

codex alimentarius commission



FOOD AND AGRICULTURE
ORGANIZATION
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WORLD
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ORGANIZATION



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Agenda Item 5

**CX/MMP 02/11
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JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON MILK AND MILK PRODUCTS

Fifth Session

Wellington, New Zealand, 8-12 April 2002

METHODS OF ANALYSIS AND SAMPLING FOR MILK PRODUCTS

Report of IDF/ISO/AOAC Working Group on Methods of Analysis and Sampling

INTRODUCTION

This document provides references for the methods of analysis and sampling needed with respect to the Codex Standards and draft and proposed draft Codex standards for milk products under consideration at the 5th Session of CCMMP.

The list of methods is provided to the Committee for consideration in Appendix I. The list contains methods recommended by the Tripartite IDF/ISO/AOAC Working Group for the draft and proposed draft standards under elaboration. After the Committee agrees to the methods, they will be forwarded to the CCMAS for endorsement. The lists are presented in the format of Volume 13 of the *Codex Alimentarius* to which the entries will ultimately be added.

The precision data for the methods are being completed by IDF, ISO and AOAC International. Details will be communicated to the Codex Secretariat before forwarding to CCMAS.

The methods (International Standards) of analysis and sampling which were published or last reviewed five years ago are reviewed systematically every year by the member bodies of IDF, ISO (ISO TC34/SC5¹) and AOAC International. After review the standard methods may be confirmed or revised.

GUIDELINES FOR SAMPLING MILK AND MILK PRODUCTS

CCMAS has made progress on its general guidelines for sampling. These guidelines relate to the requirements for statistical sampling schemes. As soon as a new draft is available from CCMAS, experts from IDF, ISO and AOAC International will work on their application to the statistical sampling of milk products.

The objective is to provide guidelines on the statistical sampling of milk products for purposes of checking the compliance of products with compositional standards. A prerequisite is an appropriate definition of limits for compositional requirements, which takes statistical aspects into consideration. This definition forms the basis for the selection/development of a statistical sampling plan allowing to check compliance. Acceptable sample sizes are an important practical aspect.

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ISO Technical Committee on Food Products / Sub Committee 5 on Milk and Milk Products

ENDORSEMENT BY CCMAS OF METHODS OF ANALYSIS PROPOSED FOR STANDARDS FOR MILK PRODUCTS (23rd Session, Budapest, Feb-Mar 2001) (See ALINORM 01/23, Appendix IV, pages 35-36)

Cheese - Dry matter (Solids, Moisture)

Endorsed Type I

CCMMP is asked what is the relationship between the results obtained by the method of drying at 102°C (that which has been adopted and endorsed) and the commonly used vacuum oven method at 88°C.

(This applies to all other provisions in the milk product standards for which oven drying at 102°C is proposed.)

Apparently comparative data are not available.

Milk & milk products – Determination of copper content

The photometric method (IDF Standard 76A) is considered old-fashioned. CCMMP is invited to consider more modern methods and, in particular, the Codex General Method, NMKL 139.

Whey powders - Lactose

Endorsed Type II

CCMAS is not satisfied that CCMMP was not willing to select one of the two methods. It is suggested that since galactose is specific to lactose, unlike glucose, the test be carried on the galactose moiety (Method B).

The experts are satisfied that working on the galactose moiety will be adequate for the purpose.

METHODS FOR STANDARDS STILL UNDER ELABORATION (23rd Session of CCMAS, Budapest, Feb-Mar 2001) (See ALINORM 01/23, Appendix IV, pages 36-38)

Cheeses, individual – Dry matter

CCMMP is asked what is the relationship between the results obtained by the method of drying at 102°C (that which has been adopted and endorsed) and the commonly used vacuum oven method at 88°C.

(This applies to all other provisions in the milk product standards for which oven drying at 102°C is proposed.)

Apparently no comparative data are available.

Creams, Whipped Creams and Fermented Creams - Milk solids-not-fat

Not endorsed

CCMAS asked why two methods have been put forward.

Since the previous sessions of both CCMMP and CCMAS a revised edition of IDF 80 / ISO 3727 has appeared. Only this method is submitted to CCMMP to be forwarded to CCMAS for endorsement.

