

REPORT NO. 881

**Food Proficiency Testing Program
Round 38 – Wheat Flour**

September 2014

ACKNOWLEDGMENTS

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1. FOREWORD

This report summarises the results of a proficiency testing program involving the analysis of wheat flour samples. It constitutes the thirty-eighth round of an ongoing series of programs involving chemical analysis of foodstuffs.

Proficiency Testing Australia (PTA) conducted the testing program in July / August 2014. 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 Dr R Hutchinson and Mr L Cleave, Global Proficiency Ltd (New Zealand). This report was authorised by Mrs F Watton, PTA Quality – Business Development Manager.

2. FEATURES OF THE PROGRAM

- (a) A total of 10 laboratories participated in the program, all of which returned results for inclusion in the final report. Laboratories from the following countries participated:

6	AUSTRALIA
2	MALAYSIA
1	NEW ZEALAND
1	DEMOCRATIC REPUBLIC OF CONGO

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.

- (b) The results reported by participants are presented in Appendix A.
- (c) Laboratories were provided with two samples of approximately 100 g of wheat flour, labelled PTA 1 and PTA 2.
- (d) Participants were requested to determine the levels of:
- Protein
 - Total Fat
 - Moisture
 - Ash
 - Dietary Fibre
 - Carbohydrate
 - Energy

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, seven samples were analysed for homogeneity by Global Proficiency Ltd (New Zealand). An additional three samples were analysed for stability 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 seven sections (A1–A7). These sections contain the analysis of results reported by laboratories for Protein, Total Fat, Moisture, Ash, Dietary Fibre, Carbohydrate and Energy.

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;
 - iv) ordered z-score charts; and
 - v) a Youden diagram of laboratories' results for the sample pair.
- (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

A uniform pair statistical design was chosen for this program. Samples PTA 1 and PTA 2 were identical for Protein, Total Fat, Moisture, Ash, Dietary Fibre, Carbohydrate and Energy.

5. OUTLIER RESULTS

Robust z-scores have been used to assess each laboratory's testing performance. When calculated from single results, z-scores are used to detect excessively large or excessively small results in comparison to the consensus value (the median). Any result with an absolute z-score greater than or equal to 3.0 (*i.e.* ≤ -3.0 or ≥ 3.0) is classified as an outlier.

For further details on the calculation and interpretation of robust z-scores, please see the *Guide to Proficiency Testing Australia 2012*, (reference [1]).

Table A: Summary Statistics for All Tests

The following table summarises the results submitted by participants for the program.

Test	Summary Statistics	Sample PTA 1	Sample PTA 2
Protein (N x 5.7) (g/100g)	Number of Results	10	10
	Median	11.32	11.34
	Normalised IQR	0.26	0.31
	Uncertainty (Median)	0.10	0.12
Moisture (g/100g)	Number of Results	10	10
	Median	13.23	13.28
	Normalised IQR	0.34	0.25
	Uncertainty (Median)	0.14	0.10
Ash (g/100g)	Number of Results	10	10
	Median	0.475	0.470
	Normalised IQR	0.029	0.015
	Uncertainty (Median)	0.011	0.006
Dietary Fibre (g/100g)	Number of Results	9	9
	Median	3.090	2.925
	Normalised IQR	0.433	0.804
	Uncertainty (Median)	0.181	0.336
Carbohydrate (g/100g)	Number of Results	8	8
	Median	70.25	70.52
	Normalised IQR	0.75	0.62
	Uncertainty (Median)	0.33	0.27
Energy (kJ/100g)	Number of Results	8	8
	Median	1466.3	1466.0
	Normalised IQR	23.7	19.0
	Uncertainty (Median)	10.5	8.4

Notes:

1. For each test, the results for all test methods were pooled and the summary statistics, above, are for the pooled results.
2. Summary statistics were calculated for the average result for each sample.
3. Summary statistics were not calculated for the Total Fat results.

4. The uncertainty of the median was calculated as: $\sqrt{\frac{\pi}{2}} \times \frac{normIQR}{\sqrt{n}}$.

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
Protein	5, 8	5, 8
Moisture	7	2, 7
Ash	-	1
Dietary Fibre	2, 10	2, 10
Carbohydrate	-	-
Energy	7	-

Note:

1. Z-scores were not calculated for the Total Fat results.

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.

