

codex alimentarius commission



FOOD AND AGRICULTURE
ORGANIZATION
OF THE UNITED NATIONS

WORLD
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Agenda Item 5

CX/MAS 10/31/5-Add.1

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Thirty-first Session

Budapest, Hungary, 8 - 12 March 2010

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS IN CODEX STANDARDS

This document contains the methods of analysis proposed by the **Codex Committee on Milk and Milk Products**.

COMMITTEE ON MILK AND MILK PRODUCTS (ALINORM 10/33/11, para. 45-62, APPENDIX III)

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

Proposed changes are shown in ~~bold strikethrough~~ for deletion and **bold underlined** for additions.

| Products | Provisions | Method | Principle | Type | CCMMP comments |
|--|---|--|---|-------------------|-----------------------|
| Milk products | Iron | NMKL 139 (1991) (Codex general method) AOAC 999.10 | Atomic absorption spectrophotometry | II | |
| Milk products | Iron | IDF 103A:1986 / ISO 6732:1985 AOAC 984.27 | Photometry (bathophenanthroline) | IV | |
| | | | <u>Inductible Couple Plasma , optical emission spectrophotometry</u> | <u>III</u> | |
| Blend of evaporated skimmed milk and vegetable fat | Total fat | ISO 1737/ IDF 13:2008 IDF 13C:1987 / ISO 1737:1999 <u>AOAC 989.05</u> | Gravimetry (Röse-Gottlieb) | IV | <i>Method update</i> |
| Blend of evaporated skimmed milk and vegetable fat | Milk solids-not-fat (MSNF) ¹ | IDF 21B:1987/ISO 6731:1989 and ISO 1737/ IDF 13:2008 IDF 13C:1987 / ISO 1737:1999 <u>AOAC 989.05</u> | Calculation from total solids content and fat contents | IV | <i>Method update</i> |
| | | | Gravimetry (Röse-Gottlieb) | <u>IV</u> | |
| Blend of evaporated skimmed milk and vegetable fat | Milk protein in MSNF ¹ | ISO 8968-1/2 IDF 20-1/2:2001 / <u>AOAC 991.20</u> | Titrimetry (Kjeldahl) | IV | |
| Reduced fat blend of evaporated skimmed milk and vegetable fat | Total fat | ISO 1737/ IDF 13:2008 IDF 13C:1987 / ISO 1737: 1999 <u>AOAC 989.05</u> | Gravimetry (Röse-Gottlieb) | IV | <i>Method update</i> |
| | | | | <u>IV</u> | |

