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Moisture content of dried milk and dried milk products – Complementary international collaborative study

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Abstract

The moisture content of dried milk and dried milk products is one of the predominant factors determining their keeping quality. ISO 5537|IDF 26 (IDF & ISO, 2004) serves as the reference method for the determination of the moisture content in all types of dried milk and is, as such, contained in Codex STAN 234-1999 (Codex Alimentarius, 2021). In order to further underpin the applicability of ISO 5537|IDF 26 to the wide range of dried milk and dried milk products, a complementary international collaborative study was conducted according to ISO 5725 part 1 (ISO, 1994) and part 2 (ISO, 2019), involving 14 laboratories in eight countries. Test samples were rennet whey powder, acid whey powder, whey permeate powder, milk permeate powder, cream powder and powdered infant formula. The results of the initial collaborative study with skim milk powders and whole milk powders (Grobecker et al., 1999) and the results of this complementary collaborative study prompt the inclusion of values of 0.15% and 0.25%, respectively for the repeatability limit and the reproducibility limit, in a revised version of ISO 5537|IDF 26 for the determination of the moisture content in dried milk and dried milk products.

Keywords

Moisture, reference method, dried milk products, international collaborative study.

Contents

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Foreword

Several parameters are monitored systematically to ensure the safety and quality of milk and milk products. Moisture content is one of them and requires a reliable analytical method to verify compliance with regulations and ensure the quality of the final product.

IDF first published its standard IDF 26 on moisture determination of dried milk back in 1964. As the standard became more used and technology developed, it was revised successively to obtain more precise results.

The standard ISO 5537 | IDF 26 is currently under revision to accommodate a wider range of products within its scope, such as rennet whey powder, acid whey powder, whey permeate powder, milk permeate powder, cream powder and powdered milk-based infant formula. It is referenced in Codex (CXS 234) to verify the provision of moisture content present in several standards for dairy products such as the Codex Standard for Milk Powders and Cream Powders (CXS 207).

As part of the revision, a collaborative study was designed and carried out to generate representative precision data for the additional dried dairy products covered by this method. This extensive task involved in total 14 laboratories worldwide.

This publication is the report documenting the additional validation studies conducted to verify the performance of the current standard. The data is of importance for practical application in routine use and to document the applicability of this standard to legislative and regulatory bodies. Data was incorporated into the revised standard ISO 5537|IDF 26, the publication of which is expected by mid-2023.

Have a good read,

Caroline Emond
April 2023

Acknowledgements

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The financial support from the Dutch National Committee of IDF for the execution of this complementary collaborative study is gratefully acknowledged.

1 The experts contributed based on their expertise and mentioning their affiliation is not an endorsement of any product, service, methodology or equipment provided or sold by their employer organisation and is not meant to provide any commercial advantage. By adding the affiliation, the IDF intends to acknowledge the support given by the employer to the experts for the IDF contribution as well as to provide transparency.

Abbreviations and acronyms

EU-DG JRC-IRMM	European Union Directorate General Joint Research Center Institute for Reference Materials and Measurements
IDF	International Dairy Federation
IHCP	Institute for Health and Consumer Protection
ISO	International Organisation for Standardization
SCAMC	Standing Committee on Analytical Methods for Composition (of the IDF)
SCSA	Standing Committee on Statistics and Automation (of the IDF)

1. Introduction

The moisture content of dried milk and dried milk products is one of the predominant factors determining their keeping quality. The rate of many potential chemical and biochemical reactions depends on the moisture content, or more precisely on the amount of 'available' or 'free' water. Therefore, limits on the moisture content of dried milk and dried milk products are frequently stated in regulations and in trade agreements. Checking compliance with stated limits requires an accurate and precise analytical method.

Reference method determination of the moisture content of food matrices relies on the principle of weight loss upon drying under controlled conditions. For this, most often, oven drying methods are applied. Influencing factors with these oven drying methods are (Walstra & Jenness, 1986; De Knegt & van den Brink, 1998):

- Evaporation of water resulting from Maillard reactions, increased with elevated temperatures;
- Loss of volatiles, present or formed during the drying process;
- Possible water uptake due to fat oxidation;
- Airflow. Intensive air ventilation is essential to obtain an accurate and uniform temperature inside the drying oven. However, the force of ventilation must be kept limited as with traditional oven drying methods the test portion is dried in an open dish;
- Drying temperature;
- Duration of drying;
- Relative humidity of the air in the testing environment.

These factors imply that the conditions during oven drying may have a significant influence on the outcome of any measurement. In other words, the moisture content of a food object is a method-defined parameter. Its determination, therefore, requires a strict standardization of the drying conditions in order to obtain accurate and precise test results.

During the 1990s, it was observed that the then reference method for the determination of moisture in dried milk, IDF 26A:1993 (IDF, 1993), exhibited unacceptable high values for repeatability and reproducibility. This initiated the quest for a more robust and precise method. It was proven that with the use of an oven with controlled airflow, a much more uniform temperature and a constant relative humidity of the drying air were achieved. Testing conditions were optimized to arrive at on average equivalent results with IDF 26A:1993 (De Knegt & van den Brink, 1998). This resulted in a new method description that was later published as ISO 5537|IDF 26:2004 (IDF & ISO, 2004).

