CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda Items 3.1, 4.1, 4.2, 5, 8, 9 **CRD19**

ORIGINAL LANGUAGE ONLY

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

43rd Session
Budapest, Hungary

13 – 18 May 2024

(Comments of Uruguay)

Agenda Item 3.1: Endorsement of methods of analysis: CCSCH and CCFO

Appendix I

We suggest (Official Analytical Methods of the American Spice Trade Association) ASTA 2.0 for moisture for all spices.

Appendix II

We suggest to include performance numerical criteria for Pb, Fe and Cu for all products.

Comments in reply to CL 2024/08-MAS

We include in Annex relevant information related with moisture in permeate and whey powder products.

Agenda Item 4.1: Review of Methods of Analysis in CXS 234: Cereal, Pulses and Legumes Workable Package

We suggest the following methods:

- ISO 2591-1 we consider is a type I method, this standard defines all the criteria to perform the test. They are general guidelines because the test conditions depend of the specification of the product.
- AACC 02-01.02 we agree with the comments done previously, AACC Method 02-01.02 is identical to AOAC 939.05
- AACC 46-11.02 In accordance with the previously detailed comments. Some other additional comments: 1- Conditions of the methods: Digestion: different catalyst mix and different digestion time: ISO 20483: at least 2 hours and AACC 46-11 90 minutes. Distillation: Different volume and preparation of the distillate collection flask. 2- Validation: The validation data in the ISO 20483 standard range between 7% and 80% protein and correspond to the matrices: wheat, corn, barley, wheat gluten, field and peas. Related with validation AACC didnt include wheat gluten proteins (the validation scope include lower levels). AACC and ISO standards are not identical
- AOAC 920.87 No validation data available for AOAC. AOAC scope are in flours. ISO 20483 scope are in cereals and byproducts. ISO 20483 have more details related with test conditions and controls, validation data and better scope. AOAC 920.87 and ISO 20483 are not identical.
- AOAC 979.09 Scope of the standard is in grains, but validated in grains that are not cereals. Validated in nusts, almonds, coconuts.

Please, let us know if you need more clarification of our comments.

Agenda Item 4.2: Review of Methods of Analysis in CXS 234: Fish and Fishery Products Workable Package

We suggest to include performance numerical criteria for metyl mercury, mercury and histamine.

We propose to retain the method of Live and raw bivalve moluscs / Paralytic sellfisch toxicity/Mousse bioassay.

Agenda Item 5: Information Document: the *General Guidelines on Sampling* (CXG 50–2004)

We agree to continue the work of the EWG and suggest to do a training in the documents and tools related.

Agenda Item 8: Harmonization of names and format for principles identified in CXS 234

We congratulate Brazil in so simple and practical approach and agree with the document presented including abbreviations.

We understand that is necessary to consider if there will be maintained the same abbreviations between languages.

Agenda Item 9: Approach for the placement of nitrogen conversion factors

We congratulate Chile and Brazil with the so clear document presented and agree with the document presented.

We understand that it is important consider the number of decimals for each factor to be consider

RELEVANT INFORMATION RELATED WITH MOISTURE IN PERMEATE AND WHEY POWDER PRODUCTS

Below we present relevant technical information to consider when confirming moisture in whey powder and permeates powder. Whey and permeate powder (annex I) are dairy products widely traded internationally.

Validation information was presented in CCMAS 42 by Argentina, Brazil and Uruguay. As CX/MAS 23/42/3 Add.1 explains:

The ISO 5537 I IDF 26 method principle of measurement is determining the moisture content by drying in a specifically designed oven at 87 °C for 5 hours while an air current of specified composition passes through the sample at a fixed flow of 33 mL/min. This method requires equipment and other specific inputs, including an oven of exclusive design with only two manufacturers in the world as described in the standard itself (annex III). Moreover, the oven is very expensive and exclusively used for dairy products.

Flow and temperature calibration capabilities and air composition are not readily available in most of the countries (annex IV), resulting in not providing metrological traceability of the determinations affecting the reliability of the results, Additionally generates costs that do not allow its applicability in all countries, a requirement for the officialization of a Codex method.

This method is exclusive, in terms of equipment and application for powdered milk products, therefore it deviates from the method selection criteria available in the Codex Procedural Manual, especially considering its practicability and applicability under normal conditions of the laboratory and the preference for methods for several groups of matrices.

