

codex alimentarius commission

FOOD AND AGRICULTURE
ORGANIZATION
OF THE UNITED NATIONS

WORLD HEALTH
ORGANIZATION

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Agenda Item 6

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JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON MILK AND MILK PRODUCTS
Fourth Session
Wellington, New Zealand, 28 February – 3 March 2000

METHODS OF ANALYSIS AND SAMPLING FOR MILK PRODUCTS¹

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 standards for milk products.

The lists of methods are provided to the Committee for consideration in Appendix I. The lists contain: methods recommended by the Tripartite Working Group for the draft and proposed draft standards under elaboration; and those for adopted standards but for which questions have been raised by the Codex Committees on Milk and Milk Products and on Methods of Analysis and Sampling (CCMAS). 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. Those methods applicable to all or most milk products are shown at the beginning of the list.

The precision data for the methods are being reviewed 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/TC 34/SC 5) and AOAC International. After review the standard methods may be confirmed or revised.

METHODS OF ANALYSIS FOR FOOD ADDITIVES AND CONTAMINANTS

ADDITIVES

IDF, ISO and AOAC International have taken note of the decisions of CCFAC concerning methods of analysis for additives (ALINORM 97/12, Appendix IV) and the criteria for prioritization and selection of methods (ALINORM 97/12, para. 28) as follows:

- "- full ADI established by JECFA;
- provisions for additives or contaminants established by Codex;
- proposed methods should have been validated;
- the use of the additive is causing or has the potential to cause problems in international trade, and;
- the additive is used in a major food or major food ingredient."

¹ This paper includes in Appendix II matters arising from the 22nd Session of the Codex Committee on Methods of Analysis and Sampling which may have implications for the work of this Committee.

Bearing in mind that virtually all of the additives listed in the standards are not used uniquely in milk products IDF, ISO and AOAC International have concerned themselves only with the requirement for pimaricin (natamycin).

CONTAMINANTS

Methods of analysis in use are those applied to all milk products. They are listed at the beginning of the table attached (except those for lead). The CCMAS recommended that Commodity Committees should select methods from the existing Codex general methods wherever possible.

OTHER PROVISIONS FOR WHICH METHODS ARE MISSING

Experts have been asked to identify methods suitable for determining the quantities specified in all such provisions. Such methods will be submitted as soon as they are available in the form meeting the requirements of the CCMAS.

MATTERS RAISED AT 3RD SESSION OF CCMMP AND 22ND SESSION OF CCMAS

IDF, ISO and AOAC International have paid particular attention to the questions raised concerning methods for analysis and sampling of milk and milk products at the 3rd Session of CCMMP (Montevideo, May 1998) and the 22nd Session of CCMAS (Budapest, November 1998) and report as follows. Some of the comments below are relevant for the methods submitted to the 4th Session of CCMMP (Wellington, Feb-Mar 2000).

PART 1 METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS FOR MILK PRODUCTS

Butter – Lead

The maximum level is 0.05 mg/kg.

AOAC method 972.25 is particularly suitable for products containing a high calcium level; it may not do for butter. AOAC 994.02 and its IUPAC equivalent may be suitable. AOAC method 972.25 has been endorsed by the CCMAS for butter, edible casein products and whey powders. The method can be withdrawn (for butter only) if the Committee so agrees.

Butter – Milkfat content, Milk-solids-non-fat-content

The CCMAS has endorsed IDF Standard 80:1997/ISO 3727:1977/AOAC 938.06 and 920.116 (gravimetric method). However, the method has been criticized as insufficiently reliable. The method is under revision and will be finalized in the beginning of 2001 or sooner.

The endorsed methods are retained for the time being pending the completion of the revision.

Cheese - Moisture content

*The CCMAS did not endorse either IDF Standard 4A:1982/ISO 5534:1985 (gravimetric method, drying at 102 °C) or AOAC 926.08 (vacuum oven method) as **only one Type I method** can be endorsed for one analyte/product combination. It asked the CCMMP to recommend only one of these two Type I methods.*

The general issue for determining moisture and dry matter in cheese is which principle to choose for the method. Vacuum oven is in use in North America. Elsewhere drying at 102°C seems more common. It can be a matter of equivalence.

