

codex alimentarius commission



FOOD AND AGRICULTURE
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Agenda Item 5a)

CX/MAS 08/29/6-Add.1

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Twenty-ninth Session

Budapest, Hungary, 10 – 14 March 2008

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS

This document contains the Methods of analysis proposed by the following Committees in Draft Standards under elaboration or as update of current methods.

- A. Codex Committee on Milk and Milk Products
- B. FAO/WHO Coordinating Committee for Asia

A. COMMITTEE ON MILK AND MILK PRODUCTS (ALINORM 08/31/11, Appendix VII)

UPDATED LIST OF METHODS OF ANALYSIS AND SAMPLING FOR CODEX STANDARDS FOR MILK AND MILK PRODUCTS

Milk and Milk Products				
Commodity	Provision	Method	Principle	Type
Milk products	Iron	IDF 103A:1986 / ISO 6732:1985¹ IDF Standard 103A:1986 ISO 6732:1985	Photometry (bathophenanthroline)	IV
Milk products (products not completely soluble in ammonia)	<i>Milk fat</i>	ISO 8262-3 IDF 124-3:2005² IDF 124-3 ISO 8262-3:2005	Gravimetry (Weibull-Berntrop)	I
Blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat ³ (MSNF)	IDF 21B:1987 / ISO 6731:1989 and IDF 13C:1987 / ISO 1737:1999	Calculation from total solids content and fat contents Gravimetry (Röse-Gottlieb)	IV
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ⁴	ISO 8968-1/2 IDF 20-1/2:2001 IDF 20-part 1 or 2:2001 ISO 8968-part 1 or 2:2001	Titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF ⁴	ISO 8968-1/2 IDF 20-1/2:2001 IDF 20-1 or 2:2001 ISO 8968-1 or 2:2001	Titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Water ⁴	ISO 5537 IDF 26:2004 IDF 26:2004 ISO 5537:2004	Gravimetry, drying at 87°C	IV
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF ⁴	ISO 8968-1/2 IDF 20-1/2:2001 IDF 20-part 1 or part 2:2001 ISO 8968-part 1 or part 2:2001	Titrimetry (Kjeldahl)	IV

¹ The format "IDF XXX:YEAR/ISO YYY:YEAR" denotes Standards that have been published separately by IDF and ISO International, but which are technically identical

² The format "ISO YYY/IDF XXX:YEAR" denotes joint ISO-IDF International Standards published by ISO.

³ Milk total solids and MSNF content include water of crystallization of lactose

⁴ Water content excluding the crystallized water bound to lactose (in fact to read moisture content)

Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water ⁵	<i>ISO 5537/IDF 26:2004</i> IDF 26:2004 ISO 5537:2004	Gravimetry, drying at 87°C	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF ⁴	<i>ISO 8968-1/2/IDF 20-1/2:2001</i> IDF 20-part 1 or part 2:2001 ISO 8968-part 1 or part 2:2001	Titrimetry (Kjeldahl)	IV
<i>Blend of sweetened condensed skimmed milk and vegetable fat</i>	<i>Sucrose</i>	<i>ISO 2911/IDF 35:2004</i> <i>Note: the scope of this method does not include this type of product. However, it is expected that the method is applicable.</i> IDF 35:2004 ISO 2911	<i>Polarimetry</i>	<i>IV</i> <i>TBE</i> <i>CCMAS</i>
Blend of sweetened condensed skimmed milk and vegetable fat	Milk solids-not-fat (MSNF) ⁴	IDF 15B:1991 / ISO 6734:1989 <i>and</i> IDF 13C:1987 / ISO 1737:1999	Calculation from total solids content and fat contents Gravimetry (Röse-Gottlieb)	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF ⁴	<i>ISO 8968-1/2/IDF 20-1/2:2001</i> IDF 20-part 1 or part 2:2001 ISO 8968-part 1 or part 2:2001	Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF ⁴	<i>ISO 8968-1/2/IDF 20-1/2:2001</i> IDF 20-part 1 or part 2:2001 ISO 8968-part 1 or part 2:2001	Titrimetry (Kjeldahl)	IV
Butter	Copper	IDF Standard 76A:1980 / ISO 5738:1980 / AOAC 960.40	Photometry, diethyldithiocarbamate	II
Butter	<i>MSNF</i>	<i>ISO 3727-2/IDF 80-2:2001</i> IDF 80-2 ISO 3727-2:2001	Gravimetry	I
Butter	<i>Milk fat</i>	<i>ISO 17189/IDF 194:2003</i> <i>or</i> <i>ISO 3727-3/IDF 80-3:2003</i> IDF 80-3 ISO 3727-3:2003	<i>Gravimetry</i> <i>Direct determination of fat using solvent extraction</i> Gravimetry - <i>Calculation from water⁵ and SNF contents</i>	<i>I</i> I <i>III</i>
Butter	Salt	<i>ISO 1738/IDF 12:2004</i> IDF 12 ISO 1738:2004	Titrimetry (Mohr: determination of chloride, expressed as sodium chloride)	II

