

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
Organization of the
United Nations



World Health
Organization

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Agenda Item 3.2

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ORIGINAL LANGUAGE

**JOINT FAO/WHO FOOD STANDARDS PROGRAMME
CODEX COMMITTEE ON METHODS OF ANALYSIS SAMPLING**

40th Session

Budapest, Hungary, 27 - 31 May 2019

**ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS AND SAMPLING PLANS
IN CODEX STANDARDS
DAIRY WORKABLE PACKAGE**

Information and comments provided by AOAC/IDF/ISO and IDF/ISO

AOAC/IDF/ISO

RECOMMENDATIONS

- Milk products – Iron: new methods proposed, and change of type of current method
- Edible casein products – Copper: new methods proposed, and change of type of current method
- Milkfat products – Copper: new methods proposed, and change of type of current method
- Milkfat products - Iron: new method proposed
- Emmental – Calcium: new methods proposed, and change of type of current method

Milk products – Iron

Commodity	Provision	Method	Principle	Type
Milk products	Iron	NMKL 139 AOAC 999.11 (Codex general method)	Atomic absorption spectrophotometry	III
<u>Milk products</u>	<u>Iron</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP mass spectrometry</u>	<u>II</u>
<u>Milk products</u>	<u>Iron</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP emission spectroscopy</u>	<u>III</u>
Milk products	Iron	AOAC 984.27	Inductively Coupled Plasma-optical emission spectrophotometry	III

AOAC/IDF/ISO propose to endorse the AOAC 2015.06 / ISO 21424 | IDF 243 (ICP-MS) as Type II and AOAC 2011.14 / ISO 15151 | IDF 229 (ICP-AES) as Type III, and changing the current type II (AOAC 999.11) to type III.

The rationale for change is the availability of validated data on several dairy matrices (the identical matrices were tested for the AOAC 2015.06 / ISO 21424 | IDF 243 and AOAC 2011.14 / ISO 15151 | IDF 229 collaborative studies and thus serve as a check on each other).

AOAC/IDF/ISO agree with removal of AOAC 984.27.

Iron	Level (mg/kg)	RSD _r (%)	RSD _R (%)	HorRat
AOAC 999.11				
Liver paste	24.3	-	11	1.12
Minced fish/milk powder	3.99	11	14	1.08
Wheat bran	124	-	11	1.43
Simulated diets	216	8.2	11	1.55
AOAC 2015.06 / ISO 21424 IDF 243				
Whole milk liquid	0.171	3.71	24.00	1.17
Whey powder	3.00	5.10	27.40	2.01
Whey protein concentrate	6.557	1.38	2.60	0.22
Whole Milk powder (as RTF 25g in 200g water)	0.274	1.37	5.53	0.28
Butter	0.279	18.70	37.90	2.07
AOAC 2011.14 / ISO 15151 IDF 229				
Whole milk liquid	0.16	16	35	1.69
Whey powder	3.38	37	59	4.4
Whey protein concentrate	6.62	3.13	16.3	1.35
Whole Milk powder (as RTF 25g in 200g water)	0.33	21	49	2.58
Butter	0.3	-	-	-

Edible casein products – Copper

Commodity	Provision	Method	Principle	Type
Edible casein products	Copper	AOAC 985.35	Atomic absorption spectrophotometry	III
Edible casein products	Copper	ISO 5738 IDF 76	Colorimetry (diethyldiethiocarbamate)	III
<u>Edible casein products</u>	<u>Copper</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP mass spectrometry</u>	<u>II</u>
<u>Edible casein products</u>	<u>Copper</u>	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP emission spectroscopy</u>	<u>III</u>

Precision figures for these methods for edible caseins products are

- *AOAC 985.35: not validated on the specific matrix; however, experts reported that it is being used for edible casein products.*
- *ISO 5738|IDF 76: Repeatability: see below table*
- *AOAC 2015.06 / ISO 21424 | IDF 243: not validated on matrix, but milk powder see below table*
- *AOAC 2011.14 / ISO 15151 | IDF 229: not validated on matrix, but milk powder see below table*

Looking at the data below, ISO 5738|IDF 76 is validated on the matrix, however experts reported it is not used and ICP is used instead. Also, the ISO 5738|IDF 76 uses potassium cyanide.