IDF/ISO/AOAC International have not been able to establish whether any cross-validation or comparison tests have been carried out. Although it is necessary to select only one Type I method for the determination of moisture in cheese, at present the selection can only be arbitrary.

Cheese – Solids content

As in the case of Cheese - moisture content, *the CCMAS did not endorse either IDF Standard 4A:1982/ISO 5534:1985 (gravimetric method, drying at 102 °C) or AOAC 926.08 (vacuum oven method) as only one Type I method can be endorsed for the one analyte/product combination. It asked CCMP to recommend only one of these two Type I methods.*

The same method is involved for moisture content in cheese and dry matter in cheese-in-brine. No cross-validation or comparison tests appear to have been carried out. Although it is necessary to select only one Type I method for the determination of solids in cheese, at present the selection can only be arbitrary.

Cheeses in Brine - Dry matter

The minimum dry matter contents are: Soft 40% ; and Semi-hard 52%.

The CCMAS did not endorse either IDF Standard 4A:1982/ISO 5534:1985 (gravimetric method, drying at 102 °C) or AOAC 926.08 (vacuum oven method) as only one Type I method can be endorsed for one analyte/product combination. It requested the CCMP to recommend only one of these two Type I methods.

IDF/ISO/AOAC International have not been able to establish whether any cross-validation or comparison tests have been carried out. Although it is necessary to select only one Type I method for the determination of dry matter in cheeses in brine, at present the selection can only be arbitrary.

Evaporated milks – Protein content

The limit for all types of evaporated milks is minimum milk protein in milk-solids-non-fat 34 % (m/m).

The CCMAS endorsed AOAC method 945.48H but not IDF 20B:1993/AOAC 991.21-23 as there can be only one Type I method and as the CCMAS was informed that the latter method has not been shown to be applicable to the commodity in question. In addition ISO/DIS 8968 was removed due to its draft status.

AOAC 945.48H refers to AOAC 991.20 for the Kjeldahl determination and this is identified with IDF Standard 20B:1993 . The methods IDF Standard 20B:1993 / AOAC 991.20-23 are identical Type I method.

Milkfat products – Antioxidants

Whether antioxidants are used or not will affect the denomination of the products covered in the standards. The maximum levels of antioxidants are as follows:

310	Propyl gallate	100 mg/kg
321	Butylated hydroxytoluene (BHT)	75 mg/kg
320	Butylated hydroxyanisole (BHA)	175 mg/kg
	Any combination of propyl gallate, BHA and BHT providing limits above are not exceeded.	200 mg/kg
306	Mixed tocopherols concentrate	500 mg/kg individually or in combination
307	α-Tocopherol	
304	Ascorbyl palmitate	
305	Ascorbyl stearate	
Antioxidant Synergists		
330	Citric acid	Limited by GMP
331	Sodium citrate	Limited by GMP

IDF Standard 165:1993, endorsed by the CCMAS, provides methods for propyl gallate, BHT, BHA. The other antioxidants are not covered by methods for the time being.

Milkfat Products - Copper content

The minimum level is 0.05 mg/kg in milkfat products and butter.

*The CCMAS endorsed AOAC 985.35 and IDF Standard 76A:1980/ISO 5738:1980/AOAC 960.40 (photometric, diethyldithiocarbamate) method) for the determination of copper at or above 5 mg/kg in milk products. However, the CCMAS did not endorse IDF Standard 76A:1980/ISO 5738:1980/AOAC 960.40 for the determination of copper at or above 0.05 mg/kg (in butter and milkfat) as it was not convinced of the applicability of the method to high-fat products. The CCMMP was requested to review the method in this respect as well as to consider the applicability of method AOAC 990.05, IUPAC Method (Pure & Appl. Chem. **60** (6))(atomic absorption spectrophotometry, graphite furnace).*

The repeatability of the method, IDF Standard 76A /ISO 5738 for butter and milkfat, $r = 0.004$ mg/kg. With such a value of r , it should be possible to determine as low as 0.05 mg/kg, if not lower.

