CODEX ALIMENTARIUS COMMISSION

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World Health Organization

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Agenda Item 4.1 **MAS-CRD/06**

ORIGINAL LANGUAGE ONLY

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS SAMPLING

REVIEW OF METHODS OF ANALYSIS IN CXS234: DAIRY WORKABLE PACKAGE (CX/MAS 21/41/4)

Comments from IDF and ISO

GROUP 1: Removal of the commodity Milk Products (see Table G1.1 in Appendix I of the eWG report)

IDF and ISO support the eWG recommendation to remove the commodity "milk products" from CXS 234.

GROUP 2: Numeric Criteria in place of Methods for Iron, Copper and Lead

IDF and ISO are of the view that Type II methods are needed for the purpose of dispute resolution and that in that case, the best candidate method must be designated as such, as per the comments and methods submitted in 2020 on page 11 of the document "Revision of the recommended methods of analysis and sampling (CXS 234-1999) - Dairy package - Comments in reply to CL 2020/029/OCS-MAS"[1].

A summary of the comments and methods is copied below for ease of reference:

Commodity	Provision	Method	Principle	Codex STAN	Proposed Type
Edible casein products	Iron	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	290	II
Edible casein products	Iron	AOAC 2011.14 / ISO 15151 | IDF 229	ICP emission spectroscopy	290	III
Milkfat products	Iron	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	280	II
Milkfat products	Copper	AOAC 2015.06 / ISO 21424 | IDF 243	ICP mass spectrometry	280	II
Milkfat products	Copper	ISO 5738|IDF 76	Photometry, diethyldithiocarbamate	280	~~II~~III
Milkfat products	Copper	AOAC 960.40	Photometry, diethyldithiocarbamate	280	IV

If the numeric criteria are endorsed, and consistent with previous AOAC/IDF/ISO submissions for new methods, IDF/ISO proposes the following methods to be listed as 'suggested methods meeting the criteria':

Milkfat Products (CXS 280-1973)

- Copper: IDF/ISO propose AOAC 2015.06 / ISO 21424 | IDF 243 (currently proposed as new type II method)
- Iron: currently no method is listed in CXS 234, and no method fitting the criteria above.

Edible Casein Products (CXS 290-1995)

- Iron: IDF/ISO proposes AOAC 2015.06 / ISO 21424 | IDF 243 (currently proposed as new type II method)

[1] http://www.fao.org/fao-who-codexalimentarius/sh-proxy/en/?lnk=1&url=https%253A%252F%252Fworkspace.fao.org%252Fsites%252Fcodex%252FMeetings%252FCX-715-41%252FWorking%2Bdocuments%202020%252Fma41_04_add1e.pdf

- Copper: IDF/ISO proposes AOAC 2015.06 / ISO 21424 | IDF 243 (currently adopted as type II method)

Numeric Criteria for lead in Butter, edible casein products and whey powders

- Butter: AOAC 972.25 listed in CXS 234, it is not fit for purpose. - No method identified that meet the proposed criteria.
- Edible casein products: currently several methods are listed as type IV in CXS 234. None fit the criteria to our knowledge. If no method fit the numeric criteria, IDF requests to keep the current methods as listed.
- Whey powders: currently no method listed in CXS 234. No identified method that meet the proposed criteria.

IDF notes that the European Committee for Standardization is currently working for a horizontal method for determination of various elements, including lead, in foods with ICPS-MS (preliminary work under CEN/TC 275/W10).

GROUP 3: ISO 5537 | IDF 26 (Dried milk — Determination of moisture content)

IDF and ISO support the eWG recommendation to retain ISO 5537 | IDF 26 as is and offers additional information in annex.

IDF and ISO support the eWG recommendation to replace water with moisture if the commodity standard could also be changed and note that footnotes are already in CXS234 to explain the discrepancy.

However in one case the footnote is not appropriate and shall be removed. There is separate provision for water in milk fat products for which CXS-234 lists ISO 5536|IDF 23 as type II method that comes with a footnote explaining that moisture content is meant. However, both the Codex standard for milkfat products (CXS 280) the IDF/ISO standard refer to water determination, therefore the note is not necessary.

Milk fat products	Water[16]	ISO 5536 | IDF 23	Titrimetry (Karl Fischer)	II

GROUP 4: AOAC 965.33 Peroxide Value of Oils and Fats ISO 3976 | IDF 74 (Milk fat — Determination of peroxide value)

IDF and ISO support the EWG recommendation to retain ISO 3976 | IDF 74 as a Type I method and AOAC 965.33 be revoked (Table G4.1 Appendix I).

GROUP 5 ISO 27205 | IDF 149 Fermented milk products – Bacterial starter cultures – Standard of identity

IDF and ISO agree that revocation of the method is not a suitable solution, and that there is discrepancy between the provision listed in CXS 234 (Microorganisms constituting the starter culture), the Provision listed in the commodity standard (CXS 243-2003) and the scope of ISO 27205 | IDF 149.