6.1 Return Rate

All ten laboratories that participated in the program submitted results. Eight of these ten laboratories (80%) provided results for all of the tests.

The return rate for all tests is as follows:

• Protein	10 out of 10	100%
• Total Fat	9 out of 10	90%
• Moisture	10 out of 10	100%
• Ash	10 out of 10	100%
• Dietary Fibre	9 out of 10	90%
• Carbohydrate	8 out of 10	80%
• Energy	8 out of 10	80%

6.2 Performance Summary

One or more statistical outliers were reported by six of the ten laboratories (60%) that returned results in this round of the Food program. The last round of the Food program where wheat flour samples were used was Round 29 (see Report No. 598). For comparison, 25% of the participants in Round 29 of the Food program reported statistical outliers.

A total of 110 results were analysed in this proficiency round. Of these results, 13 (12%) were outlier results. In Round 29 of the Food program, 6% of the total results reported were outlier results (see Report No. 598).

6.3 Protein

Of the ten laboratories that tested the samples for Protein, four used AOAC 2001.11 (Kjeldahl digestion), three used AOAC 992.23 or AOAC 990.03 (Dumas combustion), one used ISO 20483, one used an in-house method and one laboratory did not specify the method used for testing. All methods were pooled to analyse the Protein results.

Laboratories 5 and 8 reported outlier results for both samples. Laboratory 8 has reported that their outliers were due to using the incorrect conversion factor of 6.25 to calculate their Protein results, instead of using a conversion factor of 5.7.

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 Protein testing, the median and associated standard error (se) for each sample (expressed in g/100g) was as follows:

	PTA 1	PTA 2
Protein, all methods pooled	11.32 ± 0.10	11.34 ± 0.12

Figure TA-1, below, shows the distribution of all results from the methods used for Protein testing in this round.

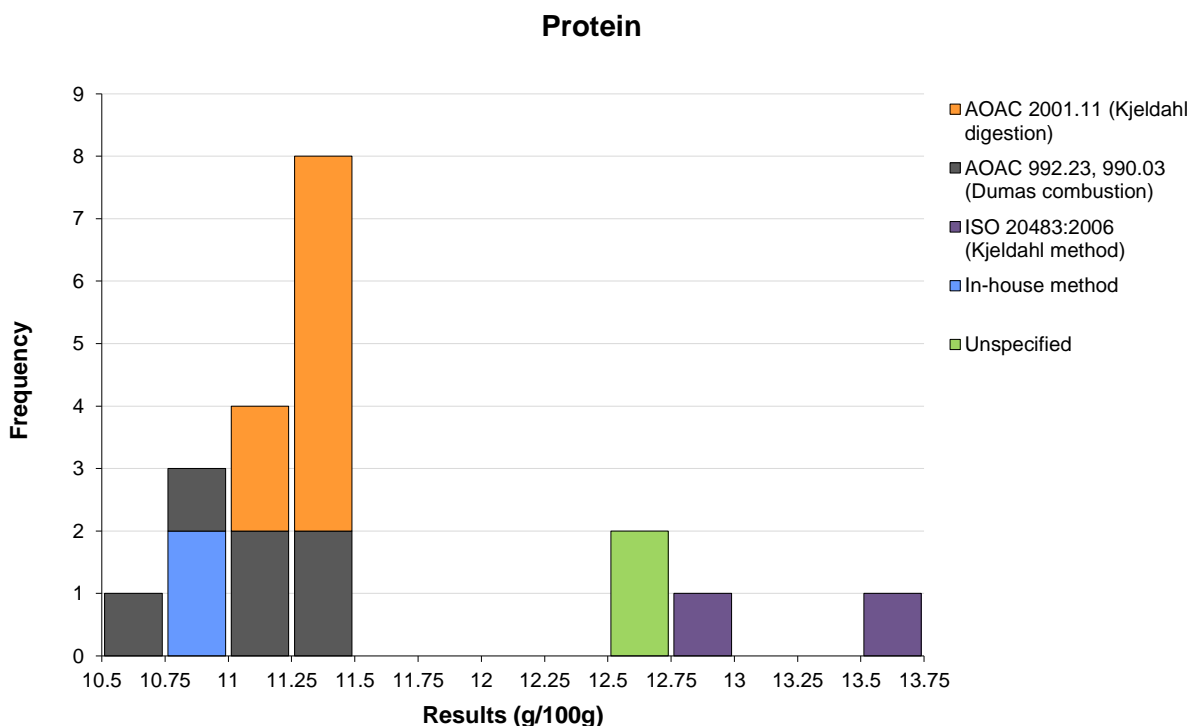


Figure TA-1. Spread of all results for Protein testing of duplicate wheat flour samples PTA 1 and PTA 2.

