

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of
the United Nations



World Health
Organization

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Agenda Item 3

CX/MAS 13/34/3-Add.2

JOINT FAO/WHO FOOD STANDARDS PROGRAMME CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Thirty-fourth Session
Budapest, Hungary, 4 - 8 March 2013

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS

EUROPEAN UNION

The European Union and its Member States (EUMS) can support endorsing the majority of the methods of analysis and sampling proposed by CCFFP, CCCF and CCPFV.

However, the EUMS is concerned that a simple reference to the General Guidelines on Sampling (CAC/GL 50-2004) is sometimes being made by Codex Committees in their Standards without defining the sampling plan to be used. This is in contrast to the detailed sampling plan for aflatoxins in dried figs, which was discussed thoroughly at the last session of CCCF.

THAILAND

Agenda Item 3: Endorsement of Methods of Analysis Provisions in Codex Standards

Thailand would like to express our appreciations to an effort for preparing the document.

In general, we agree with the methods of analysis proposed by the Codex Committees; however we would like to propose our comments on Standard for Live and Raw Bivalve Molluscs as follows:

Standard for Live and Raw Bivalve Molluscs

- I-8.6.1 Numerical Criteria Values for Biotoxins in Bivalve Molluscs

We would like to propose the amendments to I-8.6.1 Numerical Criteria Values for Biotoxins in Bivalve Molluscs as the followings;

- Saxitoxin Group

The “recovery” of Saxitoxin Group should be changed to 80-110%

- Okadaic Acid Group

It is proposed that the method performance parameters for Okadaic Acid Group should be amended as the followings:

“minimum range” should be 0.06-0.26

“precision (RSDR) ” should be 42% and

“recovery percent” should be 80-110

- Domoic Acid Group

The method performance parameters for Domoic Acid Group should be amended as the followings:

“minimum range” should be 13.9-26.1,
“recovery percent” should be 80-110 and
“precision at ML (RSDR)” should be 20.4%.

Table 1: I-8.6.1 Numerical Criteria Values for Biotoxins in Bivalve Molluscs should then read as the following table:

I-8.6.1 Numerical Criteria Values for Biotoxins in Bivalve Molluscs

Table 1

Group	Toxin	Maximum level /kg of mollusc flesh	Minimum applicable range	LOD	LOQ	Precision (RSDR)	Recovery percent
Saxitoxin (STX) Group	Total Toxicity	≤ 0.8 milligrams (2HCL) of saxitoxin equivalent	0.4 – 1.2	0.08	0.16	33%	70-120 <u>80-110</u>
Okadaic acid (OA) group	Total Toxicity	≤ 0.16 milligrams of okadaic equivalent	0.05–0.27 <u>0.06-0.26</u>	0.016	0.032	44% <u>42%</u>	70-120 <u>80-110</u>
Domoic acid (DA) group	Domoic Acid (DA)	≤ 20 milligrams domoic acid	13.2–26.8 <u>13.9-26.1</u>	2	4	22% <u>20.4%</u>	85-110 <u>80-110</u>

Comments from the IDF (International Dairy Federation) and ISO/TC34/SC5 (Subcommittee on milk and milk products of the International Organization for Standardization)

Proposed changes are shown in ~~bold strikethrough~~ for deletion and **bold underlined** for additions.

Foods for special dietary uses

Products	Provisions	Method	Principle	Type	IDF comment
Special foods	Loss on drying (milk based)	AOAC 925.23 IDF Standard 21B:1987 ISO 6731:1989 <u>ISO 6731 IDF 21:2010</u>	Gravimetry	I	Reference update
Special foods	Sodium and Potassium	ISO 8070:1987 (confirmed 1992) IDF Standard 119A:1987 <u>ISO 8070 IDF 119:2007</u>	Flame emission spectrometry <u>atomic absorption</u> spectrometry	II	Reference and principle update (as adopted for sodium and potassium in infant formula)
Infant formula	Moisture/Total Solids	AOAC 990.20 IDF 21B:1987 or ISO 6731:1989 <u>ISO 6731 IDF 21:2010</u>	Gravimetry	I	Reference update

Milk and Milk products

Products	Provisions	Method	Principle	Type	IDF comment
Edible Casein Products	Casein in protein	ISO 17997-1 IDF 29-1:2004	Titrimetry, Kjeldahl	I	This method is only valid for the determination of casein in <u>fresh raw milk</u> . While a test result will be obtained in other samples, the ‘casein’ content will not be correct due to co precipitation of denatured whey protein.