The ISO/IDF method has a LOQ of 0.05 mg/kg vs. 0.005 mg/kg for ICP-MS. The accuracy of ICP-MS is also verified by the 2011.14 data on the same samples. Also, there might be advantages to making the same method as Type II for all of these cases.

Therefore, and since milk powder is considered close enough to edible caseins, the proposal is to recommend AOAC 2015.06 / ISO 21424 | IDF 243 as Type II, and therefore ISO 5738|IDF 76 as Type III as well as AOAC 2011.14 / ISO 15151 | IDF 229.

Copper	Level (mg/kg)	RSD _r (%)	RSD _R (%)	HorRat
ISO 5738 IDF 76				
Milk	0.03	4.76	11.90	0.44
Skimmed milk	0.03	4.76	11.90	0.44
Whole milk powder	0.3	5.95	11,9	0.62
Skimmed milk powder	0.8	2.23	4.46	0.27
Caseins, caseinates and coprecipitates	1.8	2.98	5.95	0.41
Butter	0.04	3.57	17.86	0.69
Butterfat	0.04	3.57	17.86	0.69
AOAC 2015.06 / ISO 21424 IDF 243				
Whole milk liquid	0.052	0.97	5.49	0.22
Whey powder	0.345	6.31	28.59	1.52
Whey protein concentrate	1.172	1.40	4.16	0.27
Whole Milk powder (as RTF 25g in 200g water)	0.040	4.66	7.47	0.29
Butter	0.042	36	99	3.86
AOAC 2011.14 / ISO 15151 IDF 229				
Whole milk liquid	0.07	4.1	78.2	3.27
Whey powder	0.49	8.4	48.5	2.72
Whey protein concentrate	1.22	6.24	20.7	1.33
Whole Milk powder (as RTF 25g in 200g water)	0.06	20.7	71.3	2.91
Butter	0.023	-	-	-

Milkfat products – Copper

Commodity	Provision	Method	Principle	Type
Milkfat products	Copper	ISO 5738 IDF 76	Photometry, diethyldithiocarbamate	II
<u>Milkfat products</u>	<u>Copper</u>	<u>AOAC 960.40</u>	<u>Photometry, diethyldithiocarbamate</u>	<u>IV</u>

Separate lines for the current methods: Keep IDF/ISO method as type II and add AOAC as type IV.

Precision figures for these methods for copper in milkfat products are shown above.

Indeed ISO 5738 | IDF 76 has been validated butter and butterfat at level of 0.04 mg/kg. Provision for maximum level for copper in Milkfat products in STAN 280: Copper: max level of 0,05 mg/kg.

Both AOAC 2015.06 / ISO 21424 | IDF 243 and AOAC 2011.14 / ISO 15151 | IDF 229 are not fit for purpose for butter (compared to the ISO 5738 | IDF 76), which is closest to matrix Milk fat products (see validation data above).

Milkfat products – Iron

Commodity	Provision	Method	Principle	Type
<u>Milkfat products</u>	<u>Iron</u>	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP mass spectrometry</u>	<u>II</u>

AOAC/IDF/ISO propose to add an entry for iron in milkfat products. CODEX STAN 280 lists a max level of 0.2 mg/kg.

AOAC 2015.06 / ISO 21424 | IDF 243 has an LOQ of 0.073 mg/kg

<i>Iron</i>	<i>Level mg/kg</i>	<i>RSDr %</i>	<i>RSDR %</i>	<i>HorRat</i>
<i>AOAC 2015.06 / ISO 21424 IDF 243</i>				
<i>Whole milk liquid</i>	<i>0.171</i>	<i>3.71</i>	<i>37.60</i>	<i>1.82</i>
<i>Whey powder</i>	<i>3.00</i>	<i>5.10</i>	<i>27.40</i>	<i>2.01</i>
<i>Whey protein concentrate</i>	<i>6.557</i>	<i>1.38</i>	<i>2.60</i>	<i>0.22</i>
<i>Whole Milk powder (as RTF 25g in 200g water)</i>	<i>0.274</i>	<i>1.37</i>	<i>5.53</i>	<i>0.28</i>
<i>Butter</i>	<i>0.279</i>	<i>18.70</i>	<i>37.90</i>	<i>2.07</i>

Emmental – Calcium

Commodity	Provision	Method	Principle	Type
Emmental	Calcium ≥ 800mg/100g	ISO 8070 IDF 119	Flame atomic absorption	<u>III</u> IV
Emmental	Calcium ≥ 800mg/100g	<u>AOAC 2015.06 / ISO 21424 IDF 243</u>	<u>ICP mass spectrometry</u>	<u>II</u>
Emmental	Calcium ≥ 800mg/100g	<u>AOAC 2011.14 / ISO 15151 IDF 229</u>	<u>ICP emission spectroscopy</u>	<u>III</u>

AOAC/IDF/ISO propose to retype current method to type III (validated on cheese), and to add two AOAC/IDF/ISO methods as type II and type III respectively.

