

REPORT NO. 1048

**Food Proficiency Testing Program
Round 41 – Whole Milk Powder**

September 2017

ACKNOWLEDGMENTS

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1. FOREWORD

This report summarises the results of a proficiency testing program involving the analysis of whole milk powder samples. It constitutes the forty-first round of an ongoing series of programs involving chemical analysis of foodstuffs.

Proficiency Testing Australia (PTA) conducted the testing program in July / August 2017. The aim of the program was to assess laboratories' ability to competently perform the nominated tests.

The Program Coordinator was Dr M Bunt. The Technical Advisers were Ms N Rusk, Ms S Mott and Dr R Hutchinson from Global Proficiency Ltd (New Zealand). This report was authorised by Mr P Briggs, PTA General Manager.

2. FEATURES OF THE PROGRAM

- (a) A total of seven laboratories participated in the program, one of which did not return results for inclusion in the final report. Laboratories from the following countries participated:

3	AUSTRALIA
2	SINGAPORE
1	MALAYSIA
1	RUSSIA

To ensure confidential treatment of results, each laboratory was allocated a unique code number. All reference to participants in this report is by allocated code numbers. Please note that one laboratory reported more than one set of results and, therefore, this laboratory's code number (with letter) could appear several times in the same data set.

- (b) The results reported by participants are presented in Appendix A.
- (c) Laboratories were provided with two samples of approximately 100 g of whole milk powder, labelled PTA 1 and PTA 2.
- (d) Participants were requested to determine the levels of:
- Moisture;
 - Crude Protein;
 - Fat; and
 - Ash.

Laboratories were required to perform all tests using the routine test methods which would normally be used to test customer supplied samples.

- (e) Laboratories were requested to perform the tests according to the *Instructions to Participants* provided and to record the results, along with an estimate of their measurement uncertainty (MU) for each result, on the accompanying *Results Sheet*, which was distributed with the samples. Copies of these documents appear in Appendix C.
- (f) Prior to sample distribution, randomly selected samples were analysed for homogeneity by Global Proficiency Ltd (New Zealand). Stability testing was also performed on the samples by Global Proficiency Ltd (New Zealand). Based on the results of this testing, the homogeneity and stability of the samples was established (see Appendix B).

3. FORMAT OF THE APPENDICES

- (a) Appendix A is divided into four sections (A1-A4). These sections contain the analysis of results reported by laboratories for Moisture, Crude Protein, Fat and Ash.

Each section contains, where appropriate:

- i) a table of results reported by laboratories, with estimates of their MUs;
 - ii) a table of calculated z-scores and methods used;
 - iii) a listing of the summary statistics; and
 - iv) ordered z-score charts.
- (b) Appendix B contains details of the homogeneity and stability testing.
- (c) Appendix C contains copies of the *Instructions to Participants* and *Results Sheet*.

4. STATISTICAL DESIGN OF THE PROGRAM

Samples PTA 1 and PTA 2 were chosen from two batches of the Global Proficiency Ltd DairyChek Chemistry program. These were produced on the same day and both used as duplicate samples.

The summary statistics calculated for each test / sample consists of:

- *No. of Results*: the total number of results for that test/sample;
- *Median*: the middle value of the results;
- *Normalised IQR*: the normalised interquartile range of the results;
- *Uncertainty of the Median*: a robust estimate of the standard deviation of the *Median*;
- *Robust CV*: the robust coefficient of variation expressed as a percentage, *i.e.* $100 \times \text{Normalised IQR} / \text{Median}$;
- *Minimum*: the lowest laboratory result;
- *Maximum*: the highest laboratory result; and
- *Range*: the difference between the *Maximum* and *Minimum*.

The median is a measure of the centre of the data.

The normalised IQR is a measure of the spread of the results. It is calculated by multiplying the interquartile range (IQR) by a correction factor, which converts the IQR to an estimate of the standard deviation. The IQR is the difference between the upper and lower quartiles (*i.e.* the values above and below which a quarter of the results lie, respectively).

For normally distributed data, the uncertainty of the median is approximated by:

$$\sqrt{\frac{\pi}{2}} \times \frac{\text{normIQR}}{\sqrt{n}}$$

where *normIQR* is the normalised IQR and *n* is the number of results.

In order to assess laboratories' testing performance, a robust statistical approach, using z-scores, was utilised. Z-scores give a measure of how far a result is from the consensus value (*i.e.* the median), and gives a "score" to each result relative to the other results in the group.

A z-score with an absolute value less than or equal to 2.0 is considered to be satisfactory, whereas, a z-score with an absolute value greater than or equal to 3.0 is considered to be an outlier and is marked by the symbol "§". Laboratories are also encouraged to review results which have an absolute z-score value between 2.0 and 3.0 (*i.e.* $2.0 < |z\text{-score}| < 3.0$). These results are considered to be questionable results.

Ordered z-score charts indicate each laboratory's robust z-score, in order of magnitude, marked with its laboratory code number. From these charts, each laboratory can readily compare its performance relative to the other laboratories.

The ordered z-score charts in Appendix A are limited on the vertical axis to +3.0 and -3.0, so that outliers are clearly identifiable as those laboratories whose "bar" extends beyond the chart boundary.

For further details on the calculation and interpretation of robust z-scores and ordered z-score charts please see the *Guide to Proficiency Testing Australia (2016)*.

5. OUTLIER RESULTS

The following table summarises the results of the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.

Table A: Summary Statistics for All Tests

Test	Summary Statistics	PTA 1	PTA 2
Moisture (% m/m)	Number of Results	26	27
	Median	2.995	3.190
	Normalised IQR	0.022	0.115
	Uncertainty (Median)	0.005	0.028
Crude Protein (Kjeldahl) (% m/m)	Number of Results	22	22
	Median	23.740	23.675
	Normalised IQR	0.124	0.159
	Uncertainty (Median)	0.033	0.043
Crude Protein (Combustion) (% m/m)	Number of Results	5	4
	Median	n/a	n/a
	Normalised IQR	n/a	n/a
	Uncertainty (Median)	n/a	n/a
Fat (excluding Gerber) (% m/m)	Number of Results	28	26
	Median	27.825	27.925
	Normalised IQR	0.100	0.109
	Uncertainty (Median)	0.024	0.027
Ash (% m/m)	Number of Results	20	19
	Median	5.715	5.560
	Normalised IQR	0.026	0.052
	Uncertainty (Median)	0.007	0.015

Table B: Summary of Statistical Outliers

The following table lists the laboratories (by code number) that obtained outliers for each test.

Test	Sample PTA 1	Sample PTA 2
Moisture	2, 5	1, 3A, 3B, 5
Crude Protein (Kjeldahl)	6	6
Crude Protein (Combustion)		
Fat (excluding Gerber)	1, 4, 5	1, 5
Ash	1	2

Notes for Table A and Table B:

1. The summary statistics for all tests (including the number of results) and z-scores were calculated from the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.
2. For Crude Protein, the Kjeldahl and Combustion method results were analysed separately. Z-scores and summary statistics were not calculated for the Combustion method results due to an insufficient number of results reported.
3. For Fat, z-scores and summary statistics were not calculated for the Gerber method results.
4. Z-scores were calculated from the average of the duplicates for each sample.
5. Target CVs were used to calculate the z-scores for each test.

6. PTA AND TECHNICAL ADVISERS' COMMENTS

Consensus values (medians), derived from participants' results, are used as the assigned values in this program. These values are not metrologically traceable to an external reference.

The summary statistics, uncertainties of the assigned values and outliers, for each of the tests, are reported in Tables A and B above.

Complete details of the statistical analyses appear in Appendix A.

The results for all tests were seen to depend on the laboratory and / or methods used (which are only partially harmonised by IDF, ISO, AOAC). The analysis was good for showing the method differences that exist. The within-duplicate differences were relatively small in comparison. It was noted that some of the analysis methods provided were commonly used for other products (e.g. meat products, nut products, cereals and vegetable products) and may not have been appropriate for milk powders. This may have contributed to some very different results although, for Moisture, time and temperature were not enough to explain the differences seen between laboratories. The results included data imported from multiple rounds to increase the data pool and conclusions are included in the test sections following.

6.1 Return Rate

Of the seven laboratories that participated in the program, six (86%) submitted results for inclusion in the final report. The return rate for all tests is as follows:

- | | | |
|-----------------|------------|------|
| • Moisture | 6 out of 6 | 100% |
| • Crude Protein | 4 out of 6 | 67% |
| • Fat | 6 out of 6 | 100% |
| • Ash | 5 out of 6 | 83% |

6.2 Performance Summary

One or more statistical outliers were reported by all of the six laboratories that returned results in this round of the Food program. The previous milk powder round of the Food program was Round 39 (see Report No. 932 for more details). For comparison, 57% of the participants in Round 39 of the Food program reported statistical outliers.

A total of 50 results were analysed in this round of the program. Of these results, fifteen (30%) were outlier results. In Round 39 of the Food program 17% of the total results reported were outlier results (see Report No. 932).

6.3 Moisture

Of the six laboratories that tested the samples for Moisture, two laboratories tested using AOAC 984.25, including one laboratory that submitted three sets of results, one laboratory used AOAC 925.40, one laboratory used AOAC 925.45D, one laboratory used AOAC 950.46 and one laboratory used MS 1191: 1991.

The results for Moisture were pooled and analysed against the results submitted for the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.

Target CVs of 4.4% and 4.1% were used to analyse the results for samples PTA 1 and PTA 2, respectively. These target CVs were chosen to correspond to the robust standard deviations obtained for the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.