Processed fruits and vegetables

Products	Provisions	Method	Principle	Type	IDF comment
Aqueous products coconut	Total Fats	ISO 1211:1999 IDF 1D:1996 <u>ISO 1211 IDF 1:2010*</u>	Gravimetry (Röse-Gottlieb)	I	Reference update
Aqueous products coconut	Totals Solids	ISO 6731:1989 IDF 21B:1987 <u>ISO 6731 IDF 21:2010*</u>	Gravimetry	I	Reference update
Aqueous products coconut	Non-fat solids	ISO 1211:1999 IDF 1D:1996 <u>ISO 1211 IDF 1:2010*</u> And ISO 6731:1989 IDF 21B:1987 <u>ISO 6731 IDF 21:2010*</u>	Calculation: Gravimetry (Röse-Gottlieb) Gravimetry	I	Reference update
Aqueous products coconut	Moisture	ISO 6731:1989 IDF 21B:1987 <u>ISO 6731 IDF 21:2010*</u>	Gravimetry	I	Reference update

***Coconut products are not covered in the scope of the IDF/ISO standard.**

Appendix 1 for information: Changes adopted to references of IDF/ISO methods at CCMAS and CAC but not yet incorporated in CODEX STAN 234 - 2011
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Corrections are shown in ~~bold strikethrough~~ for deletion and **bold underlined** for additions, and reflect modifications adopted in 2008, 2010, 2011 and 2012 (Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008, Appendix II Alinorm 10/33/23 CCMAS 2010, Appendix II of REP 11 MAS – CCMAS 2011, para 42-43 and Appendix II of REP12MA – CCMAS 2012).

General comments

The corrections below include editorial comments that were already discussed and decided on at previous sessions of CCMAS such as:

- Correction of the ISO/IDF reference to the correct order ISO reference followed by IDF reference and year: ISO XXX|IDF YYY:YEAR
- Corrections to the principle or type of the method based on CCMAS decision
- There is an inconsistent use of the abbreviations MSNF, the fully spelled Milk Solids not fat and the associated note 15 in Codex STAN 234 (**Milk total solids and MSNF content include water of crystallization of lactose**) for the products Blends of evaporated skimmed milk, butter, creams, and whipped creams
- The note for water (note 16) is missing for butter and edible casein products, and shows as “bookmark not defined”.

MILK AND MILK PRODUCTS

<u>Commodity</u>	<u>Provisions</u>	<u>Method</u>	<u>Principle</u>	<u>Type</u>
Milk products (products not completely soluble in ammonia) <i>see Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Milk fat	IDF 124-3 ISO 8262-3:2005 <u>ISO 8262-3 IDF 124-3:2005</u>	Gravimetry (Weibull-Berntrop)	I
Blend of evaporated skimmed milk and vegetable fat <i>See Appendix II Alinorm 10/33/23 CCMAS 2010</i>	Total fat	ISO 1737 IDF 13:2008	Gravimetry (Röse-Gottlieb)	IV <u>I</u>
Blend of evaporated skimmed milk and vegetable fat <i>See Appendix II Alinorm 10/33/23 CCMAS 2010</i>	Milk solids-not-fat ^{#1} (MSNF)	ISO 6731 IDF 21:2010 and ISO 1737 IDF 13:2008	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)	I <u>I</u>