Butter	Salt	<i>ISO 15648/IDF 179:2004</i> IDF 179 ISO 15648:2004	Potentiometry (determination of chloride, expressed as sodium chloride)	III
<i>Note regarding the determination of salt content in butter: Potassium chromate used in ISO 1738/IDF 12:2004 is considered a potential carcinogen. CCMAS is requested to advise whether this method should be deleted from Codex STAN 234 or whether the hierarchy of the two salt determination methods for butter should be changed.</i>				
Butter	Vegetable fat (<i>sterols</i>)	<i>ISO 12078/IDF 159:2006</i> <i>ISO 18252/IDF 200:2006</i> ISO 17670 / IDF 202	<i>Gas liquid chromatography</i> <i>Gas liquid chromatography</i> Gas liquid chromatography	<i>II</i> <i>III</i> II
Butter	Vegetable fat	IDF Standard 32:1965 / ISO 3595:1976 (confirmed 1996)/ AOAC 955.34A	Phytosteryl acetate test	III
Butter	Water ⁵	<i>ISO 3727-1/IDF 80-1:2001</i> IDF 80 ISO 37271:2001	Gravimetry	I
Cheese	Citric acid	<i>ISO/TS 2963/IDF/RM 34:2006</i> IDF RM 34 ISO TS 2963:2006	Enzymatic method	II
Cheese	Citric acid	ISO 2963:1997 AOAC 976.15	Photometry	III
Cheese	<i>Milk fat</i>	<i>ISO 1735/IDF 5:2004</i> IDF 5 ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratslaff <i>Ratzlaff</i>)	I
Cheese	Moisture	<i>ISO 5534/IDF 4:2004</i> IDF Standard 4A:1982 ISO 5534:1985	Gravimetry, drying at 102 °C	I
Cheese (and cheese rind)	Natamycin	IDF Standard 140A:1992 / ISO 9223:1991	Molecular absorption spectrophotometry & HPLC after extraction	II
Cheeses, individual	<i>Milk fat</i> in dry matter	<i>ISO 1735/IDF 5:2004</i> IDF 5:2004 ISO 1735:2004	Gravimetry after solvent extraction	I
Cheeses in brine	<i>Milk fat</i> in dry matter (FDM)	<i>ISO 1735/IDF 5:2004</i> ⁵ IDF 5 ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratslaff <i>Ratzlaff</i>)	I

⁵ For this kind of product, repeatability and reproducibility values are given as an indication.

