

# codex alimentarius commission



FOOD AND AGRICULTURE  
ORGANIZATION  
OF THE UNITED NATIONS

WORLD  
HEALTH  
ORGANIZATION



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Agenda Item 6a)

CX/MAS 06/27/6

## JOINT FAO/WHO FOOD STANDARDS PROGRAMME

### CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Twenty-seventh Session

Budapest, Hungary, 15-19 May 2006

#### ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS

This document contains the Methods of analysis and sampling proposed by the following Committees and Task Forces in Draft Standards and Proposed Draft Standards under elaboration or as a revision of the methods included in adopted standards.

This document includes the replies of individual Committees and Task Forces to the questions from the last session(s) of CCMAS concerning specific methods of analysis under the relevant sections.

#### Part I. Methods of Analysis

- A. *Ad hoc* Intergovernmental Task Force on Fruit and Vegetable Juices
- B. Codex Committee on Cereals, Pulses and Legumes (Draft Standard for Instant Noodles)
- C. Codex Committee on Milk and Milk Products
- D. Codex Committee on Nutrition and Foods for Special Dietary Uses

#### Part II. Sampling

Codex Committee on Milk and Milk Products

**PART I. METHODS OF ANALYSIS**

**A. AD HOC INTERGOVERNMENTAL TASK FORCE ON FRUIT AND VEGETABLE JUICES<sup>1</sup>**

**General Standard for Fruit Juices and Nectars (CODEX STAN 247-2005)**

COMMODITY	PROVISION	METHOD	PRINCIPLE
Fruit Juices and Nectars	Sections 3.2 Quality Criteria and 3.3 Authenticity <sup>2</sup>	Determination of Acetic acid EN 12632 or IFU Method No 66 (1996)	Enzymatic determination
		Determination of Alcohol (ethanol) IFU Method No 52 (1983/1996)	Enzymatic determination
		Determination of anthocyanins IFU Method No 71 (1998)	HPLC
		Determination of ash in fruit products AOAC 940.26 EN 1135 (1994) IFU Method No 9 (1989)	Gravimetry
		Determination of Beet sugar in fruit juices AOAC 995.17	Deuterium NMR
		Determination of Benzoic acid as a marker in orange juice AOAC 994.11	HPLC
		Determination of C <sup>13</sup> /C <sup>12</sup> ratio of ethanol derived from fruit juices JAOAC 79, No. 1, 1996, 62-72	Stable isotope mass spectrometry
		Determination of Carbon stable isotope ratio of apple juice AOAC 981.09 - JAOAC 64, 85 (1981)	Stable isotope mass spectrometry

<sup>1</sup> ALINORM 05/28/39, Appendix II, adopted by the 28<sup>th</sup> Session of the Commission as CODEX STAN 247-2005

<sup>2</sup> **3.4 Verification of Composition, Quality and Authenticity**

Fruit juices and nectars should be subject to testing for authenticity, composition, and quality where applicable and where required. The analytical methods used should be those found in Section 9, Methods of Analysis and Sampling.

The verification of a sample's authenticity / quality can be assessed by comparison of data for the sample, generated using appropriate methods included in the standard, with that produced for fruit of the same type and from the same region, allowing for natural variations, seasonal changes and for variations occurring due to processing.

		Determination of Carbon stable isotope ratio of orange juice AOAC 982.21	Stable isotope mass spectrometry
		Determination of Carotenoid, Total/individual groups EN 12136 (1997) - IFU Method No 59 (1991)	Spectrophotometry
		Determination of Carotenoids, Total ISO 6558-2:1992	Column chromatographic separation and spectrometry
		Determination of Centrifugable pulp EN 12134 - IFU Method No 60 (1991/1998)	Centrifugation/% value
		Determination of Chloride (expressed as sodium chloride) EN12133 IFU Method No 37 (1968)	Electrochemical titrimetry
		Determination of Chloride in vegetable juice AOAC 971.27 (Codex general method) ISO 3634:1979	Titration
	Sections 3.2 Quality Criteria and 3.3 Authenticity	Determination of Essential oils AOAC 968.20 - IFU 45b	(Scott) distillation, titration
		Determination of Essential oils (in citrus fruit) ISO 1955:1982	Distillation and direct reading of the volume
		Determination of Fermentability IFU Method No 18 (1974)	Microbiological method
		Determination of Formol number EN 1133 (1994) IFU Method No 30 (1984)	Potentiometric titration
		Determination of Free amino acids EN 12742 IFU Method No 57 (1989)	Chromatography
		Determination of Fumaric acid IFU Method No 72 (1998)	HPLC
	Glucose and fructose (permitted ingredients)	Determination of glucose fructose and saccharose EN 12630 - IFU Method No 67 (1996) NMKL 148 (1993)	HPLC