Fermented milks - Lactic acid

Not endorsed

It is not clear why two methods have been put forward. CCMMP will be asked what is the function of the provision, to fix the acidity of the product or to fix the content expressed as lactic acid?

Furthermore CCMAS observes that there cannot be two Type I methods endorsed, but in submitting both IDF 150/ISO11 869 and AOAC937.05, CCMMP is asking for two Type 2s. One of them must be selected for resubmission.

Fermented milks - Dairy starter cultures of lactic acid bacteria (LAB)

Temporarily endorsed Type I

CCMMP is asked for evidence of validation of the methods. CCMMP is also asked for an opinion concerning the type of method.

Fermented milks (yoghurt) - *Streptococcus thermophilus* & *Lactobacillus delbrueckii* subsp. *bulgaricus* $\geq 10^7$ cfu/g

Temporarily endorsed Type I.

CCMMP is asked for evidence of validation of the methods. CCMMP is also asked for an opinion concerning the type of method.

Fermented milks (yoghurt) - *Streptococcus thermophilus* & *Lactobacillus delbrueckii* subsp. *bulgaricus* $\geq 10^7$ cfu/g

Temporarily endorsed Type I.

CCMMP is asked for evidence of validation of the methods. CCMMP is also asked for an opinion concerning the type of method.

Unripened Cheese Including Fresh Cheese - Dry matter [not decided (unripened / fresh cheese)] $\geq 3.5\%$ (cream cheese) and protein

Endorsement not required as the provisions have been removed from the compositional standard.

APPENDIX – METHODS REQUIRED FOR PROVISIONS IN COMMODITY STANDARDS FOR CONSIDERATION BY CCMMP

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
Dairy spreads (A-16)	Milkfat < 80% (m/m) > 10% (m/m)	-	-	-		
	Milkfat (three-quarter fat butter) < = 61 (m/m) > = 59% (m/m) (half-fat butter) < = 41% (m/m) > = 39% (m/m)	ISO 3727-3 IDF 80-3:2001 AOAC 938.06A	Calculation, determination of water & solids-non-fat, calculation of fat content	Butter methods do apply to lower fat content products such as these	III	
Fermented milks	Milkfat < = 10%	ISO 1736:2000				
	Lactic acid (fermented milk) < = 0.5% (m/m) (Yoghurt, culture-modified yoghurt, acidophilus milk) > = 0.6% (m/m) (kumys) > = 0.7% (m/m)	IDF 150:1991 ISO 11869:1997	Potentiometry, titration to pH 8.30	Question has been raised by CCMAS (see report above)	I	NE
	Lactic acid requirements as above	AOAC 937.05	Spectrophotometry (for lactic acid in milk & milk products)	Question has been raised by CCMAS (see report above)	I	NE
	Protein > = 2.7% (m/m) (except for kumys) > = [5.6%] (m/m) (concentrated fermented milk)	ISO 8968-1 IDF 20-1:2001 AOAC 991.20-23	Titrimetry (Kjeldahl)	The method is applicable for all kind of milk products but validated for milk only.	I	E
	Ethanol > = 0.5% (vol./w) (kumys)					

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
	<p>Microorganisms constituting the starter culture</p> <p>(Kefir) Lactic acid bacteria <i>Lactobacillus kefir</i> & species of <i>Leuconostoc</i>, <i>Lactococcus</i> & <i>Acetobacter</i> $\geq 10^7$ cfu/g</p> <p>Yeasts <i>Kluyveromyces marxianus</i>, <i>Saccharomyces omnisporus</i>, <i>S.cerevisiae</i> & <i>S.exiguus</i> $\geq 10^4$ cfu/g</p> <p>(Kumys) Lactic acid bacteria <i>Lactobacillus delbrueckii</i> subsp <i>bulgaricus</i> $\geq 10^7$ cfu/g</p> <p>Yeasts <i>Kluyveromyces marxianus</i> $\geq 10^4$ cfu/g</p>	IDF 149A:1997 (Annex A)	Colony count at 25°C, 30°C, 37°C and 45°C according to the starter organism in question	Question has been raised by CCMAS (see report above)	I	TE
	<p>(Yoghurt) <i>Streptococcus thermophilus</i> & <i>Lactobacillus delbrueckii</i> subsp. <i>bulgaricus</i> $\geq 10^7$ cfu/g</p>	IDF 117B:1997 ISO 7889	Colony count at 37°C	Question has been raised by CCMAS (see report above)	I	TE
	<p>(Yoghurt) <i>Streptococcus thermophilus</i> & <i>Lactobacillus delbrueckii</i> subsp. <i>Bulgaricus</i> $\geq 10^7$ cfu/g</p>	IDF 146:1991 ISO 9232	Test for identification	Question has been raised by CCMAS (see report above)	I	TE