The performance of this newly (at the time) standardized method was verified in a multi-laboratory validation and method comparison study on three whole milk powder samples and

three skimmed milk powder samples (Grobecker et al., 1999). The results of this study are summarized in Table 1.

Table 1. Results of a previous multi-laboratory comparison study in eight laboratories on three skim milk powders and three whole milk powders with three different methods for the determination of moisture/water in dried milk (Grobecker et al., 1999), all values are expressed in %.

Method	Mean	Standard deviation of repeatability (s_r)	Standard deviation of reproducibility (s_R)
IDF 26A:1993	3.28	0.067	0.142
Oven with controlled airflow (ISO 5537|IDF 26:2004)	3.20	0.048	0.066
Karl Fisher titration	3.69	0.054	0.084

From these data, it was concluded that best precision was achieved with the controlled airflow oven method and the Karl Fisher titration. Especially with reproducibility, the new controlled airflow oven method showed much better performance than IDF 26A:1993 (IDF, 1993). The calculated mean value with the controlled airflow oven method was close to the calculated mean value from the results with IDF 26A:1993 (IDF, 1993), which was in accordance with the aim during method development. Understandably, the calculated mean value for the Karl Fisher titration was higher as this method measures not only the “available water”, but all water present in the sample.

IDF and ISO therefore concluded that the new controlled airflow oven method was the best option to replace IDF 26A:1993 (IDF, 1993) as the reference method for the determination of the moisture content in dried milk. Codex Alimentarius subsequently endorsed ISO 5537|IDF 26:2004 (IDF & ISO, 2004) as a Codex Type I method for uptake in Codex Standard 234-1999 (Codex Alimentarius, 2021). Initially, this only pertained to the determination of the moisture content in (blends of) skimmed milk powder, (blends of) whole milk powder and cream powders but in later years the method was also adopted for the determination of the moisture content in whey powders and dairy permeate powders.

The adoption of the method in Codex Alimentarius for application in the additional matrices relied on single lab validation data and expert opinion. In light of the review of Codex-adopted methods of analysis and sampling in recent years, it was therefore considered appropriate to underpin the applicability of the method for the wider scope of dried milk products through a complementary international collaborative study.

2. Material and methods

The collaborative study was designed and conducted by Qlip, Zutphen (NL), in accordance with ISO 5725-1 (ISO, 1994) and ISO 5725-2 (ISO, 2019). Before being conducted, the study design was reviewed by the IDF Action Team on Statistics on Analytical Data.

2.1. Participating laboratories

The study included 14 laboratories in eight countries, which were experienced with the method. The participating laboratories are listed alphabetically in Table 2, whereas these have been named randomly A to N in the sections with the results.

Commercially available controlled airflow ovens were applied in 12 of the laboratories, whereas two laboratories worked with an in-house built controlled airflow oven, based on the available description of the apparatus in ISO 5537|IDF 26:2004 (IDF & ISO, 2004).

2.2. Test sample materials

The selected sample materials for the collaborative study were:

1. Rennet whey powder;
2. Acid whey powder;
3. Whey permeate powder;
4. Milk permeate powder;
5. Cream powder;
6. Powdered milk-based infant formula.

These materials were sourced directly from FrieslandCampina (NL), Fonterra (NL), Vreugdenhil Dairy Foods (NL) and IDH (NL). The powdered milk-based infant formula was sourced from a Dutch supermarket. Measured values for the moisture content of these samples at Qlip ranged from 1.85 to 2.41%.

Table 2. List of participating laboratories in the complementary collaborative study in alphabetical order.

Name	Country
Actalia - Poligny	France
DMK Baby Strückhausen GmbH	Germany
FrieslandCampina – LQS Leeuwarden	The Netherlands
Fonterra Co-operative Group Limited – Clandeboye Laboratory	New Zealand
Fonterra Co-operative Group Limited – Hamilton Laboratory	New Zealand
Fonterra Co-operative Group Limited – Hawera Laboratory	New Zealand
Governmental Dairy Science Laboratory – Food Chemistry Division	Ireland
ILVO – Technology and Food Science Unit	Belgium
Instytut Innowacji Przemysłu Mleczarskiego Sp. z o.o.	Poland
Japan Dairy Technical Association – Kudan-kita Laboratory	Japan
Milchwirtschaftliche Lehr- und Untersuchungsanstalt Oranienburg e. V.	Germany
MQD Institut für Analytik, Hygiene und Produktqualität GmbH	Germany
Qlip B.V.	The Netherlands
Wageningen Food Safety Research	The Netherlands

With each material, the whole batch was carefully mixed before aliquots of 50 g of each powder were transferred and packed in inert, aluminium sachets in early November 2020. These sachets were directly sealed airtight and stored at ambient temperature until being shipped to the participants in the study or being used for one of the in-house tests in the Qlip laboratory during the weeks thereafter.

The homogeneity of the samples was checked by analysis of eight sachets of each of the six samples according to ISO 5537|IDF 26:2004 at the Qlip laboratory in November 2020. The stability of the samples throughout the study was checked by comparing the results of the homogeneity testing with test results obtained on 8 December 2020, three days after the last determinations of the moisture content were made by participating laboratories.