Cottage cheese	Fat-free dry matter	ISO 5534/IDF 4:2004 IDF 5:2004 ISO 1735:2004	Gravimetry, drying at 102°C Calculation from dry matter and fat contents	IV
Cottage cheese	<i>Milk fat</i>	ISO 1735/IDF 5:2004 IDF 5 ISO 1735:2004	Gravimetry (Schmid-Bondzinski-Ratzlaff)	IV
Cottage cheese		ISO 8262-3/IDF 124-3:2005 IDF 124-3:2005 ISO 8262-3:2005	Gravimetry (Weibull-Berntrop)	
Cottage cheese	Milk fat in dry matter	ISO 8262-3/IDF 124-3:2005 IDF 126A:1988 ISO 8262-3:1987	Gravimetry (Weibull-Berntrop)	I
Cheese, unripened including fresh cheese	Protein	ISO 8968-1/IDF 20-1:2001 IDF Standard 20B:1993 AOAC 991.20-23 ISO 8968 Part I	Titrimetry, Kjeldahl	I
Cream	<i>Milk fat</i>	IDF Standard 16C:1987 / ISO 2450:1999	Gravimetry (Röse-Gottlieb)	I
Cream	Solids	IDF Standard 21B:1987 / ISO 6731:1989	Gravimetry (drying at 102°C)	I
Creams lowered in <i>milk fat</i> content	<i>Milk fat</i>	IDF Standard 16C:1987 / ISO 2450:1999 AOAC 995.19	Gravimetry	I
Creams, whipped creams and fermented creams	<i>MSNF</i>	ISO 3727-2/IDF 80-2:2001 IDF Standard 80:1977 ISO 3727:1977 AOAC 920.116	Gravimetry	I
Cream cheese	Dry matter	ISO 5534/IDF 4:2004 IDF 4:2004 ISO 5534:2004	Gravimetry drying at 102°C	IV
Cream cheese	Moisture on fat free basis	ISO 5534/IDF 4:2004 IDF 4:2004 ISO 5534:2004 and ISO 1735/IDF 5:2004 IDF 5:2004 ISO 1735:2004	Calculation from fat content and moisture content	IV
Dairy fat spreads	Total fat	ISO 17189/IDF 194:2003 IDF 194:2003 ISO 17189:2003	Gravimetry Direct determination of fat using solvent extraction	I

Dairy fat spreads	Vegetable fat (<i>sterols</i>)	ISO 12078/IDF 159:2006 ISO 18252/IDF 200:2006 IDF 54:1970 / ISO 3594:1976 IDF 32:1965 / ISO 3595:1976	Gas liquid chromatography Gas liquid chromatography Gas liquid chromatography Phytosterol acetate test	II III II III
Edible casein products	Acids, free	ISO 5547/IDF 91:2007 IDF Standard 91:1979 ISO 5547:1978	Titrimetry (aqueous extract)	IV
Edible casein products	Ash (including P ₂ O ₅)	ISO 5545/IDF 90:2007 IDF Standard 90:1979) ISO 5545:1978	Furnace, 825°C	IV
Edible Casein Products	Casein in protein	ISO 17997-1/IDF 29-1:2004 IDF Standard 29:1964	Titrimetry, Kjeldahl	I
Edible casein products	Copper	ISO 5738/IDF 76:2004 IDF 76 ISO 5738:2004	Colorimetry (diethyldithiocarbamate)	III
Edible casein products	Lactose	ISO 5548/IDF 106:2004 IDF 106 ISO 5548:2004	Photometry (phenol and H ₂ SO ₄)	IV
Edible casein products	Lead	ISO/TS 6733/IDF/RM 133:2006 IDF RM 133 ISO TS 6733: 2006	Spectrophotometry (1,5-diphenylthiocarbazone)	III
Edible casein products	<i>Milk fat</i>	ISO 5543/IDF 127:2004 ISO 5543 IDF 127: 2004	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Edible casein products	Moisture	ISO 5550/IDF 78:2006 IDF 78 ISO 5550:2006	Gravimetry (drying at 102°C)	I
Edible casein products	pH	IDF Standard 115A:1989 / ISO 5546:1979	Electrometry	IV
Edible casein products	Protein (total N x 6.38 in dry matter)	IDF Standard 92:1979 / ISO 5549:1978	Titrimetry, Kjeldahl digestion	IV
Edible casein products	Sediment (scorched particles)	ISO 5739/IDF 107:2003 IDF 107 ISO 5739:2003	Visual comparison with standard disks, after filtration	IV
Emmental	Calcium >= 800 mg/100g	ISO 8070 IDF 119:2007 ⁶	Flame atomic absorption	IV
Evaporated milks	<i>Milk fat</i>	IDF Standard 13C: 1987 / ISO 1737:1999	Gravimetry (Röse-Gottlieb)	I
Evaporated milks	Protein	ISO 8968-1/IDF 20-1:2001 AOAC 945.48H / AOAC 991.20 IDF 20B:1993	Kjeldahl, titrimetry	I
Evaporated milks	Solids, total	IDF Standard 21B:1987 / ISO 6731:1989	Gravimetry (drying at 102°C)	I