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
Cream and Prepared Creams	Milkfat ≥10% (m/m)	IDF 16C:1987 (revised to ISO 2450) ISO 2450:1999 AOAC 920.111A	Gravimetry (Röse Gottlieb)		I	E
	Milk protein ≥ 35% (m/m)	ISO 8968-1 IDF20-1:2001 AOAC 991.20	Titrimetry (Kjeldahl)	The method is applicable for all kind of milk products but validated for milk only	I	
Whey powders	Lactose (Whey powder and Acid whey powder) ≥ 61.0% (m/m)	ISO 5765-2 IDF 79-2:2002	Enzymatic, on galactose moeity		II	E
	Milk protein (Whey powder) ≥ [11.0] % (m/m) (Acid whey powder) ≥ [7.0] % m/m	ISO 8968-1 IDF 20-1:2001 AOAC 991.20	Titrimetry (modified Kjeldahl)	The method is applicable for all kind of milk products but validated for milk only	I	
	Milkfat (Whey powder, Acid whey powder) ≤ 2%* (m/m)	IDF 9C:1987 (revised to ISO 1736 ISO 1736:2000 AOAC 932.06	Gravimetry (Röse Gottlieb)		II	E
	Water (not including water of crystallization of lactose) (Whey powder) ≤ 5% (m/m) (Acid whey powder) ≤ 45% (m/m)	IDF 26A:1993		Under revision to become ISO 5537 IDF 26	I	
	Ash (Whey powder) ≥ 9.5% (m/m)	IDF 90:1979 ISO 5545:1978	Furnace, at 825°C		IV	E

* recommendation

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
	pH (in 10% solution) (Whey powder) > =5.1 (Acid whey powder) <= 5.1	IDF 115A:1989 ISO 5546:1979			II	
Individual cheeses	Milkfat in dry matter Min Max	IDF 5B:1986 ISO 1735:1987 AOAC 933.05	Gravimetry (Schmid-Bondzynski-Ratzlaff)	Under revision to become ISO 1735 IDF 5	I	E
Cheddar (C-1)	>= 1% (m/m) No restriction >= 20% (m/m) No restriction	idem				
Danbo (C-3)	>= 30% (m/m) No restriction	idem				
Edam (C-4)	>= 30% (m/m) No restriction	idem				
Gouda (C-5)	>= 30% (m/m) No restriction	idem				
Havarti (C-6)	>= 30% (m/m) No restriction	idem				
Samsøe (C-7)	>= 45% (m/m) No restriction	idem				
Emmental (C-9)	>= 30% (m/m) No restriction	idem				
Tilsiter (C-11)	>= 40% (m/m) No restriction	idem				
St Paulin (C-13)	>= 45% (m/m) No restriction	idem				
Provolone (C-15)		idem			II	
Cottage cheese (C-16) - Cottage cheese -Dry curd cottage cheese	>= 4% (m/m) No restriction None <= 4% m/m	IDF 126A:1988 ISO 8262-3:1987 AOAC –	Gravimetry (Weibull-Berntrop)			NE
Coulommiers (C-18)	>= 40% (m/m) No restriction	IDF 5B:1986 ISO 1735:1987 AOAC 933.05	Gravimetry (Schmid-Bondzynski-Ratzlaff)	Under revision to become ISO 1735 IDF 5		
Cream cheese (C-31)	>= [25/40]% (m/m) No restriction	Idem				
Camembert (C-33)	>= 30% (m/m) No restriction	Idem				
Brie (C-34)	>= 40% (m/m) No restriction	Idem				
Mozzarella - high moisture - low moisture	>= 20% (m/m) No restriction >= 2% (m/m) No restriction	Idem Idem				