Laboratory 5 reported outlier results for both samples. Laboratory 2 reported an outlier for sample PTA 1. Laboratories 1, 3A and 3B reported outliers for sample PTA 2.

The majority of data for Moisture were not within the multi-round limits. In the case of Moisture, the drying conditions are balanced between lack of moisture release under mild conditions vs excess moisture released by Maillard Browning under more severe conditions (though dish size or use of dispersing sand are extra factors). Although some drying conditions listed appear to be similar for some of the laboratories, it is notable that the results are significantly different from each other.

- For example laboratory 1 and laboratory 5 reported similar drying conditions (laboratory 1 - 103 °C / 4 hours; laboratory 5 - 105 °C / 5 hours), yet the Moisture results are significantly different from each other (laboratory 1 - 3.31% / 3.83%; laboratory 5 - 1.15% / 2.57%).
- The z-scores for sample PTA 2 appear to be a lot higher compared to sample PTA 1, with greater variability between laboratories evident for this sample.

Confidence in the medians can be expressed as the uncertainty of the median (as defined in page 3 of this report), which was calculated for each test. For the Moisture testing, the median and associated uncertainty for each sample (expressed in % m/m) was as follows:

	PTA 1	PTA 2
Moisture, all methods pooled	2.995 ± 0.005	3.190 ± 0.028

Figures TA-1 and TA-2 show the distribution of results from the methods used for Moisture testing in this round including the Global Proficiency data, and are included for interest purposes only. **Note:** For sample PTA 1, the x axis scale was not extended to the lowest reported value of 1.15%; instead, this value is shown in the 2 – 2.2% range.

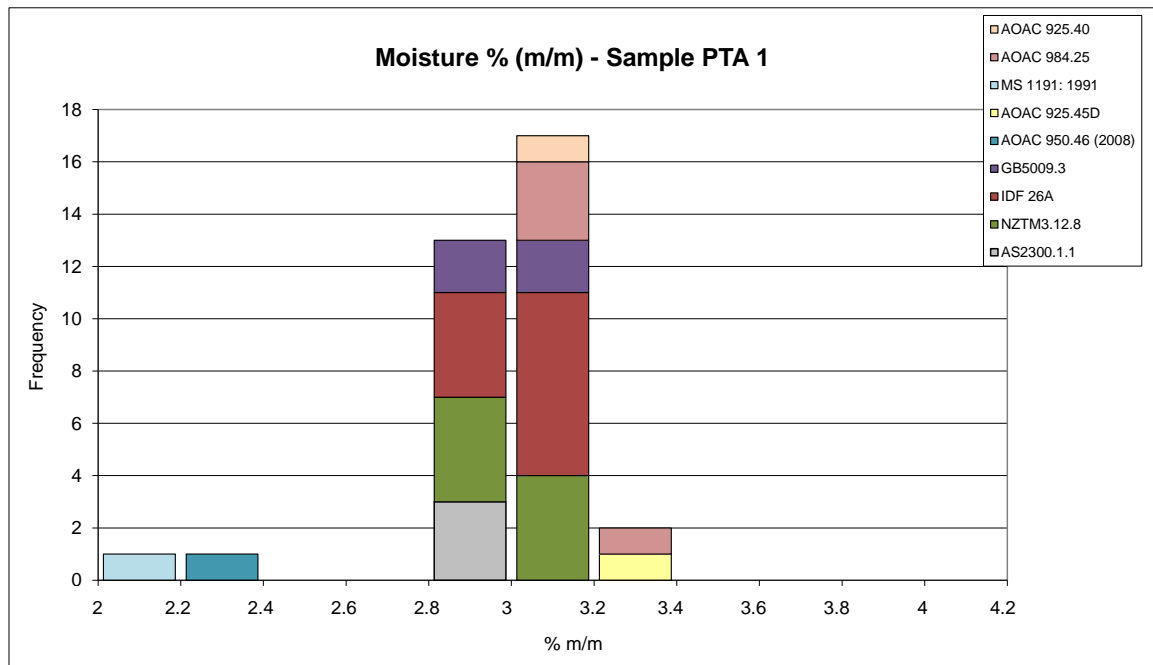


Figure TA-1. Spread of results for Moisture proficiency testing for sample PTA 1.

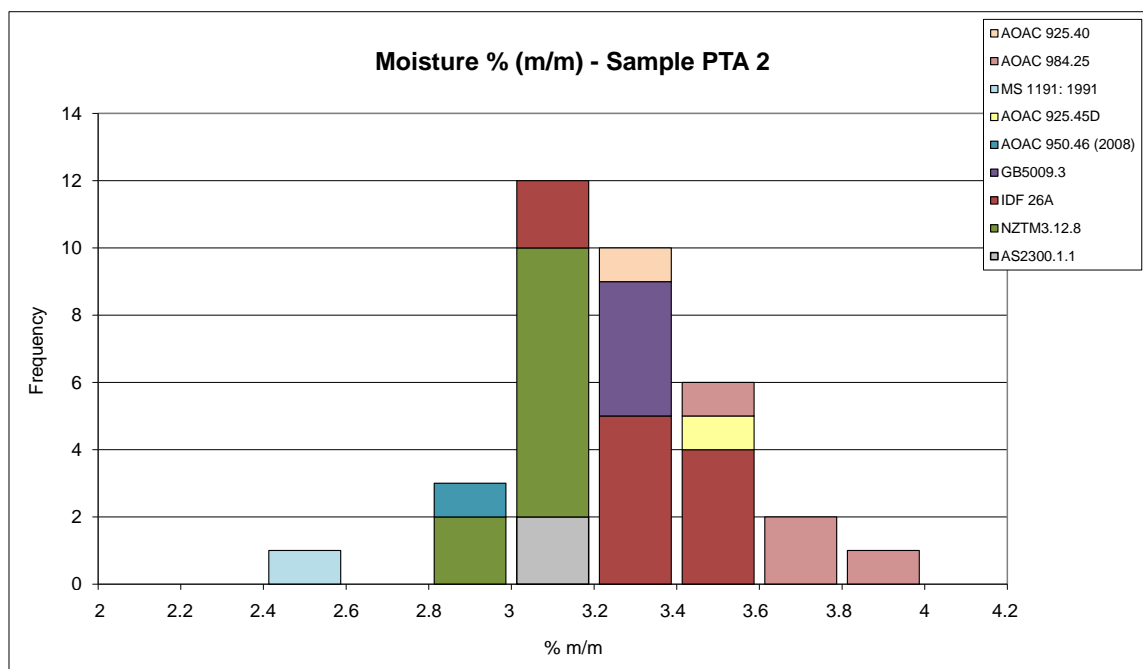


Figure TA-2. Spread of results for Moisture proficiency testing for sample PTA 2.

For this round of the program, laboratories were requested to report their MU by repeatability r and/or reproducibility R . Two laboratories reported MU for Moisture by repeatability. Two laboratories reported MU for Moisture by both repeatability and reproducibility. Two laboratories did not provide an estimate of the MU for their Moisture results by either repeatability or reproducibility.

Laboratories whose measurement uncertainty range encompassed the median or were within the uncertainty of the median range are considered an accurate representation of the laboratory's uncertainty of measurement.

6.4 Crude Protein

Four laboratories reported results for Crude Protein. One of these laboratories reported using AOAC 991.20. One laboratory tested using AOAC 992.23. One laboratory tested using SNI 01-0008. One laboratory used an in-house Kjeldahl method.

The results for Crude Protein were analysed against the results submitted for the Global Proficiency Ltd DairyChek Chemistry program, using the same samples. Laboratory 4, which tested using AOAC 992.23, was compared to the laboratories that used Combustion methods for their testing in the Global Proficiency Ltd DairyChek Chemistry program, while the other three laboratories were compared to the laboratories that used Kjeldahl methods in the Global Proficiency Ltd DairyChek Chemistry program.

The robust CVs for the Global Proficiency Ltd DairyChek Chemistry program for Crude Protein by Kjeldahl methods were 0.5% and 0.7% for samples PTA 1 and PTA 2, respectively. The robust CV of 0.5% for sample PTA 1 was considered inappropriate to analyse the results, so a target CV of 0.7% (the same CV as that obtained for sample PTA 2) was chosen to analyse the results for sample PTA 1.

Laboratory 6 reported outlier results for both samples for Crude Protein by Kjeldahl methods.

Z-scores and summary statistics were not calculated for Crude Protein by Combustion methods due to an insufficient number of results reported for the Global Proficiency Ltd DairyChek Chemistry program. However, the results reported by laboratory 4 were consistent with the results reported by the few laboratories that tested using Combustion methods in the Global Proficiency Ltd DairyChek Chemistry program.

The severity of test conditions for both the digestion and the distillation processes in the Kjeldahl test method could be examined by the use of standards such as tryptophan. At times, the use of other more difficult digestible protein sources such as nicotinamide may be used.

Figures TA-3 and TA-4 show the distribution of results from the methods used for Protein testing in this round including the Global Proficiency data (and combustion results), and are included for interest purposes only. **Note:** For sample PTA 1, the x axis scale was not extended to the highest reported value of 26.21%; instead, this value is shown in the 25.2 – 25.4% range. For sample PTA 2, the x axis scale was not extended to the highest reported value of 26.43%; instead, this value is also shown in the 25 – 25.2% range.