2.3. Sample transport

Samples were sent from Qlip by TNT FedEx Express on 16 November 2020 with accompanying health and veterinary certificates and delivered to participants between 17 November 2020 and 4 December 2020.

2.4. Instructions to the participating laboratories

Each participant was sent two test samples of each material as blind duplicates, 12 sachets in total, with a test protocol and a result sheet. The test protocol contained instructions for the handling of the sachets and the analysis of their content. Participating laboratories were instructed to store the sachets at ambient temperature, away from direct daylight, until the time of analysis. Test samples were to be analysed by a single analyst in a prescribed order to secure repeatability conditions. For the analysis of the test samples, the analyst was requested to keep strictly to the method description in ISO 5537|IDF 26:2004. Participating laboratories were requested to share information on the date of receipt, the drying temperature, the airflow rates per channel in their oven, the start and end times of drying and cooling, and any relevant observation with the receipt of the samples or during analysis.

All laboratories analysed the samples between 24 and 28 November 2020, except for two laboratories which analysed on 4 and 5 December 2020 due to delayed arrival of the samples.

2.5. Data receipt and data processing

Test data and other relevant information were returned to Qlip by all participants before 10 December 2020. The evaluation and processing of the raw data and the calculation of the precision parameters repeatability and reproducibility were all conducted by Qlip according to ISO 13528 (ISO, 2022) and ISO 5725, part 1 (ISO, 1994) and part 2 (ISO, 2019).

3. Results and discussion

3.1. General

The execution of the collaborative study went smoothly. All participating laboratories mentioned receipt of the test samples in proper order, although some laboratories did receive the test samples with up to two weeks delay. There are no indications that the delayed receipt has influenced the outcome of their measurements.

3.2. Integrity of test samples

3.2.1.Homogeneity of test samples

The results and accompanying calculations on the homogeneity of the test samples are in Table 3. For all materials, except for whey permeate powder, the criterion for sample homogeneity in ISO 13528, clause B.2.2 ($s_s < 0.3^*\sigma_{PT}$, with s_s being the estimate of the between sample standard deviation and σ_{PT} calculated from R = 0.25%), was fulfilled. The results with whey permeate powder did fulfil the expanded criterion for sample homogeneity according to ISO 13528, clause B.2.3 ($s_s < \sqrt{c}$). Thereby, it is noted that the final precision values obtained with whey permeate powder are not out of range with the precision values with the other sample materials. The homogeneity of the test samples for this collaborative study was therefore considered acceptable.

3.2.2.Stability of test samples

The results and accompanying calculations on the stability of the test samples are in Table 4. Stability tests were conducted by comparing the average result per test sample from homogeneity testing at Qlip in early November 2020 with the average of duplicate test results per test sample as obtained at Qlip on 8 December 2020, which was three days after the last measurements by a participant. Only with cream powder, the first criterion for sample stability according to ISO 13528, clause B.5.1 ($|\bar{y}_1 - \bar{y}_2| \leq 0.3 * \sigma_{PT}$, with $\bar{y}_1$ being the mean of the homegenity test results per matrix and $\bar{y}_2$ being the mean of the stability test results per matrix), was fulfilled. However, it should be noted that the measurement uncertainty in homogeneity testing as well as in the additional stability testing do contribute to the total uncertainty with stability test results. Therefore, the criterion according to ISO 13528, clause B.5.2 ($|\bar{y}_1 - \bar{y}_2| \leq 0.3 * \sigma_{PT} + 2\sqrt{u^2(\bar{y}_1) + u^2(\bar{y}_2)}$, with $u(\bar{y}_1)$ being the uncertainty of $\bar{y}_1$ and $u(\bar{y}_2)$ being the uncertainty of $\bar{y}_2$), was applied. The results for all six samples complied with this criterion. Moreover, the final precision values obtained with the test results from all

participants seem not to have been affected by issues with sample stability. Stability of the test samples throughout the study was therefore considered acceptable.

3.3. Raw data

Each of the participating laboratories reported a full set of duplicate test results for each of the test materials, resulting in 168 paired measurement results. The raw data of these individual measurements are presented in Table 5. These data were examined in accordance with ISO 5725-2 (ISO, 2019).

Table 3. Individual test results on moisture content with homogeneity testing on individual sachets of each sample material at the Qlip laboratory, calculated values for s_w, s_x and s_s and comparison of s_s against limit values as listed in ISO 13528 (ISO, 2022). s_w is within-sample standard deviation, s_x is standard deviation of sample averages and s_s is between-sample standard deviation per sample material. All values are expressed in %. Limit value is in bold and underlined typeface in case of non-compliance of the check value.