¹ Milk total solids and MSNF content include water of crystallization of lactose

| Products | Provisions | Method | Principle | Type | CCMMP comments |
|---|-----------------------------------|--|--|-----------------|---|
| Reduced fat blend of evaporated skimmed milk and vegetable fat | MSNF ¹ | IDF 21B:1987 / ISO 6731:1989 and ISO 1737 IDF 13:2008 IDF 13C:1987 / ISO1737:1999 AOAC 989.05 | Calculation from total solids content and fat contents Gravimetry (Röse-Gottlieb) | IV | <i>Method update and Principle update</i> |
| Reduced fat blend of Evaporated skimmed milk and vegetable fat | Milk protein in MSNF ¹ | ISO 8968-1/2 IDF 20-1/2:2001 / AOAC 991.20 | Titrimetry (Kjeldahl) | IV | |
| Blend of skimmed milk and vegetable fat in powdered form | Total fat | ISO 1736 IDF 9:2008 IDF 9C:1987 / ISO1736:2000 AOAC 989.05 | Gravimetry (Röse-Gottlieb) | IV | <i>Method update</i> |
| Blend of skimmed milk and vegetable fat in powdered form | Water ² | ISO 5537 IDF 26:2004 AOAC 927.05 | Gravimetry, drying at 87 C Gravimetry, drying at 100° C | IV IV | |
| Blend of skimmed milk and vegetable fat in powdered form | Milk protein in MSNF ¹ | ISO 8968-1/2 IDF 20-1/2:2001 / AOAC 991.20 | Titrimetry (Kjeldahl) | IV | |
| Reduced fat blend of skimmed milk powder and vegetable fat in powdered form | Total fat | ISO 1736 IDF 9:2008 IDF 9C:1987 / ISO 1736:2000 AOAC 989.05 | Gravimetry (Röse-Gottlieb) Gravimetry (modified Mojonier) | IV | <i>Method update</i> |
| Reduced fat blend of skimmed milk powder and vegetable fat in powdered form | Water ² | ISO 5537 IDF 26:2004 AOAC 927.05 | Gravimetry, drying at 87 °C Gravimetry, drying at 100° C | IV IV | |
| Reduced fat blend of skimmed milk powder and vegetable fat in powdered form | Milk protein in MSNF ¹ | ISO 8968-1/2 IDF 20-1/2:2001 / AOAC 991.20 | Titrimetry (Kjeldahl) | IV | |
| Blend of sweetened condensed skimmed milk and vegetable fat | Total fat | ISO 1737 IDF 13:2008 IDF 13C:1987 / ISO 1737:1999 AOAC 989.05 | Gravimetry (Röse-Gottlieb) | IV | <i>Method update</i> |
| Blend of sweetened | Sucrose | ISO 2911 IDF 35:2004 | Polarimetry | IV | |

² Water content excluding the crystallized water bound to lactose (generally known as “moisture content”)

| Products | Provisions | Method | Principle | Type | CCMMP comments |
|---|---|---|--|------------------|---|
| condensed skimmed milk and vegetable fat | | | | | |
| Blend of sweetened condensed skimmed milk and vegetable fat | Milk solids-not-fat (MSNF) ¹ | IDF 15B:1991 / ISO 6734:1989 AOAC 990.19 | Calculation from total solids content, and fat contents and sugar content | IV IV | <i>Method update and Principle update</i> |
| Blend of sweetened condensed skimmed milk and vegetable fat | Milk protein in MSNF ¹ | ISO 8968-1/2 IDF 20-1/2:2001 / AOAC 991.20 | Titrimetry (Kjeldahl) | IV | |
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat | Total fat <= 8% m/m >= 1% m/m | ISO 1737 IDF 13:2008 IDF 13C:1987 / ISO 1737: 1999 AOAC 989.05 | Gravimetry (Röse-Gottlieb) | IV IV | <i>Method update</i> |
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat | MSNF ¹ >= 20% m/m | IDF 15B:1991 / ISO 6734:1989 AOAC 990.19 | Calculation from total solids content and fat content and sugar content | IV IV | <i>Method update and Principle update</i> |
| Reduced fat blend of sweetened condensed skimmed milk and vegetable fat | Milk protein in MSNF ¹ | ISO 8968-1/2 IDF 20-1/2:2001 / AOAC 991.20 | Titrimetry (Kjeldahl) | IV | |
| Butter | Salt | ISO 1738 IDF 12:2004 AOAC 960.29 | Titrimetry (Mohr: determination of chloride, expressed as sodium chloride) | III IV | |
| Butter | Milk fat purity | ISO 17678 IDF 202:2010 | Gas liquid chromatography of triglycerides | I | <i>Method proposed for inclusion. See note below.</i> |

The method allows the user to determine whether the milk fat has been adulterated (above a certain threshold). The scope of the method is not limited to butter, but is applicable to milk fat extracted from a range of milk products. In terms of detecting adulteration practices this standard is an important method to have as practically quantitative results cannot be derived from the sterol content of vegetable fats, because they depend on the production and processing conditions. Also, the qualitative determination of foreign fat using sterols is ambiguous. Furthermore, in contrast to the sterol methods, this standard has a broader scope than just detecting adulteration with vegetable fat as it also detects adulteration with fat from animal origin.