	Sections 3.2 Quality Criteria and 3.3 Authenticity	Determination of Gluconic acid IFU Method No 76 (2001)	Enzymatic determination
		Determination of Glycerol IFU Method No 77 (2001)	Enzymatic determination
		Determination of hesperidin and naringin EN 12148 (1996) - IFU Method No 58 (1991)	HPLC
	HFCS & HIS in apple juice (permitted ingredients)	JAOAC 84, 486 (2001)	CAP GC Method
	Sections 3.2 Quality Criteria and 3.3 Authenticity	Determination of Hydroxymethylfurfural IFU Method No 69 (1996)	HPLC
		Determination of Hydroxymethylfurfural ISO 7466:1986	Spectrometry
		Determination of Isocitric acid-D IFU Method No 54 (1984)	Enzymatic determination
		Determination of Lactic acid- D and L EN 12631 (1999) IFU Method No 53 (1983/1996)	Enzymatic determination
		Determination of L-malic/total malic acid ratio in apple juice AOAC 993.05	Enzymatic determination and HPLC
		Determination of Naringin and neohesperidin in orange juice AOAC 999.05	HPLC
		Determination of pH-value EN 1132 (1994) IFU Method No 11 (1968/1989) ISO 1842: 1991	Potentiometry
		Determination of Phosphorus/Phosphate EN 1136 (1994) IFU Method No 50 (1983)	Photometric determination
		Determination of Proline EN 1141 (1994) IFU Method No 49 (1983)	Photometry

	Quinic, malic & citric acid in cranberry juice cocktail and apple juice (permitted ingredients and additives)	AOAC 986.13	HPLC
	Sections 3.2 Quality Criteria and 3.3 Authenticity	Determination of Recoverable oil AOAC 968.20 - IFU Method No 45b	Distillation and titration Scott method
		Determination of Relative density EN 1131 (1993) IFU Method No 1 (1989) & IFU Method No General sheet (1971)	Pycnometry
		Determination of Relative density IFU Method No 1A	Densitometry
		Determination of Sodium, potassium, calcium, magnesium EN 1134 (1994) IFU Method No 33 (1984)	Atomic Absorption Spectroscopy
		Determination of Sorbitol-D IFU Method No 62 (1995)	Enzymatic determination
		Determination of Stable carbon isotope ratio in the pulp of fruit juices ENV 13070 (1998) Analytica Chimica Acta 340 (1997)	Stable isotope mass spectrometry
		Determination of Stable carbon isotope ratio of sugars from fruit juices ENV 12140 Analytica Chimica Acta.271 (1993)	Stable isotope mass spectrometry
		Determination of Stable hydrogen isotope ratio of water from fruit juices ENV 12142 (1997)	Stable isotope mass spectrometry
		Determination of Stable oxygen isotope ratio in fruit juice water ENV 12141(1997)	Stable isotope mass spectrometry
		Determination of Starch AOAC 925.38 IFU Method No 73	Precipitation

	Sections 3.2 Quality Criteria and 3.3 Authenticity	Determination of Sugar beet derived syrups in frozen concentrated orange juice $\delta^{18}\text{O}$ Measurements in Water AOAC 992.09	Oxygen isotope ratio analysis
		Determination of Titrable acids, total EN 12147 (1995) IFU Method No Method No 3, (1968) ISO 750:1998	Titrimetry
		Determination of Total dry matter EN 12145 (1996) IFU Method No 61 (1991)	Gravimetric determination
		Determination of Total solids AOAC 985.26	Microwave oven drying
		Determination of Vitamin C AOAC 967.22	Microfluorometry
		Determination of Vitamin C CEN/TC275/WG9 N60	DNA

## B. CODEX COMMITTEE ON CEREALS PULSES AND LEGUMES

### Draft Standard for Instant Noodles (elaboration by correspondence)

#### Proposed Method for Determination of Moisture

The 26<sup>th</sup> Session of CCMAS in 2005 considered the methods of analysis proposed by the CCCPL in the Draft Standard for Instant Noodles which had been developed by correspondence. The Committee endorsed Section 9.3 (Extraction of oil from instant noodles), Section 9.4 (Acid Value) in the Draft Standard. However, as to Section 9.2, “moisture determination”, the Committee asked for clarification from the CCCPL on the temperature to be used in the method since there were two different proposals which would use different temperatures, one assuming drying at temperature 130°C and the other at 105°C.

The CCCPL considered the appropriate method and finally agreed to adopt the method originally proposed by Japan which uses the temperature of 105°C. The finally agreed method, as follows, with the clarification on the temperature, as provided by Japan.

#### 9.2 Determination of Moisture

##### A. Apparatus

- (a) Aluminum dish - diameter  $\geq 55\text{mm}$ , height  $\geq 15\text{mm}$ , and with **inverted tight-fitting lid**.
- (b) Air-oven - **with control accuracy  $\pm 1^\circ\text{C}$** .
- (c) Air-tight desiccator - silica gel heated at 150 °C is satisfactory drying agent.

##### B. Preparation of test sample

Remove instant noodles from package, and leave garnishing and seasoning in package. Transfer the noodles to plastic bag to prevent moisture change, and then break these into small fragments with hands or wooden hammer. Select broken noodles in the size range of 2.36 mm to 1.7 mm by using two sieves with 2.36 mm and 1.7 mm openings, and mix well. Use these noodles for test sample. If noodles are too thin to screen with sieves, cut them into 1 to 2 cm lengths, mix well, and use these cut noodles for test sample.