Commodity	Provisions	Method	Principle	Type
Reduced fat blend of evaporated skimmed milk and vegetable fat <i>See Part B - Appendix II Alinorm 10/33/23 CCMAS 2010</i>	Total fat	ISO 1737 IDF 13:2008	Gravimetry (Röse-Gottlieb)	IV I
Blend of sweetened condensed skimmed milk and vegetable fat <i>See Appendix II REP12MA – CCMAS 2012</i>	Milk solids-notfat (MSNF) ²	ISO 6734 IDF 15:2010	Calculation from total solids content, fat content and sugar content.	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat <i>See Appendix II REP12MA – CCMAS 2012.</i>	MSNF ²	ISO 6734 IDF 15:2010	Calculation from total solids content, fat content and sugar content	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form <i>Bookmark error</i>	Water ²	ISO 5537 IDF 26:2004	Gravimetry, drying at 87 °C	I
Butter	Milk solids-not-fat ¹	ISO 3727-2 IDF 80-2:2001	Gravimetry	I
Butter <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Milkfat	IDF 194 ISO 17189:2003 ISO 17189 IDF 194:2003	Gravimetry Direct determination of fat using solvent extraction	I
Butter <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Salt	IDF 179 ISO 15648:2004 ISO 15648 IDF 179:2004	Potentiometry (determination of chloride, expressed as sodium chloride)	III II
Butter <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Salt	IDF 12 ISO 1738:2004 ISO 1738 IDF 12:2004 / AOAC 960.29	Titrimetry (Mohr: determination of chloride, expressed as sodium chloride)	III III
Butter <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Water ²	IDF 80-1 ISO 3727-1:2001 ISO 3727-1 IDF 80-1:2001	Gravimetry	I
Cheese (and cheese rind) <i>See Part B - Appendix II Alinorm 10/33/23 CCMAS 2010</i>	Natamycin	ISO 9233-1 IDF 140-1:2007 ISO 9233-2 IDF 140-2:2007	Molecular absorption spectrophotometry HPLC	III II
<u>Cheeses, individual</u> <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	<u>Milk fat in dry matter</u>	<u>ISO 1735 IDF 5:2004</u>	<u>Gravimetry (Schmid-Bondzynski-Ratzlaff)</u>	<u>I</u>
Cheeses in brine <i>See Appendix III – Part D of the Alinorm</i>	Milk fat in dry matter (FDM)	IDF 5 ISO 1735:2004 ISO 1735 IDF 5:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I

Commodity	Provisions	Method	Principle	Type
<i>08/31/23 - CCMAS 2008</i>				
Creams lowered in milk fat content <i>See Part B - Appendix II Alinorm 10/33/23 CCMAS 2010</i>	Milk fat	ISO 2450 IDF 16:2008 / AOAC 995.19	Gravimetry (<u>Röse-Gottlieb</u>)	I
Creams, whipped creams and fermented creams	MSNF ¹	ISO 3727-2 IDF 80-2:2001 AOAC 920.116	Gravimetry	I
Edible casein products <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Copper	IDF 76 ISO 5738:2004 <u>ISO 5738 IDF 76:2004</u>	Colorimetry (diethyldiethiocarbamate)	III
Edible casein products <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Lactose	IDF 106 ISO 5548:2004 <u>ISO 5548 IDF 106:2004</u>	Photometry (phenol and H ₂ SO ₄)	IV
Edible casein products <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Lead	IDF/RM 133 ISO/TS 6733:2006 <u>ISO/TS 6733 IDF/RM 133:2006</u>	Spectrophotometry (1,5-diphenylthiocarbazone)	IV
Edible casein products <i>Bookmark error</i>	Water ²	ISO 5550 IDF 78:2006	Gravimetry (drying at 102 °C)	I
Edible casein products <i>See Appendix II – Part B of the Alinorm 10/33/23 - CCMAS 2010</i>	Moisture	ISO 5550 IDF 78:2006	Gravimetry (drying at 102 °C)	I
Fermented milks <i>See Appendix II REP12MA – CCMAS 2012</i>	Lactic acid (total acidity expressed as lactic acid) – Total acidity expressed as lactic acid	IDF 150:1991/ISO 11869:1997 <u>ISO/TS 11869 IDF/RM 150:2012</u>	Potentiometry, titration to pH 8.30 Spectrophotometry	IV <u>I</u>
Milk powders and cream powders <i>Bookmark error</i>	Water ²	ISO 5537 IDF 26:2004 ¹	Gravimetry (drying at 87°C)	I
Milk fat products <i>See Appendix II REP12MA – CCMAS 2012</i>	Milk fat	IDF 24:1964	Gravimetry (calculation from solids- not-fat content and water content)	IV
Processed cheese products <i>See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010</i>	Citric acid	ISO/TS 2963 IDF/RM 34:2006	Enzymatic method	IV