Sachet	Rennet whey powder		Acid whey powder		Whey permeate powder		Milk permeate powder		Cream powder		Powdered infant formula	
1	2.083	2.011	2.507	2.559	1.587	1.627	1.556	1.540	2.672	2.676	1.901	1.953
2	2.038	2.021	2.556	2.558	1.538	1.561	1.554	1.565	2.638	2.626	1.908	1.929
3	2.043	2.085	2.563	2.568	1.518	1.541	1.605	1.539	2.639	2.686	1.979	1.966
4	2.044	2.023	2.550	2.498	1.516	1.535	1.544	1.532	2.706	2.608	1.894	1.933
5	1.993	2.040	2.520	2.518	1.499	1.549	1.531	1.593	2.732	2.669	1.919	1.881
6	2.055	2.074	2.543	2.539	1.550	1.581	1.602	1.576	2.606	2.641	1.911	1.883
7	2.062	2.053	2.563	2.564	1.547	1.563	1.551	1.576	2.630	2.626	1.897	1.922
8	2.039	2.048	2.528	2.550	1.602	1.599	1.570	1.585	2.614	2.629	1.918	1.957
Mean	2.044		2.543		1.557		1.564		2.650		1.922	
s_w	0.0254		0.0193		0.0205		0.0252		0.0331		0.0240	
s_x	0.0174		0.0175		0.0325		0.0162		0.0284		0.0244	
Check value s_s	0*		0.011		0.0291		0*		0.0161		0.0176	
Limit value $0.3*\sigma_{PT}$	0.024		0.024		***0.024***		0.024		0.024		0.024	
Limit value $\sqrt{c}$	0.045		0.041		0.041		0.045		0.051		0.044	

* The estimate of the between-sample variance s_s^2 often becomes negative when s_s is much smaller than s_w. This can be expected when proficiency test items are highly homogeneous. In such case s_s is stated to be 0.

Table 4. Individual test results on moisture content with stability testing with each sample material at the Qlip laboratory, covering at least the period two weeks prior to the first determination till three days after the last determinations by participating laboratories, calculated difference of the mean values obtained at the beginning and the end of the study and comparison against limit values as calculated according to ISO 13528 (ISO, 2022). All values are expressed in %. Limit value in bold and underlined typeface in case of non-compliance of the check value.

	Rennet whey powder	Acid whey powder	Whey permeate powder	Milk permeate powder	Cream powder	Powdered infant formula
Late test result 1	2.127	2.659	1.672	1.589	2.666	1.978
Late test result 2	2.163	2.668	1.650	1.625	2.661	1.983
Mean late test result ($\bar{y}(1)$)	2.145	2.663	1.661	1.607	2.663	1.980
Mean early test result ($\bar{y}(2)$) (see Table 3)	2.044	2.543	1.557	1.564	2.650	1.922
Check value $\lvert\bar{y}(1)-\bar{y}(2)\rvert$	0.101	0.121	0.104	0.044	0.013	0.058
Limit value for $\lvert\bar{y}(1)-\bar{y}(2)\rvert$ with $R = 0.25$ (ISO 13528, B17)	**0.027**	**0.027**	**0.027**	**0.027**	0.027	**0.027**
Limit value for $\lvert\bar{y}(1)-\bar{y}(2)\rvert$ with $R = 0.25$ $r = 0.15$ (homogeneity) and *within lab* $R = 0.20$ (stability) (ISO 13528, B18)	0.135	0.135	0.135	0.135	0.135	0.135

Table 5. Raw data from replicate measurements by each participating laboratory. All listed values are expressed in %.

	Rennet whey powder		Acid whey powder		Whey permeate powder		Milk permeate powder		Cream powder		Powdered infant formula	
	Rep 1	Rep 2	Rep 1	Rep 2	Rep 1	Rep 2	Rep 1	Rep 2	Rep 1	Rep 2	Rep 1	Rep 2
Lab A	2.12	2.12	2.55	2.61	1.59	1.57	1.64	1.64	2.66	2.67	1.96	1.97
Lab B	2.15	2.19	2.66	2.65	1.60	1.72	1.64	1.68	2.64	2.63	1.98	1.92
Lab C	2.11	2.06	2.51	2.56	1.58	1.55	1.66	1.53	2.64	2.60	1.94	1.86
Lab D	2.03	2.04	2.55	2.51	1.51	1.47	1.49	1.51	2.54	2.58	1.86	1.85
Lab E	1.96	1.96	2.37	2.39	1.40	1.40	1.38	1.41	2.49	2.47	1.77	1.76
Lab F	1.85	1.86	2.31	2.33	1.42	1.39	1.31	1.32	2.29	2.30	1.61	1.63
Lab G	2.18	2.01	2.72	2.76	1.47	1.59	1.52	1.54	2.80	2.53	2.00	1.88
Lab H	2.09	2.06	2.52	2.55	1.55	1.54	1.51	1.53	2.50	2.55	1.89	1.83
Lab I	2.14	2.11	2.66	2.60	1.59	1.61	1.56	1.57	2.65	2.63	1.92	1.92
Lab J	2.00	2.03	2.61	2.56	1.50	1.53	1.50	1.50	2.58	2.63	1.84	1.89
Lab K	2.02	2.02	2.49	2.52	1.47	1.48	1.51	1.52	2.56	2.55	1.88	1.86
Lab L	2.02	2.02	2.48	2.45	1.47	1.48	1.45	1.43	2.47	2.50	1.78	1.78
Lab M	2.08	1.99	2.67	2.62	1.39	1.40	1.41	1.24	2.48	2.53	1.83	1.76
Lab N	2.37	2.41	2.70	2.77	1.91	2.12	2.03	2.26	2.85	2.97	2.19	2.15