Milkfat products – Iron

The maximum level for iron in milkfat products is 0.2mg/kg

IDF Standard 130A:1986 / ISO 6732. The repeatability for milkfat, butter and butterfat $r = 0.02$ and 0.005 respectively. With such values of r it should be possible to determine as low as 0.2 mg/kg, if not lower.

Milkfat products – Lead

See Butter-Lead above.

Milkfat products – Peroxide value

The CCMAS endorsed AOAC 965.33 for the determination of peroxide value of anhydrous milkfat. However, it did not endorse IDF Standard 74A:1991/ISO 3976:1977 (photometric, $FeCl_3/NH_4CNS$ method) recommended for all milkfat products. The CCMMP is requested by the CCMAS to consider whether AOAC 965.33 is applicable to the determination of peroxide values of other milkfat products (milkfat, butteroil, ghee, anhydrous butteroil).

Milk Powders and Cream Powder Titratable acidity

As the CCMAS has endorsed IDF Standard 86:1981, it does not appear necessary to look into the matter further.

Sweetened condensed milk - Protein content

The limit for all types of sweetened condensed milk is minimum milk protein in milk-solids-non-fat 34 % (m/m).

The CCMAS endorsed AOAC 920.115G but not *IDF 20B:1993/AOAC 991.21-23 as there can be only one Type I method and as the CCMAS was informed that the latter method has not been shown to be applicable to the commodity in question.. In addition ISO/DIS 8968 was removed due to its draft status.*

AOAC 920.115G refers to AOAC 991.20 for the Kjeldahl determination and this is identified with IDF Standard 20B:1993 . The methods, IDF Standard 20 B:1993/ AOAC 991.20-23 are identical Type I method.

Sweetened condensed milk - Solids content

*The CCMAS did not endorse either IDF Standard 15B:1991 / ISO 6734 (gravimetric, drying at 102 °C method) or AOAC 920.115D (vacuum oven method) as **only one Type I method** can be endorsed for the one analyte/product combination. It asked the CCMMP to recommend only one of these two Type I methods.*

The general issue for determining dry matter in sweetened condensed milk is which method to choose for the method. Vacuum oven is in use in North America. Elsewhere drying at 102°C seems more common. It can be a matter of equivalence.

Note that for solids in evaporated milk, CCMAS endorsed the method with drying at (98-100) °C (IDF Standard 21B / ISO 6731 / AOAC 925.23 A) rather than vacuum oven (AOAC 920.107) for which there are no precision figures for evaporated milk.

Whey powders - Lactose (expressed as anhydrous lactose)

The limit is minimum 61%.

As IDF Standard 79B:1991 / ISO/DIS 5765 provides “two methods” (enzymatic on the glucose moiety (method A) and on the galactose moiety (method B)), the CCMAS did not endorse the method and asked the CCMMP to indicate which method it prefers.

Methods A and B are complementary to each other. Nothing was mentioned about providing equal results in the collaborative study cited in the Bulletin of Int. Dairy Fed. N° 285/1993. As such the results mentioned could be used for a comparison.

Any attempt to use only one of these two methods is arbitrary.

Whey powders – Protein

The method in IDF Standard 92:1979 / ISO 5549 (titrimetric, Kjeldahl) has been endorsed by the CCMAS as a Type IV method (without precision data).

As the standard relates to casein and caseinates, it may be necessary to establish its suitability for whey powder and provide precision data. Work is currently in hand, specifically on the Dumas method.

PART 2 METHODS OF SAMPLING PROVISIONS IN CODEX STANDARDS FOR MILK PRODUCTS

Cheeses in brine

While endorsing the method, the CCMAS requested the clarification of the text in the method of sampling section of the Standard for Cheeses in Brine (cited below) as to whether it contains contradictions:

“A representative piece of cheese is placed on *a cloth or on a sheet of non-absorbent paper** for 5 to 10 minutes. A slice of 2-3 cm is cut off and sent to the laboratory in a sealed insulated box for analysis.”

**) italicized by the Codex Secretariat for clarification.*

IDF/ISO/AOAC International proposed to amend the first sentence to read: “A representative piece of cheese is placed in a cloth or on a sheet of absorbent paper for 5 to 10 min.”

The CCMMP is invited to agree to the proposed amendment.