The method of moisture that use normal pressure oven at 102 ªC is a method aligned with the criteria required to be proposed as a Codex method with the following characteristics:

- Proven safety based on performance data appropriate to the related product standard.
- Practicable and commonly used in laboratories.
- Principle of measurement of similar characteristics to the one used when establishing the technical specifications of the product standard.
- Codex general method applicable to several products.
- Equipment with greater ease of access and calibration. The temperature calibration is in a range usually used in the laboratory routine,
- It does not require specific inputs, nor does it generate waste that affects the environment.

Validation data are presented in annex II (Table 1, 2 and 3) during CCMAS 42. Additional updated information related with quality assurance tools available internationally for routine controls of laboratories are shown in Table 4 and 5 (Reference Material and Proficiency Testing). The reference method of moisture selected by most suppliers is normal pressure oven, which shows again that this method is suitable for moisture measurements of these products.

Recommendation:

Considering the results obtained and the codex selection criteria for methods of analysis available in the Procedures Manual, the performance of the methods, the limitation that have the countries to calibrate and access to the technology and inputs, the milk products experts committee involved in this study recommend the method of moisture normal pressure at 102 ºC to CCMAS.

ANNEX I – MATRIX DEFINITION IN CODEX STANDARDS

a) STANDARD FOR WHEY POWDERS CXS 289-1995

SCOPE

This standard applies to Whey Powder and Acid Whey Powder, intended for direct consumption or further processing, in conformity with the description in Section 2 of this standard.

DESCRIPTION

Whey powders are milk products obtained by drying whey or acid whey.

Whey is the fluid milk product obtained during the manufacture of cheese, casein or similar products by separation from the curd after coagulation of milk and/or of products obtained from milk. Coagulation is obtained through the action of, principally, rennet type enzymes.

Acid whey is the fluid milk product obtained during the manufacture of cheese, casein or similar products by separation from the curd after coagulation of milk and/or of products obtained from milk. Coagulation is obtained, principally, by acidification.

3. ESSENTIAL COMPOSITION AND QUALITY FACTORS

3.1 Raw materials

Whey or acid whey.

3.2 Permitted ingredients

Seed lactose in the manufacture of pre-crystallized whey powder.

3.3 Composition

Whey powder:

Criteria	Minimum content	Reference content	Maximum content
Lactose[(a)]	n.s.	61.0% (m/m)	n.s.
Milk protein[(b)]	10.0% (m/m)	n.s.	n.s.
Milk fat	n.s.	2.0% (m/m)	n.s.
Water[(c)]	n.s.	n.s.	5.0% (m/m)
Ash	n.s	n.s.	9.5% (m/m)
pH (in 10% solution)[(d)]	> 5.1	n.s.	n.s.

Acid whey powder:

Criteria	Minimum content	Reference content	Maximum content
Lactose[(a)]	n.s.	61.0% (m/m)	n.s.
Milk protein[(b)]	7.0% (m/m)	n.s.	n.s.
Milk fat	n.s.	2.0% (m/m)	n.s.
Water[(c)]	n.s.	n.s.	4.5% (m/m)
Ash	n.s.	n.s.	15.0% (m/m)
pH (in 10% solution)[(e)]	n.s.	n.s.	5.1

b) STANDARD FOR DAIRY PERMEATE POWDERS CXS 331-2017

SCOPE

This standard applies to dairy permeate powders, in conformity with the description in Section 2 of this standard, intended for further processing and/or as ingredient in other foods.

DESCRIPTION

Dairy permeate powders are dried milk productsi characterized by a high content of lactose:

a) manufactured from permeates which are obtained by removing, through the use of membrane filtration, and to the extent practical, milk fat and milk protein, but not lactose, from milk, wheyii (excluding acid whey), creamiii and/or sweet buttermilk, and/or from similar raw materials; and/or

b) obtained by other processing techniques involving removal of milk fat and milk protein, but not lactose, from the same raw materials listed under (a) and resulting in an end-product with the same composition as specified in Section 3.3.

Whey permeate powder is the dairy permeate powder manufactured from whey permeate. Whey permeate is obtained by removing whey protein, but not lactose, from whey.

