 10 countries produced data with excellent precision and accuracy. The analytical platform in the proposed method is inexpensive and the method can be used in almost any labs worldwide with basic facilities. Motivation for change For the EN 15607 method, D-biotin is extracted from a sample after an enzymatic treatment and quantified by HPLC with post column binding reaction with the fluorescent marker avidin-FTIC (Fluorescein 5-isothiocyanate). The enzymatic digestion is time consuming as it involves overnight incubation as part of the sample preparation. The avidin-FTIC is relatively expensive and the stability is questionable and it has to be monitored at frequent intervals with calibration standards to ensure the validity of the post-column reaction.
Vitamin D	EN 12821 Foodstuffs - Determination of vitamin D by high performance liquid chromatography -. Type II AOAC 992.26 (III) And	Proposal Endorse AOAC 2016.05 ISO 20636 as Type II, reclassify EN 12821 as Type III and remove AOAC 992.26 and AOAC 995.05 from CODEX STAN 234-1999. The performance of proposed AOAC 2016.05 ISO DIS 20636 (LC-MS/MS) was evaluated extensively by both Single Laboratory Validation and Multi-Laboratory Testing using a sample set of over 20 matrices encompassing a broad range of infant and follow-on formula covering a wide nutrient fortification range. 10 laboratories from six countries participated in the Multi-

	AOAC 995.05 (III)	<p>Laboratory Testing study and produced data with excellent precision and accuracy for all samples.</p> <p>Motivation for change</p> <p>This European Standard is not applicable for samples with a content of vitamin D3 and vitamin D2. This European Standard does only include measurement of vitamin D3 or vitamin D2. Whereas both vitamin D3 and vitamin D2 can be present in fortified foodstuffs. In certain parts of the world only D2 is allowed because D3 is seen as animal/meat source. Furthermore previous methods up until now used D2 as an internal standard for the determination of D3 and vice versa. This is very cumbersome and at the same time it requires additional blank determinations to ensure no natural D2 and/or D3 is present when these are add as internal standard. For this method, following sample saponification, vitamin D is extracted into organic solvent, with additional matrix separation by semi-preparative normal phase HPLC, with subsequent reversed-phase analytical HPLC. Quantitation is by use of vitamin D₂ as internal standard for vitamin D₃ analysis (vice versa for vitamin D₂ analysis). The saponification, extraction, evaporation and semi-preparative HPLC steps all contribute to a significant analysis time for this method. This method has limited sample throughput due to the serial nature of the semi-preparative step, and in a single analysis can only test a single vitamin D form (vitamin D₂ or vitamin D₃). Additionally, inter-laboratory validation for EN 12821 was performed on only two infant formula matrices.</p> <p>It is supported to remove AOAC 992.26 and AOAC 995.05 because relatively outdated methods do not use vitamin D₂ or alternatively vitamin D₃ as an internal standard. This is a 'must' if hot saponification is used.</p>
Chloride	AOAC 986.26 Type III AOAC 971.2, type II	<p>Proposal</p> <p>AOAC/ISO/IDF recommends to endorse AOAC 2016.03 ISO 21422 IDF 242 Determination of Chloride in Milk, Milk Powder, Whey Powder, Infant Formula, and Adult Nutritionals by Potentiometric Titration as Type II, and remove 971.27. AOAC 986.26 stays as type III.</p> <p>Motivation</p> <p>AOAC 986.26 does have some validation data (collaborative study) published in JAOAC 69, 777(1986). However, these data need updating with a wider range of infant formula and adult nutritional products.</p> <p>AOAC 971.27 is based on the same principle as AOAC 986.26 and was designated as Type II (reference method) for "Foods for Special Dietary uses" by CCMAS. However, the field of application of this AOAC method is "general." This method is not appropriate for infant formula and adult nutritional products, since these products are not included in the scope of application.</p>

INTERNATIONAL DAIRY FEDERATION (IDF)

Commodities	CODEX STAN	Provision	Method / version	Principle	Type	Committee	Remarks	Action suggested	IDF comment
Butter	CODEX STAN 279	Water ("Water content excluding the crystallized water bound to lactose (generally known as "moisture content")	ISO 3727 IDF 80	Gravimetry	I	CCMMP	The correct method is ISO 3727-1 IDF 80-1; The ISO method mentions moisture, non fat solids and fat content in butter	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	This method has indeed three parts. Only part 1 determines moisture (so entry in 234 should be corrected to ISO 3727-1 IDF 80-1). Part 2 determines non-fat solids, and part 3 the calculation for fat. The equivalence between moisture and water is explained in the provision column, and is important to keep.
Milk powders and cream powders	CODEX STAN 207	Water ("Water content excluding the crystallized water bound to lactose (generally known as "moisture content")	ISO 5537 IDF 26	Gravimetry (drying at 87°C)	I	CCMMP	The ISO method mentions moisture.	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The notes explains that water and moisture are equivalent: ("Water content excluding the crystallized water bound to lactose (generally known as "moisture content")
Blend of evaporated skimmed	CODEX STAN 250	Milk protein in MSNF ("Milk total solids and	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to	The WG on endorsement agreed to allocate type IV to this