Rationale:

Precision figures for these methods for calcium in cheese are summarized below.

As precision values are similar, it could be proposed to have ICP-MS as type II and the other two Type III, in line with infant formula proposal to have MS as Type II.

Calcium	Level g/kg	RSD _r %	RSD _R %	HorRat
ISO 8070 IDF 119	4.04	1.6	6.4	1.4
	2.73	2.2	7.4	1.5
AOAC 2015.06 / ISO 21424 IDF 243	4.98	1.42	5.22	1.18
AOAC 2011.14 / ISO 15151 IDF 229	4.94	1.69	3.88	0.87

IDF/ISO

APPENDIX I - list of methods that have been reviewed and proposed changes to CXS 234

PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

Commodity	Provision	Method	Principle	Type
<u>Blend of sweetened condensed skimmed milk and vegetable fat</u>	<u>Milk solids-not-fat</u> ^{Error! Bookmark not defined.} (MSNF)	<u>ISO 6734 IDF 15 and ISO 1737 IDF 13 and ISO 2911 IDF 35</u>	<u>Gravimetry, drying at 102 °C</u> <u>Calculation from total solids content, fat content and sucrose content</u> <u>Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Polarimetry</u>	<u>IV</u>
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk solids-not-fat ^{Error! Bookmark not defined.} (MSNF)	ISO 6734 IDF 15 <u>and ISO 1737 IDF 13 and ISO 2911 IDF 35</u>	Gravimetry, drying at 102 °C <u>Calculation from total solids content, fat content and sucrose content</u> <u>Gravimetry, drying at 102 °C and Gravimetry (Röse-Gottlieb) and Polarimetry</u>	IV
<i>IDF/ISO comment: harmonization of description using calculation</i>				
Butter	Vegetable fat (sterols)	ISO 12078 IDF 159	Gas chromatography	II
Butter	Vegetable fat (sterols)	ISO 18252 IDF 200	Gas chromatography	III
Cheese	Citric acid	ISO/TS 2963 IDF/RM 34	Enzymatic method	IV
<i>IDF/ISO comment: 3 lines above were missing in Appendix 1 (but are included in Appendix III)</i>				
Cheese	Milkfat	ISO 1735 IDF 5	Gravimetry (Schmid-Bondzynski-Ratzslaff)	I

Commodity	Provision	Method	Principle	Type
<u>Cheeses, individual</u>	<u>Milkfat in dry matter</u>	<u>ISO 5534 IDF 4 and ISO 1735 IDF 5</u>	<u>Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski- Ratzlaff)</u>	I
<u>Cheeses in brine</u>	<u>Milkfat in dry matter (FDM)</u>	<u>ISO 5534 IDF 4 and ISO 1735 IDF 5</u>	<u>Calculation from dry matter content and fat content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski- Ratzlaff)</u>	I
Cottage cheese	Fat-free dry matter	ISO 5534 IDF 4 and ISO 1735 IDF 5	Calculation from dry matter content and fat content Gravimetry, drying at 102 °C and Gravimetry (Schmid-Bondzynski- Ratzlaff)	I
<i>IDF/ISO comment: Harmonization of description</i>				
Dairy fat spreads	Vegetable fat (sterols)	ISO 12078 IDF 159	Gas chromatography	II
Dairy fat spreads	Vegetable fat (sterols)	ISO 18252 IDF 200	Gas chromatography	III
<i>IDF/ISO Comment: 2 lines above are included in appendix III but were missing here.</i>				
Milkfat products	Milkfat purity	ISO 17678 IDF 202	Calculation from determination of triglycerides by gas chromatography	I
<i>IDF/ISO comment: line above is included twice, one needs to be removed.</i>				
Mozzarella	Milkfat in dry matter – with high moisture	ISO 1735 IDF 5	Gravimetry after solvent extraction	I
<i>IDF/ISO comment: line above needs to be deleted, as replaced by one reflecting calculation.</i>				
<u>Sweetened Condensed Milks</u>	<u>Milk Protein in MNSFMSNFError! Bookmark not defined.</u>	<u>ISO 6734 IDF 15 and ISO 1737 IDF 13 and ISO 8968-1 IDF 20-1</u>	<u>Calculation from total solids content, fat content and protein content Gravimetry, drying at 102°C and Gravimetry (Röse-Gottlieb) and Titrimetry (Kjeldahl)</u>	I
<i>IDF/ISO comment: typo in MSNF and harmonization of description</i>				