After consultation among experts, IDF and ISO would agree with the proposal from NZ to modify the provision to 'Bacteria in fermented milk deriving (or originating) from starter culture'.

This would align with (part of) the provision in commodity standard in that it covers bacteria (but not yeasts that are used in some fermented milks), including both (a) the defined bacteria and (b) those mentioned in a label claim.

The methods listed in Annex A of ISO 27205 | IDF 149 have been validated for this purpose and so the method type could be upgraded.

GROUP 6 ISO 17678|IDF 202 Milk and milk products – Determination of milk fat purity by gas chromatographic analysis of triglycerides

IDF and ISO support the eWG recommendation to retain ISO 17678 | IDF 202 (Table 6.1 Appendix I).

Methods Not Previously Reviewed: outstanding question: Total Acidity in Fermented Milks (ISO/TS 11869 | IDF/RM 150): Should this be Type I, because of a conversion factor in the method?

There is no reproducibility data, therefore IDF and ISO recommend listing this method as type IV.

The conversion factor used relates to expressing the results in terms of lactic acid. It relates to the relative mass of lactic acid (90.08).

Annex – CCMAS Agenda item 4.1: Review of Dairy Methods – Determination of Moisture Content in Dried Milk Products Comments in CX/MAS 20/41/4 Add.1 on Group 3 – Water in milk products

Additional information from IDF and ISO

General

In CXS 234-1999 methods are listed for the determination of the moisture content[2] in dried milk products as well as methods for the determination of moisture content in other dairy matrices. The aim with this Codex provision is to determine the "free water" content, the part of the water content that is relevant for the microbiological and chemical keeping quality of dried milk products. Where the aim is to determine the total water content, it is more appropriate to apply a method based on the so-called Karl Fisher principle. Also for that purpose, a number of methods are listed in CXS 234-1999.

For the determination of the moisture content, CCMAS has since then also endorsed ISO 5537 | IDF 26:2004 as a Type I method for:

- blends of skimmed milk and vegetable fat in powdered form, in reduced fat blends of skimmed milk powder and vegetable fat in powdered form, and in milk powders and cream powders in 2006 (ALINORM 06/29/23);
- whey powders in 2008 (ALINORM 08/31/23);
- dairy permeate powders in 2018 (REP 18/MAS).

CX/MAS 20/41/4 Add.1 comments

In CX/MAS 20/41/4 Add.1 Uruguay has repeated earlier expressed concerns (see REP19/MAS) on inclusion of ISO 5537 | IDF 26:2004 in CXS 234-1999 as the Type 1 method for the determination of moisture content in dried milk products:

1) The problematic access to the equipment and test conditions defined in ISO 5537 | IDF 26:2004 in South-American countries, more specifically the limited capacity of the oven, issues with availability of supplies and specific packing, the generation of waste to the environment, and issues with demonstrating adequate air quality and air flow with use of the equipment.
2) The availability of alternative methods. In CX/MAS 20/41/4 Add. 1 a method equivalent to IDF 26A:1993 is proposed. This is supported with single lab validation data.

Reply of IDF and ISO

Ad 1) Some laboratories have built the required equipment for application of ISO 5537 | IDF 26:2004 in-house, most have purchased the vacuum drying oven from commercial suppliers. Until nowadays, the equipment is available in the market and is not expensive. Capacity can be increased by purchasing extra units. Special utensils are limited to hard polypropylene columns with polyethylene filters. These utensils are clearly described in the method and available from multiple suppliers. The method also provides guidance on controlling and checking the air flow and the air quality. IDF through its network offers to provide expert advice and guidance to users who have problems with the execution of the method.

Ad 2) ISO 5537 | IDF 26:2004 was developed more than 20 years ago to overcome the shortcomings of IDF 26A:1993. This was triggered by the observation that the then reference method for the determination of moisture in dried milk products in practice exhibited unacceptable high values for repeatability and reproducibility. Reasons for the discrepancies between laboratories was the lack of control on the critical points in the operation of drying oven methods with dried milk products:

- Intensive air ventilation is essential to obtain an accurate and uniform temperature inside the drying oven. However, the force of ventilation that can be applied is limited because with IDF 26:1993 the test portion is dried in an open dish.
- The relative air humidity in the testing environment. For instance, tests carried out showed a systematic difference of 0.3% (m/m) between the moisture content of a whole milk powder determined in winter (lower relative humidity of the air in the testing environment) and summer (higher relative humidity of the air in the testing environment.

These critical points were counteracted with the development of a vacuum drying oven, wherewith test portions of dried milk could be dried under much better standardized conditions, i.e. uniform temperature, constant air flow and relative humidity. Testing conditions were optimized to arrive at on average equivalent results with IDF 26A:1993 (de Knegt & van den Brink, 1998).

[2] Relative weight loss of a test sample upon drying until constant mass is obtained. For dried milk products this coincides with the water content excluding the crystallized water bound to lactose.

The performance of this revised method was verified in a multi-lab validation study with a comparison of the performance against IDF 26A:1993 on three skimmed milk powder samples and three whole milk powder samples (Grobecker et al., 1999). The obtained precision data for the two methods from this study are summarized in Table 1.