Sampling by attributes

The CCMAS suggested that the Standards for statistical sampling of milk and milk products be revised. Work on the revision of IDF Standard 113 has been started but is not yet finished.

Sampling by variables

Revision of IDF Standard 136A has been started but not yet finished.

APPENDIX I: METHODS OF ANALYSIS AND SAMPLING FOR MILK PRODUCTS

1. Methods for Requirements/Specifications in Draft and Proposed Draft under Elaboration (except food additives)

COMMODITY	PROVISION	METHOD ²	PRINCIPLE	NOTE ³
Milk Products	Copper <= 5 mg/kg (whey powders, edible casein products)	AOAC 985.35	Atomic absorption spectrophotometry	E/II
Milk Products	Copper <= 5 mg/kg (whey powders, edible casein products)	IDF Standard 76A:1980 ISO 5738:1980 AOAC 960.40 (Codex general method)	Photometry, diethyldithiocarbamate	E/III
Milk Products	Iron <= 20 mg/kg (spray dried whey powder, edible caseinate products except roller dried caseinates), <= 50 mg/kg (roller dried whey powder & caseinates) <= 2.0 mg/kg (butter) <= 0.2 mg/kg (milkfat products)	NMKL 139.1991 (Codex general method)		E/II
Milk Products	Iron <= 20 mg/kg (spray dried whey powder, edible caseinate products except roller dried caseinates), <= 50 mg/kg (roller dried whey powder & caseinates) <= 2.0 mg/kg (butter) <= 0.2 mg/kg (milkfat products)	IDF Standard 103A:1986 ISO 6732:1985	Photometry, bathophenanthroline	E/IV
Milk Products	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions	E/-
Milk Products	Sampling	IDF Standard 113A:1990 ISO 5538:1987	Inspection by attributes	E/-
Milk Products	Sampling	IDF Standard 136A:1992 ISO 8197:1988	General instructions	E/-

² IDF/ISO methods are reviewed systematically five years after publication or their last review. After review the standard methods may be confirmed or revised.

³ The status of endorsement (E=endorsed by the CCMAS; NE=not endorsed; blank=not yet considered by the CCMAS) and, if the method is endorsed, its Type.

COMMODITY	PROVISION	METHOD ²	PRINCIPLE	NOTE ³
Cheese (A-6, C)	Milkfat (specified in individual standards)	IDF Standard 5B:1986 ISO 1735:1987 AOAC 933.05	Gravimetry (Schmid-Bonzynski-Ratzlaff)	E/I
Cheese (A-6, C)	Moisture (specified in individual standards)	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C	NE
Cheese (A-6, C)	Moisture (specified in individual standards)	AOAC 926.08	Vacuum oven	NE
Cheese (A-6, C)	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions	E/-
Cheese (A-6, C)	Solids (specified in individual standards)	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C	NE
Cheese (A-6, C)	Solids (specified in individual standards)	AOAC 926.08	Vacuum oven	NE
Cheeses, individual (C)	Dry matter (specified in individual standards)	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C	
Cheeses, individual (C)	Milkfat in dry matter >=48 % (48-55) %	IDF Standard 5B:1986 ISO 1735:1987 AOAC 933.05	Gravimetry (Schmid-Bonzynski-Ratzlaff)	
Creams, Whipped Creams and Fermented Creams (A-9)	Milk solids-not-fat <= 20 g/kg	IDF Standard 80:1977 ISO 3727:1977 AOAC 920.116	Gravimetry	
Creams, Whipped Creams and Fermented Creams (A-9)	Milk-solid-not-fat <= 20 g/kg	IDF Standard 11A:1986	Gravimetry	
Creams, Whipped Creams and Fermented Creams (A-9)	Caseinates <= 6 g/kg	-		
Creams, Whipped Creams and Fermented Milks (A-9)	Gelatine and starches <= 6 g/kg, singly etc	-		
Cream (A-9)	Milkfat >= xx % m/m	IDF Standard 16C:1987 ISO 2450:1999 AOAC 995.19	Gravimetry	

