codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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Agenda Item 8(a) **CX/MMP 06/7/13**
January 2006

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON MILK AND MILK PRODUCTS

Seventh Session

Queenstown, New Zealand, 27 March – 1 April 2006

METHODS OF ANALYSIS AND SAMPLING FOR MILK PRODUCTS

This document is divided in three parts:

Part I

Report by IDF/ISO International Working Group on Methods of Analysis and Sampling on Review of the current methods of analysis and sampling for milk and milk products and recommendations on updates to the list of methods

1 INTRODUCTION

During its 6th Session the Codex Committee on Milk and Milk Products requested the IDF/ISO/AOAC Working Group on Methods of Analysis and Sampling to review the current methods of analysis and sampling for milk and milk products and provide recommendations on updates to the list of methods. The Committee also agreed that the report of the IDF/ISO/AOAC Working Group covering this item would be circulated and considered at its next Session.[1]

2 REPORT OF THE IDF/ISO INTERNATIONAL WORKING GROUP ON METHODS OF ANALYSIS AND SAMPLING

Following the decision on discontinuation of the former IDF/ISO/AOAC Tripartite collaboration in September 2005 the IDF/ISO International Working Group on Methods of Analysis and Sampling has reviewed the provisions relating to current ISO/IDF methods of analysis and sampling for milk and milk products as contained in CODEX STAN 234-1999 - Recommended Methods of Analysis and Sampling, Part I, Methods of Analysis and Sampling by Alphabetical Order of Commodity Categories and Names, pages 18-24.

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1 Codex ALINORM 04/27/11, para. 135 and 136

The recommendations of the IDF/ISO International Working Group on Methods of Analysis and Sampling for Milk and Milk Products are appended to this report. Proposed changes are highlighted in strikethrough text and up-dated references are highlighted in ***italics and bold***.

It is to be noted that the review of the IDF/ISO International Working Group did not include the methods of NMKL and AOAC International that are referenced in CODEX STAN 234-1999 - Recommended Methods of Analysis and Sampling, Part I, Methods of Analysis and Sampling by Alphabetical Order of Commodity Categories and Names, pages 18-24.

Milk and Milk Products				
Milk products	Iron	IDF Standard 103A:1986 ISO 6732:1985 (confirmed 1995)	Photometry (bathophenanthroline)	IV
Milk products	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-
Milk products	Sampling	~~IDF Standard 113A:1990~~ ~~ISO 5538:1987 (confirmed 1992)~~ ***IDF 113 | ISO 5538:2004***	Inspection by attributes	-
Milk products	Sampling	IDF Standard 136A:1992 ISO 8197:1988 (confirmed 1993)	Inspection by variables	-
Milk products (products not completely soluble in ammonia)	Milkfat	~~IDF Standard 126A:1988~~ ~~ISO 8262-3:1987~~ ***IDF 124-3 | ISO 8262-3:2005***	Gravimetry (Weibull-Berntrop)	I
Butter	Milk solids-not-fat	~~IDF Standard 80:1977~~ ~~ISO 3727:1977~~ ***IDF 80-2 | ISO 3727-2:2002***	Gravimetry	I
Butter	Milkfat	~~IDF Standard 80:1977~~ ~~ISO 3727:1977~~ ***IDF 80-3 | ISO 3727-3:2003***	Gravimetry	I
Butter	Salt	~~IDF Standard 12B: 1988~~ ~~ISO 1738:1997~~ ***IDF 12 | ISO 1738:2004***	Titrimetry (Mohr: determination of chloride, expressed as sodium chloride)	II
Butter	Salt	~~IDF Standard 179:1997~~ ***IDF 179 | ISO 15648:2004***	Potentiometry (determination of chloride, expressed as sodium chloride)	III
Butter	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-
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~~ ***IDF 80 | ISO 3727:2001***	Gravimetry	I
Cheese	Citric acid	~~IDF Standard 34C:1992~~ ***IDF RM 34 | ISO TS 34:2005***	Enzymic method	II
~~Cheese~~	~~Citric acid~~	~~ISO 2963:1997~~	~~Photometry~~	~~III~~
Cheese	Milkfat	~~IDF Standard 5B: 1986~~ ~~ISO 1735:1987~~ ***IDF 5 | ISO 1735:2004***	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Cheese	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-

Milk and Milk Products				
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~~ ***IDF 5 | ISO 1735:2004***	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Cheeses in brine	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-
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_2O_5)	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)~~ ***IDF 76 | ISO 5738:2004***	Colorimetry (diethyldiethiocarbamate)	III
Edible casein products	Lactose	~~IDF Standard 106:1982~~ ~~ISO 5548:1980 (confirmed 1996)~~ ***IDF 106 | ISO 5548:2004***	Photometry (phenol and H_2SO_4)	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~~ ***IDF 78 | ISO 5550:2005***	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 (confirmed1986) ISO 5549:1978 (confirmed 1993)	Titrimetry, Kjeldahl digestion	IV
Edible casein products	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-
Edible casein products	Sediment (scorched particles)	~~IDF Standard 107A:1995~~ ~~ISO 5739:1983~~ ***IDF 107 | ISO 5739:2002***	Visual comparison with standard disks, after filtration	IV
Evaporated milks	Milkfat	IDF Standard 13C: 1987 ISO 1737:1985	Gravimetry (Röse-Gottlieb)	I
Evaporated milks	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-
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~~ ***IDF 20-1 | ISO 8968-1:2001***	Titrimetry, Kjeldahl digestion	I

Milk and Milk Products				
Milk powders and cream powders	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-
Milk powders and cream powders	Scorched particles	~~IDF Standard 107A:1995~~ ~~ISO 5739:1983~~ ***IDF 107 | ISO 5739:2002***	Visual comparison with standard disks, after filtration	IV
Milk powders and cream powders	Solubility	~~IDF Standard 129A:1988~~ ~~ISO 8156:1987~~ ***IDF 129 | ISO 8156:2005***	Centrifugation	I
Milk powders and cream powders	Acidity, titratable	IDF Standard 86:1981 ***ISO 6091:1980***	Titrimetry, titration to pH 8.4	I
Milk powders and cream powders	Water	~~IDF Standard 26A:1993~~ ***IDF 26 | ISO 5537:2004***[2]	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)~~ ***IDF 6 | ISO 1740:2004***	Titrimetry	I
Milkfat products	Milkfat	IDF Standard 24:1964	Gravimetry (calculation from solids-not-fat and water content)	IV
Milkfat products	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-
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	~~IDF Standard 23A:1988~~ ***IDF 23 | ISO 5536:2002***	Titrimetry (Karl Fischer)	II
Processed cheese products	Citric acid	~~IDF Standard 34C:1992~~ ***IDF RM 34 | ISO TS 2963:2005***	Enzymic method	II
Processed cheese products	Citric acid	ISO 2963:1997	Photometry	III
Processed cheese products	Milkfat	~~IDF Standard 5B:1986~~ ~~ISO 1735:1987~~ ***IDF 5 | ISO 1735:2004***	Gravimetry (Schmid-Bondzynski- Ratzlaff)	I
~~Processed cheese products~~	~~Phosphate, added (expressed as phosphorus)~~	~~IDF Standard 51B:1991~~	~~Calculation~~	~~IV~~
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)~~ ***IDF 88 | ISO 5943:2004***	Potentionmetry (determination of chloride, expressed as sodium chloride)	II
Sweetened condensed milk	Milkfat	IDF Standard 13C: 1987 ISO 1737:1985	Gravimetry (Röse-Gottlieb)	I
Sweetened condensed milks	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-

[2] the replacing method has only been validated for milk powders, not for cream powders