milk and vegetable fat		Milk solids-not-fat (MSNF) content include water of crystallization of lactose")						paragraph 58 - REP 17/MAS	method, since no other method was identified. Ref: CRD 1 CCMAS 2010
Blend of evaporated skimmed milk and vegetable fat	CODEX STAN 250	Milk protein in MSNF ("Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose")	AOAC 991/20	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method. The Codex Stan 234 mentions incorrectly the AOAC method. The correct one is AOAC 991.20	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Blend of skimmed milk and vegetable fat in powdered form	CODEX STAN 251	Milk protein in MSNF ("Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose")	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MASs	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Blend of skimmed milk and vegetable fat	CODEX STAN 251	Milk protein in MSNF ("Milk total solids and Milk solids-not-	AOAC 991.20	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to	The WG on endorsement agreed to allocate type IV to this method, since no other

in powdered form		fat (MSNF) content include water of crystallization of lactose")						paragraph 58 - REP 17/MAS	method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Blend of sweetened condensed skimmed milk and vegetable fat	CODEX STAN 252	Milk protein in MSNF ("Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose")	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Blend of sweetened condensed skimmed milk and vegetable fat	CODEX STAN 252	Milk protein in MSNF ("Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose")	AOAC 991.20	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Edible casein products	CODEX STAN 290	Acids, free	ISO 5547 IDF 91	Titrimetry (aqueous extract)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to	The WG on endorsement agreed to allocate type IV to this method, since no other

								paragraph 58 - REP 17/MAS	method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Edible Casein Products	CODEX STAN 290	Lactose	ISO 5548 IDF 106	Photometry (phenol and H2SO4)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Edible casein products	CODEX STAN 290	pH	ISO 5546 IDF 115	Electrometry	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Edible Casein Products	CODEX STAN 290	Sediment (scorched particles)	ISO 5739 IDF 107	Visual comparison with standard	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to	The WG on endorsement agreed to allocate type IV to this method, since no other

				disks, after filtration				paragraph 58 - REP 17/MAS	method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Emmental	CODEX STAN 269	Calcium	ISO 8070 IDF 119	Flame atomic absorption	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Milk powders and cream powders	CODEX STAN 207	Scorched particles	ISO 5739 IDF 107	Visual comparison with standard disks, after filtration	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Reduced fat blend of evaporated skimmed	CODEX STAN 250	Milk protein in MSNF ("Milk total solids and Milk solids-not-	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to	The WG on endorsement agreed to allocate type IV to this method, since no other

milk and vegetable fat		fat (MSNF content include water of crystallization of lactose")						paragraph 58 - REP 17/MAS	method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Reduced fat blend of evaporated skimmed milk and vegetable fat	CODEX STAN 250	Milk protein in MSNF ("Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose")	AOAC 991.20	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	CODEX STAN 251	Milk protein in MSNF ("Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose")	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Reduced fat blend of skimmed milk powder	CODEX STAN 251	Milk protein in MSNF ("Milk total solids and Milk solids-not-	AOAC 991.20	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to	The WG on endorsement agreed to allocate type IV to this method, since no other

and vegetable fat in powdered form		fat (MSNF) content include water of crystallization of lactose")						paragraph 58 - REP 17/MAS	method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	CODEX STAN 252	Milk protein in MSNF ("Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose")	ISO 8968-1 IDF 20-1	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	OK
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	CODEX STAN 252	Milk protein in MSNF ("Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose")	AOAC 991.20	Titrimetry (Kjeldahl)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Whey powders	CODEX STAN 289	Ash	ISO 5545 IDF 90	Gravimetry (ashing at 825 °C)	IV	CCMMP	This provision has no type II method	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified.

									Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010
Whey powders	CODEX STAN 289	Moisture, "Free"	ISO 2920 IDF 58	Gravimetry (drying at 88 °C ±2°C)	IV	CCMMP	This provision has no type II method. The Stan 289 doesn't mention "Moisture free", just "Water". The ISO method mentions dry matter in whey cheese.	Submit this information to IDF, according to paragraph 58 - REP 17/MAS	The WG on endorsement agreed to allocate type IV to this method, since no other method was identified. Type IV methods could be listed only when no other alternative existed. Ref: CRD 1 CCMAS 2010. The current version of STAN 234 correctly lists water with a note explaining the equivalence between moisture and water.
Whey powders	Codex Stan 289	Copper	AOAC 985.35	Atomic absorption spectrophotometry	II	CCMMP	Methods applicated for Baby Foods/Infant Formula, Pet Foods, Baby Foods/Enteral Products. The CODEX STAN 289 does not mention this provision.	Revoke this provision/ method, since there is no reference in Codex Stan 289	