| Products | Provisions | Method | Principle | Type | CCMMP comments |
|--|-------------------------------|--|---|-------------|--|
| Cheese (and cheese rind) | Natamycin | ISO 9233-1 IDF 140-1:2007 | Molecular absorption spectrophotometry | III | Type update (Response to CCMAS. ALINORM 08/31/23 para. 59) |
| | | ISO 9233-2 IDF 140-2:2007 | HPLC | II | Type update (Response to CCMAS. ALINORM 08/31/23 para. 59) |
| <u>Cheese</u> | <u>Sodium chloride</u> | <u>ISO 5943 IDF 88:2006</u> | <u>Potentiometry (determination of chloride, expressed as sodium chloride)</u> | II | <i>Method proposed for inclusion</i> |
| Cottage cheese | Fat-free matter | dry ISO 5534 IDF 4:2004 <u>and ISO 1735 IDF 5:2004</u> | Gravimetry, drying at 102 °C | IV | <i>Method update and Principle update</i> |
| | | | <u>Gravimetry (Schmid-Bondzynski-Ratzlaff)</u> | IV | |
| | | | <u>AOAC 926.08 and AOAC 933.05</u> | IV | |
| | | | <u>Gravimetry, drying at 102 °C (vacuum oven)</u> | IV | |
| | | | <u>Gravimetry (modified Mojonier)</u> | IV | |
| | | | Calculation from dry matter <u>content</u> and fat contents | | |
| Cottage cheese | Milk fat | ISO 1735 IDF 5:2004 | Gravimetry (Schmid-Bondzynski-Ratzlaff) | IV | |
| | | <u>AOAC 933.05</u> | <u>Gravimetry (modified Mojonier)</u> | IV | |
| | | ISO 8262-3 IDF 124-3:2005 | Gravimetry (Weibull-Berntrop) | IV | |
| Cheese, unripened including fresh cheese | Protein | ISO 8968-1/ <u>2</u> IDF 20-1/ <u>2</u> :2001/AOAC 991.20 and 991.23 | Titrimetry, Kjeldahl | I | <i>Method update</i> |
| Cream and prepared creams | Milk protein | ISO 8968-1/ <u>2</u> IDF 20-1/ <u>2</u> :2001/AOAC 991.20 | Titrimetry (Kjeldahl) | I | <i>Method update</i> |
| Cream | Milk fat | <u>ISO 2450 IDF 16:2008</u> ISO 16C:1987 / ISO 2450:1999 | Gravimetry (Röse-Gottlieb) | I | <i>Method update</i> |
| Creams lowered in milk fat content | Milk fat | <u>ISO 2450 IDF 16:2008</u> ISO 16C:1987 / ISO 2450:1999 AOAC 995.19 | Gravimetry | I | <i>Method update</i> |

| Products | Provisions | Method | Principle | Type | CCMMP comments |
|---|--|--|--|----------------------------------|--|
| Cream cheese | Dry matter | ISO 5534 IDF 4:2004 <u>AOAC 926.08</u> | Gravimetry drying at 102 °C (<u>forced air oven</u>) <u>Gravimetry drying at 100 °C (vacuum oven)</u> | IV <u>IV</u> | |
| Cream cheese | Moisture on fat free basis | ISO 5534 IDF 4:2004 <u>AOAC 926.08</u> | Gravimetry drying at 102°C (<u>forced air oven</u>) <u>Gravimetry drying at 100 °C (vacuum oven)</u> | IV <u>IV</u> | |
| | | ISO 1735 IDF 5:2004 <u>AOAC 933.05</u> | Gravimetry (Schmid-Bondzynski-Ratzlaff) <u>Gravimetry (modified Mojonner)</u> | IV <u>IV</u> | |
| | | | Calculation from fat content and moisture content | III | |
| <u>Dairy fat spreads</u> | <u>Milk fat purity</u> | <u>ISO 17678 IDF 202:2010</u> | <u>Gas liquid chromatography of triglycerides</u> | <u>I</u> | <i>Method proposed for inclusion See also comments above for Butter</i> |
| Edible casein products | Acids, free | ISO 5547 IDF 91:2008 7 | Titrimetry (aqueous extract) | IV | <i>Method update</i> |
| Edible casein products <u>(rennet casein)</u> | Ash (including P ₂ O ₅) | ISO 5545 IDF 90:2008 7 | Gravimetry, ashing at 825 °C | IV <u>I</u> | <i>Product update and Type update. This should be Type I as the precision figures are available in the standard.</i> |
| <u>Edible casein products (acid casein)</u> | <u>Fixed ash (including P₂O₅)</u> | <u>ISO 5544 IDF 89:2008</u> | <u>Gravimetry, ashing at 825 °C</u> | <u>I</u> | <i>Method Proposed for inclusion: see comment below.</i> |