The robust CVs of 2.3% and 2.7% for the two samples compare well with the values of 2.7% and 2.4% obtained in Round 29 of the Food program (see Report No. 598).

For this proficiency round, laboratories were requested to report their MU by repeatability (r) and / or reproducibility (R). Two laboratories reported MU for Protein by repeatability, and two by reproducibility. Four laboratories reported both repeatability and reproducibility MU for Protein. Two laboratories did not provide an estimate of the MU for their Protein results.

6.4 Total Fat

Nine laboratories tested the samples for Total Fat. Of these laboratories, four tested using acid hydrolysis, three tested using Soxhlet extraction, one tested using an in-house method, while one laboratory did not specify the method used for testing. Simple solvent extractions (without acid hydrolysis) cannot extract bound fats from materials such as wheat flour. This results in differences between results for the different types of methods. These differences become insignificant in fat measurements on materials with higher fat contents. The results for Total Fat, therefore, should be statistically evaluated by method used. Unfortunately, there were not enough results reported using any particular method to calculate z-scores.

Even though z-scores could not be calculated for the Total Fat results this round, laboratory 7 has reported unusually high results for both samples. These results should be investigated.

Figure TA-2, below, shows the distribution of all results from the methods used for Total Fat testing in this round.

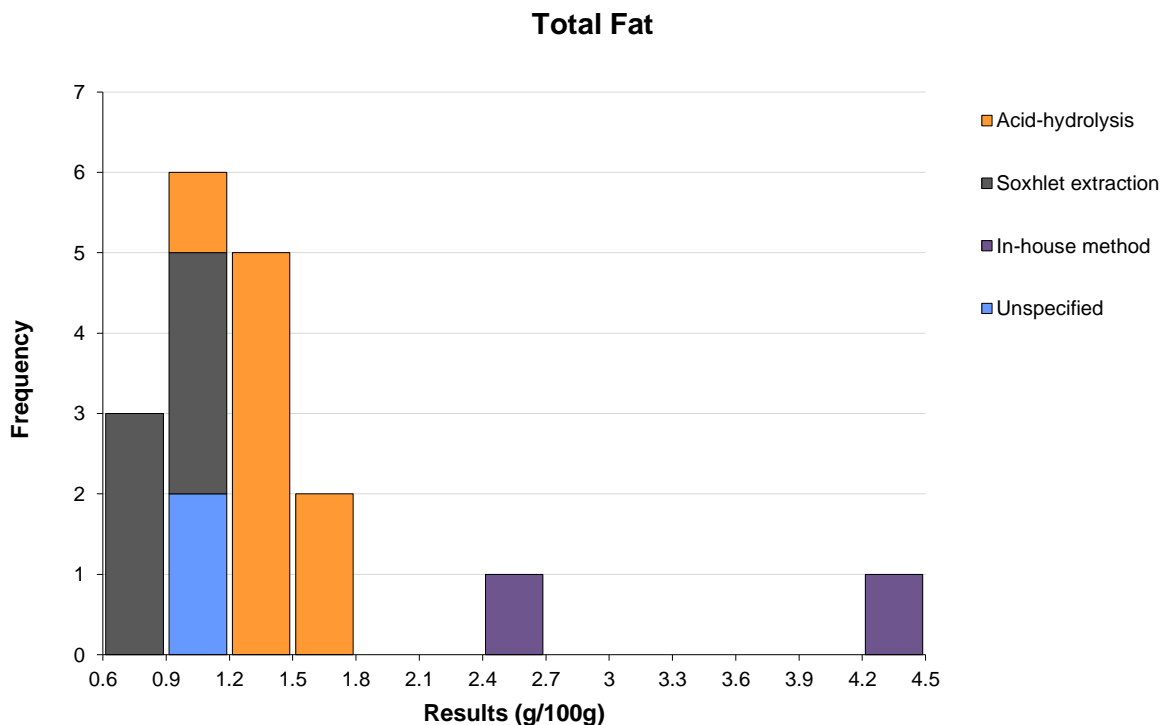


Figure TA-2. Spread of all results for Total Fat testing of duplicate wheat flour samples PTA 1 and PTA 2.

