

codex alimentarius commission



FOOD AND AGRICULTURE
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Agenda Item 8 (a)

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME CODEX COMMITTEE ON MILK AND MILK PRODUCTS

Sixth Session

Auckland, New Zealand, 19 – 23 April 2003

METHODS OF ANALYSIS AND SAMPLING FOR MILK AND MILK PRODUCTS

Comments submitted in response to CL 2002/11-MMP, Part C

The following comments were received from IDF/ISO/AOAC International Working Group on Methods of Analysis and Sampling

1 INTRODUCTION

This document provides recommendations for the methods of analysis and sampling (MAS) needed for Codex Standards and draft and proposed draft Codex standards for milk products under consideration at the 6th Session of CCMMP. It also provides answers to questions raised by CCMAS.

2 MAS FOR DRAFT, AND PROPOSED DRAFT, STANDARDS

The list of methods is provided to the Committee for consideration in Appendix 1. The list contains MAS recommended by the Tripartite IDF/ISO/AOAC International Working Group for the draft and proposed draft standards under elaboration.

Recommendation:

CCMMP is invited to:

- (a) refer the methods that relate to standards recommended for adoption at step 8 and that are not already endorsed to CCMAS for endorsement and for inclusion in Volume 13.
- (b) hold other methods for later referral.
- (c) note that a method is not available for the ratio of whey protein to casein in whey cheese.

3 QUESTIONS REFERRED TO CCMMP BY CCMAS (ALINORM 03/23, Appendix VI, Part C)

The 5th Session of CCMMP noted a series of questions concerning methods of analysis related to milk and milk products arising from the 23rd Session of the Codex Committee on Methods of Analysis and Sampling (CCMAS) (ALINORM 01/23, Appendix IV). However, due to time constraints, the specific requests of the CCMAS were not considered.

The 24th Session of CCMAS noted that there was no reply from CCMMP to some earlier questions and did not endorse the methods concerned, pending further clarification from the Committee. The questions are in ALINORM 03/23, Appendix VI, Part C, and are repeated below, with responses from the IDF/ISO/AOAC International Working Group. The conclusions are included in Appendix 1 of this report.

Recommendation:

CCMMP is invited to forward the responses below to CCMAS.

FERMENTED MILKS**Lactic acid - Standards concerned: IDF 150:1991 and ISO 11869:1997**

CCMAS requests CCMMP to indicate whether the IDF method of lactic acid determines total acidity or lactic acid as in the provision.

IDF/ISO/AOAC International answer:

These methods do not measure lactic acid, but titratable acidity and express the result as lactic acid.

Lactic acid - Standards concerned: AOAC 937.05 and AOAC 947.05

CCMAS requests CCMMP to clarify what type of method is requested since there cannot be two type II methods.

IDF/ISO/AOAC International answer:

Like IDF 150, AOAC 947.05 is a Type I method. It determines titratable acidity and expresses the results as lactic acid. AOAC 937.05 is an older method using spectrophotometry. This should be a Type III method.

Microorganisms constituting the starter culture - Standards concerned: IDF 149A:1997

CCMAS requests CCMMP to clarify whether a collaborative study has been performed and what the type of the method is.

IDF/ISO/AOAC International answer:

The method is a Type I method. No collaborative study has been carried out. As a consequence no results are available. The Annex of the standard was prepared on the basis of methods of analyses published in scientific references. A questionnaire was also circulated among IDF/ISO/AOAC International joint action team members and comments were taken into consideration when drafting the standard (Questionnaire 1496/D of 28th May 1996).

Yoghurt

Streptococcus thermophilus & Lactobacillus delbrueckii subsp. Bulgaricus $\geq 10^7$ cfu/g - Standards concerned IDF 117B: 1997 and ISO 7889.

CCMAS requests CCMMP to clarify whether a collaborative study has been performed and the type of the method.

IDF/ISO/AOAC International answer:

The method is a Type I method. A comprehensive interlaboratory test was carried out in 1978 to determine the suitability of the following culture media : skim milk, MRS and M17 media acidified at pH 5.4, Lee's medium, LAB medium, LS-differential medium (see IDF Standard 117A:1988 for method references).

The interlaboratory test was carried out on 30 samples of yogurt purchased on the local market of different countries and involved the following countries:

Italy, United Kingdom, Switzerland, Australia, Germany, Japan, Belgium.

The study has not been published in a peer-reviewed scientific journal.

Only regular reports have been drawn up by Prof. Accolas who at that time was both the IDF/ISO/AOAC E44 group Chairman and the coordinator of the interlaboratory study. Unfortunately, after so many years, it is no longer impossible to publish the ring test results, because Prof. Accolas has passed away many years ago and data are no longer available.

Note: A joint ISO 7889 / IDF 117:2003 has been published. It is recommended that this new standard be included in the provision.

Streptococcus thermophilus & Lactobacillus delbrueckii subsp. Bulgaricus $\geq 10^7$ cfu/g - Standards concerned IDF 146: 1991 and ISO 9232.

CCMAS requests CCMMP to clarify whether a collaborative study has been performed and the type of the method.