From these data it was concluded that best precision was achieved with the revised method, which was subsequently published as ISO 5537 | IDF 26:2004. Especially with reproducibility ISO 5537 | IDF 26:2004 showed much more favorable performance than IDF 26A:1993. The calculated mean value with ISO 5537 | IDF 26:2004 was close to the calculated mean value from the results with IDF 26A:1993, which was in accordance with the aim during method development.

In addition to this earlier study IDF and ISO/TC 34/SC 5 have during 2020 conducted a complementary multi-laboratory validation study on the performance of ISO 5537|IDF 26:2004 with whey powder, dairy permeate powder, cream powder and powdered infant formula. The results of this study are summarized in Table 2.

The results show that obtained values for repeatability and reproducibility with these other dried dairy matrices are in the same range as with the earlier study on skimmed milk powder and whole milk powder and underpin the applicability of the method for the full range of dried milk products.

A publication on this complementary multi-laboratory validation study is in preparation. The results will be included in ISO 5537 | IDF 26 in the near future.

Recommendation to CCMAS41

To support the eWG recommendation to keep ISO 5537 | IDF 26:2004 as Codex Type I method for checking compliance with existing Codex provisions on the moisture content in the full range of dried milk products.

References

Grobecker, K.H., Rückold, S. & Anklam, E. 1999. Determination of the water content in milk powder: Report of a collaborative study performed in the period June-July 1999. European Commission Report (August 1999), EU-DG JRC-IRMM & IHCP.

International Dairy Federation. 1993. IDF Provisional Standard 26A:1993. Dried Milk and Dried Cream. Determination of Water Content.

International Organization for Standardization/International Dairy Federation. 2004. ISO 5537 | IDF 26 - Dried milk – Determination of Moisture Content (Reference Method).

Knegt, R.J. & van den Brink, H. 1998. Improvement of the Drying Oven Method for the Determination of the Moisture Content of Milk Powder. Int. Dairy J. 8, p 733-738.

Table 1. Results of a multi-lab comparison study with ISO 5537 | IDF 26:2004 and IDF 93A:1993 on 3 skimmed milk powders (SMP) and 3 whole milk powders (WMP) as conducted by Grobecker et al., 1999.

	ISO 5537 | IDF 26:2004						IDF 26A:1993					
	SMP 1	SMP 2	SMP 3	WMP 1	WMP 2	WMP 3	SMP 1	SMP 2	SMP 3	WMP 1	WMP 2	WMP 3
Number of participating labs after eliminating outliers	8	8	8	8	8	8	8	8	8	8	8	8
Mean value, % m/m	3.62	3.57	3.93	3.16	2.52	2.38	3.72	3.74	4.02	3.21	2.57	2.44
Repeatability standard deviation s_r% m/m	0.052	0.085	0.053	0.035	0.045	0.049	0.081	0.092	0.082	0.057	0.069	0.080
Coefficient of variation of repeatability, %	1.44	2.38	1.34	1.11	1.80	2.06	2.18	2.46	2.04	1.78	2.68	3.28
Repeatability limit, r (2,8*s_r), % m/m	0.146	0.238	0.148	0.098	0.126	0.137	0.227	0.258	0.230	0.160	0.193	0.224
Reproducibility standard deviation s_R% m/m	0.058	0.097	0.074	0.060	0.055	0.098	0.177	0.175	0.167	0.157	0.155	0.150
Coefficient of variation of reproducibility, %	1.61	2.69	1.89	1.89	2.19	4.11	4.76	4.68	4.15	4.89	6.03	6.15
Reproducibility limit, R (2,8*s_R), % m/m	0.162	0.272	0.207	0.168	0.154	0.274	0.496	0.490	0.468	0.440	0.434	0.420

Table 2. Results of a multi-lab validation study with ISO 5537 | IDF 26:2004 on rennet whey powder (RWP), acid whey powder (AWP) whey permeate powder (WPP),), milk permeate powder (MPP), cream powder (CP) and powdered infant formula (PIF) as conducted by IDF/ISO in 2020. To be published.

	ISO 5537 | IDF 26:2004					
	RWP	AWP	WPP	MPP	CP	PIF
Number of participating labs after eliminating outliers	11	12	12	12	11	12
Mean value, % m/m	2.06	2.57	1.52	1.52	2.57	1,87
Repeatability standard deviation s_r% m/m	0.026	0.030	0.037	0.046	0.024	0.039
Coefficient of variation of repeatability, %	1.27	1.16	2.46	3.01	0.93	2.07
Repeatability limit, r (2,8*s_r), % m/m	0.073	0.083	0.104	0.128	0.067	0.109
Reproducibility standard deviation s_R% m/m	0.064	0.098	0.083	0.102	0.068	0.072
Coefficient of variation of reproducibility, %	3.10	3.82	5.44	6.71	2.66	3.85
Reproducibility limit, R (2,8*s_R), % m/m	0.179	0.274	0.231	0.285	0.191	0.202