CODEX ALIMENTARIUS COMMISSION

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda Item 4.2

MAS44/CRD12
April 2025
ORIGINAL LANGUAGE ONLY

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

44th Session

Virtual

5 – 8 May and 14 May 2025

COMMENTS OF IDF/ISO

While all information is explained in detail in document CX/MAS 25/44/5, IDF and ISO wish to bring the following points to the attention of CCMAS delegates in support of the proposal 2, that is not to endorse the 102°C method for determination of moisture in whey powders as a type IV method, for both technical and procedural reasons:

For technical reasons:

- The 102 °C method was assessed not being fit for purpose more than 20 years ago, and replaced by a much more solidly performing, easy to operate, fit for purpose alternative in ISO 5537|IDF 26, a short video to explain how to operate is available in English and Spanish.
- Whereas information on performance and trueness is available from the study conducted by Grobecker et al [1999] for whole milk powder and skim milk powder, this is lacking for whey powders. The outcome of an evaluation of Proficiency Testing data from the Latin American countries is inconclusive and lacks information on trueness.
- The 102 °C method is considered unfit for purpose for the moisture determination in whey powders because of the specific issues with these types of matrices. These matrices have a much higher lactose content than whole milk powder and skim milk powder. Moreover, glucose and galactose may be present. These factors make these matrices more prone to Maillard reactions upon heating. This is visible through browner colorization during the drying process (see pictures in Annex). Moreover, with drying temperatures above 90°C, lactose starts to lose its water of crystallization. These phenomena were already observed by Heinrich (1970) and where with the development of ISO 5537|IDF 26 also the reason to choose for a mild drying temperature (87 °C) and closely controlled humidity control. It also explains the poorer precision of the 102 °C method in all dairy matrices as compared to ISO 5537|IDF 26 and in matrices with elevated lactose content in more particular.
- It should be noted that, when agreed between trade partners, they are free to apply the 102°C method as a routine method to verify compliance with stated limits for moisture content between them. For that, there is no need to have the 102°C method contained in the *Recommended methods of analysis and sampling* (CXS 234-1999). In case of dispute, it is widely agreed that ISO 5537|IDF 26 as a Codex Type I method is the method of choice.

For procedural reasons:

- Endorsement of the method as Type IV, *in this case with only inconclusive validation data,* raises issues of coexistence of Type I and Type IV methods and creates a precedent for the future, as no criteria have been set for such situation, which may give a wrong impression of equivalence.
- The definition of a Tentative method (Type IV) is “A Type IV method is a method which has been used traditionally or else has been recently introduced but for which the criteria required for acceptance by the Committee on Methods of Analysis and Sampling have not yet been determined.”
- The 102°C method is not a recent method, but an old method which was revoked in because not fit for purpose.

Finally, IDF wishes to have incorrect precision values from the 2022 LAC Proficiency Test data for AWP presented in CX/MAS 23/42/3 Add.1 and MAS43/CRD19 amended based on the recalculations by the Expert Working Group.

References:

Grobecker et al. 1999. Determination of the water content in milk powder: Report of a collaborative study performed in the period June-July 1999. European Commission Report, EU-DG JRC-IRMM & IHCP.

Heinrich, C. 1970. Thermogravimetrische Untersuchungen zur Wassergehaltsbestimmung von Milchpulver. Milchwissenschaft 25 p. 387-391 (English translation available).

Annex

Pictures of 2 different samples of whey powders after:

1 **Drying at 87°C for 5 h (according to ISO 5537 IDF 26)**

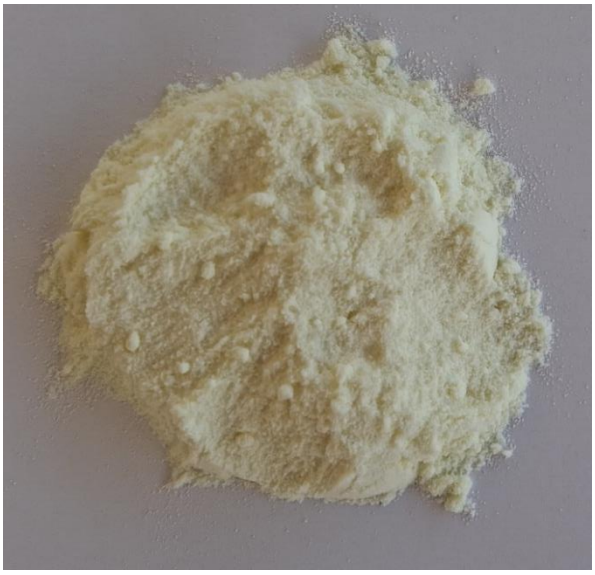
2 **Drying at 102°C for 5 h (2 h + 3 sequences of 1 h, according to IDF 26 A:1993)**

We can observe a significant colour difference between the 2 drying processes.

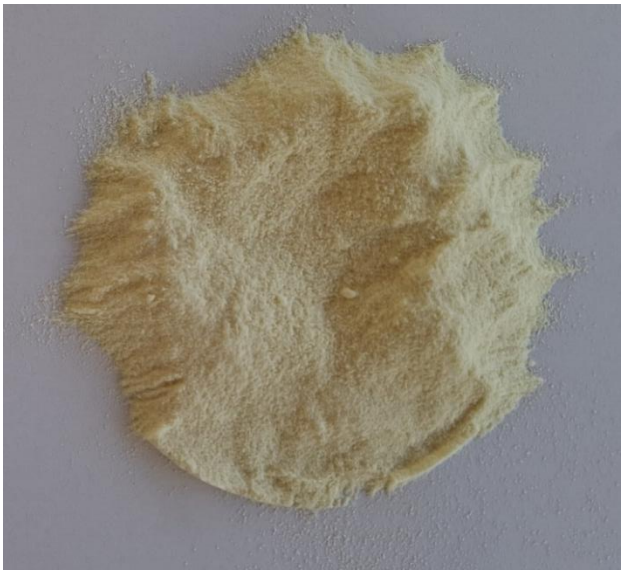
102°C process has a more yellow color, linked to the reactiveness of glucose and galactose content (maillard reaction).

SAMPLE 1

1



2



SAMPLE 2

1

2