Milk permeate powder is the dairy permeate powder manufactured from milk permeate. iv

3. ESSENTIAL COMPOSITION AND QUALITY FACTORS

3.1 Raw materials Dairy permeate powders: Milk permeate, whey permeate, cream permeate, sweet buttermilk permeate and/or similar lactose-containing milk products Whey permeate powder: Whey permeate Milk permeate powder: Milk permeate

3.2 Permitted ingredients Seed lactosev in the manufacture of pre-crystallized products.

3.3 Composition

Criteria	Dairy permeate powder	Whey permeate powder	Milk permeate powder
Minimum lactose, anhydrous[(a)] (m/m)	76.0%	76.0%	76.0%
Maximum nitrogen (m/m)	1.1%	1.1%	0.8 %
Maximum milk fat (m/m)	1.5%	1.5%	1.5%
Maximum ash (m/m)	14.0%	12.0%	12.0%
Maximum moisture[(b)] (m/m)	5.0%	5.0%	5.0%

ANNEX II - VALIDATION DATA

a) COLABORATORY STUDY 2022

During 2022 a collaborative study was performed by several laboratories in Latin American Countries to evaluate alternative methods of ISO 5537 I IDF 26, for the purpose of evaluating their performance data and compliance with the General Criteria for the selection of methods of analysis, established in the Codex Procedural Manual. The collaborative study was designed considering a moisture method with a vacuum oven (AOAC 927.05) and a moisture method in a normal oven at 102 ºC (Annex I) in the following matrices: Whey powder (AWP), Whey protein concentrate (RWP). Additional complementary data related with the ISO 5537 I IDF 26 are obtained.

These studies were coordinated by the National Metrology Institute of Uruguay (LATU, ISO/IEC 17043 accredited) under the ISO 5725 series) within the framework of ISO/IEC 17043, with the participation of 12 laboratories from Brazil, Uruguay, Panama, Argentina and Costa Rica. The samples were obtained from Global Proficiency (New Zealand) except for Powdered milk mix with vegetable fat that was prepared by LATU.

Results of this collaborative study are presented to CCMAS in CX/MAS 23/42/3 Add.1 May 2023 and shown in Table 1 for moisture method in a normal oven at 102 ºC in the following matrices: Whey powder (AWP), Whey protein concentrate (RWP). Additional complementary data related with the ISO 5537 I IDF 26 (Table 2).

A comparison of reference material used are shown in Table 3.

Table 1-Results of a collaborative study of several laboratories carried out with a moisture method in an oven at normal pressure (102 ± 2) ° C

Attribute	RWP	AWP
Number of participants after removing outliers	9	8
Average value, % m/m	4,67	1,63
Repeatability standard deviation, s_r, % m/m	0,063	0,072
Repeatability coefficient of variation, %	1,35	4,45
Repeatability limit, r , $(2.8\ s_r)$, % m/m	0,177	0,203
Reproducibility standard deviation, s_R, % m/m	0,119	0,141
Reproducibility Coefficient of Variation, %	2,55	8,68
Reproducibility limit, R , $(2.8\ s_R)$, % m/m	0,334	0,395

Table 2-Results of a collaborative study of several laboratories carried out with ISO 5537 I IDF 26: 2004 (Informative data)

Attribute	RWP	AWP
Number of participants after removing outliers	2	2
Average value, % m/m	4,803	1,862
Repeatability standard deviation, s_r, % m/m	0,038	0,034
Repeatability coefficient of variation, %	0,80%	1,81
Repeatability limit, r , $(2.8\ s_r)$, % m/m	0,107	0,094
Reproducibility standard deviation, s_R, % m/m	0,031	0,127
Reproducibility Coefficient of Variation, %	0,64	6,80
Reproducibility limit, R , $(2.8\ s_R)$, % m/m	0,087	0,354

Table 3 – Reference material description- Comparison with reference material

Reference Material				Moisture 102 ºC (normal pressure)	
Matrix	Assigned Value % m/m	Acceptance Range % m/m	Sample Provider	Average value % m/m	% with respect to the assigned value
RWP	4,70	4,49 – 4,91	Global Proficiency	4,67	99,4
AWP	1,64	1,60 – 1,68	Global Proficiency	1,63	99,4

The international availability of reference material and proficiency testing currently available is described in Tables 4 and 5.