Milk and Milk Products				
Whey cheese	Dry matter	~~IDF Standard 58:1970 (confirmed 1993)~~ ~~ISO 2920:1974 (confirmed 1996)~~ ***IDF 58 | ISO 2920:2004***	Gravimetry (drying at 88±2°C)	IV
Whey cheese	Milkfat (in dry matter)	IDF Standard 59A:1986 ISO 1854:1987	Gravimetry (Röse-Gottlieb)	I
Whey cheese	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-
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)~~ ***IDF 76 | ISO 5738:2004***	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)~~ ***IDF 58 | ISO 2920:2004***	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
Whey powders	Sampling	~~IDF Standard 113A:1990~~ ~~ISO 5538:1987 (confirmed 1992)~~ ***IDF 113 | ISO 5538:2004***	Inspection by attributes	-
Whey powders	Sampling	~~IDF Standard 50C:1995~~ ~~ISO 707:1997~~ ***IDF 50 | ISO 707:2005***	General Instructions for obtaining a sample from a bulk	-
Yoghurt products	*Lactobacillus bulgaricus* & *Streptococcus thermophilus*	~~IDF Standard 117A:1988~~ ***IDF 117 | ISO 7889:2003***	Colony count at 37°C	
Yoghurt products	*Lactobacillus bulgaricus* & *Streptococcus thermophilus*	~~IDF Standard 146:1991~~ ***IDF 146 | ISO 9232:2003***	Test for identification	
Yoghurt products	Solids, Total	~~IDF Standard 151:1991~~ ***IDF 151 | ISO 13580:2005***	Gravimetry (drying at 102°C)	I

Part II

Report by IDF/ISO International Working Group on Methods of Analysis and Sampling on Methods of analysis required in the Draft Codex Standards for Milk and Milk Products currently being elaborated by CCMMP

1 INTRODUCTION

During its 6th Session the Codex Committee on Milk and Milk Products requested the IDF/ISO/AOAC Working Group on Methods of Analysis and Sampling to prepare a list of methods of analysis required in the Draft Codex Standards for milk and milk products currently being elaborated by CCMMP on the basis of the information received. The Committee also agreed that the report of the IDF/ISO/AOAC Working Group covering this item would be circulated and considered at its next Session.[3]

2 REPORT OF THE IDF/ISO INTERNATIONAL WORKING GROUP ON METHODS OF ANALYSIS AND SAMPLING

Following the decision on discontinuation of the former IDF/ISO/AOAC Tripartite collaboration in September 2005 the IDF/ISO International Working Group on Methods of Analysis and Sampling has prepared a list of methods of analysis required in the Draft Codex Standards for milk and milk products currently being elaborated by CCMMP on the basis of the information received. The list is appended to this report. It also includes comments of the experts of the IDF/ISO International Working Group on Methods of Analysis and Sampling.

[3] Codex ALINORM 04/27/11, para. 135 and 136

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
Blend of evaporated skimmed milk and vegetable fat (at Step 5)	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 solids-not-fat* (MSNF)	>= 17.5% m/m	IDF 21B:1987| ISO 6731:1989 IDF 13C:1987 | ISO 1737:1999	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)		I	E 22 CCMAS
	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
Blend of evaporated partly 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
	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 (for evaporated milk)
	Milk protein in MSNF*	>= 34% m/m in the MSNF	IDF 20-1:2001| ISO 8963-1:2001	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
Blend of skimmed milk and vegetable fat in powdered form (at Step 5)	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	I	

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

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
					is applicable.		
	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-pat 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	
Blend of partly 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	

* Water excluding the crystallized water bound to lactose (in fact to read moisture content)
* 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	
Blend of sweetened condensed skimmed milk and vegetable fat **(at Step 5)**	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 solids-not-fat[*] (MSNF)	>= 20% m/m	IDF 21B:1987| ISO 6731:1989 IDF 13C:1987| ISO 1737:1999	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)		I	E 22 CCMAS
	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

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

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
Blend of sweetened condensed partly 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
	MSNF * >= 20% m/m	>= 20% m/m	IDF 21B:1987| ISO 6731:1989 IDF 13:1987 | ISO1737:1999	Calculation from total solids and fat contents		I	E 22 CCMAS (for evaporated 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
Cheddar (C-1) (applies, *mutatis mutandis*, to Danbo (C-3), Edam (C-4), Gouda (C-5), Havarti (C-6), Samsø (C-7), Emmental (C-9), Tilsiter (C-11), St Paulin (C-13), Provolone (C-15), Coulommiers (C-18), **(Cheddar and Danbo at Step 5, all others at Step 4)**	Milkfat in dry matter (FDM)	>= 22% m/m Reference level [48-60%] m/m The above is the requirement for Cheddar only. The other named cheeses have different and in a number of cases more complicated, requirements in this regard.	IDF 5:2004| ISO 1735:2004	Gravimetry (Schmid-Bondzinski-Ratzlaff)	ISO 5:2004/ISO 1735:2004 measures fat and when DM is measured using IDF 4:2004/ISO 5534:2004 FDM is then calculated using the values obtained from the above.	I	E 22 CCMAS

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
	Dry matter according to FDM	FDM 22-30% m/m >=49% FDM 30-40% m/m >=53% FDM 40-48% m/m >=57% FDM 48-60% m/m >=61% FDM >60% m/m >=66%	IDF 4:2004| ISO 5534:2004	Gravimetry, drying at 102°C		I	E 23 CCMAS
Emmental (C-9) only (cheese ready for sale) (at Step 4)	Propionic acid >=150mg/100g				No specific IDF/ISO method for measurement of propionic acid in cheese exists		
Emmental (C-9) (at Step 4)	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 well. (Note: experience with the dry ash method suggests there may sometimes be some loss of minerals). 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.	III	

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
Cottage cheese (C-16) (at Step 4)	Milkfat	>= 0% m/m Reference level 4-5% m/m	IDF 124-3:2005 | ISO 8262-3:2005 IDF 5:2004 | ISO 1735:2004	Gravimetry (Weibull-Berntrop)	Use IDF 5 except when the lactose content >5% of non fat solids in which case IDF 124-3:2005 should be used. The scope of the methods does not include this type of product. However, it is expected that the methods are applicable.	I	***? Item is in progression for publication as joint IS IDF 124-3 | ISO 8262-3 in 2005***
	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 as appropriate) the FFDM may be obtained through calculation.	I	E 23 CCMAS
Coulommiers (C-18) (at Step 4)	Milkfat in dry matter	40% level 40% to 50% = >40% but < 50% Reference level 42% = > 50% but < 60% Reference level 46% =>60% Reference level 52%	IDF 5:2004| ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff	ISO 5:2004/ISO 1735:2004 measures fat and when DM is measured using IDF 4:2004/ISO 5534:2004 FDM is then calculated using the values obtained from the above.	I	22 CCMAS

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
	Dry matter	40% reference level 40% to 50%	IDF 4:2004| ISO 5534:2004	Gravimetry Drying at 102°C		I	23 CCMAS
Cream cheese (C-31) (at Step 4)	Milk fat in dry matter	25% Reference Level 60-70%	IDF 5:2004| ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff	ISO 5:2004/ISO 1735:2004 measures fat and when DM is measured using IDF 4:2004/ISO 5534:2004 FDM is then calculated using the values obtained from the above.	I	E 22 CCMAS
	Moisture on fat free basis	67% Reference level not specified	IDF 4:2004| ISO 5534:2004 IDF 5:2004 | ISO 1735:2004	Calculation from fat content and moisture content Gravimetry (Schmid-Bondzynski-Ratzlaff	The scope of the methods does not include this type of product. However, it is expected that the methods are applicable.		E 22 CCMAS E 23 CCMAS
	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 does not include this type of product. However, it is expected that the methods are applicable.	I	E 23 CCMAS
Camembert (C-33) (at Step 4)	Milkfat in dry matter	Minimum content 30% Reference level 45-55%	IDF 5:2004 | ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff		I	E 23 CCMAS
	Dry matter	=>30% but < 40% reference level 38% =>30% but <45% reference level 41% =>45 but <55% reference level 43%	IDF 4:2004| ISO 5534:2004	Gravimetry Drying at 102°C		I	E 22 CCMAS