Both methods ISO 5545|IDF 90:2008 and ISO 5544|IDF 89:2008 are needed to cover the full range of products covered under “Edible casein products”. The adopted method ISO 5545|IDF 90:2008 is suitable for caseins obtained by rennet precipitation and of caseinate, except ammonium caseinate. ISO 5544|IDF 89 needs to be used for acid caseins, ammonium caseinates and their mixtures with rennet casein and with caseinates of unknown type. Magnesium acetate is added to fix the P₂O₅; Magnesium acetate is not needed for rennet caseins as there is enough minerals in this type of product to bind the phosphorus.

| Products | Provisions | Method | Principle | Type | CCMMP comments |
|--|---|--|---|------------------|--|
| Edible casein products | Moisture <u>Water²</u> | ISO 5550 IDF 78:2006 | Gravimetry (drying at 102 °C) | I | <i>Provision update: The Codex standard for Edible casein products refers to water, with a note stating that “The water does not include the water of crystallization of lactose”. Hence the provision “moisture” must be changed to “Water” with a reference to the footnote.</i> |
| Edible casein products | Lead | NMKL 139 (1991) (Codex general method) / AOAC 999.10 | Atomic absorption spectrophotometry | III IV | |
| Evaporated milks | Milk fat | ISO 1737 IDF 13:2008 IDF 13C: 1987 / ISO 1737:1999 | Gravimetry (Röse-Gottlieb) | I | <i>Method update</i> |
| Evaporated milks | Protein | ISO 8968-1/2 IDF 20-1/2:2001 AOAC 945.48H / AOAC 991.20 | Kjeldahl, titrimetry | I | <i>Method update</i> |
| Fermented milks | Protein | ISO 8968-1/2 IDF 20-1/2:2001 AOAC 991.20 | Titrimetry (Kjeldahl) | I | <i>Method update</i> |
| Fermented milks | Milk fat | ISO 1211 IDF 1:2010 IDF 1D:1996 / ISO 1211:1999 / AOAC 905.02 | Gravimetry | I | <i>Method update</i> |
| Fermented milks – Yoghurt and yoghurt products | <i>Lactobacillus delbrueckii</i> subsp. <i>bulgaricus</i> & <i>Streptococcus thermophilus</i> | ISO 7889 IDF 117:2003 | Colony count at 37 °C | I | |
| Fermented milks | Lactic acid (total acidity expressed as lactic acid) | IDF 150:1991 / ISO 11869:1997 | Potentiometry, titration to pH 8.30 Spectrophotometry | IV-I | <i>Type update: This method does not have precision figures. Hence, it needs to be a Type IV method.</i> |
| Fermented milks | Microorganisms constituting | ISO 27205 IDF 149:2010 IDF 149A:1997 (Annex A) | Colony count at 25 °C, 30 °C, 37 °C and 45 °C according to the starter organism | IV | <i>Method update</i> |

