

CODEX ALIMENTARIUS COMMISSION



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Agenda Item 4.2

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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

DETERMINATION OF MOISTURE CONTENT IN WHEY POWDER – STATISTICAL ANALYSIS OF DATA ON THE 102°C and ISO 5537 | IDF 26 METHODS

(Submitted by Australia)

The CCMAS members are invited to review the following data and summaries as provided to the expert working group (ExWG) on the 102°C and ISO 5537 | IDF 26 methods for the determination of moisture content in whey powder, to allow their own statistical assessment and conclusions. Noting the intention here is not to change the recommendation of the CCMAS44 virtual working group (VWG), just so members obtain a more complete picture of the validation data provided to the ExWG and process of statistical assessment Australia undertook.

Tabulation of the Whey powder – moisture methods validation data provided:

Table 1 Tabulating only Acid Whey Powder (AWG) moisture 102°C method data as provided by Uruguay to ExWG using Global Proficiency QC Material 11650-QC-PR9669 in the 2022 LAC Collaborative study

Laboratory	Replicate 1	Replicate 2	Replicate 3	Replicate 4	Replicate 5
1	1.593	1.606	1.612	1.603	1.665
2	1.518	1.509	1.390	1.371	1.426
3	1.575	1.569	1.546	1.576	1.598
4	1.551	1.732	1.715	1.672	1.955
5	1.586	1.583	1.551	1.650	1.593
6	1.550	1.510	1.555	1.536	1.463
7	1.611	1.628	1.626	1.615	1.618
8	2.379	2.326	2.358	2.358	2.385

Table 2 Tabulating only Acid Whey (AWP) moisture by ISO 5537 | IDF 26 method data provided to ExWG based on International Dairy Federation. (2023). Moisture content of dried milk and dried milk products – Complementary international collaborative study (Bulletin of the IDF No. 524/2023).

Laboratory	Replicate 1	Replicate 2
1	2.550	2.610
2	2.660	2.650
3	2.510	2.560
4	2.550	2.510
5	2.370	2.390
6	2.310	2.330
7	2.720	2.760
8	2.520	2.550
9	2.660	2.600
10	2.610	2.560
11	2.490	2.520
12	2.480	2.450
13	2.670	2.620
14	2.700	2.770

Australia wanted to use a well-established, fit-for purpose, and readily available analysis and decided to use for this purpose the AOAC Interlaboratory Study Workbook – Blind Replicates¹. The initial statistical analysis is summarised in Table 3 below.

Table 3 - Blind replicate statistical analysis of IDF 524-AWP plus Uruguay AWP data

Attribute	Symbol	IDF 524-AWP	Uruguay-AWP
Total number of laboratories	p	14	8
Total number of replicates	Sum(n(L))	28	40
Overall mean of all data (grand mean)	XBARBAR	2.560	1.682
Repeatability standard deviation	s(r)	0.0307	0.0617
Reproducibility standard deviation	s(R)	0.1226	0.2917
Repeatability relative standard deviation	RSD(r)	1.20	3.67
Reproducibility relative standard deviation	RSD(R)	4.79	17.35
HORRAT value		1.38	4.69

We assessed the IDF 524-AWP data and no outliers were identified, however for the Uruguay-AWP data the Cochran's test highlights 'Lab 4' with the maximum within lab variance, and thus an outlier. We then moved to the Single Grubbs Test where the result was 'Significant' and highlighting 'Lab 8' as the 'highest average' and giving 74.2% of the '% decrease in standard deviation', and consistently comparatively higher than data from the other laboratories. We would normally ask Lab 4 & Lab 8 'if according to their internal controls the method was operating within specification', but as a 'reviewer' this is not within our purview).