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
		=> 55% reference level 48%					
Brie (C-34) (at Step 4)	Milkfat in dry matter	Minimum content 40% Reference level 45-55%	IDF 5:2004 | ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff		I	
	Dry matter	=>40% but < 45% reference level 42% =>45% but <55% reference level 43% =>55 but <60% reference level 48% => 60% reference level 51%	IDF 4:2004| ISO 5534:2004	Gravimetry Drying at 102°C		I	E 22 CCMAS
Mozzarella (at Step 4)	Milkfat in dry matter – with high moisture	Minimum 20% reference 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	
	Milkfat in dry matter – with low moisture	Minimum 18% reference 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	
	Dry matter	=>18% but <30% reference level with low moisture 34% =>20% but <30% reverence level with high moisture 24%	IDF 4:2004| ISO 5534:2004	Gravimetry Drying at 102°C		I	E 22 CCMAS

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
		=>30% but <40% reference level with low moisture 39% reference level with high level moisture 26% =>40% but < 50% reference level with low moisture 42% reference level with high moisture 29% => 45% but <50% reference level with low moisture 45% reference level with high moisture 31% =>50% but < 60% reference level with low moisture 47% reference level with high moisture 34% =>60% but <85% reference level with low moisture 53% reference level with high moisture 38%					

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
Provolone (C-15) ***Mozzarell***a	Fibrous texture with long stranded parallel-oriented protein fibres	Pasta filata processing			No IDF/ISO method available		
Whey cheeses (at Step 5)	Total fat		IDF 59A:1986| ISO 1854:1999	Gravimetry (Röse Gottlieb)		I	E 22 CCMAS
Whey cheeses by concentration (part of the above standard)	Total fat		IDF 59A:1986| ISO 1854:1999	Gravimetry (Röse Gottlieb)			
Whey cheeses by coagulation (part of the above standard)	Total fat		IDF 5:2004| ISO 1735:2004				
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
Whey cheeses by coagulation (part of the above standard)	Dry matter (total solids)		IDF 4:2004|ISO 5534:2004		The scope of the method does not include this type of product. However, it is expected that the method is applicable.	I	
	Ratio whey protein to casein to exceed that of milk				No IDF/ISO method available		
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		
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 |	Calculation from fat content and dry matter content	Applicable only to whey cheese made by concentration		

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
			ISO 2920:2004				
Skimmed whey cheese (part of above standard)	Fat on the dry basis	Less than 10%	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		
Processed cheese (Step 2, current draft of IDF DG leader)	Milk fat in dry matter	Maximum 75%	IDF 5:2004 | ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff)	IDF 5:2004/|ISO 1735:2004 [or IDF 124-1/3:2005/ISO 8262-1/3:2005] measures fat. When DM is measured by IDF 4:2004/ISO 5534:2004 FDM may be obtained by calculation	I	E 22 CCMAS
	Dry matter	=>30% but <50% reference level 34% =>50% but <75% reference level 50% <30% reference level 29%	IDF 4:2004| ISO 5534:2004	Gravimetry Drying at 102°C	Applicability of this method has not been checked for this type of product.		E 23 CCMAS
Processed cheese qualified as "spreadable" (part of above standard)	Milk fat in dry matter	Maximum 75%	IDF 5:2004 | ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff)	Use IDF 5 except when the lactose content >5% of non fat solids in which case IDF 124-3:2005 should be used.	I	?
	Dry matter	=>30% but <50% reference level 30% =>50% but <75% reference level 40%	IDF 4:2004| ISO 5534:2004	Gravimetry Drying at 102°C	Applicability of this method has not been checked for this type of product.	I	E 23 CCMAS

Commodity	Provision	Requirement	Method	Principle	Comments	Type	Status
		<30% reference level 25%					
Protein		Declare milk protein content where consumers would be misled if omitted	IDF/RM 25: 2005 | ISO/TS 17837:2005	Kjeldahl Method	This method measures total protein and does not specifically measure milk protein	I	
Dairy spreads (at Step 3)	Milk fat<) 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	Direct determination of fat using solvent extraction		I	
Fermented Milk Drinks Provisions (at Step 3)	Dairy ingredients	=>40%			No IDF/ISO method available		

Part III

Report by IDF/ISO International Working Group on Methods of Analysis and Sampling on Recommendations for sampling plans for milk products on the basis of the Codex General Guidelines on Sampling

1. INTRODUCTION

1. At its 6th Session in 2004 the Codex Committee on Milk and Milk Products requested the IDF/ISO/AOAC Working Group on Methods of Analysis and Sampling to prepare recommendations for sampling plans for milk products on the basis of the General Guidelines on Sampling, recently finalised by the CCMAS. The Committee also agreed that the report of the IDF/ISO/AOAC Working Group covering this item would be circulated and considered at its next Session.[4]

2. SUMMARY REPORT OF THE IDF/ISO INTERNATIONAL WORKING GROUP ON METHODS OF ANALYSIS AND SAMPLING

2. Following the decision on discontinuation of the former IDF/ISO/AOAC Tripartite collaboration in September 2005 the IDF/ISO International Working Group on Methods of Analysis and Sampling has undertaken to examine the issues involved with the implementation of the sampling plans contained in the Codex Guidelines. The general conclusion from this review is that there are several issues; which prevent the Codex Guidelines from being applied immediately to the assessment of conformance in milk products. These issues include a lack of definition of the required stringency for sampling plans, the application of sampling plans for lots of discrete items to product which is a bulk material, and the presence of significant measurement error associated with the testing of many Codex parameters in milk products.

3. RECOMMENDATIONS OF THE IDF/ISO INTERNATIONAL WORKING GROUP ON METHODS OF ANALYSIS AND SAMPLING

3. Considering that principal reason for the failure to identify any suitable sampling plans for general use by CCMMP is the presence of significant measurement error, it is recommended that the matter of sampling plans in the presence of significant measurement error be referred to an expert group of statisticians to see whether they can find a solution.

4. If CCMMP wants to implement sampling plans in the shorter term, there are several options are available, as discussed in the review. However these options all suffer from deficiencies, particularly concerning their validity or their stringency. Unless one is able to compensate for the effect of significant measurement error, there is potential for the stringency of sampling plans to be seriously eroded, and users should be made aware of these risks before using any of these options.

5. Possible options discussed in the review are:

- Ignore measurement error and use the sampling plans in the Codex Guidelines
- Use the methods of the recently developed ISO 22110/IDF207 Standard, to carry out a verification that [nominal] assessments made by the supplier and the 'consumer' are consistent, considering the known measurement errors. The disadvantage with this option is that it requires co-operation between the supplier and the consumer and that the outcome is inconclusive in the event of a disagreement, without any mechanism to determine which party is the more correct.
- Use methods based on measurement uncertainty considerations. These methods are applied mostly using single sample assessments, but while they could be regarded as valid, they are quite weak for detecting anything except large amounts of product out of specification.