| Products | Provisions | Method | Principle | Type | CCMMP comments |
|--|--|---|--|-------------------------------|--|
| | the starter culture | | in question | | |
| <u>Fermented milks</u> | <u>Lactobacillus acidophilus</u> | <u>ISO 20128 IDF 192:2006</u> | <u>Colony count at 37 °C</u> | <u>I</u> | <i>Method proposed for inclusion: The Codex standard 243 has a provision for Lactobacillus acidophilus in Acidophilus Milk</i> |
| <u>Fermented milks</u> | <u>Colony-forming units of yeasts and/or moulds</u> | <u>ISO 6611 IDF 94:2004</u> | <u>Colony-count at 25 °C</u> | <u>IV</u> | <i>Method proposed for inclusion: The Codex standard 243 has a provision for yeasts in connection with Kefir and Kumys.</i> |
| Milk powders and cream powders | Milk fat | <u>ISO 1736 IDF 9:2008</u> IDF 9C:1987 / ISO1736:2000 | Gravimetry (Röse-Gottlieb) | I | <i>Method update</i> |
| Milk powders and cream powders | Protein (in MSNF ¹) | ISO 8968-1/2 IDF 20-1/2:2001 / <u>AOAC 991.20</u> | Titrimetry, Kjeldahl digestion | I | <i>Method update</i> |
| Milk powders and cream powders | Solubility <u>Index</u> | ISO 8156 IDF 129:2005 | Centrifugation | I | <i>Provision update</i> |
| Milk powders and cream powders | Water ² | ISO 5537 IDF 26:2004 ³ | Gravimetry (drying at 87 102 °C) | I <u>IV</u> | <i>Principle update and Type update: the method has been validated on milk powders so it should be Type I instead of Type IV</i> |
| Milk fat products | Milk fat | IDF 24:1964 | Gravimetry (calculation from solids-not-fat <u>content</u> and water content) | IV | <i>Principle update</i> |
| <u>Milk fat products</u> | <u>Milk fat purity</u> | <u>ISO 17678 IDF 202:2010</u> | <u>Gas liquid chromatography of triglycerides</u> | <u>I</u> | <i>Method proposed for inclusion. See note below</i> |
| <i>The method allows the user to determine whether the milk fat has been adulterated (above a certain threshold). The scope of the method is not limited to butter, but is applicable to milk fat extracted from a range of milk products. In terms of detecting adulteration practices this standard is an important method to have as practically quantitative results cannot be derived from the sterol content of vegetable fats, because they depend on the production and processing conditions. Also, the qualitative determination of foreign fat using sterols is ambiguous. Furthermore, in contrast to the sterol methods, this standard has a broader scope than just detecting adulteration with vegetable fat as it also detects adulteration with fat from animal origin.</i> | | | | | |

³ The method has only been validated for milk powders, not for cream powders

| Products | Provisions | Method | Principle | Type | CCMMP comments |
|---|---|--|--|-----------------|---|
| Milk fat products | Water | ISO 5536 IDF 23:2002 29 | Titrimetry (Karl Fischer) | II | <i>Method update</i> <i>Note that in the provision there is no reference to footnote 2, which is correct as Milk fat products contain a negligible amount of MSNF (and thus lactose), and the method measures all water including any water of crystallization of lactose.</i> |
| Milk products obtained from fermented milks heat-treated after fermentation | Protein | ISO 8968-1 2 IDF 20-1 2:2001 AOAC 991.20- 23 | Titrimetry (Kjeldahl) | I | <i>Method update</i> |
| Mozzarella | Milk fat in dry matter – with high moisture | ISO 1735 IDF 5:2004 <u>AOAC 933.05</u> | Gravimetry after solvent extraction | IV <u>IV</u> | <i>Method update and Type update</i> |
| Mozzarella | Milk fat in dry matter – with low moisture | ISO 1735 IDF 5:2004 <u>AOAC 933.05</u> | Gravimetry after solvent extraction | IV <u>IV</u> | <i>Method update and Type update</i> |
| Processed cheese products | Citric acid | ISO/TS 2963 IDF/RM 34:2006 | Enzymatic method | IV | |
| Processed cheese products | Citric acid | AOAC 976.15 | Photometry | III | |
| Processed cheese products | Milk fat | ISO 1735 IDF 5:2004 | Gravimetry (Schmid – Bondzynski-Ratzlaff) | I | |
| Processed cheese products | Phosphate, added (expressed as phosphorus) | IDF 51B:1991 | Calculation from phosphorus content and nitrogen content | IV | <i>Principle update</i> |
| Processed cheese products | Phosphorus | IDF 33C:1987 / ISO 2962:1984 | Spectrophotometry (molybdate-ascorbic acid) | II | |
| Processed cheese products | Salt | ISO 5943 IDF 88:20064 | Potentiometry (determination of chloride, expressed as sodium chloride) | II | <i>Method update</i> |