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
Individual cheeses	Dry matter (Total solids) In the range of:					
Cheddar (C-1)	$\geq 42-64\%$ (m/m)	IDF 4A:1982 ISO 5534:1985 AOAC- all methods differ	Gravimetry, drying at 102°C	Question has been raised by CCMAS (see report above).	II	E
Danbo (C-3)	$\geq 41-57\%$ (m/m)	Idem		Items under revision to become ISO 5534 IDF 4		
Edam (C-4)	$\geq 47-58\%$ (m/m)	Idem				
Gouda (C-5)	$\geq 48-62\%$ (m/m)	Idem				
Havarti (C-6)	$\geq 46-58\%$ (m/m)	Idem				
Samsøe (C-7)						
- Samsøe	$\geq 46-59\%$ (m/m)	Idem				
- Mini-Samsøe	$\geq 46-57\%$ (m/m)	Idem				
Emmental (C-9)	$\geq 60-63\%$ (m/m)	Idem				
Tilsiter (C-11)	$\geq 49-61\%$ (m/m)	Idem				
St Paulin (C-13)	$\geq 44-54\%$ (m/m)	Idem				
Provolone (C-15)	$\geq 51-56\%$ (m/m)	Idem				
Cottage cheese (C-16)						
- Cottage cheese	$\geq 20\%$ (m/m)	Idem				
- Dry curd cottage cheese	$\geq 24\%$ (m/m)	Idem				
Coulommiers (C-18)	In the range of: $\geq 42-52\%$ (m/m)	Idem				
Cream cheese (C-31)	$\geq 25\%$	Idem				
Camembert (C-33)	In the range of: $\geq 38-51\%$ (m/m)	Idem				
Brie (C-34)	$\geq 42-51\%$ (m/m)	Idem				
Mozzarella						
- high moisture	$\geq 24-38\%$ (m/m)	Idem				
- low moisture	$\geq 31-53\%$ (m/m)	Idem				

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
Cream cheese	Moisture on a fat-free basis ≥ 67% (m/m)	IDF 4A:1982 ISO 5534:1985 AOAC – All methods IDF 5B:1986 ISO 1735:1987 AOAC 933.5	Calculation, determination of fat and water content, calculation fat-free basis	Methods under revision		
[Sweetened condensed milk with vegetable fat/Blend of sweetened condensed milk with vegetable fat]	Total fat ≥ [7-8] % (m/m)	IDF 13C:1987 (revised to ISO 1737) ISO 1737:1999 AOAC 920.115F	Gravimetry (Röse Gottlieb)		II	E
	Milk solids-not-fat (including water of crystallization of lactose) ≥ 20% (m/m)	IDF 15B:1991 ISO 6734:1989 ISO 1737:1999	Calculation, Determination of water content and fat content, calculation of solids-non-fat content	Question has been raised by CCMAS (see report above)	I	E
	Milk protein in milk solids-not-fat ≥ 34% (m/m)	AOAC 920.115G	Titrimetry (Kjeldahl)			
[Evaporated skimmed milk with vegetable fat/Blend of evaporated skimmed milk with vegetable fat]	Total fat ≥ [6-8] % (m/m)	IDF 13C:1987 (revised to 1737) ISO 1737:1999 AOAC 945.48G	Gravimetry (Röse Gottlieb)		II	E
	Milk solids-not-fat (including water of crystallization of lactose) ≥ [17.5-20] % (m/m)	IDF 21B:1987 ISO 6731:1989 AOAC 925.23A ISO 2450:1999	Calculation, determination of water content and fat content, calculation of solids-non-fat content			
	Milk protein in milk solids-not-fat = 34% (m/m)	AOAC 945.48H	Titrimetry (Kjeldahl)		I	E

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
[Skimmed milk powder with vegetable fat/Blend of skimmed milk powder with vegetable fat] (for both skimmed milk product and partly skimmed milk product)	Total fat ≥ 26% (m/m)	IDF 9C (revised to ISO 1736) ISO 1736:2000 AOAC 932.06	Gravimetry (Röse Gottlieb)			
	Water* ≤ 5% (m/m)	Under revision to become ISO 5537 IDF 26	Gravimetry, drying at 102°C	Method is for total solids		
	Milk protein in milk solids-not-fat*	ISO 8968-1+3:IDF 20-1+3:2001 AOAC 930.29A	Titrimetry (Kjeldahl)	The method is applicable for all kind of milk products but validated for milk only	I	E

* milk solids and milk solids-not-fat including water of crystallization of lactose