3.4. Data analysis

3.4.1.Consistency of data

As a first step, the consistency of the data was evaluated using Mandel statistics. Mandel *h* values indicate how close a sample test result obtained in one laboratory is to the average of all sample test results obtained at the other laboratories. Mandel *k* values indicate the consistency in the repeatability standard deviation in one laboratory with the average of the repeatability standard deviations in the other laboratories. The calculated Mandel *h* and *k* values are listed in Tables 6 and 7. Data are indicated in bold and underlined typeface where Mandel values exceeded the critical value with 14 laboratories at a significance level of 1%. Graphical presentations for Mandel *h* and *k* values are in Figures 1 and 2 with samples 1 to 6 being rennet whey powder, acid whey powder, whey permeate powder, milk permeate powder, cream powder and powdered infant formula, respectively.

Mandel *h* values indicated relatively high-test sample results for laboratory N, with the critical value being exceeded for five out of six test samples. Relatively low-test sample results were apparent with some samples for laboratory F, but without exceeding a critical value. Mandel *k* values indicated a significant deviation of results with four different test samples reported by laboratories G and N, two test samples for each laboratory.

3.4.2.Outlier identification

Statistical outliers were identified by applying Cochran and Grubbs tests with a significance level of 1% (outlier) and 5% (straggler), see Tables 8 and 9. Outliers (o) and stragglers (s) are indicated in bold and underlined typeface. Two outliers were detected with test sample results for laboratory G using the Cochran test, this is confirmation of the earlier presented high Mandel *k* values with these results, see Table 7. These test sample results were therefore excluded from further statistical evaluation. Subsequently, two further outliers and two stragglers were identified with the Grubbs test, all for laboratory N. The outliers were also discarded from the data set.

As observed from applying Mandel h statistics, laboratory N clearly obtained much higher test results than all other laboratories. It now shows also multiple Cochran stragglers and Grubbs outliers and stragglers. When asked, laboratory N noted that according to their internal controls the method was operating within specifications. No possible cause for the higher test results as compared to other laboratories could be identified. Still, considering the number and degree of deviations in the sample test results of this laboratory, it was decided to discard their results from further calculations of the precision parameters.

Mandel *h* statistics already indicated that the sample test results of laboratory F were comparatively lower than from the other laboratories. Although the critical value was not exceeded, Grubbs

values were relatively high. Upon further inquiry, laboratory F showed proof of a possible issue with the temperature control of the equipment, likely having resulted in a drying temperature about 2 °C lower than prescribed in ISO 5537|IDF 26. It was therefore decided to also discard their sample test results from the dataset before final calculation of the precision parameters.

Table 6. Mandel h values, lower and upper critical values with 14 laboratories are –2.30 and 2.30 (P<0.01).

Sample	Rennet whey powder	Acid whey powder	Whey permeate powder	Milk permeate powder	Cream powder	Powdered infant formula
Lab A	0.40	0.17	0.22	0.47	0.61	0.72
Lab B	0.82	0.79	0.73	0.57	0.39	0.60
Lab C	0.11	-0.21	0.12	0.24	0.28	0.20
Lab D	-0.30	-0.25	-0.36	-0.23	-0.16	-0.16
Lab E	-0.92	-1.49	-0.94	-0.75	-0.75	-0.88
Lab F	-1.82	-1.99	-0.91	-1.17	-2.09	-2.06
Lab G	0.20	1.49	-0.11	-0.08	0.61	0.52
Lab H	0.03	-0.21	-0.01	-0.13	-0.42	-0.12
Lab I	0.45	0.58	0.34	0.10	0.43	0.36
Lab J	-0.47	0.21	-0.20	-0.23	0.17	-0.08
Lab K	-0.43	-0.45	-0.46	-0.15	-0.20	-0.04
Lab L	-0.43	-0.79	-0.46	-0.52	-0.71	-0.76
Lab M	-0.30	0.70	-0.97	-1.10	-0.57	-0.64
Lab N	**2.65**	1.45	**3.01**	**2.97**	**2.41**	**2.37**

Table 7. Mandel k values, critical value with 14 laboratories is 2.39 (P<0.01).

Sample	Rennet whey powder	Acid whey powder	Whey permeate powder	Milk permeate powder	Cream powder	Powdered infant formula
Lab A	0.000	1.387	0.268	0.000	0.118	0.192
Lab B	0.700	0.231	1.607	0.466	0.118	1.153
Lab C	0.875	1.156	0.402	1.515	0.473	1.538
Lab D	0.175	0.925	0.536	0.233	0.473	0.192
Lab E	0.000	0.462	0.000	0.350	0.236	0.192
Lab F	0.262	0.324	0.308	0.105	0.165	0.269
Lab G	**2.975**	0.925	1.607	0.233	**3.192**	2.306
Lab H	0.525	0.694	0.134	0.233	0.591	1.153
Lab I	0.525	1.387	0.268	0.117	0.236	0.000
Lab J	0.525	1.156	0.402	0.000	0.591	0.961
Lab K	0.000	0.694	0.134	0.117	0.118	0.384
Lab L	0.000	0.694	0.134	0.233	0.355	0.000
Lab M	1.575	1.156	0.134	1.981	0.591	1.345
Lab N	0.700	1.618	**2.813**	**2.680**	1.418	0.769