#### C. Determination

##### 1. Fried noodles

In cooled and weighed dish (with **lid**), previously heated to **105°C**, weigh ca 2 g well-mixed test portion to 1mg. Uncover test portion and dry dish, **lid**, and contents 2 h in oven provided with opening for ventilation and maintained at **105°C**. (The 2 h drying period begins when oven temperature is actually 105 °C.) After drying period, cover dish while still in oven, transfer to desiccator, and weigh to 1 mg soon after reaching room temperature. Report loss in weight as moisture (indirect method).

##### 2. Non-fried noodles

For non-fried noodles, follow the directions for fried noodles, but dry test portion for 4 h.

#### D. Calculation

Calculate using the following equations.

$$\text{Moisture (\%)} = \frac{\{\text{g test portion before drying} - \text{g test portion after drying}\}}{\text{g test portion before drying}} \times 100$$

#### **Clarification to the question on the temperature raised by the CCMAS (reply to the CCMAS)**

#### **Comparison of the Japanese method with other proposed methods**

##### ① **AACC Method (44-15A) (Moisture-Air Oven Methods)**

AACC Method (44-15A) requires drying cereal products for 60min at 130°C±1°C after air-drying (until the moisture content reaches less than 10%) when the moisture content is at or above 13%(two-step), and the same drying process at 130°C without air-drying when the moisture content is below 13%. AACC Method also requires drying corn and soybean which contain high fat for 72 hours at 103°C±1 °C.

Figure 1 and Table 1 show moisture contents of fried noodles using air oven method by drying at 130°C and 105°C (which Japan proposed). Fried noodles generally contain heat-labile substances and drying at 130°C may cause reaction of intrinsic substances. Drying at 130°C may also increase their decomposition and/or evaporation resulting in over-estimation of moisture content. For this reason, Japan proposes a method which employs one-step drying for 2 hours at 105°C±1°C. According to Student's t-test, there is a significant difference between the results obtained by drying at 130°C and those at 105°C (significance level P=0.005, P (T<=t) 3.79E-08).

Non-fried noodles have a lot of moisture content compared with fried noodles. Figure 2 shows moisture contents of non-fried noodles by drying at 130°C and 105°C using air oven method. Figure 2 also shows moisture contents at 99°C±1°C using vacuum oven methods (AOAC 925.09). By drying for 8 hours at 99°C using vacuum oven method, the moisture content reached a constant value 11.5%. The almost same moisture content was obtained by drying at 105°C for 4 hours. Although the almost same moisture content was obtained drying at 130°C for 1 hour, the method was lacking for robustness as the non-fried noodles was in process of water evaporation. Considering these results, Japan proposes a method which employs one-step drying for 4 hours at 105°C±1°C. For non-fried noodles containing 13% or more moisture, the method proposed by Japan is easier than AACC Method (44-15A) with sufficiently low RSDr at 0.2% (n=5) as shown Table 3.

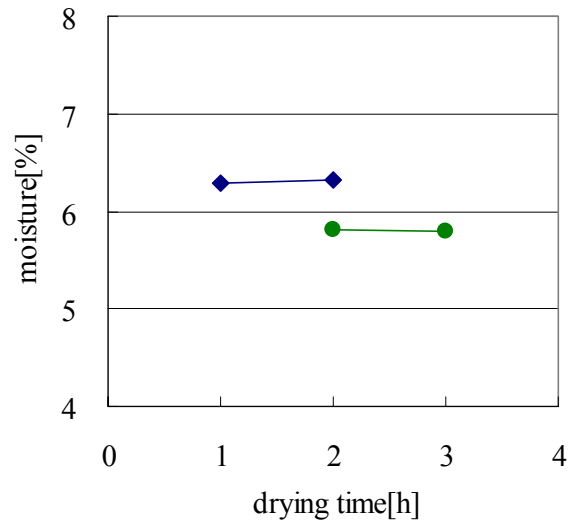


Figure 1 Moisture(%) of fried noodle

◆ 130°C ● 105°C

Table 1 Moisture(%) of fried noodle

drying time(h)	130°C moisture(%)	RSD %	105°C moisture(%)	RSD %
1	6.3	0.7		
2	6.3	0.6	5.8	0.6
3			5.8	0.7



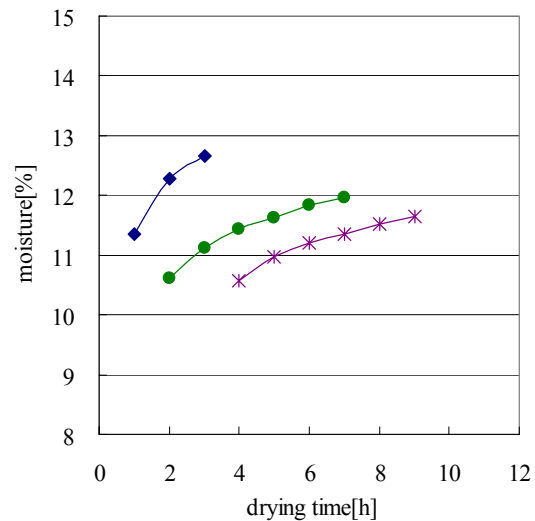


Figure 2 Moisture(%) of non-fried noodle (made from flour)