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

6.5 Moisture

Of the ten laboratories that tested the samples for Moisture, six used AOAC 925.10, one used AOAC 930.15, one used ISO 712: 2009, one used an in-house method and one laboratory did not specify the method used for testing.

The time and temperatures used for Moisture determination ranged between 15 hours at 70 °C to one hour at 130 °C. The majority of laboratories dried at 130 - 135 °C for between 1 - 2.5 hours.

All methods were pooled to analyse the Moisture results.

Laboratory 7 reported outlier results for both samples. 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 Moisture testing, the median and associated standard error (se) for each sample (expressed in g/100g) was as follows:

	PTA 1	PTA 2
Moisture, all methods pooled	13.23 ± 0.14	13.28 ± 0.10

Figure TA-3, below, shows the distribution of results from the methods used for Moisture testing in this round.

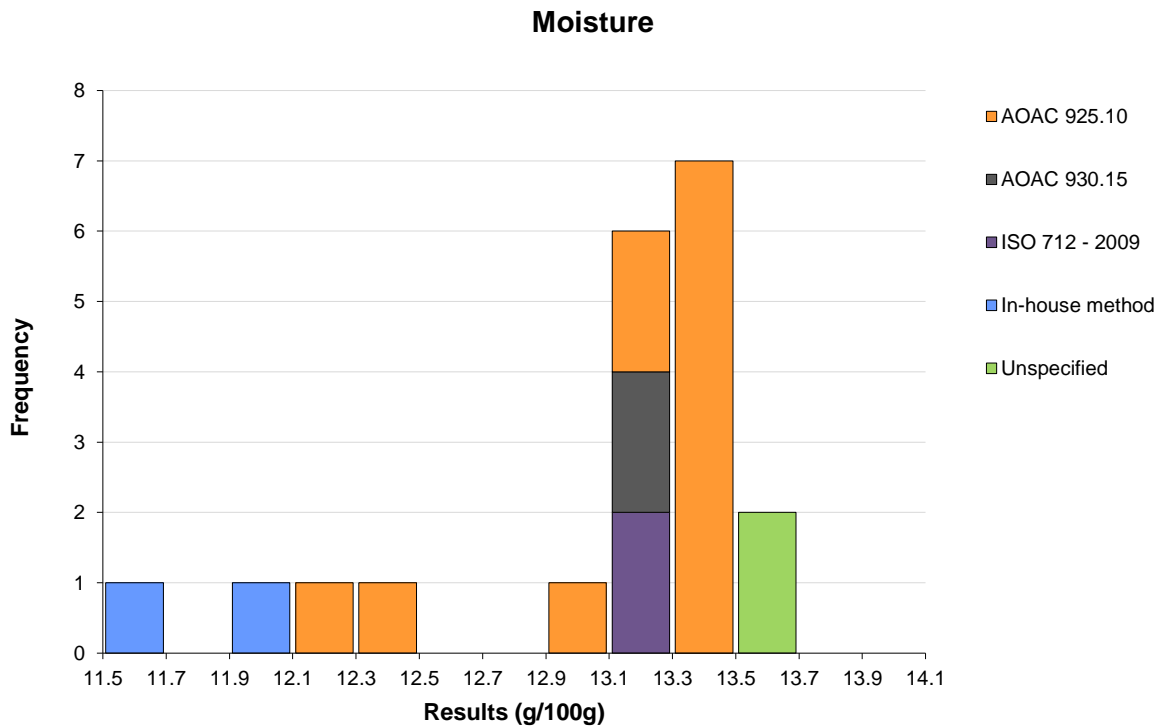


Figure TA-3. Spread of all results for Moisture testing of duplicate wheat flour samples PTA 1 and PTA 2.

The robust CVs of 2.6% and 1.9% for the two samples are lower than the values of 5.3% and 5.8% obtained in Round 29 of the Food program (see Report No. 598).