APPENDIX II - List of methods and comments where multiple comments were received and final format requires further clarification

Blend of skimmed milk and vegetable fat in powdered form	Water ¹	ISO 5537 IDF 26	Gravimetry, drying at 87 °C	
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water ^{Error! Bookmark not defined.}	ISO 5537 IDF 26	Gravimetry, drying at 87 °C	

ISO 5537 | IDF 26 had only been validated for whole and skimmed milk powder and in the 2010 CCMMP Final report this method was referred to CCMAS as TYPE IV.

The 2010 CCMAS Final Report endorsed the method for this commodity as TYPE I and IDF expert review has found that method is applicable to many powders and would support matrix extension. This has been captured in the Update and captured here for information.

IDF/ISO comments: applicability on blends of skimmed milk and vegetable fat in powdered form is confirmed upon based on gained experience with the method with other dairy powders and expert judgement.

<u>Cottage cheese (for samples containing lactose up to 5%)</u>	<u>Milkfat in dry matter</u>	<u>ISO 5534 IDF 4 and ISO 1735 IDF 5</u>	<u>Calculation from dry matter content Gravimetry, drying at 102°C and Gravimetry (Schmid-Bondzynski-Ratzlaff)</u>	<u> </u>
<u>Cottage cheese (for samples containing lactose over 5% or with non-dairy ingredients)</u>	<u>Milkfat in dry matter</u>	<u>ISO 5534 IDF 4 and ISO 8262-3 IDF 124-3</u>	<u>Calculation from dry matter content Gravimetry, drying at 102°C and Gravimetry (Weibull-Berntrop)</u>	<u> </u>

Reviewer \question about matrix, but the method should extend to the matrix.

IDF/ISO comments: IDF confirms that 2 methods are necessary to cover the scope of the commodity standard for cottage cheese (STAN 273).

¹ Water content excluding the crystallized water bound to lactose (generally known as “moisture content”)

APPENDIX III - List containing proposed edits/actions that require further decisions**PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES**

Commodity	Provision	Method	Principle	Type
Milk and Milk Products				
Creams, Whipped Creams and Fermented Creams	Milk solids-not-fat (MSNF)	ISO 3727-2 IDF 80-2 AOAC 920.116	Gravimetry	I
The ISO/IDF method and the AOAC method are not identical and AOAC recommends removal of AOAC 920.116. ISO 3727-2 IDF 80-2 has not been validated for these matrices, ISO 3727-2 IDF 80-2 has been validated for butter.				
IDF/ISO comment: while the method has not been validated on creams, the method has demonstrated to perform well on creams.				
Fermented milks	Lactobacillus acidophilus	ISO 20128 IDF 192	Colony count at 37 °C	I
Reviewer (Hungary) recommends change to the provision, but unclear exactly what should be written?				
IDF/ISO comments: Provision is as in STAN 243, and IDF confirms it is fit for purpose.				
Fermented milks	Milkfat	ISO 1211 IDF 1 / AOAC 989.05	Gravimetry (Röse-Gottlieb)	I
Two methods are not identical, so one will have to be removed and the other retained. Both methods are fit for purpose.				
IDF/ISO recommend retaining ISO 1211 IDF 1 which has been revised in 2010, whereas AOAC 989.05 was last revised in 1996.				
Whey powders	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
AOAC recommends removal, a new method will need to be identified as a replacement.				
<u>Whey powders</u>	<u>Lead</u>	<u>ISO/TS 6733 IDF/RM 133</u>		<u>IV</u>
IDF/ISO recommend ISO/TS 6733 IDF/RM 133 as a Type IV.				
Codex limit for milk and secondary milk products (STAN 193) is 0.02 mg/kg.				
This Technical Specification describes a method for the quantitative determination of the total lead content in milk and milk products. The detection limit of the method is 0,001 mg/kg for liquid products and 0,01 mg/kg for solid products if dry ashing is applied.				