IDF/ISO/AOAC International answer:

The method is a Type I method. Two interlaboratory tests were carried out in 1982 (pilot test) and 1984 (Ring test) respectively. Both tests took into consideration pure strains of *S. thermophilus* and *L. delbrueckii* subsp. *bulgaricus* provided for by each member. The last study involved 8 different laboratories from IDF member countries (Czechoslovakia, Denmark, France, Italy, Spain, Switzerland, UK and Israel). The methods applied for the classification of the different strains are described in the Standard. The ring test results have never been published but the standard was drawn up based on this data. A summary of results is given in the Minutes of the group meeting in Milan, 11 March 1985.

Note: Joint ISO 9232 / IDF 146:2003 has been published. It is recommended that this standard is included in the provision.

INDIVIDUAL CHEESES

Dry matter (Total solids) - Standards concerned: IDF 4A: 1982, ISO 5534: 1985 and AOAC 926.08

CCMAS requests CCMMP to clarify the difference in results with the previous method

IDF/ISO/AOAC International answer:

The situation here is a bit confusing. First, according to the 1994 edition of Vol 13, IDF 4A and ISO 5534 were endorsed as methods for total solids in cheese, so perhaps the current status needs to be checked. Second, it's not clear what CCMAS means by the "previous method". It is assumed that the question refers to a comparison between the IDF/ISO methods and the AOAC method. The Tripartite is not aware of comparative data available for these methods. However, from a technical viewpoint we would like to point out that both the previous method and the recommended methods evaporate volatiles from the product which are then expressed as moisture. The 102 °C oven method could possibly give moisture results that are slightly too high as at that temperature there could possibly be some browning of the sample, which is an indication of a reaction between lactose and protein, which possibly could lead to some loss of lactose-bound water. However, it is considered that this is unlikely to be a significant issue because cheese contains very little lactose. To emphasise this point, it is further noted that the 102 °C method is used for a wide range of dairy products including such products as whole milk powder and skim milk powder with typical levels of lactose of 35% and 50% respectively. The previous method (vacuum oven method) could lead to results that are too low because not all types of vacuum oven allow for circulating fresh air, which means that there is a risk of saturation of the atmosphere inside the oven. Hence, the recommended method is the preferred method.

APPENDIX 1 METHODS OF ANALYSIS AND SAMPLING FOR MILK PRODUCTS

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
Cheese (A-6) (adopted Codex standard)	Protein	ISO 8968-1/IDF 20-1:2001 AOAC 930.29	Titrimetry (Kjeldahl)	The method is applicable for all kind of milk products but validated for milk only. ISO 8968-3 IDF 20-3:2004 will be also be applicable when published	I	
Fermented milks (adopted Codex standard)	Lactic acid (fermented milk) < = 0.5% (m/m) (Yoghurt, alternate culture yoghurt, acidophilus milk) > = 0.6% (m/m) (kumys) > = 0.7% (m/m)	IDF 150:1991 ISO 11869:1997; AOAC 947.05	Potentiometry, titration to pH 8.30; Titrimetric using phenolphthalein	Response to CCMAS: the method measures titratable acidity. The results are expressed as lactic acid IDF 150 and ISO 11869 are under revision and the scope will be extended to include milk and fermented milks and will be published jointly.	I	NE 23 CCMAS 24 CCMAS
	Lactic acid requirements as above	AOAC 937.05	Spectrophotometry (for lactic acid in milk & milk products)	Response to CCMAS: This should be a Type III method	III	TE 23 CCMAS
Cheeses, individual (at Step 3)	Milkfat in dry matter	IDF Standard 5B:1986 ISO 1735:1987 AOAC 933.05	Gravimetry (Schmid-Bondzynski-Ratzlaff)		I	E 23 CCMAS and 22 CCMAS
Cheeses, individual (at Step 3) Cheddar (C-1) Danbo (C-3) Edam (C-4) Gouda (C-5) Havarti (C-6) Samsoe (C-7)	Dry matter (Total solids) > = 49% (m/m) > = 41% (m/m) > = 47% (m/m) > = 48% (m/m) > = 46% (m/m) > = 46% (m/m)	IDF 4A:1982 ISO 5534:1985; AOAC 926.08 Idem Idem Idem Idem Idem	Gravimetry, drying at 102 °C; Vacuum oven at 100 °C	Response to CCMAS: the IDF and ISO methods are the preferred methods. Methods are under revision.	I	E 23 CCMAS NE at 22 CCMAS