[4] Codex ALINORM 04/27/11, para. 135 and 136

- Use methods based on EC2535/2001. The principal disadvantage of this method is that it relies on long term estimates of process standard deviations provided by the producer and calculated assuming that processes are stable.

Adoption of any of these options should be considered only as a temporary measure, while a more valid approach is sought that provides acceptable risks of incorrect compliance decisions.

6. If it doesn't prove possible to find sampling plans to enforce conformity to the desired levels of stringency, then CCMMP may have to consider the following:

- Relaxation of the stringency relating to commodity standards so that the desired stringency can be achieved with the test methods and sampling plans available.
- Redefining existing standards in terms of parameters which meet the scope of the Codex Guidelines.
- Removal of those standards altogether.

7. CCMMP should convene some forum to decide on the stringency appropriate to assessments of conformity in relation to the standards for milk products.

8. Further work needs to be done to address the issue of the application of the Codex Guidelines to lots consisting of continuous product, for example sampling plans could be selected on the basis of typical lot sizes for the product concerned.

9. The reasons leading to these conclusions are summarised below and discussed more fully in an attached technical discussion.

3.1 SAMPLE SIZE VERSUS LOT SIZE

10. Traditionally, sampling plans, including those in the Codex Guidelines, have assumed that lots consist of discrete units. However this constraint is unnecessary; sampling plans for inspection by attributes and inspection by variables apply equally to products which are a continuum (bulk material).

11. Published sampling schemes have also presented sampling plans chosen according to the size of the lot inspected. The designers of these schemes have deliberately, but arbitrarily, increased sample size with lot size in order to reduce the chance of making an incorrect decision on larger lots, where the cost of an incorrect decision is greater. However selection of a sampling plan in terms of lot size, and the increasing sample size in relation to lot size, is not generally suitable for milk products where the sizes of lots, in terms of the numbers of packages they contain, is determined by the final use of the product and not necessarily by the quantity of product in the lot itself.

12. The Codex Guidelines do contain sampling plans on the sampling of bulk materials, taken from ISO/FDIS 10725 and ISO11648-1. However these sampling plans are used to assess conformance of the *average* level of some attribute to a specified value. While this is important in many contractual arrangements, these sampling plans are generally not suitable for Codex purposes, where conformity to a lower or an upper specification limit is required.

3.2 STRINGENCY

13. Currently, most Codex standards specify only limits, with no information on how product is to be assessed for conformance to those limits, or the stringency required of those assessments. This has lead to a variety of interpretations and ad hoc procedures carrying with them risks that assessments of the same product may not be consistent, or that consumers or producers may be placed at unnecessary risk.

14. It was intended that this situation would be overcome by the development of the Codex Guidelines, in that they would provide a template for the selection of sampling plans to be used in conjunction with Codex standards.

15. To select a sampling plan for a particular application, a Commodity Committee must first make a decision about the stringency required of that plan, that is the inherent risks of accepting product of poor quality and of rejecting product of acceptable quality. Commodity Committees, CCMMP included, still need to make policy decisions on what risks are acceptable before any formal sampling plans can be applied.

3.3 MEASUREMENT ERROR

16. In general, unless specific allowances can be made, use of an imprecise test method will increase the risks of failing product of acceptable quality product and of accepting poor quality product. The Codex Guidelines are applicable only when measurement error is less than 30% of the total variation. However it is apparent from a limited review of data, that many methods used to test Codex parameters in milk products would not meet this requirement. Although a number of solutions have been suggested to this problem, there does not appear to be any general statistical methodology in the literature, when inter-laboratory test error is significant, that will deliver a prescribed stringency.

17. While the issues relating to stringency and the use of sampling plans for lots not consisting of discrete units could be resolved relatively easily, say by policy decisions and using nominal lot sizes respectively, there do not appear to be any general sampling plans that control risks satisfactorily in the presence of significant between laboratory measurement error.

Annex 1

Technical Discussion

Introduction

This annex discusses the CCMAS Guidelines on Sampling and identifies issues with the implementation of the sampling plans contained in those guidelines. The general conclusion is that the Codex Guidelines are not immediately applicable to the assessment of conformance in milk products, particularly because the presence of significant between laboratory measurement error for many Codex parameters assessed in dairy products would appear to render the Codex Guidelines unusable. Further, there does not seem to be any simple way of overcoming this problem, to design a sampling plan to control risks to prescribed levels, in the presence of significant measurement error.

Definitions

The following definitions are useful in any discussion on sampling plans.

Sample (ISO3534-1977; 3.2)

One or more items taken from a population [lot] and intended to provide information on the population and possibly serve as a basis for a decision on the population, or the process which provided it.

Sampling Plan (ISO3534-1977; 4.30)

A plan according to which one or more samples are taken in order to obtain information and possibly reach a decision [about the lot or the process which produced it].

Acceptance Sampling Plan (ISO3534-1977; 4.37)

A sampling plan intended to determine the acceptance or the rejection of a lot.

This is the type of sampling plan referred to in the Codex Procedural Manual. Note that these sampling plans specify not only the number of samples taken, and possibly how those samples are to be taken, but also criteria for the acceptance of the lot, based on the test results obtained from testing of the samples.

Operating Characteristic Curve for a sampling plan (ISO3534-1977: 4.44)

A curve showing, for a given sampling plan, the probability of acceptance of a lot as a function of its actual quality.

Producer's Risk (ISO3534-1977: 4.45)

A point on the operating characteristic curve corresponding to a predictable and usually low probability of rejection. This probability of rejection is called the ***producer's risk****.*

Consumer's Risk (ISO3534-1977: 4.46)

A point on the operating characteristic curve corresponding to a predictable and usually low probability of acceptance. This probability is then called the ***consumer's risk*** *and the corresponding lot quality is called the* ***limiting quality (LQ).***

Acceptable Quality Level (ISO3534-1977: 4.51) (AQL)

A quality level which in a sampling plan corresponds to a specified but relatively high probability of acceptance.

Inspection by Attributes

A method which consists in taking note, for every item of a population or of a sample taken from this population, of the presence or absence of a certain qualitative (attribute) and in counting how many items have or do not have this characteristic

Inspection by Variables

A method which consists in measuring a quantitative characteristic for each item of a population or of a sample taken from this population..

Codex Guidelines on Sampling

The Codex Guidelines on Sampling (CGS) is a compilation of material from various ISO and other standards concerned with the assessment of conformity of products to specified limits. The guidelines deal with the control of both the average level and the percentage non-conforming, although the latter situation is the more common within CCMMP.

The purpose of the CGS is to make available, in a relatively succinct and accessible document, a catalogue of valid acceptance sampling plans for use in Codex Commodity Standards for the assessment of conformity of products to those standards.

Indeed the Codex Procedural Manual states:

Codex methods of sampling are designed to ensure that fair and valid sampling procedures are used when food is being tested for compliance with a particular Codex commodity standard.

The appropriate Codex Commodity Committee should indicate:

1. *the basis on which the criteria in the Codex Commodity Standards have been drawn up*
   - *every item in a lot*[5]
   - *or a specified high proportion shall comply with the precision of the standard*
   - *or whether the average of a set of samples extracted from a lot must comply and if so, whether a minimum or maximum tolerance is given*
2. *The sampling protocol may include the following information:*
   - *The statistical criteria to be used for acceptance or rejection of the lot on the basis of the sample*
   - *The procedures to be adopted in cases of dispute*

The guidelines also contain a certain amount of explanatory material relating to the underlying statistical theory and to implementation of sampling plans.

Why Use Statistical Sampling Methods?

The main aim of any product inspection plan is to ensure that the consumer receives the quality required, while remembering that financial resources are not unlimited and that the cost of the product must reflect the cost of inspection as well as the cost of production.