⁶ Draft International Standard

Fermented milks	Milk fat	IDF ID:1996 / ISO 1211:1999 / ISO 1211:1999 IDF ID:1996 AOAC 905.02	Gravimetry	I
<i>Fermented milks</i>	<i>Milk fat</i>	<i>IDF 116A:1987</i>	<i>Gravimetry</i>	
<i>Note: IDF 116A:1987 describes a method for the determination of fat content in milk-based edible ices and ice mixes. The scope of this method does not include fermented milks. CCMAS should advise whether this method is fit for purpose.</i>				
<i>Fermented milks - Yoghurt and yoghurt products</i>	<i>Lactobacillus delbrueckii subsp bulgaricus & Streptococcus thermophilus</i>	<i>ISO 7889/IDF 117:2003</i>	<i>Colony count at 37°C</i>	<i>I</i>
<i>Fermented milks - Yoghurt and yoghurt products</i>	<i>Lactobacillus delbrueckii subsp bulgaricus & Streptococcus thermophilus</i>	<i>ISO 9232/IDF 146:2003</i>	<i>Test for identification</i>	<i>I</i>
Milk powders and cream powders	<i>Milk fat</i>	IDF Standard 9C:1987 / ISO 1736:2000	Gravimetry (Röse-Gottlieb)	I
Milk powders and cream powders	Protein (in <i>MSNF</i>)	ISO 8968-1/IDF 20-1:2001 IDF 20-1 ISO 8968-1:2001	Titrimetry, Kjeldahl digestion	I
Milk powders and cream powders	Scorched particles	ISO 5739/IDF 107:2003 IDF 107 ISO 5739:2003	Visual comparison with standard disks, after filtration	IV
Milk powders and cream powders	Solubility	ISO 8156/IDF 129:2005 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 ⁵	ISO 5537/IDF 26:2004⁷ IDF 26 ISO 5537:2004	Gravimetry (drying at 102°C)	IV
<i>Milk fat</i> products	Antioxidants (phenolic)	IDF Standard 165:1993	Reversed phase gradient liquid chromatography	II
<i>Milk fat</i> Products	Copper	ISO 5738/IDF 76:2004 / IDF Standard 76A:1980/ISO 5738:1980 /AOAC 960.40	Photometry, diethyldithiocarbamate	II
<i>Milk fat</i> products	Fatty acids, free (expressed as oleic acid)	ISO 1740/IDF 6:2004 IDF 6 ISO 1740:2004	Titrimetry	I
<i>Milk fat</i> products	<i>Milk fat</i>	IDF Standard 24:1964	Gravimetry (calculation from solids-not-fat and water content)	IV
<i>Milk fat</i> Products	Peroxide value (expressed as meq. of oxygen/kg fat)	ISO 3976/IDF 74:2006 AOAC 965.33	Photometry Titrimetry	III I

⁷ The replacing method has only been validated for milk powders, not for cream *powders*