◆ 130°C ● 105°C \* 99°C(Vacuum Oven method )

Table 2 Moisture(%) of non-fried noodle(made from flour)

drying time(h)	99°C(Vacuum Oven method)		130°C		105°C	
	moisture(%)	RSD%	moisture(%)	RS D%	moisture(%)	RS D%
1	-	-	11.4	0.6	-	-
2	-	-	12.3	0.3	10.6	0.3
3	-	-	12.7	0.3	11.1	0.3
4	10.6	0.7	-	-	11.4	0.2
5	11.0	0.6	-	-	11.6	0.2
6	11.2	0.6	-	-	11.8	0.2
7	11.4	0.5	-	-	12.0	0.2
8	11.5	0.4	-	-	-	-
9	11.6	0.4	-	-	-	-

Table3 Moisture(%) of bean starch vermicelli

99°C(Vacuum Oven)		130°C		105°C	
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drying time(h)	moisture(%)	RSD%	moisture(%)	RSD%	moisture(%)	RSD%
1			14.0	0.4		
2			14.9	0.2	13.4	0.1
3			15.3	0.2	14.0	0.1
4	13.1	0.8			14.3	0.2
5	13.5	0.5			14.5	0.2
6	13.8	0.4			14.7	0.2
7	14.0	0.3				
8	14.1	0.3				
9	14.3	0.2				
10	14.4	0.2				

## ② AOAC Method 925.10

AOAC method 925.10 has been developed for the determination of moisture in flour but not for that in instant noodles. The AOAC method requires drying flour for 60min at 130°C±3°C. Japan proposes a method which employs one-step drying for 2 hours at 105°C±1°C for fried noodles, and a method which employs one-step drying for 4 hours at 105°C±1°C for non-fried noodles. These methods proposed by Japan are more appropriate for the determination of moisture in instant noodles without over-estimation of moisture content described above.

## C. CODEX COMMITTEE ON MILK AND MILK PRODUCTS<sup>3</sup>

### Part A – Methods of analysis for standards currently being elaborated

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
Blend of evaporated skimmed milk and vegetable fat (at Step 8)	Total fat	>=7.5% m/m	IDF 13C:1987   ISO 1737:1999	Gravimetry (Röse-Gottlieb)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	E 22 CCMAS (milk fat in evaporated milk)
	Milk solids-not-fat* (MSNF)	>= 17.5% m/m	IDF 21B:1987   ISO 6731:1989	Calculation from total solids content and fat		I	E 22 CCMAS (evaporated milk)

<sup>3</sup> ALINORM 06/29/11, Appendix XXVI

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
			IDF 13C:1987   ISO 1737:1999	content Gravimetry (Röse-Gottlieb)			
	Milk protein in MSNF*	>=34% m/m in the MSNF	IDF 20-part 1 or 2:2001   ISO 8963-part 1 or 2:2001	Titrimetry (Kjeldahl)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	E 23 CCMAS (evaporated milk)
Reduced fat blend of evaporated skimmed milk and vegetable fat (part of above standard)	Total fat	<=7.5% m/m >= 1% m/m	IDF 13C:1987   ISO 1737: 1999	Gravimetry (Röse-Gottlieb)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	E 22 CCMAS (milk fat in evaporated milk)
	MSNF *	>= 19% m/m	IDF 21B:1987   ISO 6731:1989  IDF 13C:1987   ISO1737:1999	Calculation from total solids and fat contents		I	E 22 CCMAS (evaporated milk)
	Milk protein in MSNF*	>= 34% m/m in the MSNF	IDF 20-1 or 2:2001   ISO 8963-1 or 2:2001	Titrimetry (Kjeldahl)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	E 23 CCMAS (evaporated milk)
<b>Blend of skimmed milk and vegetable fat in powdered form (at Step 8)</b>	Total fat	>=26% m/m	IDF 9C:1987   ISO1736:1999	Gravimetry (Röse-Gottlieb)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	
	Water**	<= 5% m/m	IDF 26:2004	Gravimetry,	The scope of the method does	I	

\* Milk total solids and Milk solids-not-fat content include water of crystallization of lactose

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
			ISO 5537:2004	drying at 87°C	not include this type of product. However, it is expected that the method is applicable.  For WMP and SMP this method was found to produce results that were not significantly different from those produced by IDF26A:1993		
	Milk protein in MSNF*	>= 34% m/m in the MSNF	IDF 20-part 1 or part 2:2001   ISO 8961-part 1 or part 2:2001	Titrimetry (Kjeldahl)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form (part of above standard)	Total fat	<=26% m/m >= 1.5% m/m	IDF 9C:1987   ISO 1736:1999	Gravimetry (Röse-Gottlieb)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	