COMMODITY	PROVISION	METHOD ²	PRINCIPLE	NOTE ³
Creams Lowered in Milkfat Content	Milkfat >= 10 %	IDF Standard 16C:1987 ISO 2450:1999 AOAC 995.19	Gravimetry	
Creams, Whipped creams and Fermented Creams (A-9)	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions	
Dairy Spreads	Milkfat (59-61) %	IDF Standard 80:1977 ISO 3727:1977 AOAC 938.06	Gravimetry	
Edible Casein Products (A-18)	Ash (including P ₂ O ₅) >= 7.5 % (rennet casein), <= 2.5 % (acid casein)	IDF Standard 90:1979 ISO 5545:1978	Furnace, 825 °C	E/IV
Edible Casein Products (A-18)	Casein in protein >= 95 %	IDF Standard 29:1964	Titrimetry, Kjeldahl	
Edible Casein Products (A-18)	Free acid <= 0.27 ml 0.1 N NaOH/g	IDF Standard 91:1979 ISO 5547:1978	Titrimetry, aqueous extract	E/IV
Edible Casein Products (A-18)	Lactose <= 1.0 %	IDF Standard 106:1982 ISO 5548:1980	Photometry, phenol and H ₂ SO ₄	E/IV
Edible Casein Products (A-18)	Lead <= 1 mg/kg	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	E/II
Edible Casein Products (A-18)	Lead <= 1 mg/kg	IDF Standard 133A:1992	Spectrometry, 1,5-diphenylthiocarbazone	E/III
Edible Casein Products (A-18)	Lead <= 1 mg/kg	AOAC 982.23 (Codex general method)	Anodic Stripping Voltammetry	E/III
Edible Casein Products (A-18)	Lead <= 1 mg/kg	NMKL 139.1991 (Codex general method)	Atomic absorption spectrophotometry	E/III
Edible Casein Products (A-18)	Milkfat <= 2.0 %	IDF Standard 127A:1988 ISO 5543:1986	Gravimetry (Schmidt-Bondzynski-Ratslaff)	E/I
Edible Casein Products (A-18)	Moisture <= 12 % (rennet casein & acid casein), <= 8 % (caseinates)	IDF Standard 78C:1991 ISO 5550:1978	Gravimetry, drying at 102 °C	E/I
Edible Casein Products (A-18)	pH <= 7.5 (caseinates)	IDF Standard 115A:1989 ISO 5546:1979	Electrometry	E/IV

COMMODITY	PROVISION	METHOD ²	PRINCIPLE	NOTE ³
Edible Casein Products (A-18)	Protein (total N x 6.38 in dry matter) >= 84 % (rennet casein), >= 90 % (acid casein), >= 88 % (caseinates)	IDF Standard 92:1979 ISO 5549:1978	Titrimetry, Kjeldahl	E/IV
Edible Casein Products (A-18)	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions	E/-
Edible Casein Products (A-18)	Sediment (scorched particles) (in 25 g) <= 15 mg (rennet casein), <= 22.5 mg (acid casein, spray dried caseinates), <= 81.5 mg (roller dried caseinates)	IDF Standard 107A:1995 ISO 5739:1983	Visual comparison with standard disks, after filtration	E/IV
Fermented Milks (A-11)	Milk solids-not-fat (level not specified)	-		
Fermented Milks (A-11)	Protein in milk solids-not-fat >= 34 % (except for kumys)	-		
Fermented Milks (A-11)	Lactic acid >= 0.6 % (m/m) (yoghurt, acidophilus milk, cultured milk, cultured buttermilk, fermented milk containing bifidobacteria, kefir), >= 0.7 % (m/m) (kumys)	IDF Standard 150:1991 ISO 11869:1997	Potentiometry	
Fermented Milks (A-11)	Lactic acid >= 0.6 % (m/m) (yoghurt, acidophilus milk, cultured milk, cultured buttermilk, fermented milk containing bifidobacteria, kefir), >= 0.7 % (m/m) (kumys)	AOAC 937.05	Spectrophotometric (for lactate acid in milk & milk products)	
Fermented Milks (A-11)	Protein >= 2.8 % (m/m) (except for kumys)	IDF Standard 20B:1993 ISO DIS 8968 AOAC 991.20-23	Titrimetry (Kjeldahl)	
Fermented Milks (A-11)	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions	
Fermented Milks (Acidophilus milk) (A-11)	<i>Lactobacillus acidophilus</i> >= 10 ⁷ cfu/g	-		