<i>Milk fat</i> products	Vegetable fat (sterols)	<i>ISO 12078/IDF 159:2006</i> <i>ISO 18252/IDF 200:2006</i>	<i>Gas liquid chromatography</i> <i>Gas liquid chromatography</i>	<i>II</i> <i>III</i>
		<i>IDF Standard 54:1979 / ISO 3594:1976</i>	<i>Gas liquid chromatography</i>	<i>II</i>
<i>Milk fat</i> products	<i>Vegetable fat</i>	<i>IDF Standard 32:1965 / ISO 3595:1976</i>	<i>Phytosteryl acetate test</i>	<i>III</i>
<i>Milk fat</i> products	Water ⁵	<i>ISO 5536/IDF 23:2002</i> <i>IDF 23 ISO 5536:2002</i>	Titrimetry (Karl Fischer)	II
<i>Milk fat</i> products (anhydrous <i>milk fat</i>)	Peroxide value	<i>ISO 3976/IDF 74:2006</i> <i>AOAC 965.33</i>	<i>Photometry</i> <i>Titrimetry</i>	<i>III</i>
Milk products obtained from fermented milks heat-treated after fermentation	Protein	<i>ISO 8968-1/IDF 20-1:2001</i> <i>IDF Standard 20B:1993</i> <i>ISO 8968 Part 1</i> AOAC 991.20-23	Titrimetry (Kjeldahl)	I
Mozzarella	<i>Milk fat</i> in dry matter – with high moisture	<i>ISO 1735/IDF 5:2004</i> <i>IDF 5:2004 </i> <i>ISO 1735:2004</i>	Gravimetry after solvent extraction	IV
Mozzarella	<i>Milk fat</i> in dry matter – with low moisture	<i>ISO 1735/IDF 5:2004</i> <i>IDF 5:2004 </i> <i>ISO 1735:2004</i>	Gravimetry after solvent extraction	IV
Processed cheese products	Citric acid	<i>ISO/TS 2963/IDF/RM 34:2006</i> <i>IDF RM 34 ISO TS 2963:2006</i>	Enzymatic method	II
Processed cheese products	<i>Milk fat</i>	<i>ISO 1735/IDF 5:2004</i> <i>IDF 5:2004 </i> <i>ISO 1735:2004</i>	Gravimetry (Schmid- Bondzynski- Ratzlaff)	I
Processed cheese products	Phosphate, added (expressed as phosphorus)	IDF <i>Standard</i> 51B:1991	Calculation	IV
Processed cheese products	Phosphorus	IDF <i>Standard</i> 33C:1987 / ISO 2962:1984	Spectrophotometry (molybdate-ascorbic acid)	II
Processed cheese products	Salt	<i>ISO 5943/IDF 88:2004</i> <i>IDF 88 ISO 5943:2004</i>	Potentiometry (determination of chloride, expressed as sodium chloride)	II
Sweetened condensed milk	<i>Milk fat</i>	IDF <i>Standard</i> 13C: 1987 / ISO 1737:1999	Gravimetry (Röse-Gottlieb)	I
Sweetened and Condensed Milks	Protein	<i>ISO 8968-1/IDF 20-1:2001 /</i> AOAC 945.48H / AOAC 991.20 <i>IDF 20B:1993</i>	Kjeldahl, titrimetry	I
Sweetened Condensed Milks	Solids	IDF <i>Standard</i> 15B:1991 / ISO 6734:1989	Gravimetry, drying at 102 °C	I

Whey cheeses by concentration	Milk fat	IDF 59A:1986 ISO 1854:1999	Gravimetry (Röse Gottlieb)	I
Whey Cheese	Dry matter (for denomination)	IDF 58 ISO 2920:2004	Gravimetry, drying at 88 °C	I
Whey cheeses by concentration	Dry matter (total solids)	IDF 58 ISO 2920:2004	Gravimetry, drying at 88 °C	I
Whey cheeses by coagulation	Dry matter (total solids)	IDF 4:2004 ISO 5534:2004	Gravimetry, Drying at 102°C	IV
Whey cheese	Fat on the dry basis	IDF 59 A:1986 / ISO 1854:1999 and IDF 58:2004 ISO 2920:2004	Calculation from fat content and dry matter content	I
Whey cheese	Milk fat (in dry matter)	IDF standard 59A:1986 / ISO 1854:1999	Gravimetry (Röse-Gottlieb)	I
Whey cheeses including whey cheeses by concentration	Total fat	IDF 59A:1986 / ISO 1854:1999	Gravimetry (Röse-Gottlieb)	I
Whey cheeses by coagulation	Total fat	IDF 5:2004 ISO 1735:2004	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Creamed whey cheese	Fat on the dry basis	IDF 59 A: 1986 / ISO 1854: 1999 and IDF 58:2004 ISO 2920:2004	Calculation from fat content and dry matter content	I
Skimmed whey cheese	Fat on the dry basis	IDF 59 A:1986 / ISO 1854:1999 and IDF 58:2004 ISO 2920:2004	Calculation from fat content and dry matter content	I
Whey powders	Ash	ISO 5545/IDF 90:2007 IDF Standard 90:1979 ISO 5545:1978	Furnace, 825°C	IV
Whey powders	Copper	ISO 5738/IDF 76:2004 IDF 76 ISO 5738:2004	Photometry (diethyldiethiocarbamate)	III
Whey Powders	Lactose	ISO 5765-1/2/IDF 79-1/2:2002 IDF 79B:1991	Enzymatic method: Part 1 - Glucose moiety or Part 2 - Galactose moiety glucose moiety (method A), galactose moiety (method B)	II
Whey powders	Milk fat	IDF Standard 9C:1987 / ISO 1736:2000	Gravimetry (Röse-Gottlieb)	I
Whey powders	Moisture, "Free"	ISO 2920/IDF 58:2004 IDF 58 ISO 2920:2004	Gravimetry (drying at 88 °C ±2°C)	IV