\* Milk total solids and Milk solids-not-fat content including water of crystallization of lactose

\*\* Water content excluding the crystallized water bound to lactose (in fact to read moisture content)

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
	Water**	<= 5% m/m	IDF 26:2004   ISO 5537:2004	Gravimetry, drying at 87°C	The scope of the method does not include this type of product. However, it is expected that the method is applicable. For WMP and SMP this method was found to produce results that were not significantly different from those produced by IDF26A:1993	I	
	Milk protein in MSNF*	>= 34% m/m in the MSNF	IDF 20-part 1 or part 2:2001   ISO 8961-part 1 or part 2:2001	Titrimetry (Kjeldahl)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	
<b>Blend of sweetened condensed skimmed milk and vegetable fat (at Step 8)</b>	Total fat	>=[7-8%] m/m	IDF 13C:1987   ISO 1737:1999	Gravimetry (Röse-Gottlieb)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	E 22 CCMAS (milk fat in sweetened condensed milk)
	Milk solids-not-fat* (MSNF)	>= 20% m/m	IDF 15B:1991   ISO 6734:1989  IDF 13C:1987   ISO 1737:1999	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)		I	E 23 CCMAS (solids in sweetened condensed milk) E 22 CCMAS (milk fat in sweetened condensed milk)

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\*Milk total solids and Milk solids-not-fat content include water of crystallization of lactose

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
	Milk protein in MSNF*	>=34% m/m in the MSNF	IDF 20-part1 or part 2:2001   ISO 8963-part 1 or part 2:2001	Titrimetry (Kjeldahl)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	E 23 CCMAS (sweetened condensed milk)
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat (part of above standard)	Total fat <= 8% m/m >= 1% m/m	<= 8% m/m >= 1% m/m	IDF 13C:1987   ISO 1737: 1999	Gravimetry (Röse-Gottlieb)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	E 22 CCMAS (milk fat in sweetened condensed milk)
	MSNF * >= 20% m/m	>= 20% m/m	IDF 15B:1991   ISO 6734:1989 IDF 13:1987   ISO1737:1999	Calculation from total solids and fat contents		I	E 23 CCMAS (solids in sweetened condensed milk) E 22 CCMAS (milk fat in sweetened condensed milk)
	Milk protein in MSNF*	>= 34% m/m in the MSNF	IDF 20-part 1 or part 2:2001   ISO 8963-part 1 or part 2:2001	Titrimetry (Kjeldahl)	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	E 23 CCMAS (sweetened condensed milk)
<b>Individual Cheeses</b>	Milkfat in dry matter (FDM)		IDF 5:2004   ISO 1735:2004			I	E CCMAS 22 and 23 as IDF 5B:1986, ISO 1735:1987 AOAC 933.0; reference to be updated
<b>Emmental (C-9) (at Step 8)</b>	Calcium >= 800mg/100g		IDF 154: 1992   ISO 8070:1987	Flame atomic absorption	The scope of the method does not include this type of product. However, when using either a dry ashing or an acid digest preparation it is expected to work for cheese as	III	

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
					well. (Note: experience with the dry ash method suggests there may sometimes be some loss of minerals). <sup>4</sup>		
<b>Cottage cheese (C-16) (at Step 5/8)</b>	Milkfat	>= 0% m/m Reference level 4-5% m/m	IDF 5:2004   ISO 1735:2004  IDF 124-3:2005   ISO 8262-3:2005	Gravimetry (Schmid-Bondzinski-Ratzlaff)  Gravimetry (Weibull-Berntrop)	Use IDF 5 2004 / ISO 1735:2004 except when the lactose content >5% of non fat solids in which case IDF 124-3:2005 /ISO 8262-3:2005 should be used. The scope of the methods do not include this type of product. However, it is expected that the methods are applicable.	I	
	Fat-free dry matter	>=18% m/m	IDF 4:2004   ISO 5534:2004	Gravimetry, drying at 102°C	IDF 4:2004   ISO 5534:2004 measures DM when used with IDF 5:2004   ISO 5534:2004 (or IDF 124-3:2005 / ISO 8262-3:2005 as appropriate) the FFDM may be obtained through calculation.	I	
<b>Cream cheese (C-31) (at Step 5/8)</b>	Moisture on fat free basis	67% Reference level not specified	IDF 4:2004   ISO 5534:2004 and IDF 5:2004   ISO 1735:2004	Calculation from fat content and moisture content	The scope of the methods do not include this type of product. However, it is expected that the methods are applicable.	I	E 23 CCMAS for Cheese (with previous reference)
	Dry matter	22% restricted by the MMFB reference level not specified	IDF 4:2004   ISO 5534:2004	Gravimetry Drying at 102°C	The scope of the methods do not include this type of product. However, it is expected that the methods are applicable.	I	E 27 CCMAS for individual cheeses
<b>Mozzarella (at Step 5/8)</b>	Milkfat in dry matter –	Minimum 20% reference level	IDF 5:2004   ISO 1735:2004		The scope of the method does not include this type of	I	

<sup>4</sup> IDF 154 is an old and provisional standard and will be deleted when IDF 119/ISO 8070 is published.