¹ The method has only been validated for milk powders, not for cream powders

Commodity	Provisions	Method	Principle	Type
Processed cheese products <i>See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010</i>	Citric acid	AOAC 976.15	Photometry	III
Processed cheese products <i>See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010</i>	Milkfat	ISO 1735 IDF 5:2004	Gravimetry (Schmid-Bondzynski-Raztlaff)	I
Processed cheese products <i>See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010</i>	Phosphate, added (expressed as phosphorus)	IDSF 51B:1991	Calculation from phosphorous and nitrogen content	IV
Processed cheese products <i>See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010</i>	Phosphorus	IDF 33C:1987/ISO 2962:1984	Spectrophotometry (molybdate-ascorbic acid)	II
Processed cheese products <i>See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010</i>	Salt	ISO 5943 IDF 88:2004	Potentionmetry (determination of chloride, expressed as sodium chloride)	II
Sweetened condensed milk <i>See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010</i>	Milk fat	ISO 1737:1999 ISO 1737 IDF 13:2008	Gravimetry (Röse-Gottlieb)	I
Whey Cheese <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Dry matter (for denomination)	IDF 58 ISO 2920:2004	Gravimetry, drying at 88 °C	I
Whey cheeses by concentration <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Dry matter (total solids)	IDF 58 ISO 2920:2004	Gravimetry, drying at 88 °C	I
Whey cheeses by coagulation <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Dry matter (total solids)	IDF 4:2004 ISO 5534:2004	Gravimetry, Drying at 102°C	IV
Whey cheese <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Fat on the dry basis	IDF 59 A:1986 / ISO 1854:1999 and IDF 58:2004 ISO 2920:2004	Calculation from fat content and dry matter content	I

Commodity	Provisions	Method	Principle	Type
Whey cheese <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Milkfat (in dry matter)	IDF standard 59A:1986 / ISO 1854:1999	Gravimetry (Röse-Gottlieb)	I
Whey cheeses including whey cheeses by concentration <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Total fat	IDF 59A:1986 / ISO 1854:1999	Gravimetry (Röse-Gottlieb)	I
Whey cheeses by coagulation <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Total fat	IDF 5:2004+ ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Creamed whey cheese <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Fat on the dry basis	IDF 59 A: 1986 / ISO 1854:1999 and IDF 58:2004+ ISO 2920:2004	Calculation from fat content and dry matter content	I
Skimmed whey cheese <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	Fat on the dry basis	IDF 59 A:1986 / ISO 1854:1999 and IDF 58:2004+ ISO 2920:2004	Calculation from fat content and dry matter content	I
<u>Whey cheeses by coagulation</u> <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	<u>Milk fat</u>	<u>ISO 1735 IDF 5:2004</u>	<u>Gravimetry (Schmid-Bondzynski-Ratzlaff)</u>	<u>I</u>
<u>Whey cheeses by coagulation</u> <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	<u>Milk fat in dry matter</u>	<u>ISO 1735 IDF 5:2004 and ISO 5534 IDF 4:2004</u>	<u>Calculation from fat content and dry matter content Gravimetry (Schmid-Bondzynski-Ratzlaff) Gravimetry, drying at 102 °C</u>	<u>I</u>
<u>Whey cheeses by concentration</u> <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	<u>Milk fat</u>	<u>ISO 1854 IDF 59:2008</u>	<u>Gravimetry (Röse Gottlieb)</u>	<u>I</u>
<u>Whey cheeses by concentration</u> <i>See Appendix III – Part D of the Alinorm 08/31/23 - CCMAS 2008</i>	<u>Milk fat in dry matter</u>	<u>ISO 1854 IDF 59:2008 and ISO 2920 IDF 58:2004</u>	<u>Calculation from fat content and dry matter content Gravimetry (Röse Gottlieb) Gravimetry, drying at 88 °C</u>	<u>I</u>
Yoghurt products <i>See Part B - Appendix II Alinorm 10/33/23</i>	Lactobacillus bulgaricus &	IDF 117+ISO 7889:2003	Colony count at 37°C	

Commodity	Provisions	Method	Principle	Type
- CCMAS 2010	<i>Streptococcus thermophilus</i>			
Yoghurt products See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010	<i>Lactobacillus bulgaricus</i> & <i>Streptococcus thermophilus</i>	IDF 146 ISO 9232:2003	Test for identification	
Yoghurt products See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010	Solids, Total	IDF 151 ISO 13580:2005	Gravimetry (drying at 102°C)	I
Yoghurt See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010	<i>Streptococcus thermophilus</i> & <i>Lactobacillus delbrueckii</i> subsp. <i>bulgaricus</i> ≥ 10 ⁷ cfu/g	ISO 7889/IDF 117:2003	Colony count at 37°C	I
Yoghurt See Part B - Appendix II Alinorm 10/33/23 - CCMAS 2010	<i>Streptococcus thermophilus</i> & <i>Lactobacillus delbrueckii</i> subsp. <i>bulgaricus</i> ≥ 10 ⁷ cfu/g	ISO 9232/IDF 146:2003	Test for identification: morphological, cultural and biochemical characteristics	I