Whey powders	Protein (total N x 6.38)	IDF Standard 92:1979 / ISO 5549:1978	Titrimetry, Kjeldahl digestion	IV
Whey powders	Water (not including water of crystallization of lactose)	ISO 5537/IDF 26:2004 / IDF 26A:1993 AOAC 927.05	Gravimetry	I
Yoghurt products	Lactobacillus bulgaricus & Streptococcus thermophilus	IDF 117 ISO 7889:2003	Colony count at 37°C	I
Yoghurt products	Lactobacillus bulgaricus & Streptococcus thermophilus	IDF 146 ISO 9232:2003	Test for identification	I
Yoghurt products	Solids, Total	IDF 151 ISO 13580:2005	Gravimetry (drying at 102°C)	I
Yoghurt	Streptococcus thermophilus & Lactobacillus delbrueckii subsp. bulgaricus ≥ 10 ⁷ cfu/g	ISO 7889/IDF 117:2003	Colony count at 37°C	I
Yoghurt	Streptococcus thermophilus & Lactobacillus delbrueckii subsp. bulgaricus ≥ 10 ⁷ cfu/g	ISO 9232/IDF 146:2003	Test for identification: morphological, cultural and biochemical characteristics	I

CHANGES TO CODEX STAN 234 PART 1-B, P. 47-48

METHODS OF SAMPLING BY ALPHABETICAL ORDER OF COMMODITY CATEGORIES AND NAMES

Milk and Milk Products	Methods of Sampling	Notes
Butter	ISO 707/IDF 50	General Instructions for obtaining a sample from a bulk
Cheese	ISO 707/IDF 50	General Instructions for obtaining a sample from a bulk
Cheeses in brine	ISO 707/IDF 50	General Instructions for obtaining a sample from a bulk
Edible casein products	ISO 707/IDF 50	General Instructions for obtaining a sample from a bulk
Creams, Whipped creams and Fermented Creams	IDF Standard 50C:1995 ISO 707:1997 AOAC 968.12	General instructions
Fermented Milks		
Evaporated milks	ISO 707/IDF 50	General Instructions for obtaining a sample from a bulk
Milk powders and cream powders	ISO 707/IDF 50	General Instructions for obtaining a sample from a bulk
Milkfat products	ISO 707/IDF 50	General Instructions for obtaining a sample from a bulk
Sweetened condensed milks	ISO 707/IDF 50	General Instructions for obtaining a sample from a bulk
Whey cheese	ISO 707/IDF 50	General Instructions for obtaining a sample from a bulk
Whey powders	IDF 113 ISO 5538:2004	Inspection by attributes

Whey powders	ISO 707/IDF 50	General Instructions for obtaining a sample from a bulk
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⁹ Draft standard which is publicly available

B. FAO/WHO COORDINATING COMMITTEE FOR ASIA (ALINORM 07/30/15, Appendix III)

Draft Standard for Ginseng Product

COMMODITY	PROVISION	METHOD	PRINCIPLE
Ginseng Product	Moisture	AOAC 925.45	vacuum drying
Ginseng Product	Solids	AOAC 925.45 and calculated by subtracting the content of water from 100%.	calculation
Ginseng Product	Ash	AOAC 923.03	Gravimetry
Ginseng Product	Water-insoluble Solids	described in Annex A	
Ginseng Product	Water-saturated 1-butanol extracts	described in Annex B	
Ginseng Product	Ginsenosides Rb1 and Rf	described in Annex C	

Determination of Water-insoluble Solid Content

Place ca 1 g sample in 25 ml centrifugal tube with constant weight. Add 15 ml of distilled water and dissolve the sample. Centrifuge for 15 min at 3000 rpm and discard supernatant. Repeat twice this centrifugation. Dry centrifugal tube and residue to constant weight at 105°C. Report results in percent.

$$\text{water-insoluble solid content (\%)} = (W1-W0)/ S \times 100$$

S: weight of sample (g)

W1: weight of centrifugal tube and residue after drying (g)

W0: weight of centrifugal tube (g)

* The method mentioned in Annex A is stipulated in the Korean Food Standards Law and modifies the "AOAC Official Method 950.66."