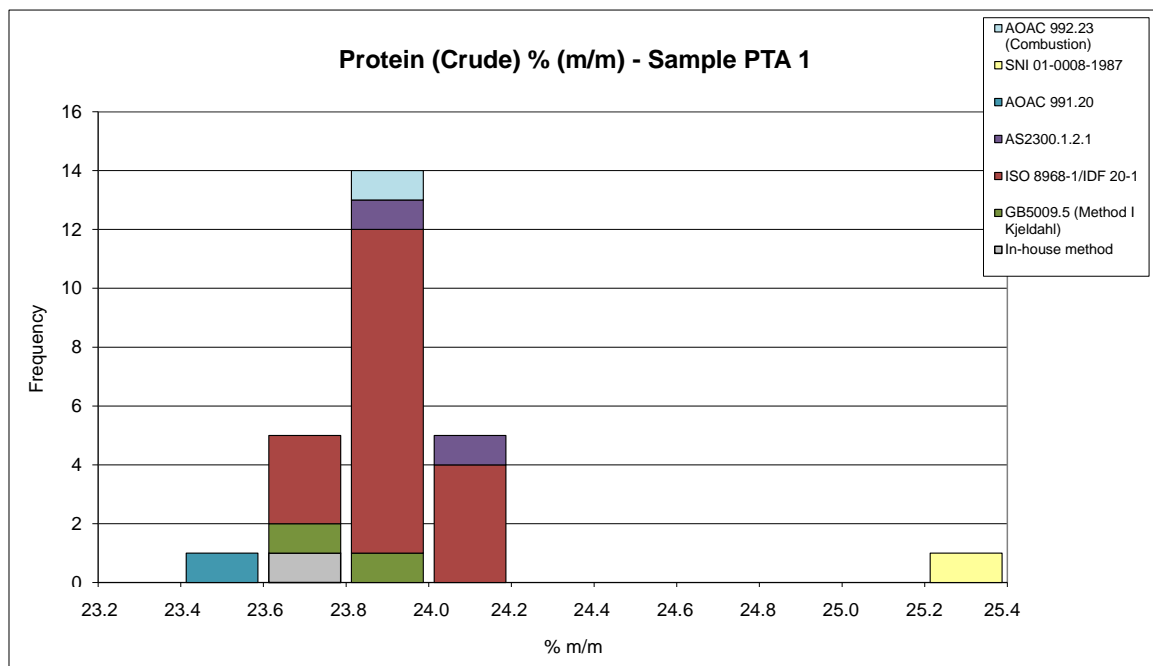


Figure TA-3. Spread of results for Crude Protein proficiency testing via Kjeldahl for sample PTA 1.

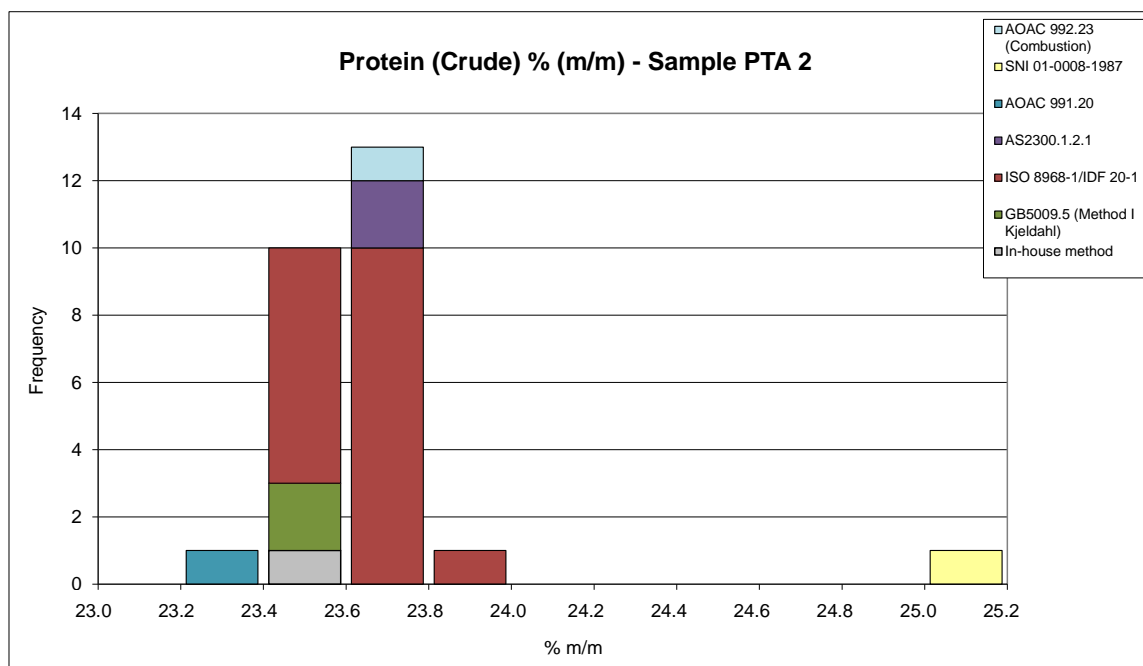


Figure TA-4. Spread of results for Crude Protein proficiency testing via Kjeldahl for sample PTA 2.

Confidence in the medians can be expressed as the uncertainty of the median (as defined in page 3 of this report), which was calculated for each test. For the Crude Protein testing by Kjeldahl methods, the median and associated uncertainty for each sample (expressed in % m/m) was as follows:

	PTA 1	PTA 2
Crude Protein, Kjeldahl methods	23.740 ± 0.033	23.675 ± 0.043

For this round of the program, laboratories were requested to report their MU by repeatability *r* and/or reproducibility *R*. One laboratory reported MU for Crude Protein by repeatability. Two laboratories reported MU for Crude Protein by both repeatability and reproducibility. One laboratory did not provide an estimate of the MU for their Crude Protein results by either repeatability or reproducibility.

Laboratories whose measurement uncertainty range encompassed the median or were within the uncertainty of the median range are considered an accurate representation of the laboratory's uncertainty of measurement.

6.5 Fat

Six laboratories tested the samples for Fat. One laboratory submitted three sets of results for the AOAC 2003.06 method. One laboratory submitted results for the AOAC 960.39 method. One laboratory tested using ISO 1736. One laboratory tested using ISO 2446. One laboratory tested using AACC 30-10.01. One laboratory tested using an in-house modified Mojonnier method.

The results for Fat were analysed against the results submitted for the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.

Target CVs of 2.0% were used to analyse the results for both samples. This target CV was chosen to correspond to the robust CVs obtained for Round 39 of the Food program (see Report No. 932).

Z-scores were not calculated for the laboratory that used the Gerber method, ISO 2446, as all other laboratories used an RG method.

Laboratories 1 and 5 reported outlier results for both samples. Laboratory 4 reported an outlier for sample PTA 1. All results were lower than the assigned values.

Confidence in the medians can be expressed as the uncertainty of the median (as defined in page 3 of this report), which was calculated for each test. For the Fat testing, the median and associated uncertainty for each sample (expressed in % m/m) was as follows:

	PTA 1	PTA 2
Fat, excluding Gerber methods	27.825 ± 0.024	27.925 ± 0.027

Figures TA-5 and TA-6 show the distribution of results from the methods used for Fat testing in this round including the Global Proficiency data (and Gerber Fat results), and are included for interest purposes only. **Note:** For sample PTA 1, the x axis scale was not extended to the lowest reported value of 13.09%; instead, this value is shown in the 25.8 – 26% range. For sample PTA 2, the x axis scale was not extended to the lowest reported value of 11.03%; instead, this value is also shown in the 25.8 – 26% range.

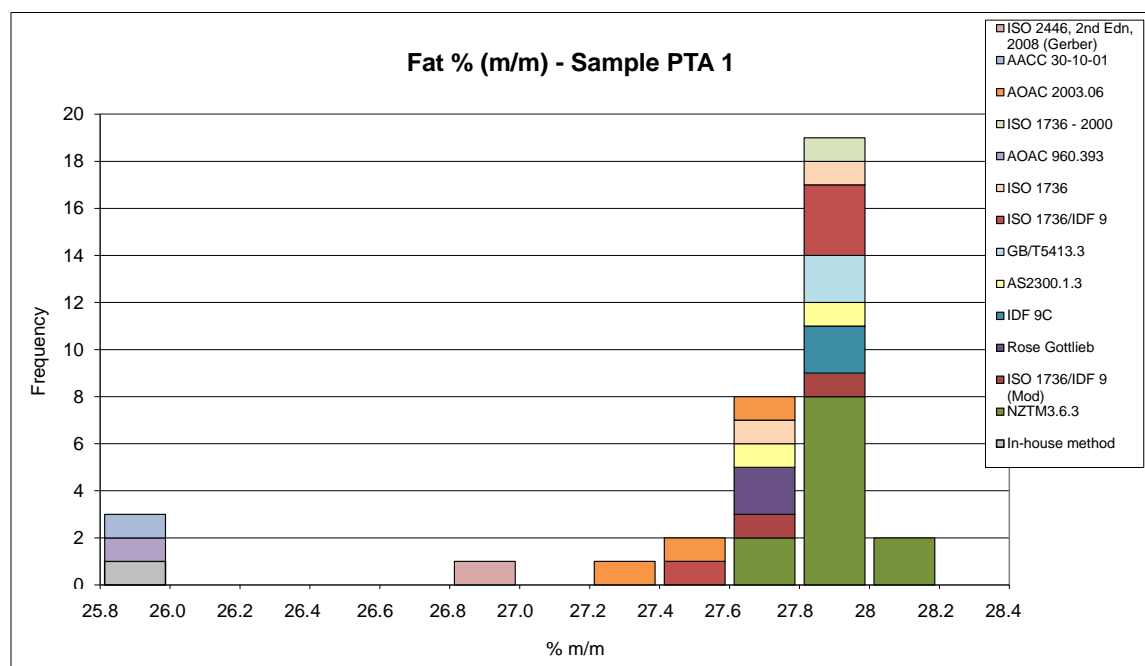


Figure TA-5. Spread of results for Fat proficiency testing of sample PTA 1.