COMMODITY	PROVISION	METHOD ²	PRINCIPLE	NOTE ³
Fermented Milks (Cultured Milk, Cultured Buttermilk) (A-11)	Mesophilic lactic acid producing bacteria, either single culture or mixed cultures $\geq 10^7$ cfu/g or, in the case of bifidobacteria,	-		
Fermented Milks (Fermented Milk containing Bifidobacteria) (A-11)	Bifidobacteria $\geq 10^6$ cfu/g	-		
Fermented Milks (Kefir) (A-11)	<i>Kluyveromyces marxianus</i> , <i>Saccharomyces omnisporus</i> , <i>S. cerevisiae</i> & <i>S. exiguus</i> $\geq 10^4$ cfu/g	-		
Fermented Milks (Kefir) (A-11)	<i>Lactobacillus kefir</i> and species of <i>Leuconostoc</i> , <i>Lactococcus</i> & <i>Acetobacter</i> $\geq 10^7$ cfu/g	-		
Fermented Milks (Kumys) (A-11)	<i>Kluyveromyces marxianus</i> $\geq 10^4$ cfu/g	-		
Fermented Milks (Kumys) (A-11)	<i>Lactobacillus delbrueckii</i> subsp. <i>Bulgaricus</i> $\geq 10^7$ cfu/g	-		
Fermented Milks (A-11)	Dairy starter cultures of lactic acid bacteria (LAB)	IDF Standard 149A:1997 (Annex A)	Colony count at 25 °C, 30 °C, 37 °C & 45 °C according to the starter organism in question	
Fermented Milks (Yoghurt) (A-11)	<i>Streptococcus thermophilus</i> & <i>Lactobacillus delbrueckii</i> subsp. <i>Bulgaricus</i> $\geq 10^7$ cfu/g	IDF Standard 117B:1997 ISO DIS 7889	Colony count at 37°C	
Fermented Milks (Yoghurt) (A-11)	<i>Streptococcus thermophilus</i> & <i>Lactobacillus delbrueckii</i> subsp. <i>bulgaricus</i> $\geq 10^7$ cfu/g	IDF Standard 146:1991 ISO CD 9232	Test for identification	

COMMODITY	PROVISION	METHOD ²	PRINCIPLE	NOTE ³
Milk Products obtained from Fermented Milks Heat-Treated after Fermentation (A-11)	Milk solids-not-fat (no level specified)	-		
Milk Products obtained from Fermented Milks Heat-Treated after Fermentation (A-11)	Protein in milk solids-not-fat $\geq 34\%$	-		
Milk Products obtained from Fermented Milks Heat-Treated after Fermentation (A-11)	Solids-not-fat (no level specified)	-		
Milk Products obtained from Fermented Milks Heat-Treated after Fermentation (A-11)	Protein $\geq 2.8\%$ (m/m)	IDF Standard 20B:1993 ISO DIS 8968 AOAC 991.20-23	Titrimetry (Kjeldahl)	
Milk Products obtained from Fermented Milks Heat-Treated after Fermentation (A-11)	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions	E/-
Processed Cheese Products (A-8)	Dry matter $\geq 20\%$	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C	
Processed Cheese Products (A-8)	Dry matter $\geq 20\%$	AOAC 926.08	Gravimetry, vacuum oven	
Processed Cheese Products (A-8)	Gelatin and starch ≤ 10 g/kg singly or combined and/or in combination with stabilizers/thickeners (processed cheese preparations)	AOAC 940.24 (cottage cheese)		