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
Emmental (C-9) Tilsiter (C-11) St Paulin (C-13) Provolone (C-15) Cottage cheese (C-16)	> = 60% (m/m) > = 49% (m/m) > = 44% (m/m) > = 51% (m/m) Fat-free dry matter >= 18% (m/m)	Idem Idem Idem Idem Dry matter (IDF 4A:1982; ISO 5534:1985; AOAC 926.08) Fat (IDF 126A:1988; ISO 8262-3:1987)	Calculation: determination of fat content and moisture content, calculation fat- free basis		I	E 24 CCMAS
Coulommiers (C-18)	Dry matter (Total solids) > = 42% (m/m)	IDF 4A:1982 ISO 5534:1985; AOAC 926.08				
Cream cheese (C-31) Camembert (C-33) Brie (C-34) Mozzarella - high moisture - low moisture	> = 22% > = 38% (m/m) > = 42% (m/m) > = 24% (m/m) > = 34% (m/m)	Idem Idem Idem Idem Idem				
Cream cheese (at Step 3)	Moisture on a fat-free basis > = 67% (m/m)	IDF 4A:1982 ISO 5534:1985 AOAC 926.08 IDF 5B:1986 ISO 1735:1987 AOAC- 933.05	Calculation: determination of fat content and moisture content, calculation fat- free basis	Methods are under revision	I	
Emmental (at Step 3)	Propionic acid ≥ 150 mg/100 g					

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
	Calcium ≥ 800 mg/100 g					
[Sweetened condensed milk with vegetable fat/Blend of sweetened condensed milk with vegetable fat] (at Step 3)	Total fat > = [7–8]% (m/m)	ISO 1737:1999 AOAC 920.115F	Gravimetry (Röse Gottlieb)		I	22 CCMAS
	Sampling	IDF 50C:1995 ISO 707:1997 AOAC 970.27	General instructions for obtaining a sample from a bulk	IDF 50C & ISO 707 are under revision to become joint ISO 707 IDF 50		
	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		I	E 23 CCMAS for Sweetened Condensed Milk
	Milk protein in milk solids-non-fat > = 34% (m/m)	AOAC 920.115G ISO 8968-1 IDF 20-1 :2001	Titrimetry (Kjeldahl)	ISO 8968-3 IDF 20-3:2004 will also be applicable when published	I	E 23 CCMAS for Sweetened Condensed Milk (previous edition of method)
[Evaporated skimmed milk with vegetable fat/Blend of evaporated skimmed milk with vegetable fat] (at Step 3)	Total fat > = [6–8]% (m/m)	ISO 1737:1999	Gravimetry (Röse Gottlieb)		I	E 22 CCMAS (previous edition of method)
	Milk solids-not-fat	IDF 21B:1987	Calculation,		I	E

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
	(including water of crystallization of lactose) > = [17.5–20]% (m/m)	ISO 6731:1989 ISO 2450:1999	determination of water content and fat content, calculation of solids-non-fat content			22 CCMAS (for evaporated milk E
	Milk protein in milk solids-non-fat + 34% (m/m)	AOAC 945.48H ISO 8968-1 IDF 20-1:2001	Titrimetry (Kjeldahl)	The method is applicable for all kinds of milk products but validated for milk only. ISO 8968-3 IDF 20-3:2004 will also be applicable when published	I	E 23 CCMAS (previous edition of method)
	Sampling	IDF 50C:1995 ISO 707:1997 AOAC 970.27	General instructions for obtaining a sample from a bulk	IDF 50C & ISO 707 are under revision to become joint ISO 707 IDF 50		
[Skimmed milk powder with vegetable fat/Blend of skimmed milk powder with vegetable fat] (for both skimmed milk product and partly skimmed milk product) (at Step 3)	Total fat > = 26% (m/m)	IDF 9C (revised to ISO 1736) ISO 1736:2000 AOAC 932.06	Gravimetry (Röse Gottlieb)		I	
	Water < = 5% (m/m)	ISO 5537/ IDF 26A:1993	Gravimetry, drying at 102°C	Method is for total solids	I	
	Milk protein in milk solids-non-fat	ISO 8968-1 IDF 20-1:2001 AOAC 930.29	Titrimetry (Kjeldahl)	The method is applicable for all kinds of milk products but validated for milk only. ISO 8968-3/IDF 20-3:204 will also be applicable when published	I	
	Sampling	IDF 50C:1995 ISO 707:1997		IDF 50C & ISO 707 are under revision to become		

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE	TYPE	STATUS
		AOAC 970.28		joint ISO 707 IDF 50		
Dairy spreads (at Step 3)	Milk fat < 80% (m/m) > 10% (m/m) Milk fat (three-quarter fat butter) < = 61% (m/m) > = 59% (m/m) (half-fat butter) < = 41% (m/m) > = 39% (m/m)	ISO 17189 IDF 194:2003	Direct determination of fat using solvent extraction		I	
	[Salt]					
	[Dry matter]					
Processed cheese (at Step 3)	Milkfat	IDF 5B:1986 ISO 1735:1987 AOAC 933.05	Gravimetry (Schmid- Bondzynski-Ratzlaff)		I	E
	Protein	IDF 25:1964	Titrimetry (Kjeldahl)	The method is applicable to processed cheese	I	
	Dry matter	IDF 4A:1982 ISO 5534:1985; AOAC 926.08	Gravimetry, drying at 102°C; Vacuum oven at 100 ° C	Methods are under revision	I	E 22 CCMAS
Whey cheese (at Step 3)	Total fat	IDF 59A:1986	Gravimetry (Röse Gottlieb)		I	E 22 CCMAS
	Dry matter (total solids)	IDF 58:1970 ISO 2920:1974	Gravimetry, drying at 88 °C		I	E 23 CCMAS
	Ratio whey protein to casein			No method available		