Determination of water-saturated 1-butanol extracts

1. Preparation of water-saturated 1-butanol

Mix 1-butanol with water in separatory funnel in the ratio of 70:30 and shake it vigorously. Let stand until the upper and lower phases are separated. Discard lower layer (water layer).

2. Analysis method

2.1 Dried Ginseng

Weigh ca 5 g test portion, ground to pass 80 mesh or finer sieve, into 250 ml erlenmeyer flask and reflux with 50 ml water saturated 1-butanol on a water bath at 80°C for 1 hour. Decant 1-butanol into another 250 ml erlenmeyer flask. Repeat twice the above extraction. Combine the solvent and filter into a 250 ml separatory funnel. Add 50 ml of distilled water. Shake and stand until the upper and lower layer are separated completely into two layers. Collect 1-butanol layer (upper layer) in an evaporation flask, vacuum-evaporate to dryness. Add 50 ml of diethyl ether, re-flux it on a water bath approximately at 46°C for 30 minutes, and decant the diethyl ether. Dry flask and contents to constant weight at 105°C. Report increase in weight flask as "1-butanol extracts in ginseng". Express the result as mg per gram on dried ginseng.

$\text{water-saturated 1-butanol extracts(mg/g)} = (A-B)/ S$

S: weight of sample (g)

A: weight of flask after concentrating and drying extracts (mg)

B: weight of flask (mg)

2.2 Ginseng Extract (including a powered type)

Place 1~2 g sample in 250 ml erlenmeyer flask, dissolve in 60ml water and transfer into separating funnel. Add 60ml of diethyl ether. Shake and stand until the upper and lower layer are separated. Collect lower layer and extract with 60 ml water saturated 1-butanol for three times. Combine the solvent into a 250 ml separatory funnel. Add 50 ml of distilled water. Shake and stand until the upper and lower layer are separated completely into two layers. Collect 1-butanol layer (upper layer) in an evaporation flask with constant weight, vacuum-evaporate to dryness. Dry flask and contents to constant weight at 105°C. Report increase in weight flask as "1-butanol extracts in ginseng extract". Express the result as mg per gram on ginseng extract.

References

1. Planta medica, vol 25, pp 194-202, 1974
2. Chem. Pharm Bull., vol 14, pp 595-600, 1966
3. Korean J. Ginseng Sci., 10(2), pp 193-199, 1986

Annex C

Identification of ginsenosides Rb1 and Rf

Ginsenosides in ginseng products can be identified either by Thin Layer Chromatography(TLC) or High Performance Liquid Chromatography(HPLC).

1. Preparation of sample solution

Dilute the dried 1-butanol extract of Annex B with ten-fold volume of methanol, dissolve completely, and filter through 0.45 µm membrane filter.

2. Preparation of standard solution

Dissolve standard ginsenosides, such as ginsenoside-Rb1 and -Rf, in methanol to make a 1% solution and filter through 0.45 µm membrane filter.

3. Identification

3.1 Thin Layer Chromatography

Spot 2-5 µl of the standard and sample solutions, as indicated in the above, on TLC plate (silica gel), previously dried at 110°C for 15 minutes in dry oven. Develop with an upper solution of 1-butanol:ethylacetate:water (5:1:4, v/v/v) or a lower solution of chloroform:methanol:water (65:35:10, v/v/v). Spray 10% sulfuric acid or 30% sulfuric acid-ethanol solution over TLC plate and oven dry it at 110°C for 5-10 minutes to reveal its color. Identify the ginsenosides of Ginseng products by comparing the Rf values and colors with those of standard ginsenosides.

3.2 High Performance Liquid Chromatography

Prepare standard and sample solutions, as indicated in the above. Analyze ginsenoside with HPLC depending upon the operating condition. Identify ginsenosides of sample by comparing retention times of peaks with those of the standard.

Operating condition

Column: NH2 column, µ-Bondapak C18 column, carbohydrate analyzing column or equivalent

Detector: UV (203 nm) or ELSD

Eluent: UV: acetonitrile: water (30:70, v/v)

ELSD: acetonitrile: water: isopropanol (94.9:5.0:0.1, v/v/v)

Flow rate: 1.0 ml/min ~ 2.0 ml/min

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