COMMODITY	PROVISION	METHOD ²	PRINCIPLE	NOTE ³
Processed Cheese Products (A-8)	Milkfat (dry basis) (no level specified)	IDF Standard 5B:1986 ISO 1735:1987 AOAC 933.05	Gravimetry (Schmid-Bonzynski-Ratzlaff)	
Sweetened Yoghurt (A-11)	Ethanol ≥ 0.5 % (v/w) (kumys)	-		
Unripened Cheese Including Fresh Cheese	Gelatine and starch ≤ 5 g/kg	-		
Unripened Cheese Including Fresh Cheese	Dry matter [not decided (unripened/fresh cheese)] ≥ 3.5 % (cream cheese)	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C	
Unripened Cheese Including Fresh Cheese	Moisture on fat free basis > 67 % (m/m)	-		
Unripened Cheese Including Fresh Cheese	Dry matter [not decided (unripened/fresh cheese)] ≥ 35 % (m/m), < Restricted by the MMFB	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C	
Unripened Cheese Including Fresh Cheese	Dry matter [not decided (unripened/fresh cheese)] ≥ 3.5 % (cream cheese)	AOAC 926.08	Gravimetry, vacuum oven	
Unripened Cheese Including Fresh Cheese	Milkfat in dry matter ≥ 60 % (cream cheese)	-		
Unripened Cheese Including Fresh Cheese	Protein ≥ 60 % (in milkfat free dry matter without addition of foods and flavouring substances)	IDF Standard 20B:1993 ISO DIS 8968 AOAC 991.20/920.123	Titrimetry, Kjeldahl	
Whey Powders (A-15)	Ash ≤ 9.5 % (whey powder), ≤ 15.0 % (acid whey powder)	IDF Standard 90:1979 ISO 5545:1978	Furnace, 825 °C	E/IV
Whey Powders (A-15)	Lactose (expressed as anhydrous lactose) ≥ 61.0 %	IDF Standard 79B:1991 ISO CD 5765	Enzymatic method; Glucose moiety (method A), Galactose moiety (method B)	NE
Whey Powders (A-15)	Lead ≤ 1 mg/kg	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	E/II
Whey Powders (A-15)	Milkfat ≤ 2 %	IDF Standard 9C:1987 ISO 1736:1985 AOAC 932.06	Gravimetry (Röse-Gottlieb)	E/I

COMMODITY	PROVISION	METHOD ²	PRINCIPLE	NOTE ³
Whey Powders (A-15)	Moisture, "Free" <= 5.0 % (whey powder), <= 4.5 % (acid whey powder)	IDF Standard 58:1970 ISO 2920:1974	Gravimetry, drying at 88 °C	E/IV
Whey Powders (A-15)	pH (in 10 % solution) > 5.1 (whey powder), <= 5.1 (acid whey powder)	-		
Whey Powders (A-15)	Protein (total N x 6.38) >= 11 % (whey powder), >= 10 % (acid whey powder)	IDF Standard 92:1979 ISO 5549:1978	Titrimetry, Kjeldahl	E/IV
Whey Powders (A-15)	Sampling	IDF Standard 113A:1990 ISO 5538:1987	Inspection by attributes	E/-
Whey powders (A-15)	Sampling	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions	E/-

2. Methods for adopted Codex Standards for which questions have been raised by the CCMMP or CCMAS (except food additives)⁴

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE ⁵
Milk Products	Copper <=0.05 mg/kg (butter, milkfat products)	AOAC 985.35	Atomic absorption spectrophotometry	⁶
Milk Products	Copper <= 0.05 mg/kg (whey powders, edible casein products)	IDF Standard 76A:1980 ISO 5738:1980 AOAC 960.40 (Codex general method)	Photometry, diethyldithiocarbamate	NE
Cheeses in Brine (208)	Dry matter >= 40 % (soft), >= 52 % (semi-hard)	IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C	NE
Cheeses in Brine (208)	Dry matter >= 40 % (soft), >= 52 % (semi-hard)	AOAC 926.08	Gravimetry, vacuum oven	NE
Milkfat Products (A-2)	Propyl gallate, BHT, BHA (use or non-use)	IDF Standard 165:1993	Reversed phase gradient light chromatography	E/II

⁴ See also Part 1 of the body of the text.

⁵ The status of endorsement (E=endorsed by the CCMAS; NE=not endorsed; blank=not yet considered by the CCMAS) and, if the method is endorsed, its Type.

⁶ Endorsed for the level <=5 mg/kg.