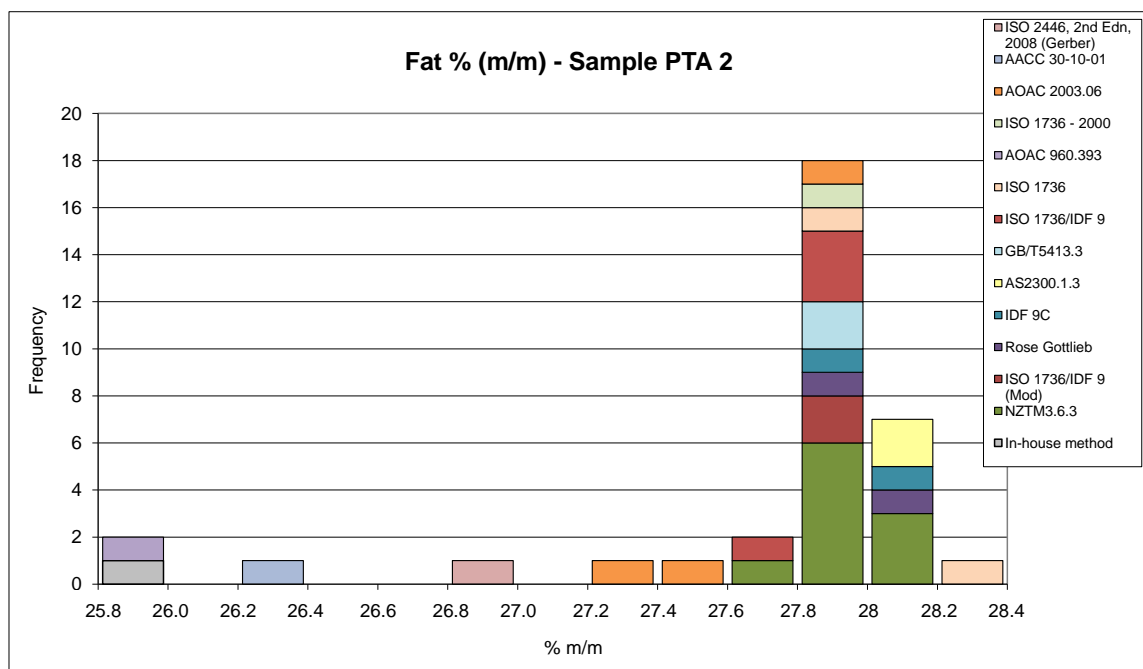


Figure TA-6. Spread of results for Fat proficiency testing of sample PTA 2.

For this round of the program, laboratories were requested to report their MU by repeatability r and/or reproducibility R . Two laboratories reported MU for Fat by repeatability. Two laboratories reported MU for Fat by both repeatability and reproducibility. Two laboratories did not provide an estimate of the MU for their Fat results by either repeatability or reproducibility.

Laboratories whose measurement uncertainty range encompassed the median or were within the uncertainty of the median range are considered an accurate representation of the laboratory's uncertainty of measurement.

6.6 Ash

Of the five laboratories that tested the samples for Ash, three laboratories tested using AOAC 930.30, one laboratory tested using AACC 08-01.01 and one laboratory used an in-house method.

The results for Ash were pooled and analysed against the results submitted for the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.

Target CVs of 1.1% and 1.2% were used to analyse the results for samples PTA 1 and PTA 2, respectively. These target CVs were chosen to correspond to the robust standard deviations obtained for the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.

Laboratory 1 reported an outlier result for sample PTA 1. Laboratory 2 reported an outlier for sample PTA 2.

Confidence in the medians can be expressed as the uncertainty of the median (as defined in page 3 of this report), which was calculated for each test. For the

Ash testing, the median and associated uncertainty for each sample (expressed in % m/m) was as follows:

	PTA 1	PTA 2
Ash, all methods pooled	5.715 ± 0.007	5.560 ± 0.015

Figures TA-7 and TA-8 show the distribution of results from the methods used for Ash testing in this round including the Global Proficiency data, and are included for interest purposes only.

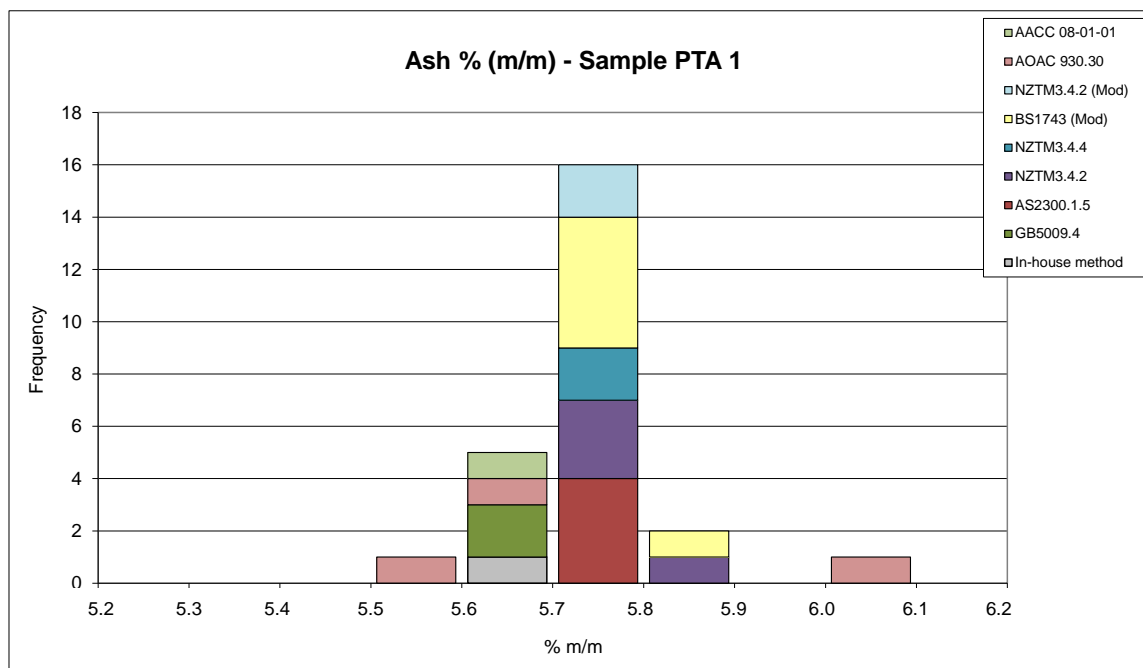


Figure TA-7. Spread of results for Ash proficiency testing of sample PTA 1.

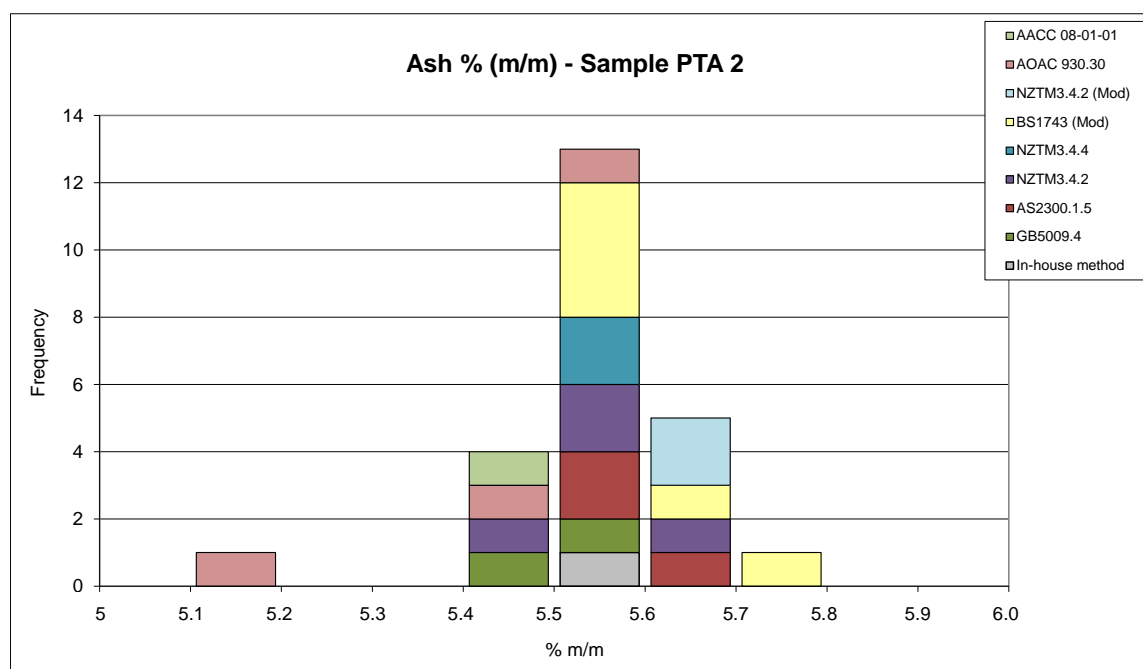


Figure TA-8. Spread of results for Ash proficiency testing of sample PTA 2.

For this round of the program, laboratories were requested to report their MU by repeatability r and/or reproducibility R . One laboratory reported MU for Ash by repeatability. Two laboratories reported MU for Ash by both repeatability and reproducibility. Two laboratories did not provide an estimate of the MU for their Ash results by either repeatability or reproducibility.