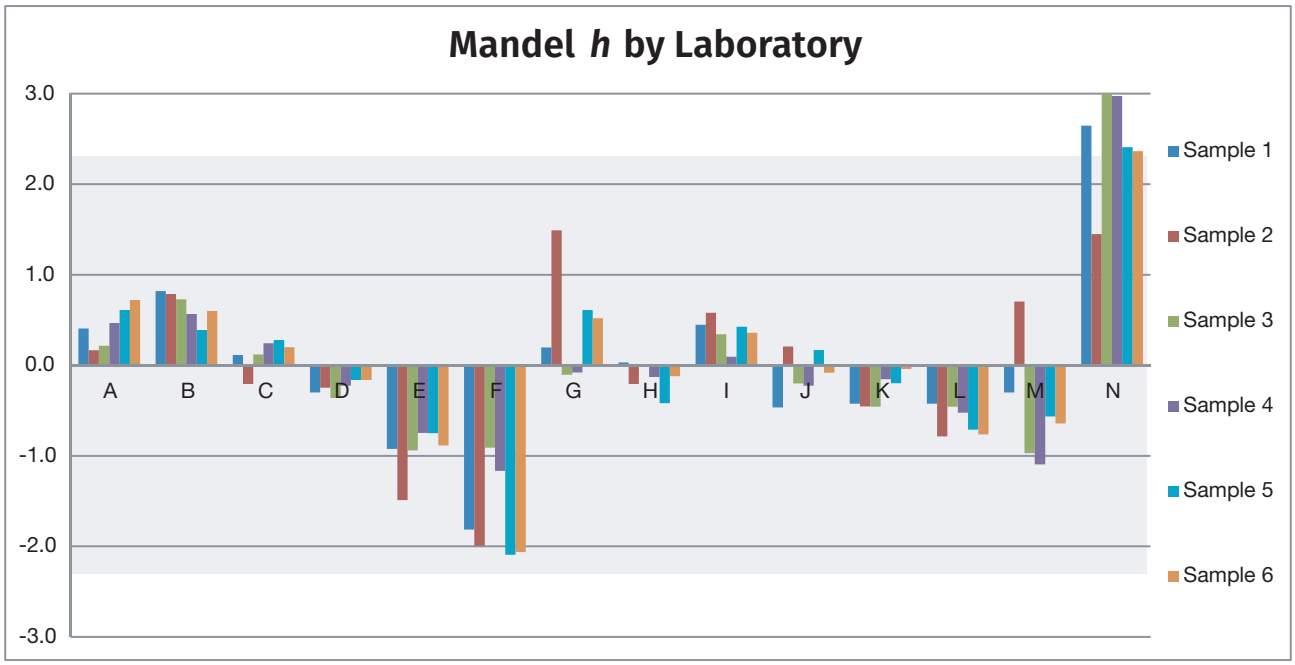

Figure 1. Mandel h values by laboratory.

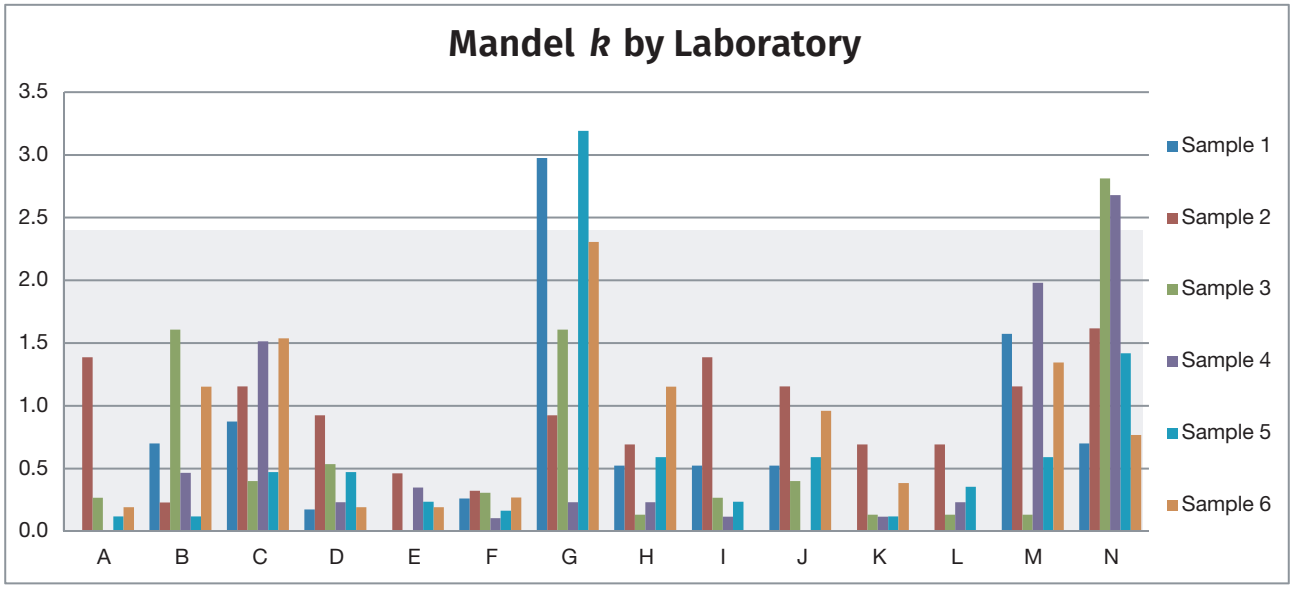

Figure 2. Mandel k values by laboratory.