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE ⁵
Milkfat Products (A-2)	Peroxide value (expressed as milliequivalents of oxygen/kg fat) <= 0.3 (anhydrous milkfat, anhydrous butteroil), <= 0.6 (milkfat, butteroil, ghee)	IDF Standard 74A:1991 ISO 3976:1977	Photometry, FeCl ₃ /NH ₄ CNS	NE
Sweetened Condensed Milks (A-4)	Solids >= 28 % (sweetened condensed milk), >= 24 % (sweetened condensed skimmed milk, sweetened condensed partly skimmed milk)	IDF Standard 15B:1991 ISO 6734:1989	Gravimetry, drying at 102 °C	NE
Sweetened Condensed Milks (A-4)	Solids >= 28 % (sweetened condensed milk), >= 24 % (sweetened condensed skimmed milk, sweetened condensed partly skimmed milk)	AOAC 920.115D	Gravimetry, vacuum oven	NE

3. Methods established for food additives

COMMODITY	PROVISION	METHOD	PRINCIPLE	NOTE ⁷
Cheese and Processed Cheese Products	Citric acid	IDF Standard 34C:1992	Enzymatic	E/II
Cheese and Processed Cheese Products	Citric acid	ISO 2963:1997 AOAC 976.15	Photometry	E/II
Cheese and Cheese Rind	Pimaricin (Natamycin) 2 mg/dm ² surface. Absent at 5 mm depth	IDF Standard 140A:1992 ISO 9233:1991	Molecular absorption spectrometry & HPLC (extraction)	E/II
Processed Cheese Products	Added phosphate (expressed as phosphorus)	IDF Standard 51B:1991	Calculation	
Processed Cheese Products	Citrate emulsifying agents	IDF Standard 52A:1992 ISO 12082:1997	Calculation from citric acid & lactose contents	
Processed Cheese Products	Phosphorus	IDF Standard 33C:1987 ISO 2962:1984 AOAC 990.24	Spectrophotometry, molybdate-ascorbic acid	

⁷ The status of endorsement (E=endorsed by the CCMAS; NE=not endorsed; blank=not yet considered by the CCMAS) and, if the method is endorsed, its Type.

**APPENDIX II: MATTERS ARISING FROM THE 22ND SESSION OF THE
CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
WHICH MAY HAVE IMPLICATIONS FOR THE WORK OF THE COMMITTEE**
(Prepared by the Codex Secretariat)

1. ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING IN DRAFT STANDARDS FOR MILK PRODUCTS⁸ (ALINORM 99/23, APPENDIX III)

The Committee on Methods of Analysis and Sampling considered for endorsement methods of analysis contained in the Draft Standards for Butter, Milkfat Products, Evaporated Milks, Sweetened Condensed Milks, Milk Powders and Cream Powder, Cheese, Whey Cheese, and Cheeses in Brine, and those applicable to all or most milk products, submitted by this Committee. It endorsed many of them but did not endorse several Type I methods where there were more than one Type I methods submitted and it was not clear which one is most suitable.

The Committee endorsed all methods of sampling submitted but it requested clarification on the wording concerning the sampling of cheeses in brine.

2. GENERAL MATTERS REGARDING SELECTION OF METHODS OF ANALYSIS AND THEIR SUBMISSION FOR ENDORSEMENT

- i. The Committee recommended that commodity committees should select method from the existing Codex general methods wherever possible and use the SI unit system in the specifications of Codex standards (ALINORM 99/23, paras 61-62)
- ii. The Committee asked the commodity committees to provide information as required by the Checklists contained in Volume 13 of the *Codex Alimentarius* and the *Codex Alimentarius Commission Procedural Manual*, when they send methods of analysis and sampling to the Committee (ALINORM 99/23, para. 60)
- iii. The Committee agreed that it would have no objection to the use of proprietary methods, provided that similar results were available by from other method(s)(ALINORM 99/23, para. 8)

3. REQUEST FOR INFORMATION (ALINORM 99/23, PARAS 9-13)

In the process of elaborating General Guidelines on Sampling, the Committee had discussions on the use of a “statistical” approach as opposed to “practical” approach. The Committee agreed that information should be sought from commodity committees on the acceptance of the statistical approach to sampling when defining compliance with the specifications in Codex standards.

⁸ See the body of this document