Laboratories whose measurement uncertainty range encompassed the median or were within the uncertainty of the median range are considered an accurate representation of the laboratory's uncertainty of measurement.

6.7 Measurement Uncertainty

For this round of the program, laboratories were requested to report their MU by repeatability r and/or reproducibility R for each test result. The proportion of MU estimates returned for each individual test is as follows:

<u>Test</u>	<u>Repeatability (r)</u>	<u>Reproducibility (R)</u>
Moisture	4 out of 6 67%	2 out of 6 33%
Crude Protein	3 out of 4 75%	2 out of 4 50%
Fat	4 out of 6 67%	2 out of 6 33%
Ash	3 out of 5 60%	2 out of 5 40%

Participants were also asked to describe the method used for estimating their MU. Only one laboratory provided this information, which can be found in Table C below.

Table C: Method of Measurement Uncertainty Estimation

Lab Code	Method
6	Reference MU: JCGM 100 - 2008.

7. REFERENCES

1. *Guide to Proficiency Testing Australia (2016)*. (This document is located on the PTA website at www.pta.asn.au under Programs / Documents).
2. AOAC 984.25 (1984) *Moisture (loss of mass on drying) in frozen french-fried potatoes. Convection method.*
3. AOAC 925.40 (1925) *Loss on drying (moisture) in nuts and nut products.*
4. AOAC 950.46 (2008) *Moisture in Meat.*
5. MS 1191 (1991) *Cereal-based snack foods – Specification.*
6. AOAC 991.20 (1994) *Nitrogen (Total) in Milk - Kjeldahl Methods.*
7. AOAC 992.23 (1998) *Crude protein in cereal grains and oilseeds. Generic combustion method.*
8. SNI 01-0008 (1987) *Palm kernel oil cakes.*
9. AOAC 2003.06 (2006) *Crude Fat in Feeds, Cereal, Grains and Forages – Randall/Soxtec/Hexanes Extraction-Submersion Method.*
10. AOAC 960.39 (1960) *Fat (crude) or ether extract in meat.*
11. AACC Method 30-10.01. *Crude Fat in Flour, Bread, and Baked Cereal Products Not Containing Fruit.*
12. ISO 1736 (2008) *Dried milk and dried milk products – Determination of fat content – Gravimetric method (Reference method).*
13. ISO 2446 (2008) *Milk – Determination of fat content.*
14. AOAC 930.30 (1930) *Ash of Dried Milk.*
15. AACC Method 08-01.01. *Ash Content in Flour and Bread – Basic Method.*

APPENDIX A

Summary of Results

Section A1

Moisture

A1.1

Whole Milk Powder – Moisture (% m/m) Results and Measurement Uncertainty

Lab Code	Sample PTA 1				Sample PTA 2			
	Result 1	Result 2	MU r (±)	MU R (±)	Result 1	Result 2	MU r (±)	MU R (±)
1	3.34	3.27	-	-	3.82	3.84	-	-
2	2.40	2.37	0.04	-	2.84	2.82	0.03	-
3A	3.16	3.17	-	-	3.68	3.70	-	-
3B	3.13	3.07	-	-	3.67	3.64	-	-
3C	3.05	3.10	-	-	3.64	3.50	-	-
4	3.22	3.24	0.3	0.6	3.57	3.55	0.3	0.6
5	1.13	1.16	0.02	0.04	2.61	2.52	0.05	0.09
6	3.12	3.14	0.12	-	3.20	3.27	0.12	-

Whole Milk Powder – Moisture (% m/m) Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code	Temp (°C)	Time (hrs)
	Average	Z-Score	Average	Z-Score			
1	3.31	2.35	3.83	4.85 §	2	103	4
2	2.39	-4.62 §	2.83	-2.73	5	102	16.50
3A	3.17	1.29	3.69	3.79 §	2	103 ± 2	16
3B	3.10	0.80	3.66	3.52 §	2	103 ± 2	16
3C	3.08	0.61	3.57	2.88	2	103 ± 2	16
4	3.23	1.78	3.56	2.80	5	100	16
5	1.15	-14.02 §	2.57	-4.73 §	5	105	5
6	3.13	1.02	3.24	0.34	5	105 ± 2	2

Method Codes

1 = AS2300.1.1	0
2 = AOAC 984.25	4
3 = AOAC 927.05	0
4 = IDF 26A	0
5 = Other	4

A1.2

Summary Statistics

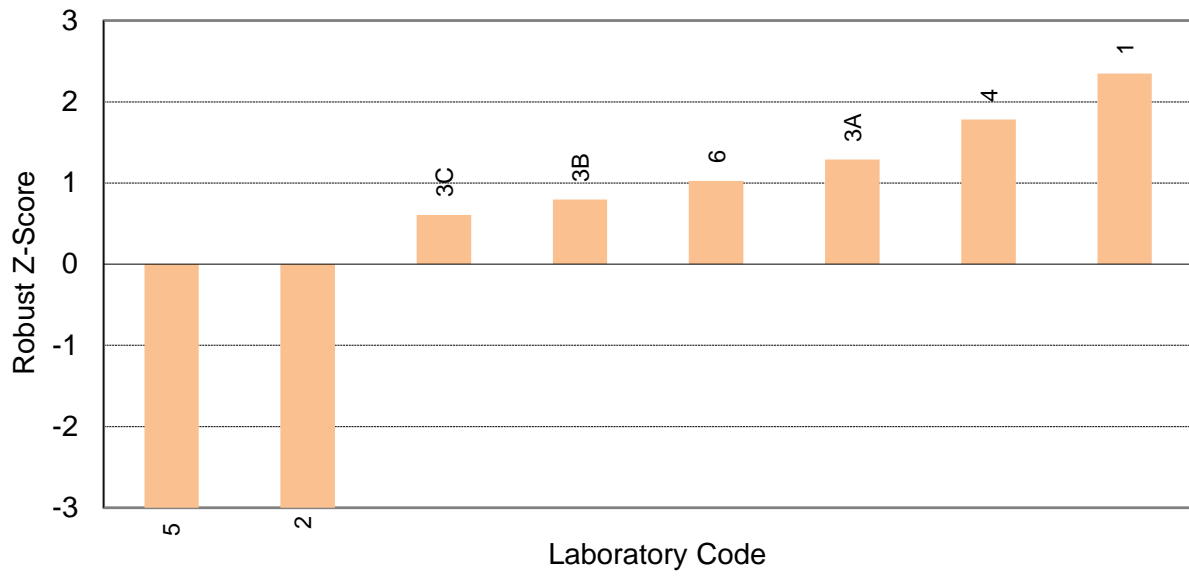
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	26	27
Median	2.995	3.190
Normalised IQR	0.022	0.115
Uncertainty (Median)	0.005	0.028
Robust CV	0.7%	3.6%
Target SD	0.132	0.132
Target CV	4.4%	4.1%
Minimum	2.80	2.91
Maximum	3.16	3.45
Range	0.36	0.54

Notes:

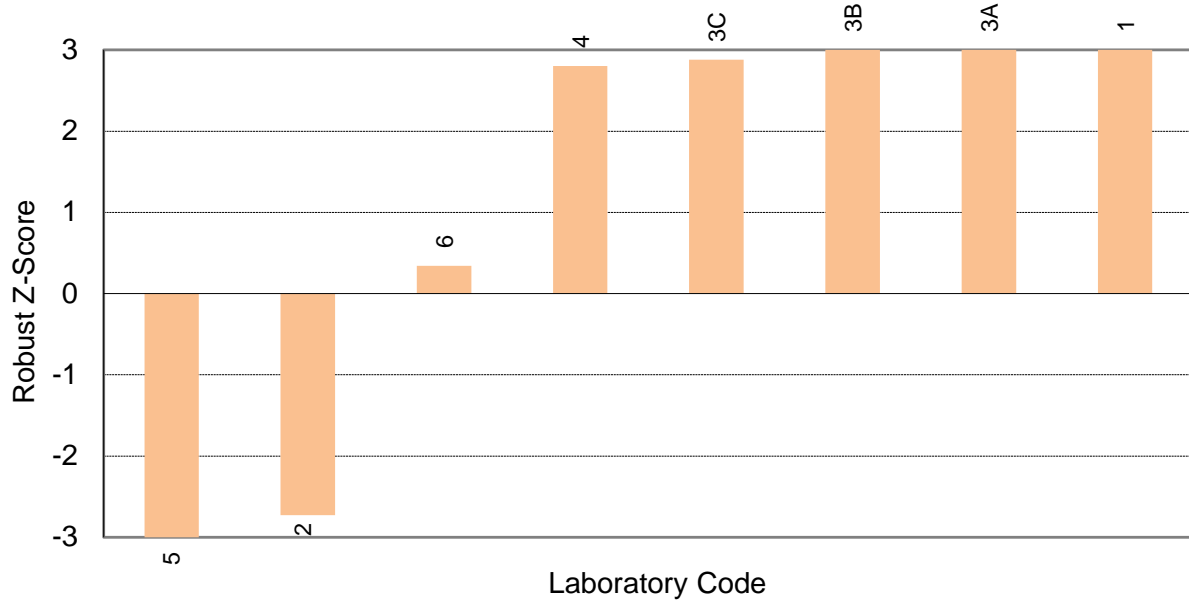
1. § denotes an outlier (i.e. $|z\text{-score}| \geq 3.0$).
2. Z-scores were calculated for the average result for each sample.
3. Z-scores and summary statistics (including the number of results) for Moisture were calculated from the results for the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.
4. A target CV was used to calculate the robust z-scores for both samples.
5. The target SD was obtained for each sample by multiplying the target CV by the median. These values were used to calculate the z-scores. For more information on the use of target CVs to calculate z-scores, please see the Guide to Proficiency Testing Australia (2016).
6. Laboratory 2 used AOAC 950.46 for their Moisture testing.
7. Laboratory 4 used AOAC 925.45D for their Moisture testing.
8. Laboratory 5 used MS 1191: 1991 for their Moisture testing.
9. Laboratory 6 used AOAC 925.40 for their Moisture testing.