Table 8. Calculated Cochran values, critical values with 14 laboratories are 0.492 (P<0.05) for stragglers and 0.599 (P<0.01) for outliers.

Sample	Rennet whey powder	Acid whey powder	Whey permeate powder	Milk permeate powder	Cream powder	Powdered infant formula
Lab A	0.000	0.137	0.005	0.000	0.001	0.003
Lab B	0.035	0.004	0.185	0.016	0.001	0.095
Lab C	0.055	0.095	0.012	0.164	0.016	0.169
Lab D	0.002	0.061	0.021	0.004	0.016	0.003
Lab E	0.000	0.015	0.000	0.009	0.004	0.003
Lab F	0.005	0.007	0.007	0.001	0.002	0.005
Lab G	**0.632 (o)**	0.061	0.185	0.004	**0.728 (o)**	0.380
Lab H	0.020	0.034	0.001	0.004	0.025	0.095
Lab I	0.020	0.137	0.005	0.001	0.004	0.000
Lab J	0.020	0.095	0.012	0.000	0.025	0.066
Lab K	0.000	0.034	0.001	0.001	0.001	0.011
Lab L	0.000	0.034	0.001	0.004	0.009	0.000
Lab M	0.177	0.095	0.001	0.280	0.025	0.129
Lab N	0.035	0.187	**0.565 (s)**	**0.513 (s)**	0.144	0.042

Table 9. Calculated Grubbs values (one outlying observation), critical values with 14 laboratories are 2.507 (P<0.05) for stragglers and 2.755 (P<0.01) for outliers, critical values with 13 laboratories are 2.462 (P<0.05) for stragglers and 2.699 (P<0.01) for outliers.

Sample	Rennet whey powder	Acid whey powder	Whey permeate powder	Milk permeate powder	Cream powder	Powdered infant formula
Lab A	0.421	0.172	0.224	0.485	0.668	0.748
Lab B	0.836	0.816	0.756	0.588	0.444	0.623
Lab C	0.130	0.214	0.124	0.254	0.332	0.206
Lab D	0.286	0.257	0.375	0.236	0.116	0.168
Lab E	0.910	1.545	0.974	0.776	0.713	0.918
Lab F	1.804	2.069	0.945	1.212	2.078	2.142
Lab G		1.546	0.109	0.081		0.539
Lab H	0.046	0.214	0.009	0.133	0.377	0.127
Lab I	0.462	0.602	0.357	0.099	0.481	0.373
Lab J	0.452	0.215	0.209	0.236	0.220	0.085
Lab K	0.411	0.472	0.475	0.158	0.153	0.043
Lab L	0.411	0.815	0.475	0.545	0.676	0.793
Lab M	0.286	0.730	1.008	1.137	0.526	0.668
Lab N	**2.665 (s)**	1.503	**3.119 (o)**	**3.086 (o)**	**2.496 (s)**	2.454

Table 10. Means of duplicates for final calculation of the precision parameters. All values expressed in %.

Sample	Rennet whey powder	Acid whey powder	Whey permeate powder	Milk permeate powder	Cream powder	Powdered infant formula
Lab A	2.12	2.58	1.58	1.64	2.67	1.97
Lab B	2.17	2.66	1.66	1.66	2.64	1.95
Lab C	2.09	2.54	1.57	1.60	2.62	1.90
Lab D	2.04	2.53	1.49	1.50	2.56	1.86
Lab E	1.96	2.38	1.40	1.40	2.48	1.77
Lab F						
Lab G		2.74	1.53	1.53		1.94
Lab H	2.08	2.54	1.55	1.52	2.53	1.86
Lab I	2.13	2.63	1.60	1.57	2.64	1.92
Lab J	2.02	2.59	1.52	1.50	2.61	1.87
Lab K	2.02	2.51	1.48	1.52	2.56	1.87
Lab L	2.02	2.47	1.48	1.44	2.49	1.78
Lab M	2.04	2.65	1.40	1.33	2.51	1.80
Lab N						

Table 11. Absolute differences between duplicates for final calculation of the precision parameters. All values expressed in %.