| Products | Provisions | Method | Principle | Type | CCMMP comments |
|--|--------------------------|---|--|------------------------------|--|
| Sweetened condensed milk | Milk fat | ISO 1737 IDF 13:2008 IDF 13C:1987 / ISO 1737:1999 | Gravimetry (Röse-Gottlieb) | I | Method update |
| Sweetened and Condensed Milks | Protein | ISO 8968-1/2 IDF 20-1/2:2001 / AOAC 945.48H / AOAC 991.20 | Kjeldahl, titrimetry | I | Products update and Method update |
| Whey cheeses by concentration | Milk fat | ISO 1854 IDF 59:2008 IDF 59A:1986 / ISO 1854:1999 | Gravimetry (Röse Gottlieb) | I | Method update |
| Whey cheeses by concentration | Milk fat in dry matter | ISO 1854 IDF 59:2008 IDF 59A:1986 / ISO 1854:1999 and ISO 2920 IDF 58:2004 | Gravimetry (Röse Gottlieb) Gravimetry, drying at 88 °C Calculation from fat content and dry matter content | I I | Method update |
| Whey powders | Ash | ISO 5545 IDF 90:20087 | Furnace, 825 °C | IV | Method update |
| Whey powders | Milk fat | ISO 1736 IDF 9:2008 IDF 9C:1987 / ISO 1736:2000 | Gravimetry (Röse-Gottlieb) | I | Method update |
| Whey powders | Milk protein | ISO 8968-1/2 IDF 20-1/2:2001 / AOAC 991.20 | Titrimetry (modified Kjeldahl) | I | Method update |
| Whey powders | Protein (total N x 6.38) | ISO 8968-1/2 IDF 20-1/2:2001 / AOAC 991.20 IDF 92:1979 / ISO 5549:1978 | Kjeldahl, titrimetry Titrimetry, Kjeldahl digestion | I IV | Method update, Principle update And type update. |
| Whey powders | Water ² | ISO 5537 IDF 26:2004 / AOAC 927.05 | Gravimetry (drying at 87 ¹⁰² °C) | I | Provision update (note) and Principle update |

PART B – METHODS OF SAMPLING BY ALPHABETICAL ORDER OF COMMODITY CATEGORIES AND NAMES

| Commodity Standard | Method of Sampling | Notes | CCMMP comments |
|-------------------------------|--|---|-----------------------|
| Milk and Milk products | | | |
| Milk products | IDF 50 ¹⁰ ISO 707 ISO 707 IDF 50:2008 | General instructions for obtaining a sample from a bulk | <i>Method update</i> |
| Milk products | IDF 113 ISO 5538:2004 ISO 5538 IDF 113:2004 | Inspection by attributes | <i>Method update</i> |
| Milk products | IDF standard 136A:1992 ISO 8197:1988 | Inspection by variables | <i>Method update</i> |

¹⁰~~Draft standard which is publicly available~~