A1.3

Moisture (% m/m) - Sample PTA 1



Moisture (% m/m) - Sample PTA 2



Section A2

Crude Protein

A2.1

Whole Milk Powder – Crude Protein (% m/m) Results and Measurement Uncertainty

Lab Code	Sample PTA 1				Sample PTA 2			
	Result 1	Result 2	MU r (±)	MU R (±)	Result 1	Result 2	MU r (±)	MU R (±)
2	23.40	23.33	0.10	-	23.27	23.21	0.09	-
4	23.73	23.73	0.15	0.3	23.60	23.67	0.15	0.3
5	23.73	23.46	0.14	0.27	23.35	23.73	0.19	0.38
6	26.19	26.23	-	-	26.40	26.46	-	-

Whole Milk Powder – Crude Protein (Kjeldahl) (% m/m) Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code
	Average	Z-Score	Average	Z-Score	
2	23.37	-2.35	23.24	-2.73	7
5	23.60	-0.91	23.54	-0.85	7
6	26.21	15.46 §	26.43	17.29 §	7

Whole Milk Powder – Crude Protein (Combustion) (% m/m) Average Results and Methods

Lab Code	Sample PTA 1	Sample PTA 2	Method Code
	Average	Average	
4	23.73	23.64	7

Method Codes

1 = AS2300.1.2.1	0
2 = AS2300.1.2.1 Appendix A	0
3 = ISO8968-1/IDF20-1	0
4 = IDF 20B	0
5 = AOAC 991.23	0
6 = AOAC 930.29a	0
7 = Other	4

A2.2

Summary Statistics (Kjeldahl)

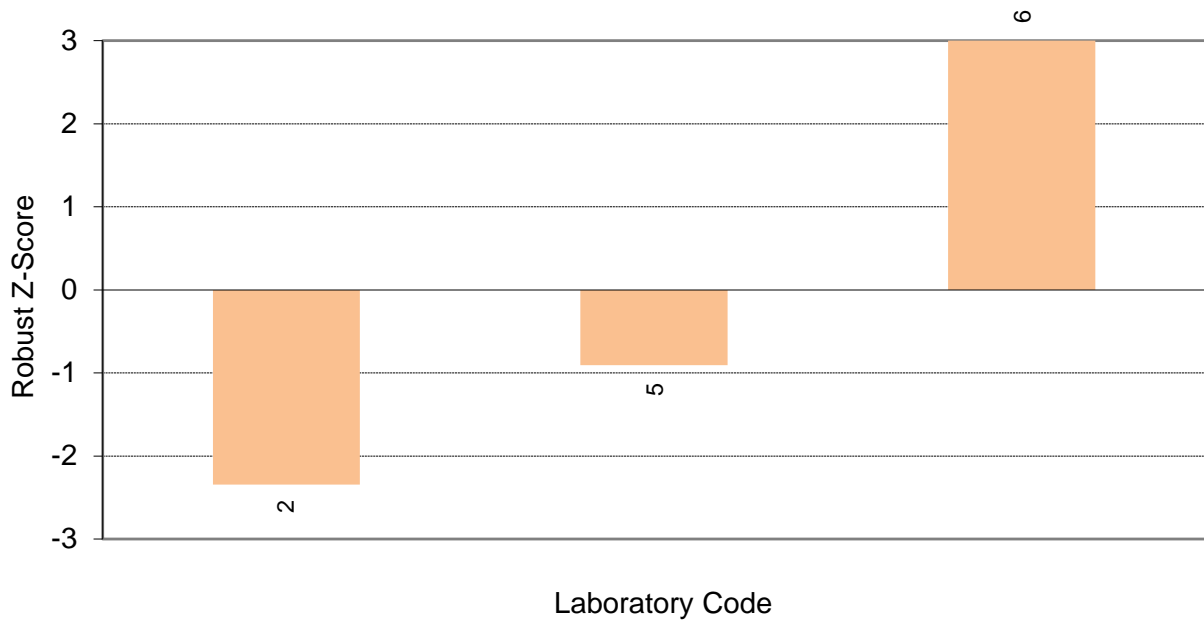
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	22	22
Median	23.740	23.675
Normalised IQR	0.124	0.159
Uncertainty (Median)	0.033	0.043
Robust CV	0.5%	0.7%
Target SD	0.160	0.159
Target CV	0.7%	0.7%
Minimum	23.54	23.46
Maximum	23.90	23.80
Range	0.36	0.34

Notes:

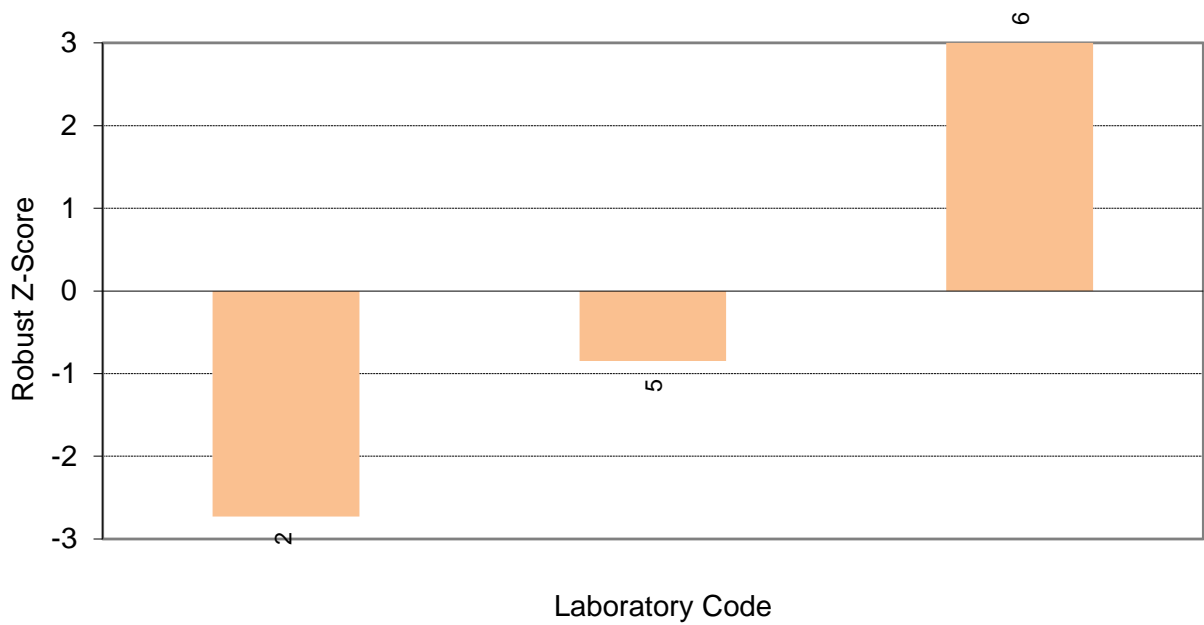
1. § denotes an outlier (i.e. $|z\text{-score}| \geq 3.0$).
2. Z-scores were calculated for the average result for each sample.
3. The Kjeldahl and Combustion method results were analysed separately.
4. Z-scores and summary statistics (including the number of results) for Crude Protein by Kjeldahl methods were calculated from the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.
5. Z-scores and summary statistics were not calculated for Crude Protein by Combustion methods due to an insufficient number of results reported for the Global Proficiency Ltd DairyChek Chemistry program.
6. A target CV was used to calculate the robust z-scores for both samples.
7. The target SD was obtained for each sample by multiplying the target CV by the median. These values were used to calculate the z-scores. For more information on the use of target CVs to calculate z-scores, please see the Guide to Proficiency Testing Australia (2016).
8. Laboratory 2 used AOAC 991.20 for their Crude Protein testing.
9. Laboratory 4 used AOAC 992.23 for their Crude Protein testing.
10. Laboratory 5 used an in-house Kjeldahl method for their Crude Protein testing.
11. Laboratory 6 used SNI 01-0008 for their Crude Protein testing.

