

CODEX ALIMENTARIUS COMMISSION



Food and Agriculture
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Organization

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Agenda Item 3

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

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ENDORSEMENT OF METHODS OF ANALYSIS AND SAMPLING PLANS FOR PROVISIONS IN CODEX STANDARDS

(Submitted by International Dairy Federation (IDF)/International Organization for Standardization (ISO) in response to the determination of moisture in dried milk products proposed by Argentina, Brazil and Uruguay)

IDF and ISO acknowledge the extensive work provided by the delegations of Argentina, Brazil and Uruguay on determination of moisture in dried milk products contained in the Appendix II of the document CX/MAS 23/42/3 Add.1.

IDF/ISO notes the following:

- 2 methods have been evaluated, one with the application of a vacuum oven according to AOAC 927.05 and the other one with the application of a normal oven at 102°C (resembling the method according to provisional standard IDF 26A: 1993, which was withdrawn in 2004). The 2 methods were multi-lab tested in 4 to 10 laboratories in 5 countries on milk protein concentrate, skimmed milk powder, whey powder, whey protein concentrate, whole milk powder, infant formula and powdered milk mix with vegetable fat. We note that no matrix representing dairy permeates powders has been tested in this study.
- We understand the method using a normal oven at 102°C, described in Annex II with Appendix II in CX/MAS 23/42/3 Add.1 is proposed as Codex type I for dried milk matrices.

We invite all CCMAS delegates to a thorough read of the IDF/ISO¹ submission, contained in the same document, which describes the history in method development and the process that led to the replacement of IDF 26A: 1993 (normal oven at 102 °C) with ISO 5537|IDF 26:2004 (controlled air flow oven at 87 °C) as Codex type I reference method, initially for dried milk but later also for a number of dried milk products.

Moreover, we refer to the report on a recently executed multi-lab validation trial in which the favourable performance of ISO 5537|IDF 26:2004 for the determination of moisture in a range of dried milk products was further underpinned, [accessible for free here](#). This complementary multi-lab validation trial was conducted by an EN ISO/IEC 17043 accredited laboratory with participation of 14 laboratories in 8 countries in full conformity with ISO 5725-2 and related ISO standards.

In particular, we want to highlight the following points:

- The proposed method using a normal oven at 102 °C is not internationally standardized,
- Reported repeatability values r in Table 2 for the different matrices range from 0,105 till 0,203 g/100 g, whereas with ISO 5537|IDF 26 values between 0,067 till 0,238 g/100 g were obtained. Reported reproducibility values R for the proposed method range from 0,169 till 0,519 g/100 g, whereas with ISO 5537|IDF 26 values between 0,154 and 0,296 g/100 g were obtained. This underlines the favourable performance of ISO 5537|IDF 26 as compared to oven drying at 102 °C in terms of between lab variability, as was already concluded from the method comparison study by Grobecker et al, European Commission Report (August 1999) EU-DG JRC-IRMM & IHCP.

¹ To date IDF represents 40 member countries, [ISO/TC 34/SC 5](#) includes 30 participating member countries and 45 observing countries

- The assigned values for the Global Proficiency reference samples were also based on 102°C methods, so the comparison is against a like method rather than the current ISO 5537|IDF 26. Two of the samples also have assigned values for 'Moisture Reference Dryer' which is ISO 5537|IDF 26. The improved precision is again demonstrated by the acceptance ranges being narrower. For the skimmed milk powder (SMP) , 3.88 – 3.97 compared to 3.77 – 4.00. For the whole milk powder (WMP), 2.78 – 2.90 compared to 2.63 – 2.84. We also note that samples of whey protein concentrate (RWP), milk protein concentrate (MPP) do not relate to Codex product standards and are not within the scope of ISO 5537|IDF 26.
- Global Proficiency (New Zealand) operates an Inter laboratory Comparison Programme in which six SMP and six WMP samples are assessed every 2 months. Within the programme, the samples can be tested for both 102°C and ISO 5537|IDF 26 moisture. The results for these parameters demonstrate the precision advantage of the ISO 5537|IDF 26 as the rolling reproducibility, calculated from the last 24 sample reproducibility values, is significantly lower, i.e. Feb 2023 WMP 0.0935 vs. 0.257 and SMP 0.117 vs. 0.28. Furthermore, the forwarded data for the proposed method indicate rather variable bias between the mean results for each of the sample types and methods.
- The better performance of ISO 5537|IDF 26 can be explained from the further standardization of the execution of the method, the optimized air circulation system and the controlled relative humidity of the drying air. The relevance of these critical factors can be underpinned with published experimental data, see De Knecht & van den Brink, Int. Dairy J. (8) 1998, 733-738.
- Drying with 87 °C in a controlled air flow oven as with ISO 5537|IDF 26 implies more mild drying conditions with less risk of Maillard reactions, loss of volatiles and fat oxidation as compared to the proposed method. This is particularly true for high lactose content powders,
- ISO 5537|IDF 26 provides the details to create the drying apparatus without having to purchase the purpose made ovens. However, most laboratories implementing the method choose to use a commercially available controlled air flow oven for convenience. The cost of purchase of such an oven and utensils with a lifetime up to 20 years is below € 5 000. These controlled air flow ovens are now widely in use in dairy laboratories worldwide, as was also apparent from the broad participation in the recently conducted IDF/ISO complementary multi-lab validation trial;
- There is no specific air composition required. ISO 5537|IDF 26 makes use of slightly pressurized normal air. The equipment to check the air flow rate is easily available;
- The consumables (frits, tubes etc) can be reused many times. The waste generated is trivial and can be recycled (polyethylene or polypropylene).
- Thusfar, ISO 5537|IDF 26 has only been validated for dairy matrices. We do not rule that the method principle may also bring favourable precision with the determination of moisture content in other dried food commodities as compared to currently applied methods.

The primary aim for identifying a method as type I (or II when relevant) in the Codex Standard 234 is to have a precise and reliable method that allows for comparison of results with high confidence, in order to serve as a non-bias tool for resolving a trade dispute.

Therefore, it is only needed that a few laboratories involved with control testing are equipped for such a method (e.g. HPLC for other analysis). IDF/ISO acknowledges that alternative methods may be preferred at a national level or for routine purposes. However, for dispute resolution in (international) trade, IDF/ISO, based on worldwide expert opinions and gained experience during the past 20 years, reiterates its recommendation to preserve ISO 5537|IDF 26 as Codex type I method for the determination of moisture content for the following products:

| Commodity | Provision | Method | Principle | Type | Codex STAN |
|---|------------------|-------------------|-----------------------------|------|------------|
| Blend of skimmed milk and vegetable fat in powdered form | Water (Moisture) | ISO 5537 IDF 26 | Gravimetry, drying at 87 °C | I | 251 |
| Reduced fat blend of skimmed milk powder and vegetable fat in | Water (Moisture) | ISO 5537 IDF 26 | Gravimetry, drying at 87 °C | I | 251 |

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|--------------------------------|------------------|-------------------|-----------------------------|---|-----|
| powdered form | | | | | |
| Dairy permeate powders | Moisture | ISO 5537 IDF 26 | Gravimetry, drying at 87 °C | I | 331 |
| Milk powders and cream powders | Water (Moisture) | ISO 5537 IDF 26 | Gravimetry, drying at 87 °C | I | 207 |
| Whey powders | Water (Moisture) | ISO 5537 IDF 26 | Gravimetry, drying at 87 °C | I | 289 |