[5] In principle, such an outcome is not achievable by sampling methods, since not every item in a lot is inspected. It is assumed that this might refer to ‘every item in the sample’, that is to attributes sampling plans having zero acceptance numbers.

However manufacturing processes and test methods are variable – two samples taken from the same run of the same manufacturing process generally have different chemical or microbiological 'compositions', and repeated measurements made on the same sample inevitably vary. As a consequence, there is inherent uncertainty associated with any decision made using sampling and testing of product, which leads to risks that incorrect decisions might be made concerning the conformity of product to specification. As a result of this uncertainty, it is not possible to provide a 100% guarantee using sampling methods that all product within a lot complies to a specification - there must always be some risk.

However having acknowledged that there are risks, by using statistical methods sampling plans can often be developed that make allowances for sampling and measurement error, to accept or reject product with no more than a prescribed level of risk considered appropriate for the situation.

This implies that unless statistical principles are employed in the design of sampling plans, users of such plans may be vulnerable to uncalculated and possibly unjustifiably high risks.

Attributes versus Variables

There are basically two types of sampling plans – inspection by attributes where measured outcomes are considered on a pass/fail, presence /absence or similar basis, and inspection by variables where a decision is made according to the value of some statistic, usually calculated from the sample mean and standard deviation of the testing data. This document discuses only the latter situation, which is considered most appropriate for the assessment of conformance of compositional parameters. While some Codex standards already include details of sampling plans based on inspection by attributes, that type of sampling plan is also susceptible to measurement error, and cannot be applied universally.

Issues

There are several technical and practical reasons which might prevent the Codex Guidelines on Sampling from being applied immediately to milk products. These issues are discussed below. Some solutions are offered but in other cases further investigation will need to be carried out before a satisfactory solution can be proposed.

In this discussion the term ***producer*** is used to refer to the producer himself or any agency representing or acting on behalf of the producer, and similarly the term ***consumer*** is used to refer to the consumer or any agency acting on behalf of the consumer, including regulatory agencies.

Stringency

Traditionally most Codex standards specify only limits, with no information on how product is to be assessed for conformance to those limits, or the stringency required of those assessments. This has lead to a variety of interpretations and ad hoc procedures, carrying with them risks that assessments of the same product may not be consistent, or that consumers or producers may be placed at unnecessary risk.

It was intended that this situation would be overcome by the development of the Codex Guidelines, in that they would provide a template for the selection of sampling plans to be used in conjunction with Codex standards.

To select a sampling plan for a particular application, a Commodity Committee must first make a decision about the stringency required of that plan, that is of the inherent risks of accepting product of poor quality and of rejecting product of acceptable quality. These risks are usually specified by two parameters, the Acceptable Quality Level (AQL) and the Limiting Quality (LQ), respectively.

Most published standards and CGS present sampling plans classified according to:

- Sample Size
- Inspection Level
- Acceptable Quality Level

It seems doubtful that consumers, wishing to keep things simple, would implement the switching rules that appear in CGS and in the standards. Indeed, these rules seem far more suited to the control of outgoing quality by a producer, causing tightened (i.e. more stringent) inspection with the onset of deteriorating quality, and a possible relaxation following a long, continuous period of good quality.

It is common practice to specify only a single risk, usually an Acceptable Quality Level, for the inspection of a continuing series of lots from a single manufacturer, or a Limiting Quality for the inspection of isolated lots.

IDF113A and IDF136A provide the following guidance:

A major defect is one that is likely to make the product unfit for use, i.e. in the case of milk products, unfit for sale to the consumer. A major defect would result in the product spoiling or becoming unfit for sale or processing. Examples include

a) Composition defect, where this would affect keeping quality;

b) Contamination with inhibitory substances

A minor defect is a failure to comply with a specification, but which does not make the unit unfit for use and sale, nor cause it to spoil; for example, a unit, the chemical composition or net content of which falls outside, but close to, a specification limit

Sampling plans for major defects shall be selected from the tables in ISO3951 using an AQL of not more than 6.5%.

Sampling plans for minor defects shall be selected from the tables in ISO3951 using an AQL of not more than 10%.

ISO3951 says that the choice of the inspection level and AQL is governed by a number of factors, but is mainly a balance between the total cost of inspection and the consequences of nonconforming items passing into service.

IDF136A provides sampling plans for AQLs for percentages nonconforming of 1%, 1.5%, 2.5%, 4%, 6.5% and 10%.

ISO3951 provides sampling plans for AQLs for percentages nonconforming of 0.10%, 0.15%, 0.25%, 0.40%, 0.65%, 1%, 1.5%, 2.5%, 4% , 6.5% and 10%.

CGS contains sampling plans for AQLs for percentages nonconforming of 0.65%, 2.5% and 6.5%.

Sample Size versus Lot Size

Traditionally, sampling plans have been documented for lots assumed to consist of discrete units. However this constraint is unnecessary, and sampling plans for inspection by attributes and inspection by variables apply equally to products which are a continuum.

Published sampling schemes have also presented sampling plans chosen according to the size of the lot inspected. The designers of these schemes have deliberately, but arbitrarily, increased sample size with lot size in order to reduce the chance of making an incorrect decision on larger lots, where the cost of an incorrect decision will be greater. However this approach is not strictly necessary and is not generally suitable for milk products where the size of the lot, in terms of the number of packages it contains, is determined by the final use of the product and not by the quantity of product in the lot itself.

Bulk Materials

CGS contains sampling plans on the sampling of bulk materials, taken from ISO/FDIS 10725 and ISO11648-1. However these sampling plans are used to assess conformance of the *average* level of some attribute to a specified value. While this is important in many contractual arrangements, these sampling plans are generally not suitable for Codex purposes, where conformity to a lower or an upper specification limit is assessed.

Issues for Producers

It is anticipated that sampling plans adopted by CCMMP will be used primarily by regulatory agencies wishing to assess conformity of imported products against Codex and other standards. In any case, it is generally not appropriate for producers and consumers to use exactly the same sampling plans to assess the same product. This is because in practice the true amount of product out of specification will be unknown, but at any quality level there is a chance that a lot might be accepted. In the worst case situation, a lot which has a 50% chance of being accepted will have a 50% chance of being rejected when re-inspected by a consumer using the same sampling plan.

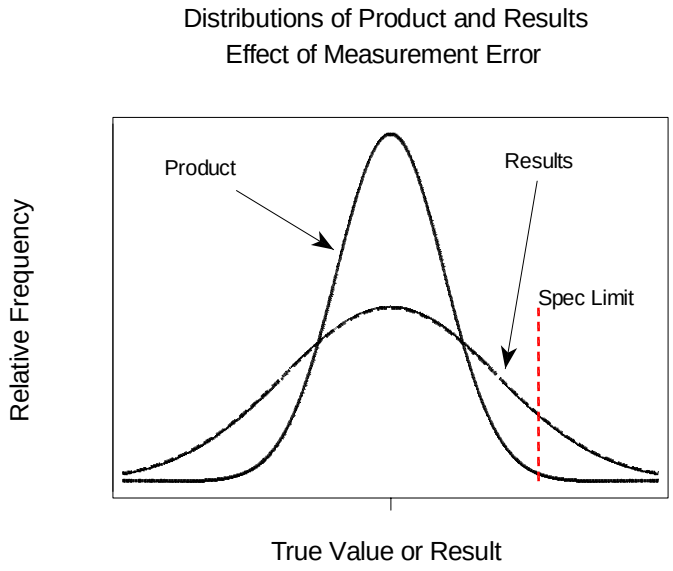
This raises the following issues:

- Producers must be aware of the methods customers will use to assess their products, including the stringency of those assessments
- Either suitable sampling plans need to be prepared for producers to enable them to control risks satisfactorily, or guidance must be given for producers to enable them to develop suitable sampling plans of their own.