Sample	Rennet whey powder	Acid whey powder	Whey permeate powder	Milk permeate powder	Cream powder	Powdered infant formula
Lab A	0.00	0.06	0.02	0.00	0.01	0.01
Lab B	0.04	0.01	0.12	0.04	0.01	0.06
Lab C	0.05	0.05	0.03	0.13	0.04	0.08
Lab D	0.01	0.04	0.04	0.02	0.04	0.01
Lab E	0.00	0.02	0.00	0.03	0.02	0.01
Lab F						
Lab G		0.04	0.12	0.02		0.12
Lab H	0.03	0.03	0.01	0.02	0.05	0.06
Lab I	0.03	0.06	0.02	0.01	0.02	0.00
Lab J	0.03	0.05	0.03	0.00	0.05	0.05
Lab K	0.00	0.03	0.01	0.01	0.01	0.02
Lab L	0.00	0.03	0.01	0.02	0.03	0.00
Lab M	0.09	0.05	0.01	0.17	0.05	0.07
Lab N						

Table 12. Summary statistics and calculated precision values from the complementary interlaboratory study.

Sample	Rennet whey powder	Acid whey powder	Whey permeate powder	Milk permeate powder	Cream powder	Powdered infant formula
Number of participating laboratories	14	14	14	14	14	14
Number of outliers	1	0	1	1	1	0
Number of accepted results	11	12	12	12	11	12
Mean value	2.06	2.57	1.52	1.52	2.57	1.87
Repeatability standard deviation, s_r (%)	0.026	0.030	0.037	0.046	0.024	0.039
Repeatability relative standard deviation, RSDs_r (%)	1.27	1.16	2.46	3.01	0.93	2.07
Repeatability limit, r [$r = 2.8 \times s_r$] (%)	0.073	0.083	0.104	0.128	0.067	0.109
Reproducibility standard deviation, s_R (%)	0.064	0.098	0.083	0.102	0.068	0.072
Reproducibility relative standard deviation, RSDs_R (%)	3.10	3.82	5.44	6.71	2.66	3.85
Reproducibility limit, R [$R = 2.8 \times s_R$] (%)	0.179	0.274	0.231	0.285	0.191	0.202

Table 13. Summary statistics and calculated precision values from the initial interlaboratory study (Grobecker et al., 1999).

Sample	Skim milk powder (1)	Skim milk powder (2)	Skim milk powder (3)	Whole milk powder (1)	Whole milk powder (2)	Whole milk powder (3)
Number of accepted results	8	8	8	8	8	8
Mean value	3.62	3.57	3.93	2.52	3.16	2.38
Repeatability standard deviation, s_r (%)	0.052	0.085	0.053	0.045	0.035	0.049
Repeatability relative standard deviation, RSDs_r (%)	1.44	2.38	1.34	1.80	1.11	2.06
Repeatability limit, r [$r = 2.8 \times s_r$] (%)	0.146	0.238	0.148	0.126	0.084	0.137
Reproducibility standard deviation, s_R (%)	0.058	0.096	0.074	0.055	0.060	0.098
Reproducibility relative standard deviation, RSDs_R (%)	1.61	2.69	1.89	2.19	1.89	4.11
Reproducibility limit, R [$R = 2.8 \times s_R$] (%)	0.162	0.296	0.207	0.154	0.168	0.274

3.4.3.Statistical evaluation of the results

The means and difference of test results after removal of outliers and 'abnormal' results are presented in Tables 10 and 11. A summary of all obtained statistical parameters from this study is contained in Table 12, including calculated values for repeatability standard deviation (s_r), reproducibility standard deviation (s_R), repeatability limit (r) and reproducibility limit (R). The contents of this table are to become part of the annexes within the revised version of ISO 5537|IDF 26.

Currently stated values for r and R in ISO 5537|IDF 26 (IDF & ISO, 2004) are 0.15% and 0.20%. These are based on the outcome of a collaborative study with samples from three skim milk powders and three whole milk powders (Grobecker et al., 1999), see Table 13.

The obtained values for r and R per sample from this complementary study are well in alignment with the values for whole milk and skim milk powder in the initial study. Averaging the obtained precision values per sample of the initial study and this complementary study results in values of 0.12% for r and 0.22% for R.

From the results of the two studies, it appears that the stated value for the repeatability limit r in ISO 5537|IDF 26 can also be considered applicable for the additional dried matrices. However, the stated value for the reproducibility limit R seems to overestimate the performance of the method to some extent, both for skim milk powder and whole milk powder as well as for the additionally examined dried matrices. It is therefore proposed to amend the value for the reproducibility limit in the new draft of the standard from 0.20% to 0.25%.

3.5. Other remarks from participating laboratories

Two laboratories reported problems with measuring the airflow in each channel of the drying apparatus. Further guidance on this is included in the new version of the standard.

4. Conclusion

The applicability of ISO 5537|IDF 26 to the wide range of dried milk and dried milk products was underpinned with the results of a complementary international collaborative study with 14 laboratories in eight countries. Test samples were rennet whey powder, acid whey powder, whey permeate powder, milk permeate powder, cream powder and powdered infant formula. Based on the results of the initial collaborative study (Grobecker et al., 1999) and the results of this complementary study, the values for the repeatability limit and the reproducibility limit can be defined as:

Repeatability: The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will, in not more than 5% of the cases, be greater than 0.15%.

Reproducibility: The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will, in not more than 5% of the cases, be greater than 0.25%.

Those limits are expressed for the 95% probability level and may not be applicable to ranges and matrices other than those studied.

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