IDF 119/ISO 8070 has been submitted to an interlab validation study extending the field of application to Ca and Mg and to “other milk products” including cheese in 2004. Precision figures were satisfactory.

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
	with high moisture	40%-50%			product. However, it is expected that the method is applicable.		
	Milkfat in dry matter – with low moisture	Minimum reference 18% level 40%-50%	IDF 5:2004   ISO 1735:2004		The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	
<b>Whey cheeses (at Step 8) including Whey cheeses by concentration</b>	Total fat		IDF 59A:1986 ISO 1854:1999	Gravimetry (Röse Gottlieb)		I	CCMAS 22: same IDF method; ISO 1854: 1987; AOAC 974.09 as milk fat (dry basis)
Whey cheeses by coagulation (part of the above standard)	Total fat		IDF 5:2004   ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff)		I	
Whey cheeses by concentration (part of the above standard)	Dry matter (total solids)		IDF 58:2004 ISO 2920:2004	Gravimetry, drying at 88 °C		I	E 23 CCMAS with previous reference
Whey cheeses by coagulation (part of the above standard)	Dry matter (total solids)		IDF 4:2004 ISO 5534:2004	Gravimetry, Drying at 102°C	The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	
Whey cheese (part of above standard)	Fat on the dry basis	Minimum 10% and < 33%	IDF 59 A:1986   ISO 1854:1999 and IDF 58:2004   ISO 2920:2004	Calculation from fat content and dry matter content	Applicable only to whey cheese made by concentration	I	
Creamed whey cheese (part of above standard)	Fat on the dry basis	Minimum 33%	IDF 59 A: 1986   ISO 1854: 1999 and IDF 58:2004   ISO 2920:2004	Calculation from fat content and dry matter content	Applicable only to whey cheese made by concentration	I	
Skimmed whey	Fat on the	Less than 10%	IDF 59 A:1986	Calculation from	Applicable only to whey	I	



Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
cheese (part of above standard)	dry basis		ISO 1854:1999 and IDF 58:2004   ISO 2920:2004	fat content and dry matter content	cheese made by concentration		
<b>Dairy fat spreads (at Step 5/8)</b>	Milk fat  (three-quarter fat butter)  (half-fat butter)	< 80% (m/m) > = 10% (m/m)  < = 62% (m/m) > = 60% (m/m)  < = 41% (m/m) > = 39% (m/m)	IDF 194:2003   ISO 17189:2003	Gravimetry Direct determination of fat using solvent extraction		I	
	Vegetable fat		IDF 54:1970   ISO 3594 (confirmed 1996) IDF 32:1965   ISO 3595:1976 (confirmed 1996)	Gas liquid chromatography  Phytosterol acetate test	These methods are likely to be superseded by IDF 202   ISO 17678 "Milkfat - Detection of foreign fats by GLC analysis of triglycerides"Reference method)", which IDF/ISO expect to publish by 2007	II  III	

**Part B - Updated list of methods of analysis for Codex Standards for milk products**

Milk products	Iron	IDF Standard 103A:1986 ISO 6732:1985 (confirmed 1995)	Photometry (bathophenanthroline)	IV
Milk products (products not completely soluble in ammonia)	Milkfat	<del>IDF Standard 126A:1988</del> <del>ISO 8262-3:1987</del> <b>IDF 124-3 / ISO 8262-3:2005</b>	Gravimetry (Weibull-Berntrop)	I
Butter	Milk solids-not-fat	<del>IDF Standard 80:1977</del> <del>ISO 3727:1977</del> <b>IDF 80-2 / ISO 3727-2:2002</b>	Gravimetry	I
Butter	Milkfat	IDF Standard 80:1977 ISO 3727:1977 <b>IDF 80-3 / ISO 3727-3:2003</b>	Gravimetry	I
Butter	Salt	IDF Standard 12B: 1988 ISO 1738:1997 <b>IDF 12 / ISO 1738:2004</b>	Titrimetry (Mohr: determination of chloride, expressed as sodium chloride)	II
Butter	Salt	IDF Standard 179:1997 <b>IDF 179 / ISO 15648:2004</b>	Potentiometry (determination of chloride, expressed as sodium chloride)	III
Butter	Vegetable fat	IDF Standard 54:1970 ISO 3594:1976 (confirmed 1996)	Gas liquid chromatography	II
Butter	Vegetable fat	IDF Standard 32:1965 ISO 3595:1976 (confirmed 1996)	Phytosteryl acetate test	III
Butter	Water	IDF Standard 80:1977 ISO 3727:1977 <b>IDF 80 / ISO 3727:2001</b>	Gravimetry	I
Cheese	Citric acid	<del>IDF Standard 34C:1992</del> <b>IDF RM 34 / ISO TS 34:2005</b>	Enzymic method	II
<del>Cheese</del>	<del>Citric acid</del>	<del>ISO 2963:1997</del>	<del>Photometry</del>	<del>III</del>
Cheese	Milkfat	IDF Standard 5B: 1986 ISO 1735:1987 <b>IDF 5 / ISO 1735:2004</b>	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Cheese (and cheese rind)	Natamycin	IDF Standard 140A:1992 ISO 9223:1991 (confirmed 1996)	Molecular absorption spectrophotometry & HPLC after extraction	II