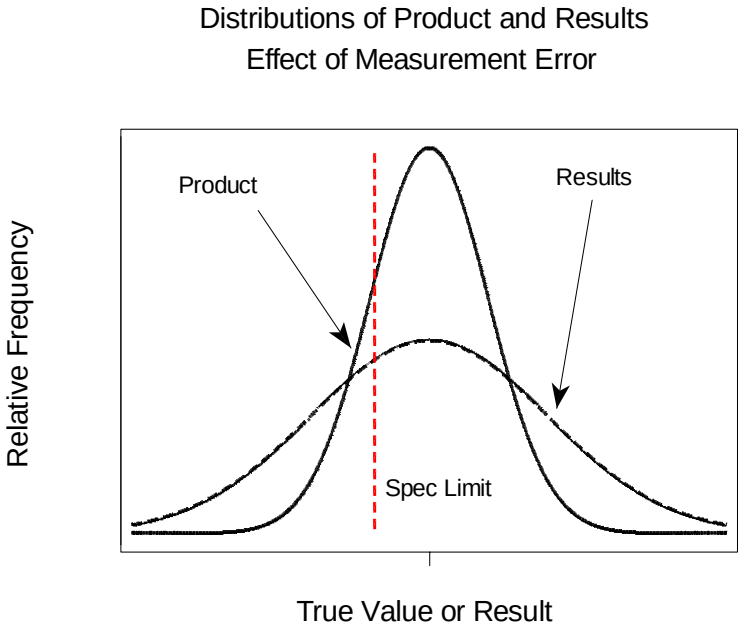
The latter issue has been highlighted in drafts of the Codex Disputes Procedure, where rules developed to resolve disputes included considerations which might not have normally been taken into account in a producer's assessment of the product prior to shipment.

Measurement Error

In general, unless specific allowances can be made, use of an imprecise test method will increase the risks of failing product of acceptable quality product and of accepting poor quality product. The first figure shows that additional variation due to repeatability type error of the test method causes the proportion of results above the upper specification limit to appear greater than the amount of product exceeding the limit.



The second figure shows the converse situation where the product is largely out of specification against the upper limit, but measurement error will make it appear that a greater proportion of results will appear in spec than is actually the case.



In most cases, given that reproducibility of any test method is usually considerably greater than its repeatability, the inter-laboratory component of test error will be substantial. This means that the results of any laboratory will be naturally biased with respect to the true values and against the results obtained by another laboratory.

CGS are applicable only when measurement error is less than 30% of the total variation (as measured by standard deviations). Note this equivalent to the ratio of the measurement error standard deviation to the process standard deviation not exceeding 300%, approximately.

Sampling Plans in the Presence of Significant Measurement Error

As above, CGS are applicable only when measurement error is less than 30% of the total variation. However it is apparent from a limited review of data, that many methods used to test Codex parameters in milk products would not meet this requirement. Although there does not appear to be any general statistical methodology in the literature for the situation when inter-laboratory test error is significant, a number of possible solutions have been suggested to this problem:

A. Ignore the measurement error and accept the higher risks that may be present.
B. Use sampling plans that do not allow for measurement error but which have greater levels of stringency than required to compensate for extra risks incurred due to measurement error.
C. Use the methods of the recently developed ISO/DIS 22110/IDF207 standard to perform a verification that the producer's and consumer's assessments are consistent, based on known testing performance.
D. Use the methods as in EC 2535/2001 developed originally to assess conformance of the fat content of butter.
E. Use of single result assessments

These options are considered in the appendix with the conclusion that, unless the biases caused by inter-laboratory test error can be determined, there appears to be no simple way of constructing a valid sampling plan when between-laboratory measurement error is not negligible, let alone a plan delivering a prescribed stringency. However, even if the biases are known, the presence of other biases due to run to run variation across batches of tested samples within a laboratory may still be significant enough to cause problems.

Appendix: Options for Sampling Plans in the Presence of Significant Measurement Error

The problem of assessing conformance in the presence of measurement error has been studied extensively in the case where measurements are contaminated by a single source of test error described by repeatability-type variation.

However as mentioned above, many test methods employed in the assessment of dairy products have a significant inter-laboratory component of measurement error, compared to process variation, as well as repeatability error. There do not appear to be any published methods for the design of sampling plans, to control the producer's and/or consumer's risks to prescribed levels, in these situations.

The parameter σ_L.

The inter-laboratory component of measurement error, described by the parameter σL, causes a uniform but random bias at any laboratory, affecting all results produced by that laboratory using the method. σL is the between laboratory component of the reproducibility standard deviation σR. The quantities σL and σR are usually estimated as part of validation studies.

However many validation studies are based on data from relatively few laboratories, but even with the usual minimum of eight laboratories, estimates of σL could be quite uncertain. In the worst case situation with eight laboratories, the uncertainty (95% confidence interval) of the estimate of σL , relative to its true value, could be as much as ± 50%.[6]

Of course repeatability and reproducibility variation are not the only components of measurement error, there is also between [testing] run or batch variation, which causes a constant bias on each test result in a batch of tested samples. This type of error is not usually estimated in validation studies, but exists and may have a material effect on conformance decisions.

It appears that σL has a crucial role on the performance of any sampling plan in the presence of significant inter-laboratory test error, whether risks are controlled to prescribed levels or not. This is because in inspection by variables plans, where decisions are made based on functions of means and standard deviations, terms involving the process and repeatability standard deviations tend to be divided by the square root of the number of samples, whereas terms involving σL are not. This also raises the important issue that a laboratory would have to be sure that its performance conformed to σL, before using that value in any acceptance criterion, not something that a laboratory can do in isolation of other laboratories.

The 30% Rule

The sampling plans in the Codex guidelines were considered to be applicable provided measurement error did not exceed 30% of the total variation. This rule was derived by considering that the standard deviation of test results will be only minimally greater than the true process standard deviation when the 30% condition applies.

Ownership of Information

In practice, a *consumer* will not have access to information about a manufacturer's process variability unless they have received large quantities of the product in the past and have tested it extensively. This raises the question as to how a *consumer* can determine whether the condition on the measurement error (30%) is satisfied to decide whether CGS are applicable.

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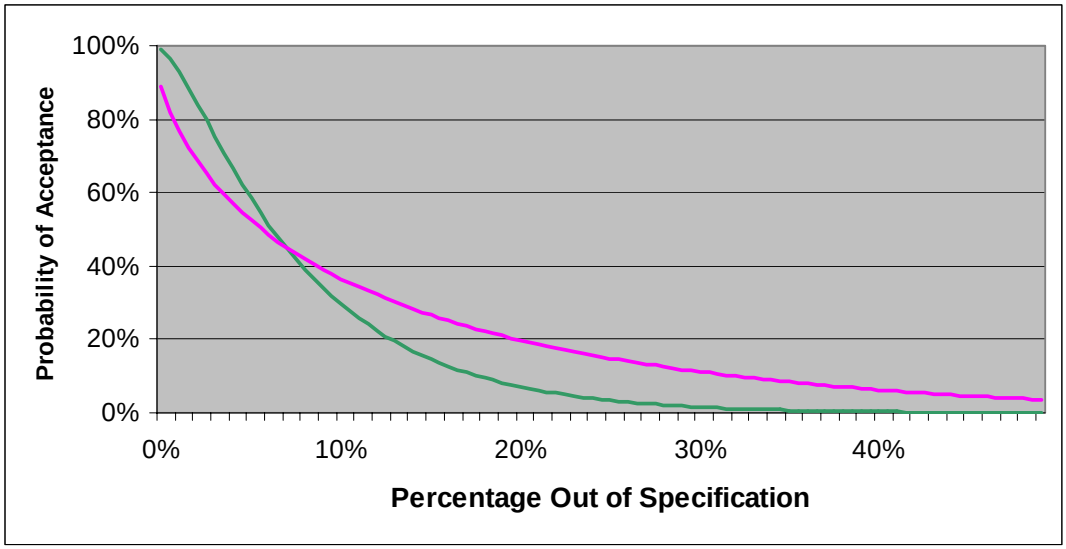
[6] Based on the result quoted in Box, Hunter & Hunter, *Statistics for Experimenters*, Wiley 1978, that the 95% confidence interval, expressed as a percentage, for an estimated standard deviation s in terms of the true value σ is $+200/(\sqrt{2n})$ where n is the number of 'degrees of freedom'.