APPENDIX IV - List of methods not reviewed by any member of the EWG

<i>Commodity</i>	<i>Provision</i>	<i>Method</i>	<i>Principle</i>	<i>Type</i>
Milk and Milk Products				
Milk and Milk Products	Melamine	ISO/TS 15495 IDF/RM 230	LC-MS/MS	IV
Milk and Milk Products	Melamine	EN 16858:2017	LC-MS/MS	II
<i>IDF/ISO recommends a validated method as type II, EN 16858:2017 Foodstuffs - Determination of melamine and cyanuric acid in foodstuffs by liquid chromatography and tandem mass spectrometry. The EN method is under IDF/ISO consideration to become an international standard and will be brought to CCMAS when published.</i>				
Butter	Milkfat	ISO 17189 IDF 194	Gravimetry Direct determination of fat using solvent extraction	I
<i>IDF/ISO confirm line above (provision in STAN 279)</i>				
Butter	Salt	ISO 15648 IDF 179	Potentiometry (determination of chloride, expressed as sodium chloride)	II
<i>IDF/ISO confirm line above (provision in STAN 279)</i>				
Butter	WaterError! Bookmark not defined.	ISO 37274-1 IDF 80-1	Gravimetry	I
<i>IDF/ISO confirm line above (provision in STAN 279)</i>				
Dairy fat spreads	Total fat	ISO 17189 IDF 194	Gravimetry Direct determination of fat using solvent extraction	I
<i>I IDF/ISO confirm line above (provision in STAN 253)</i>				
Edible casein products	Acids, free	ISO 5547 IDF 91	Titrimetry (aqueous extract)	IV
<i>IDF/ISO confirm line above (provision in STAN 290)</i>				
Edible casein products	Lactose	ISO 5548 IDF 106	Photometry (phenol and H ₂ SO ₄)	IV
<i>IDF/ISO confirm line above (provision in STAN 290)</i>				
Edible casein products	Milkfat	ISO 5543 IDF 127	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
<i>IDF/ISO confirm line above (provision in STAN 290) and suggestion correction of typo on 'Ratzlaff'.</i>				
Edible casein products	pH	ISO 5546 IDF 115	Electrometry	IV
<i>IDF/ISO confirm line above (provision in STAN 290) no validation data referenced in standard</i>				

<i>Commodity</i>	<i>Provision</i>	<i>Method</i>	<i>Principle</i>	<i>Type</i>
Milk and Milk Products				
Fermented milks	Dry matter (total solids) ^{Error!} Bookmark not defined.	ISO 13580 IDF 151	Gravimetry (drying at 102 °C)	I
IDF/ISO confirm line above. No provision for milk solids in STAN 243 but in GSLPF (STAN 1)				
Fermented milks	Total acidity expressed as percentage of lactic acid	ISO/TS 11869 IDF/RM 150	Potentiometry, titration to pH 8.30	I
IDF/ISO confirm line above (provision in STAN 243)				
Fermented milks	Microorganisms constituting the starter culture	ISO 27205 IDF 149 (Annex A)	Colony count at 25 °C, 30 °C, 37 °C and 45 °C according to the starter organism in question	IV
IDF/ISO confirm line above (provision in STAN 243)				
Milk powders and cream powders	Scorched particles	ISO 5739 IDF 107	Visual comparison with standard disks, after filtration	IV
IDF/ISO confirm line above (provision in STAN 207)				
Milk powders and cream powders	Solubility Index	ISO 8156 IDF 129	Centrifugation	I
IDF/ISO confirm line above (provision in STAN 207)				
Whey powders	Moisture, "Free"	ISO 2920 IDF 58	Gravimetry (drying at 88°C ±2°C)	IV
IDF/ISO recommend deletion of this line. There is already a line/method for determination of water in whey powders. The method ISO 2920 IDF 58 is for determination of dry matter in whey cheese, but milkfat in dry matter is also covered by another line.				