Cheeses in brine	Milkfat in dry matter	IDF Standard 5B:1986 ISO 1735:1987 <b>IDF 5 / ISO 1735:2004</b>	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Cream	Milkfat	IDF Standard 16C:1987 ISO 2450:1985	Gravimetry (Röse-Gottlieb)	I
Cream	Solids	IDF Standard 21B:1987 ISO 6731:1989	Gravimetry (drying at 102°C)	I
Edible casein products	Acids, free	IDF Standard 91:1979 (confirmed 1986) ISO 5547:1978 (confirmed 1993)	Titrimetry (aqueous extract)	IV
Edible casein products	Ash (including P <sub>2</sub> O <sub>5</sub> )	IDF Standard 90:1979 (confirmed 1986) ISO 5545:1978	Furnace, 825°C	IV
Edible casein products	Copper	IDF Standard 76A:1980 ISO 5738:1980 (confirmed 1995) <b>IDF 76 / ISO 5738:2004</b>	Colorimetry (diethyldiethiocarbamate)	III
Edible casein products	Lactose	IDF Standard 106:1982 ISO 5548:1980 (confirmed 1996) <b>IDF 106 / ISO 5548:2004</b>	Photometry (phenol and H <sub>2</sub> SO <sub>4</sub> )	IV
Edible casein products	Lead	IDF Standard 133A:1992	Spectrophotometry (1,5-diphenylthiocarbazone)	III
Edible casein products	Milkfat	IDF Standard 127A:1988 ISO 5543:1986 (confirmed 1996)	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Edible casein products	Moisture	IDF Standard 78C:1990 ISO 5550:1978 <b>IDF 78 / ISO 5550:2005</b>	Gravimetry (drying at 102°C)	I
Edible casein products	pH	IDF Standard 115A:1989 ISO 5546:1979 (confirmed 1996)	Electrometry	IV
Edible casein products	Protein (total N x 6.38 in dry matter)	IDF Standard 92:1979 (confirmed 1986) ISO 5549:1978 (confirmed 1993)	Titrimetry, Kjeldahl digestion	IV
Edible casein products	Sediment (scorched particles)	IDF Standard 107A:1995 ISO 5739:1983 <b>IDF 107 / ISO 5739:2002</b>	Visual comparison with standard disks, after filtration	IV
Evaporated milks	Milkfat	IDF Standard 13C: 1987 ISO 1737:1985	Gravimetry (Röse-Gottlieb)	I
Evaporated milks	Solids	IDF Standard 21B:1987 ISO 6731:1989	Gravimetry (drying at 102°C)	I

Milk powders and cream powders	Milkfat	IDF Standard 9C: 1987 ISO 1736:1985	Gravimetry (Röse-Gottlieb)	I
Milk powders and cream powders	Protein (in milk solids-not-fat)	IDF Standard 20B:1993 <b>IDF 20-1 / ISO 8968-1:2001</b>	Titrimetry, Kjeldahl digestion	I
Milk powders and cream powders	Scorched particles	IDF Standard 107A:1995 ISO 5739:1983 <b>IDF 107 / ISO 5739:2002</b>	Visual comparison with standard disks, after filtration	IV
Milk powders and cream powders	Solubility	IDF Standard 129A:1988 ISO 8156:1987 <b>IDF 129 / ISO 8156:2005</b>	Centrifugation	I
Milk powders and cream powders	Acidity, titratable	IDF Standard 86:1981 <b>ISO 6091:1980</b>	Titrimetry, titration to pH 8.4	I
Milk powders and cream powders	Water	<del>IDF Standard 26A:1993</del> <b>IDF 26 / ISO 5537:2004<sup>5</sup></b>	Gravimetry (drying at 102°C)	IV
Milkfat products	Antioxidants (phenolic)	IDF Standard 165:1993	Reversed phase gradient liquid chromatography	II
Milkfat products	Fatty acids, free (expressed as oleic acid)	IDF Standard 6B:1989 ISO 1740:1991 (confirmed 1996) <b>IDF 6 / ISO 1740:2004</b>	Titrimetry	I
Milkfat products	Milkfat	IDF Standard 24:1964	Gravimetry (calculation from solids-not-fat and water content)	IV
Milkfat products	Vegetable fat (sterols)	IDF Standard 54:1979 ISO 3594:1976 (confirmed 1996)	Gas liquid chromatography	II
Milkfat products	Vegetable fat	IDF Standard 32:1965 ISO 3595:1976 (confirmed 1996)	Phytosteryl acetate test	III
Milkfat products	Water	<del>IDF Standard 23A:1988</del> <b>IDF 23 / ISO 5536:2002</b>	Titrimetry (Karl Fischer)	II
Processed cheese products	Citric acid	<del>IDF Standard 34C:1992</del> <b>IDF RM 34 / ISO TS 2963:2005</b>	Enzymic method	II
Processed cheese products	Citric acid	ISO 2963:1997	Photometry	III
Processed cheese products	Milkfat	IDF Standard 5B:1986 ISO 1735:1987 <b>IDF 5 / ISO 1735:2004</b>	Gravimetry (Schmid- Bondzynski-Ratzlaff)	I
<del>Processed cheese products</del>	<del>Phosphate, added (expressed as phosphorus)</del>	<del>IDF Standard 51B:1991</del>	<del>Calculation</del>	<del>IV</del>