One solution would be for consumers to require producers to provide measures of the variability of their processes. However there is a danger that a consumer, possibly being mistrustful of information supplied by a producer, may feel obliged to verify the data and impose constraints on such parameters over and above what a Codex Commodity Standard actually requires. This could lead to increases in the *Producer's Risk* by requiring compliance to declared process parameters, for example, as well as to the legal limits. Another consideration is that producers might be reluctant to release this information about their processes.

Evaluation of Options for Sampling Plans in the Presence of Significant Measurement Error
Option A: Ignoring Measurement Error

The following graph shows the Operating Characteristic curves for the inspection by variables sampling plan based on n = 10 samples and an acceptability constant of k = 1.5. The steeper curve represents the OC curve when no measurement error is present, while the shallower curve shows the performance of the same plan in the presence of measurement error. It is assumed that the process standard deviation of σ = 0.2, and repeatability and reproducibility standard deviations σ_r = 0.05 and σ_R = 0.15 respectively[7].



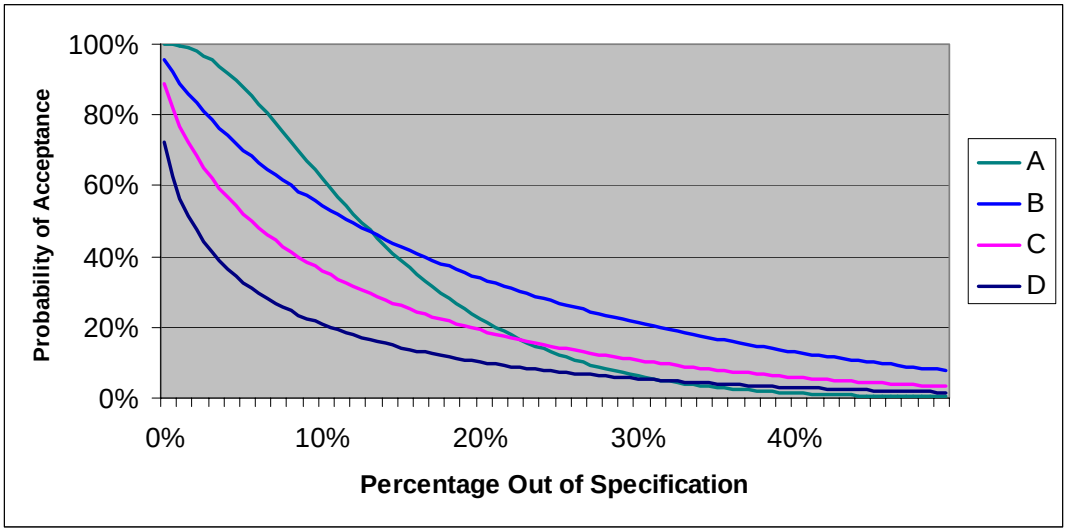
This graph shows an increase in both producer's and consumer's risks caused by the measurement error. Obviously a decision would have to be made whether these increases in risks were acceptable, but such decisions would have to be made on a case by case basis, considering the variation of the product and the measurement errors in each application of the sampling plan. This approach does not seem to lend itself to use in generic standards.

Option B: Use of Greater Stringency to Compensate for Measurement Error

One option considered was to use sampling plans with a greater stringency to compensate for the effects of measurement error. The following example, using sampling plans from CGS (Table 17, p80), shows that this does not work in this case – there is an increase in producer's risk and in some cases in consumer's risk, but the risk profiles do not resemble that of the original error-free sampling plan.

Curve A shows the OC curve for the inspection by variables sampling plan (with known standard deviation) for AQL = 6.5%, i.e. n = 15 and k = 1.13, when no measurement error is present. Curve B shows the OC in the situation with the same measurement error, described above.

[7] The Operating Characteristics of the sampling plans in the presence of measurement error were evaluated using the methods described by Christie (2002).



Curves C and D are the OC curves (in the presence of measurement error) for the more stringent plans appearing in the same section of the table in CGS, namely AQL = 2.5% (n = 11, k = 1.51) and AQL 0,.65% (n = 8, k = 1.96) respectively.

This shows that increasing the nominal stringency of a sampling plan does not appear a satisfactory method for overcoming measurement error, at least in this instance. At best, one faces the same issues described above, of deciding on a case by case basis whether the increase in risks to both the consumer and the producer would be acceptable.

Option C: Use Methods of EC2535/2001

This regulation was introduced by the EC to assess conformance of the fat content of butter. Under this regulation, butter was considered to comply with an upper fat limit, say *U*, provided:

$$\bar{x} \le U - 1.645\sigma + 1.645\sqrt{\sigma_L^2 + \frac{\sigma^2}{n} + \frac{\sigma_r^2}{n}}$$

where

$\bar{x}$ is the average fat result, determined from n samples.

σ is the 'typical process standard deviation', declared by the producer

σ_r is the repeatability standard deviation of the test method for fat, estimated from validation data

σ_R is the reproducibility standard deviation of the test method for fat, estimated from validation data

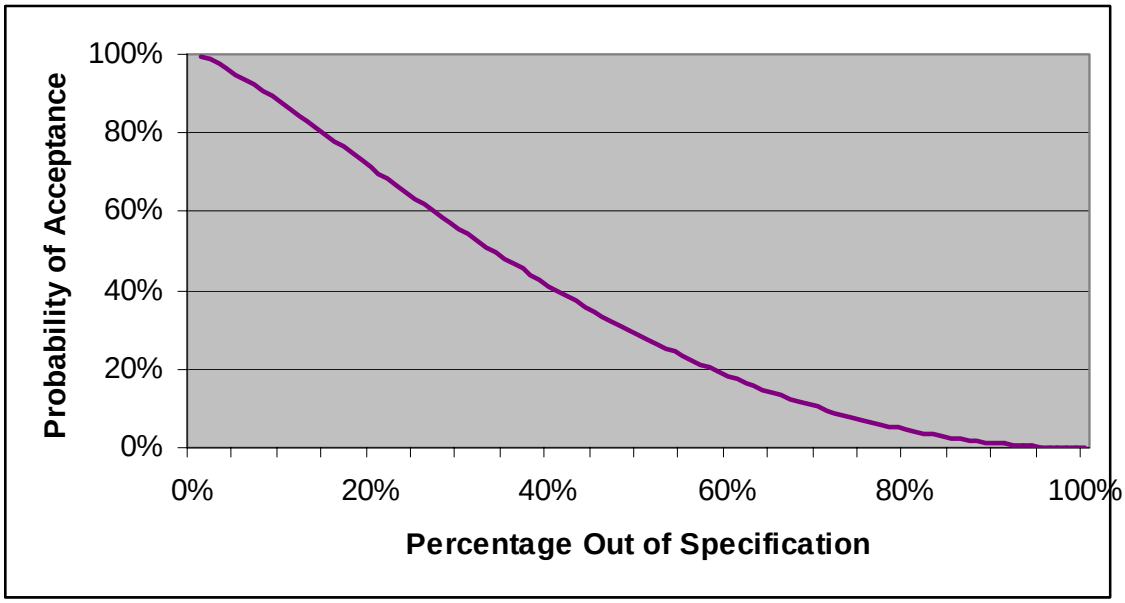
σ_L is the between laboratory component of the reproducibility standard deviation, calculated using $\sigma_R^2 = \sigma_L^2 + \sigma_r^2$