Based on this data Australia's suggestion is that Tables 1, 2, & 3 as previously presented in [ma43 CRD19](#) on pages 4 and 5 should be amended as follows based on the above assessment and to include the data provided for the IDF 524-AWP (2023). Corresponding data also appeared in [CX/MAS 23/42/3 Add.1](#) page 9: 'Table 2- Results of a collaborative study of several laboratories carried out with a moisture method in an oven at normal pressure (102 ± 2) ° C'. Note, the scope of the ExWG was specifically for the Whey powder data assessment, so no review of RWP or other dried milk and dried milk product data was undertaken.

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	AWP
Number of participants after removing outliers	9	8 incl. outliers	8 6 excl. outliers
Average value, % m/m	4,67	1,63 1.68	1,63 1.56
Repeatability standard deviation, S_r, % m/m	0,063	0,072 0.062	0,072 0.038
Repeatability coefficient of variation, %	1,35	4,45 3.67	4,45 2.41
Repeatability limit, r , (2.8 S_r) , % m/m	0,177	0,203 0.173 00.1731730.105	0,203 0.105
Reproducibility standard deviation, S_R, % m/m	0,119	0,144 0.292	0,144 0.076

¹ See <https://www.aoac.org/resources/aoac-international-interlaboratory-study-workbook-blind-replicates/> downloaded 16/07/2024.

Attribute	RWP	AWP	AWP
Reproducibility Coefficient of Variation, %	2,55	8,68 <u>17.4</u>	8,68 <u>4.85</u>
Reproducibility limit, R , (2.8 S _R) , % m/m	0,334	0,395 <u>0.817</u>	0,395 <u>0.212</u>

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

Attribute	RWP	AWP	<u>IDF AWP study 2023</u>
Number of participants after removing outliers	2	2	<u>13</u>
Average value, % m/m	4,803	1,862	<u>2.57</u>
Repeatability standard deviation, S _r , % m/m	0,038	0,034	<u>0.030</u>
Repeatability coefficient of variation, %	0,80%	1,81	<u>1.16</u>
Repeatability limit, r , (2.8 S _r) , % m/m	0,107	0,094	<u>0.083</u>
Reproducibility standard deviation, S _R , % m/m	0,031	0,127	<u>0.098</u>
Reproducibility Coefficient of Variation, %	0,64	6,80	<u>3.82</u>
Reproducibility limit, R , (2.8 S _R) , % m/m	0,087	0,354	<u>0.274</u>

Table 3 — Reference material description- Comparison with reference material

Reference Material			Moisture 102 °C (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 <u>1.56</u>	99,4 <u>95.1</u>

During the ExWG a presentation was provided with the following 2022 LAC Collaborative summary table 4 (below) for Moisture Content of Whey Powder V01 241214LF emailed on 14 Dec 2024 by Uruguay, which differed significantly to the summary data submitted previously to CCMAS for AWP in CX/MAS 23/42/3 Add.1 page 10 Table 2, and MAS43/CRD19 page 5 Table 2. However, this latter Uruguay's statistical summary provided on 14 Dec 24 is in better agreement with the statistical assessments of the other group members.

Table 4 — Statistical summary and calculated accuracy values from the Collaboratory study. Method: Normal pressure oven at 102°C – CXS 234 Appendix III before (A) and after the elimination of outliers results (B dicarting Lab 3, B* dicarting Lab 3&5)

Attribute	A	B	B*
Number of participants after removing outliers	8	7	6
Average value, % m/m	1,682	1,584	1,561
Repeatability standard deviation, S_r, % m/m	0,062	0,065	0,038
Repeatability coefficient of variation, %	3,670	4,127	2,408
Repeatability limit, r, (2.8 S_r), % m/m	0,173	0,183	0,105
Reproducibility standard deviation, S_R, % m/m	0,286	0,088	0,068
Reproducibility Coefficient of Variation, %	17,03	5,530	4,348
Reproducibility limit, R, (2.8 S_R), % m/m	0,802	0,245	0,190

Recommendation:

When assessing individually or comparing the Whey Powder moisture methods validation of the ISO 5537 | IDF 26 and the Normal pressure (102±2°C) method provided in CXS 234 Appendix XI. The above assessment(s) should be considered.