Table 4 – Proficiency Testing providers in whey powder and permeate powder

Product	**Provider procedence**	**International recognition**	**Assigned value Method**
RWP	Germany	Accredited to ISO/IEC 17043	Mean of participants
	New Zeland	Accredited to ISO/IEC 17043	Normal pressure oven 102°C
	France	Accredited to ISO/IEC 17043	Mean of participants
	United Kindgtom	Accredited to ISO/IEC 17043	Mean of participants
AWP	Germany	Accredited to ISO/IEC 17043	Mean of participants
	New Zeland	Accredited to ISO/IEC 17043	Normal pressure oven 102°C
	France	Accredited to ISO/IEC 17043	Mean of participants
	United Kindgtom	Accredited to ISO/IEC 17043	Mean of participants

Table 5 – Reference Materials providers in whey powder and permeate powder

Product	**Provider procedence**	**International recognition**	**Assigned value Method**
RWP	Germany	In accordance with ISO 17034	oven 87 °C normal pressure oven 102 °C
	New Zeland	Accredited to ISO 17034	Normal pressure oven 102°C
	France	In accordance with ISO 17034	Normal pressure oven 102 ºC IDF 26A:1993 / ISO 5537|FIL 26
	United Kindgtom	Not available	Not available
AWP	Germany	In accordance with ISO 17034	Water
	New Zeland	Accredited to ISO 17034	Normal pressure oven 102°C
	France	Not available	Not available
	United Kindgtom	Not available	Not available

ONE SINGLE VALIDATION DATA

Validation data for a single laboratory accredited ISO/IEC 17025 were shown in Table 6.

Table 6 – Proficiency testing data for moisture in permeates and whey powder in normal pressure oven at 102 °C during three years

Product	PT provider procedence	Assigned Value	Z score	Bias % m/m	% of bias	SR % m/m
WPR	United Kindgtom	2,61	-0,4	-0,053	-2,03	0,13
WPR	United Kindgtom	2,40	-0,32	-0,057	-2,37	0,18
WPR	France	1,44	1,61	0,32		0,20
WPR	France	1,52	1,68	0,35		0,21
AWP	United Kindgtom	5,68	0,19	0,14	2,46	0,74

b) VALIDATION DATA FOR ISO 5537 I IDF 26 : 2004 PUBLISHED BY ISO/IDF ON CCMAS 41 MEETING MAS/CDR06

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

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	ISO 5537 | IDF 26:2004					
	RWP	AWP	WPP	MPP	CP	PIF
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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
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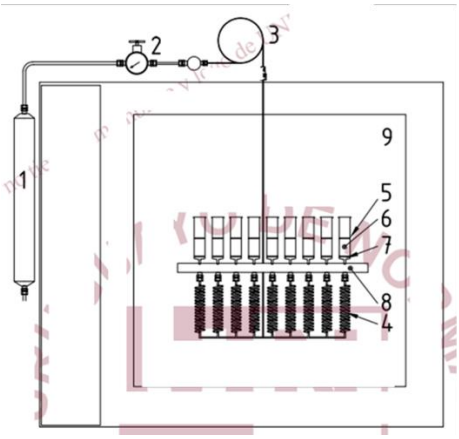
Coefficient of variation of reproducibility, %	3.10	3.82	5.44	6.71	2.66	3.85
Reproducibility limit, R (2,8*s_R), % m/m	0.179	0.274	0.231	0.285	0.191	0.202

ANNEX III – Annex A (ISO 5537 I IDF 26)



1 tube of polycarbonate (5.2.4)
2 constant pressure regulator (5.2.3)
3 restrictor (5.2.3)
4 copper tube (5.2.2)
5 filter of polyethene (5.4)
6 container (5.4)
7 filter of polyethene (5.4)
8 metal block (5.2.1)

- Copper tubes, of length 1 500 mm, of internal diameter 2 mm, connected to the metal block in the drying oven.
- Tube, made of polycarbonate, of length 350 mm, of diameter 40 mm, filled with silica gel with hygrometric indicator. The silica gel should have been dried at 150 °C for more than 12 h before use.
- Columns, made of hard polypropylene (Phenomenex 1213–10211)1), of length 90 mm, of internal diameter 20 mm, provided with two polyethene filters (Phenomenex 1212–1023), narrowed towards one end to fit onto the block.
- Synthetic stoppers, of adequate sizes to fit with the columns, made of soft polyethylene (Emergo 20273 B198 and 20371 U1)

ANNEX IV - CALIBRATION CAPABILITIES – DEW POINT AND HUMIDITY - BIPM

https://www.bipm.org/kcdb/cmc

COUNTRY	NUMBER OF COUNTRIES
AFRIMET	1
APMP	7
COOMET	1
EURAMET	25
SIM	6