<sup>5</sup> the replacing method has only been validated for milk powders, not for cream powders

Processed cheese products	Phosphorus	IDF Standard 33C: 1987 ISO 2962:1984 (confirmed 1994)	Spectrophotometry (molybdate-ascorbic acid)	II
Processed cheese products	Salt	IDF Standard 88A:1979 ISO 5943:1988 (confirmed 1996) <b>IDF 88 / ISO 5943:2004</b>	Potentionmetry (determination of chloride, expressed as sodium chloride)	II
Sweetened condensed milk	Milkfat	IDF Standard 13C: 1987 ISO 1737:1985	Gravimetry (Röse-Gottlieb)	I
Whey cheese	Dry matter	IDF Standard 58:1970 (confirmed 1993) ISO 2920:1974 (confirmed 1996) <b>IDF 58 / ISO 2920:2004</b>	Gravimetry (drying at 88±2°C)	I
Whey cheese	Milkfat (in dry matter)	IDF Standard 59A:1986 ISO 1854:1987	Gravimetry (Röse-Gottlieb)	I
Whey powders	Ash	IDF Standard 90:1979 (confirmed 1986) ISO 5545:1978	Furnace, 825°C	IV
Whey powders	Copper	IDF Standard 76A:1980 ISO 5738:1980 (confirmed 1995) <b>IDF 76 / ISO 5738:2004</b>	Photometry (diethyldiethiocarbamate)	III
Whey powders	Milkfat	IDF Standard 9C:1987 ISO 1736:1985	Gravimetry (Röse-Gottlieb)	I
Whey powders	Moisture, "Free"	IDF Standard 58:1970 (confirmed 1993) ISO 2920:1974 (confirmed 1996) <b>IDF 58 / ISO 2920:2004</b>	Gravimetry (drying at 88±2°C)	IV
Whey powders	Protein (total N x 6.38)	IDF Standard 92:1979 (confirmed 1986) ISO 5549:1978 (confirmed 1978)	Titrimetry, Kjeldahl digestion	IV
Yoghurt products	<i>Lactobacillus bulgaricus</i> & <i>Streptococcus thermophilus</i>	<del>IDF Standard 117A:1988</del> <b>IDF 117 / ISO 7889:2003</b>	Colony count at 37°C	
Yoghurt products	<i>Lactobacillus bulgaricus</i> & <i>Streptococcus thermophilus</i>	<del>IDF Standard 146:1991</del> <b>IDF 146 / ISO 9232:2003</b>	Test for identification	
Yoghurt products	Solids, Total	<del>IDF Standard 151:1991</del> <b>IDF 151 / ISO 13580:2005</b>	Gravimetry (drying at 102°C)	I

#### D. CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES (GLUTEN FREE FOODS)

##### Draft Revised Standard for Gluten-Free Foods (at Step 6)

The 26<sup>th</sup> Session of the CCMAS endorsed temporarily the R5 ELISA method for the determination of gluten as a Type I method.

COMMODITY	PROVISION	METHOD	PRINCIPLE	TYPE	STATUS
Gluten-free foods	Gluten	Enzyme-Linked Immunoassay R5 Mendez (ELISA) Method	Immunoassay	I	TE

References: *Eur J Gastroenterol Hepatol* 2003; 15: 465-474

The CCMAS is invited to consider the status of the R5 Mendez (ELISA) Method. Updated information on this method is distributed separately for information.

## PART II. SAMPLING

### COMMITTEE ON MILK AND MILK PRODUCTS : Updated list of methods of sampling for Codex Standards for milk products

Milk products	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk
Milk products	Sampling	IDF Standard 113A:1990 ISO 5538:1987 (confirmed 1992) <b>IDF 113 / ISO 5538:2004</b>	Inspection by attributes
Milk products	Sampling	IDF Standard 136A:1992 ISO 8197:1988 (confirmed 1993)	Inspection by variables
Butter	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk
Cheese	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk
Cheeses in brine	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk
Edible casein products	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk
Evaporated milks	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk

Milk powders and cream powders	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk
Milkfat products	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk
Sweetened condensed milks	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk
Whey cheese	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk
Whey powders	Sampling	IDF Standard 113A:1990 ISO 5538:1987 (confirmed 1992) <b>IDF 113 / ISO 5538:2004</b>	Inspection by attributes
Whey powders	Sampling	IDF Standard 50C:1995 ISO 707:1997 <b>IDF 50 / ISO 707:2005</b>	General Instructions for obtaining a sample from a bulk