A2.3

Crude Protein (Kjeldahl) (% m/m) - Sample PTA 1



Crude Protein (Kjeldahl) (% m/m) - Sample PTA 2



Section A3

Fat

A3.1

Whole Milk Powder – Fat (% m/m) Results and Measurement Uncertainty

Lab Code	Sample PTA 1				Sample PTA 2			
	Result 1	Result 2	MU r (±)	MU R (±)	Result 1	Result 2	MU r (±)	MU R (±)
1	25.80	26.07	-	-	25.45	25.40	-	-
2	27.93	27.96	0.52	-	27.91	27.98	0.52	-
3A	27.69	27.76	-	-	27.83	27.79	-	-
3B	27.55	27.48	-	-	27.66	27.48	-	-
3C	27.27	27.22	-	-	27.10	27.50	-	-
4	25.34	25.86	0.2	0.5	26.22	26.47	0.2	0.5
5	12.89	13.29	0.20	0.40	11.06	10.99	0.04	0.07
6	26.99	26.96	0.02	-	26.96	26.94	0.02	-

Whole Milk Powder – Fat (% m/m) Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code
	Average	Z-Score	Average	Z-Score	
1	25.94	-3.40 §	25.43	-4.48 §	6
2	27.95	0.22	27.95	0.04	6
3A	27.73	-0.18	27.81	-0.21	4
3B	27.52	-0.56	27.57	-0.64	4
3C	27.25	-1.04	27.30	-1.12	4
4	25.60	-4.00 §	26.35	-2.83	6
5	13.09	-26.48 §	11.03	-30.26 §	6
6	26.98	-	26.95	-	6

Method Codes

1 = AS2300.1.3	0
2 = AOAC 989.05	0
3 = AOAC 932.06	0
4 = AOAC 2003.06	3
5 = IDF 9C	0
6 = Other	5

A3.2

Summary Statistics (excluding Gerber)

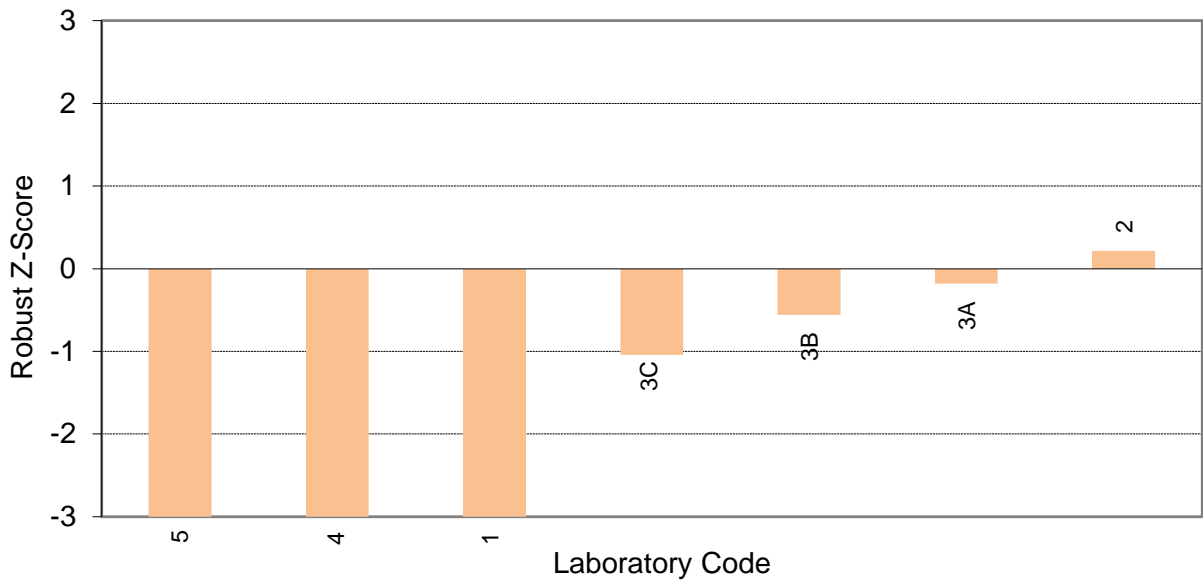
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	28	26
Median	27.825	27.925
Normalised IQR	0.100	0.109
Uncertainty (Median)	0.024	0.027
Robust CV	0.4%	0.4%
Target SD	0.557	0.559
Target CV	2.0%	2.0%
Minimum	27.45	27.76
Maximum	28.06	28.22
Range	0.61	0.46

Notes:

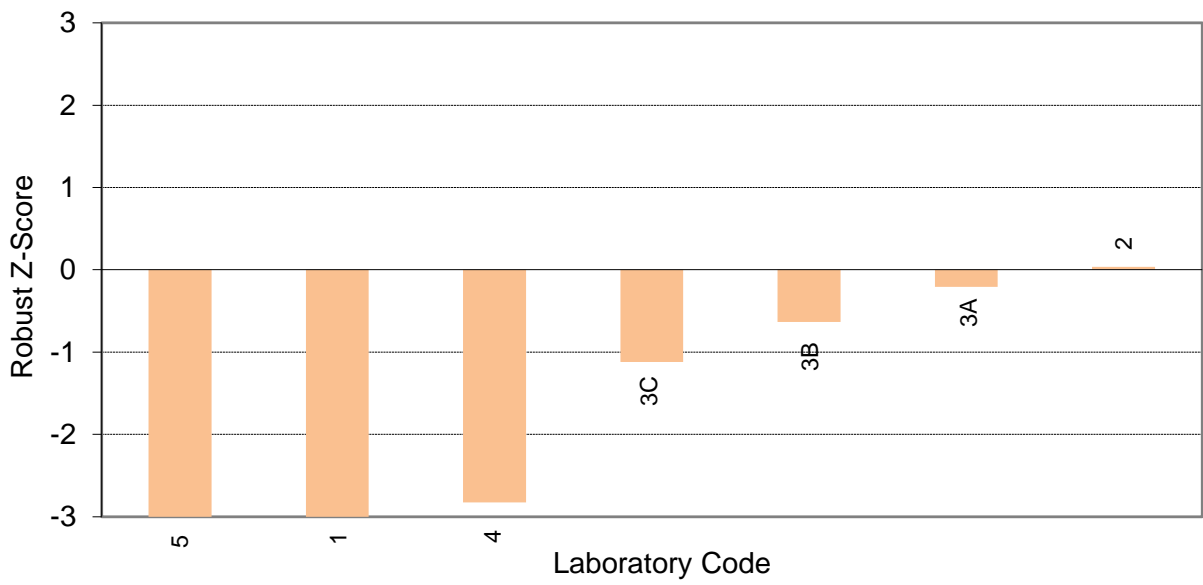
1. § denotes an outlier (i.e. $|z\text{-score}| \geq 3.0$).
2. Z-scores were calculated for the average result for each sample.
3. Z-scores and summary statistics (including the number of results) for Fat were calculated from the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.
4. Z-scores were not calculated for laboratory 6, which used a Gerber method.
5. A target CV was used to calculate the robust z-scores for both samples.
6. The target SD was obtained for each sample by multiplying the target CV by the median. These values were used to calculate the z-scores. For more information on the use of target CVs to calculate z-scores, please see the Guide to Proficiency Testing Australia (2016).
7. Laboratory 1 used AOAC 960.39 for their Fat testing.
8. Laboratory 2 used ISO 1736 for their Fat testing.
9. Laboratory 4 used AACC 30-10.01 for their Fat testing.
10. Laboratory 5 used an in-house modified Mojonnier method for their Fat testing.
11. Laboratory 6 used ISO 2446 for their Fat testing.

A3.3

Fat (% m/m) - Sample PTA 1



Fat (% m/m) - Sample PTA 2



Section A4

Ash

A4.1

Whole Milk Powder – Ash (% m/m) Results and Measurement Uncertainty

Lab Code	Sample PTA 1				Sample PTA 2			
	Result 1	Result 2	MU r (±)	MU R (±)	Result 1	Result 2	MU r (±)	MU R (±)
1	6.03	5.97	-	-	5.60	5.40	-	-
2	5.59	5.55	0.01	-	5.08	5.17	0.01	-
4	5.65	5.64	0.1	0.17	5.49	5.50	0.1	0.17
5	5.69	5.70	0.01	0.01	5.56	5.55	0.01	0.01
6	5.62	5.60	-	-	5.48	5.50	-	-

Whole Milk Powder – Ash (% m/m) Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code	Temp (°C)	Time (hrs)
	Average	Z-Score	Average	Z-Score			
1	6.00	4.38 §	5.50	-0.92	4	550	4
2	5.57	-2.23	5.13	-6.69 §	4	550	16.50
4	5.65	-1.08	5.50	-1.00	5	580	16
5	5.70	-0.31	5.56	-0.08	5	550	2
6	5.61	-1.62	5.49	-1.08	4	550	2

Method Codes

1 = AS2300.1.5	0
2 = AOAC 945.45	0
3 = AOAC 942.05	0
4 = AOAC 930.30	3
5 = Other	2

A4.2

Summary Statistics

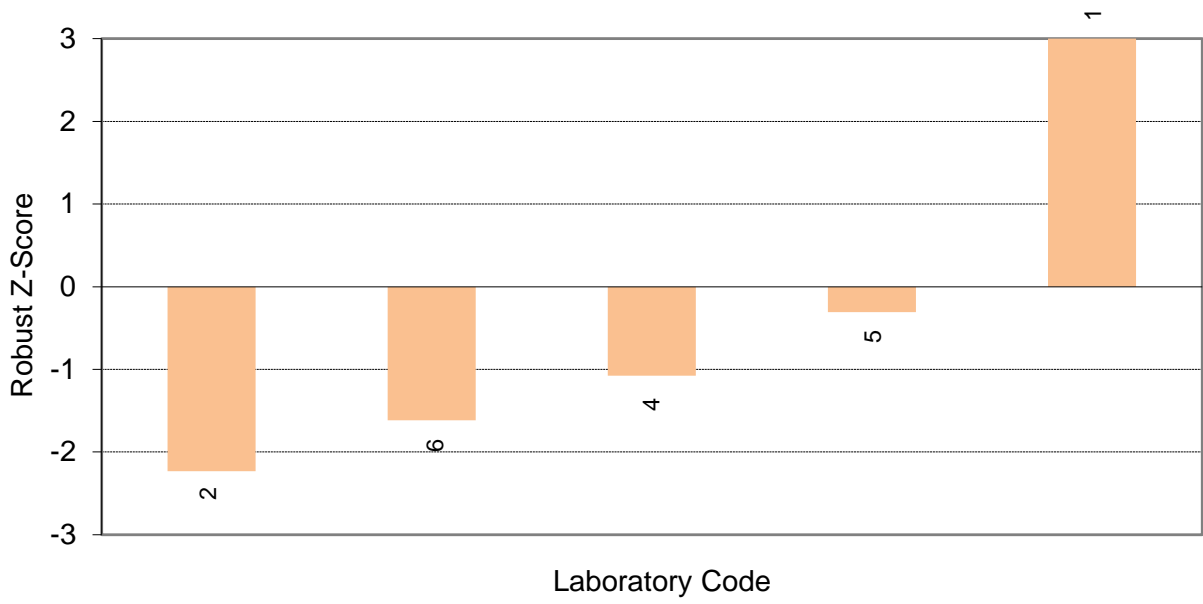
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	20	19
Median	5.715	5.560
Normalised IQR	0.026	0.052
Uncertainty (Median)	0.007	0.015
Robust CV	0.5%	0.9%
Target SD	0.065	0.065
Target CV	1.1%	1.2%
Minimum	5.65	5.41
Maximum	5.82	5.70
Range	0.17	0.29