However this scheme appears to have some shortcomings:

- It relies on the producer supplying a value of the typical standard deviation supplied value for the manufacturing process. As mentioned above, it is to be expected that, in many cases, producers may be reluctant to provide such information about their processes.
- Lots are checked for conformity to this standard deviation, as described elsewhere in EC2535/2001, in addition to the assessment of conformance against the upper fat limit. The measures taken in the event of the failure of this check, to replace the typical standard deviation by the sample estimate for the lot, have the potential to increase the producer's risk [of unjustified failure] considerably.

- The scheme has an AQL of 5% - there is a 5% chance of failure when 5% of the product in a lot is out of specification. This quality level is not consistent with those available in CGS, namely 0.65%, 2.5% or 6.5%. Further, this AQL is only nominal as no allowance is made for the bias caused by the inter-laboratory component of measurement error.

The following OC shows the performance of this sampling plan, with process and measurement error standard deviations typical of what occurs for fat in butter, assuming that the bias caused by inter-laboratory test error is zero.



Option D: Use Methods of ISO 22110/IDF 207

This standard, currently being finalised, acknowledges the presence of random bias due to inter-laboratory test error, and adopts a different approach. Rather than an assessment of the product, this standard proposes a verification, to check whether independent assessments of the product made by the producer and the consumer, are consistent, considering the known test errors. The assessments made by the producer and consumer are based on the quantities $\bar{x} \pm k \cdot s$, so do not allow for inter-laboratory test error or explicitly for intra-laboratory error.

While this proposal provides a way of overcoming the intractability of establishing sampling plans allowing for significant between laboratory measurement error, it appears to suffer from some disadvantages:

- The method requires co-operation between the consumer and the producer for its implementation. This co-operation may not exist, or might not be convenient to arrange.
- The comparative assessments made by the producer and consumer in this method are only nominal, as they ignore the presence of measurement error.
- The outcome of the verification is inconclusive in terms of the status of the product. The conclusion is that the assessments made by the two parties are consistent or not.
- There is no mechanism, statistical or otherwise, provided for further investigation, to determine which party is the more correct.

Option E: Use of Single Result Assessments

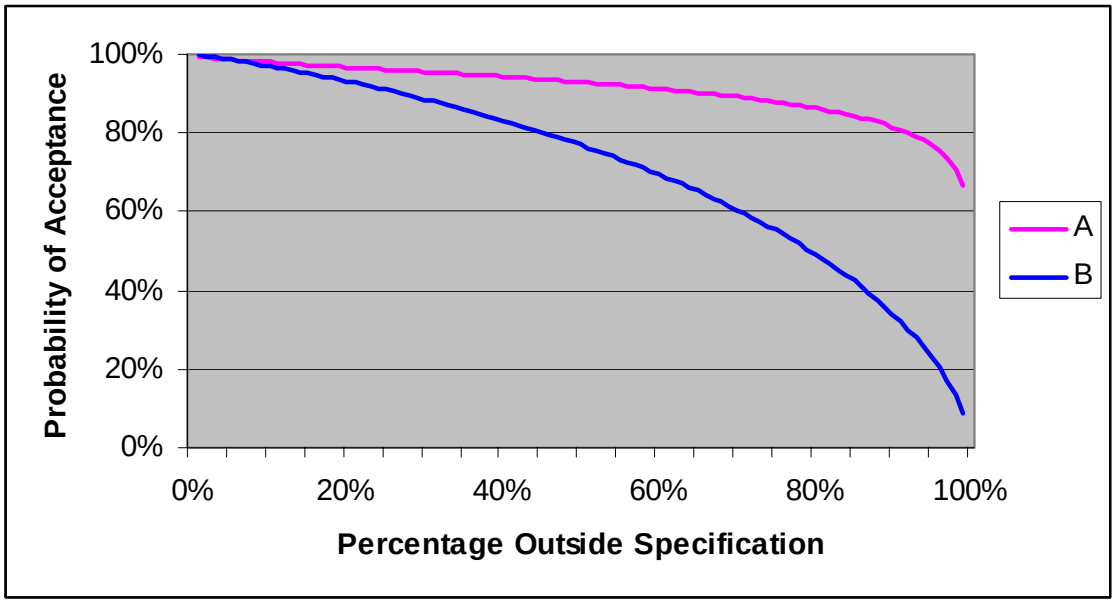
Although they do not appear in either CGS or ISO, the use of single result assessments has become increasingly common in recent times, particularly as a result of work carried out concerning measurement uncertainty.

In this approach, product is considered nonconforming if a single test result lies outside a specification limit by an amount more than can be reasonably accounted for by the measurement uncertainty of the test method. This is often referred to as the "beyond reasonable doubt" approach.

However there are several issues relating to the stringency of single sample assessments, and their consistency with CGS:

- Unlike the sampling plans in CGS, these plans have not been designed to deliver a prescribed stringency - a check needs to be made to ensure that the plans have a stringency acceptable to users.
- Unlike CGS, the choice of a stringency is removed from users – generally [extended] measurement uncertainty is pre-defined.
- Measurement uncertainty is always taken into account, so these schemes are not consistent with CGS, which requires that measurement error is considered only when significant.
- Sampling error is ignored even when it is significant, but in these cases the allowance made for measurement uncertainty will serve as an additional allowance for product conformance.

The following Operating Characteristics show the performance of a single sample assessment scheme:



Curve A reflects a situation with low product variation but high test variation, whereas Curve B shows the converse, high product variation but low test variation.

Irrespective of whether measurement error is significant or not, the sampling plan has a high chance of accepting product containing a high proportion out-of-specification.

Issues surrounding the stringency could, in principle, be reduced by setting the cut-off to a value below the [upper] specification limit, designed to control the consumer's risk. However this would cause an increased risk to the producer and seems undesirable, as:

- In rejecting product from a reputable source as non-compliant, the onus is normally considered to lie on the consumer to prove that the product is non-compliant, rather than on the producer to prove that it is compliant, and
- As the choice of sampling schemes, sample sizes and analytical test methods are normally under the control of the consumer, inadequacies in these should be paid for in terms of consumer's risk rather than producer's risk.

These two principles are consistent with the Codex principle that fair procedures should be used for the assessments of foods.

References

ISO/DIS 22110/IDF 207 Milk and milk products — Sampling — Inspection by variables in the presence of measurement error

ISO 10725:2000 (E) Acceptance sampling procedures for the inspection of bulk materials

ISO2859-1:1999 Sampling procedures for inspection by attributes – Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection.

ISO8197/IDF113A Milk and Milk Products Sampling Inspection by Attributes

ISO8197/IDF136A Milk and Milk Products Sampling Inspection by Variables

ISO3951:1989 (E) Sampling procedures and charts for inspection by variables for percent nonconforming

ISO3534:1977 (E/F) Statistics – Vocabulary and Symbols

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