Notes:

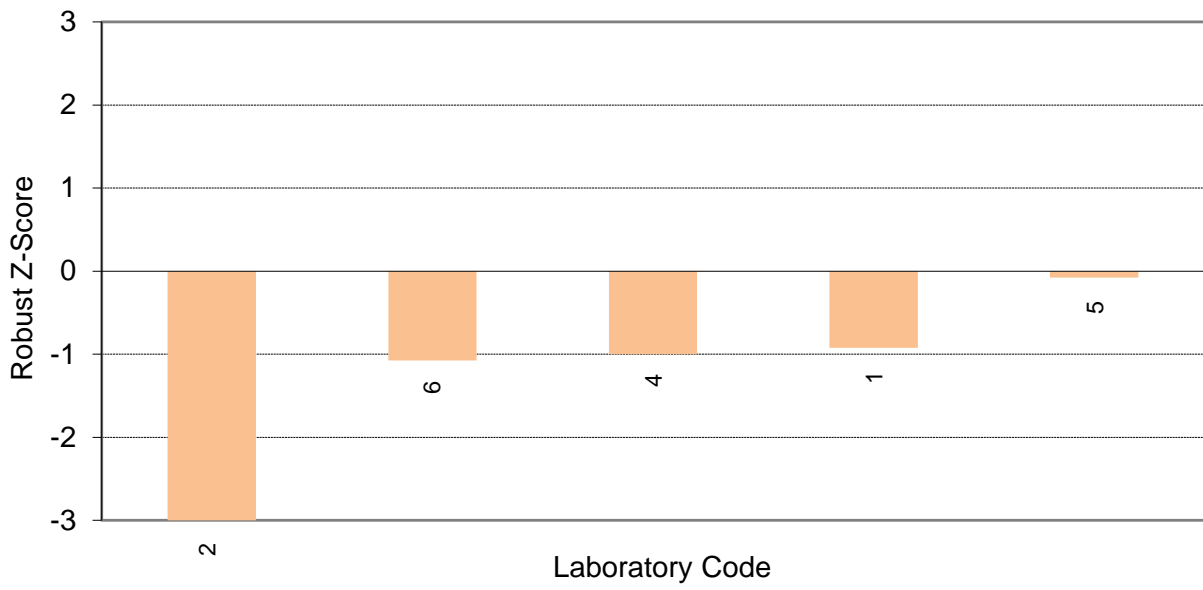
1. § denotes an outlier (i.e. $|z\text{-score}| \geq 3.0$).
2. Z-scores were calculated for the average result for each sample.
3. Z-scores and summary statistics (including the number of results) for Ash were calculated from the results for the Global Proficiency Ltd DairyChek Chemistry program, using the same samples.
4. A target CV was used to calculate the robust z-scores for both samples.
5. The target SD was obtained for each sample by multiplying the target CV by the median. These values were used to calculate the z-scores. For more information on the use of target CVs to calculate z-scores, please see the Guide to Proficiency Testing Australia (2016).
6. Laboratory 4 used AACC 08-01.01 for their Ash testing.
7. Laboratory 5 used an in-house method for their Ash testing.

A4.3

Ash (% m/m) - Sample PTA 1



Ash (% m/m) - Sample PTA 2



APPENDIX B

Homogeneity and Stability Testing

B1.1

Homogeneity Testing

Prior to distribution eight samples of whole milk powder, from the batch used for sample PTA 1, were tested for homogeneity by Global Proficiency Ltd (New Zealand). Each sample was tested in duplicate for Crude Protein. The results of the homogeneity testing appear in the following table.

Whole Milk Powder – Crude Protein (% m/m)		
Sample No.	Result A	Result B
260513	23.75	23.82
260141	23.64	23.59
260428	23.53	23.60
260516	23.62	23.61
260397	23.57	23.60
260486	23.56	23.50
260136	23.60	23.57
260434	23.61	23.59

Analysis of this data indicated that the samples were sufficiently homogeneous and, therefore, any participant results identified as outliers cannot be attributed to sample variability.

Stability Testing

Three samples from the batch used for sample PTA 1 were tested for stability by Global Proficiency Ltd (New Zealand). The results, below, indicated that the samples were sufficiently stable for use in this program.

Whole Milk Powder – Crude Protein (% m/m)		
Sample No.	Result A	Result B
260120	23.63	23.58
260509	23.57	23.59
260430	23.59	23.52

APPENDIX C

Instructions to Participants

and

Results Sheet

PROFICIENCY TESTING AUSTRALIA

Food Proficiency Testing Program
Round 41, July 2017

INSTRUCTIONS TO PARTICIPANTS

To ensure that the results of this program can be analysed correctly, participants are asked to note carefully:

- 1) Two samples of whole milk powder (each approximately 100 g), labelled PTA 1 and PTA 2, have been provided for compositional analysis. These samples are provided in foil laminated sachets and should be stored below 30 °C prior to testing. These samples may be tested for some, or all of the following tests, according to each laboratory's requirements.
- 2) The following tests are to be performed on each sample in **duplicate** and the **results reported**:
 - Moisture
 - Protein (Crude)
 - Fat
 - Ash
- 3) The tests may commence as soon as samples are received. Analysts should be aware of analyte stability and perform tests in an appropriate order. The conversion factor to be used for reporting protein is $N \times 6.38$.
- 4) Report results on the attached **Results Sheet** to the specified number of decimal places. Results should not be reported as "greater than" or "less than", as such data cannot be statistically analysed.
- 5) Please identify the methods used on the **Results Sheet**, using the Method Codes listed on Page 2 of these instructions. Laboratories should use the routine test methods which would normally be used to test customer supplied samples.
- 6) Laboratories are also requested to calculate and report an estimate of measurement uncertainty (MU) for each reported measurement result. All estimates of measurement uncertainty must be given as a 95% confidence interval (coverage factor $k \approx 2$).
- 7) Return **Results Sheets**, either by mail, facsimile or email to:

Mark Bunt Proficiency Testing Australia PO Box 7507 Silverwater NSW 2128 AUSTRALIA	Telephone: +61 2 9736 8397 (1300 782 867) Fax: +61 2 9743 6664 Email: mbunt@pta.asn.au
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All results should arrive at the above address by no later than **Friday 4 August 2017**. Results reported later than this date may not be analysed in the final report.

C1.2

PROFICIENCY TESTING AUSTRALIA

Food Proficiency Testing Program Round 41, July 2017



METHOD CODES

Analysis	Method	Code
Moisture (% m/m)	AS2300.1.1	1
	AOAC 984.25	2
	AOAC 927.05	3
	IDF 26A	4
	Other (please specify)	5
Protein (Crude) (% m/m)	AS2300.1.2.1	1
	AS2300.1.2.1 Appendix A	2
	ISO8968-1/IDF20-1	3
	IDF 20B	4
	AOAC 991.23	5
	AOAC 930.29a	6
	Other (please specify)	7
Fat (% m/m)	AS2300.1.3	1
	AOAC 989.05	2
	AOAC 932.06	3
	AOAC 2003.06	4
	IDF 9C	5
	Other (please specify)	6
Ash (% m/m)	AS2300.1.5	1
	AOAC 945.45	2
	AOAC 942.05	3
	AOAC 930.30	4
	Other (please specify)	5

C2.1

PROFICIENCY TESTING AUSTRALIA

Food Proficiency Testing Program

Round 41, July 2017



RESULTS SHEET

Laboratory Code:

Date Samples Received: _____

Temperature on Arrival: _____

Test	Sample 1				Sample 2				Date Tested	Method Code
	Result 1	Result 2	MU (\pm) r	MU (\pm) R	Result 1	Result 2	MU (\pm) r	MU (\pm) R		
Moisture* (0.01% m/m)										
Protein (Crude) (N x 6.38) (0.01% m/m)										
Fat (0.01% m/m)										
Ash** (0.01% m/m)										

* Please specify the temperature/time of moisture determination: _____ $^{\circ}$ C/ _____ hours.** Please specify the temperature/time of ashing: _____ $^{\circ}$ C/ _____ hours.

Report results for Fat as % fat in sample, not as fat in dry matter.

Report measurement uncertainty (MU) by repeatability r and/or reproducibility R.

All estimates of MU must be given as a 95% confidence interval (coverage factor $k \approx 2$).

Please state below the method used to determine the MU (e.g. GUM (bottom up), proficiency trial data, in-house precision data, Horwitz equation, "best guess", etc.)

Print Name: _____ Signature & Date: _____

----- End of report -----