For this proficiency round, laboratories were requested to report their MU by repeatability (r) and / or reproducibility (R). Two laboratories reported MU for Moisture by repeatability, and two by reproducibility. Four laboratories reported both repeatability and reproducibility MU for Moisture. Two laboratories did not provide an estimate of their MU for this test.

6.6 Ash

Ten laboratories tested the samples for Ash. Of these, five used AOAC 923.03, one used AACCI Method 08-01.01, one used ISO 2171: 2007, two used in-house methods and one laboratory did not specify the method used for testing.

The temperatures used for ashing ranged between 525 °C and 600 °C. The time for ashing ranged between three to 20 hours.

All methods were pooled to analyse the Ash results.

Laboratory 1 reported an outlier result for sample PTA 2. There were no outliers reported for sample PTA 1.

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 standard error (se) for each sample (expressed in g/100g) was as follows:

	PTA 1	PTA 2
Ash, all methods pooled	0.475 ± 0.011	0.470 ± 0.006

Figure TA-4, below, shows the distribution of results from the methods used for Ash testing in this round.

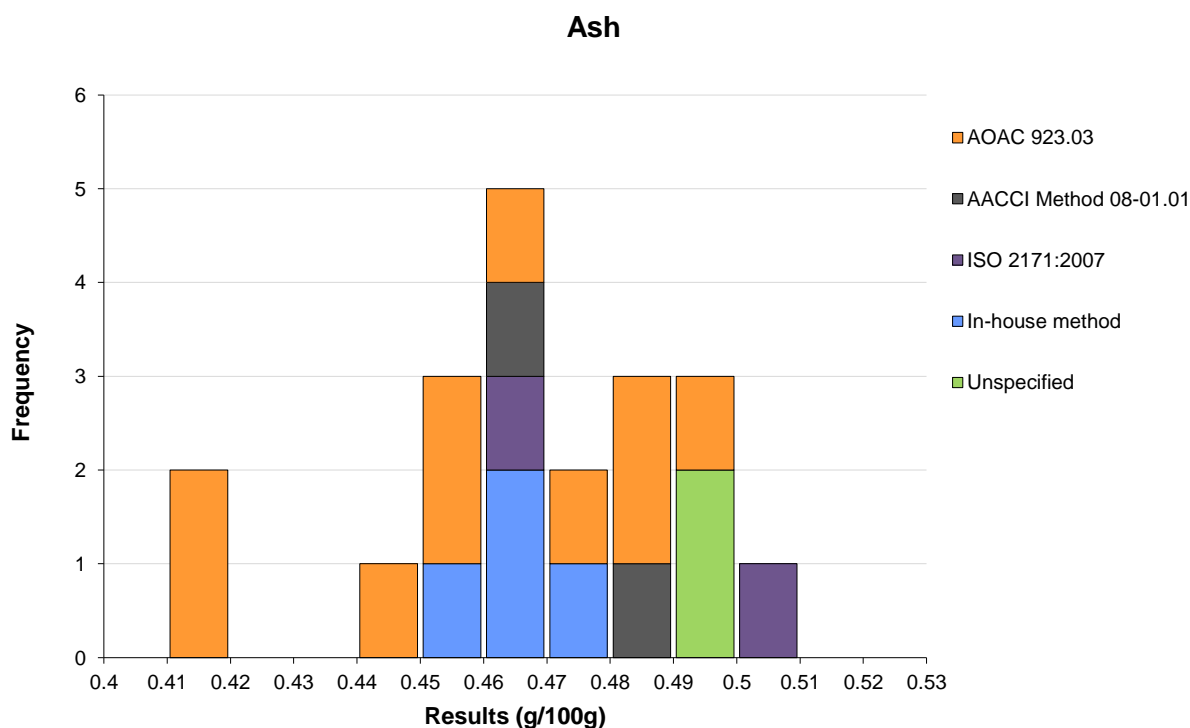


Figure TA-4. Spread of all results for Ash testing of duplicate wheat flour samples PTA 1 and PTA 2.

The robust CVs of 6.1% and 3.2% for the two samples are higher than the values of 3.2% and 3.2% obtained in Round 29 of the Food program (see Report No. 598).

For this proficiency round, laboratories were requested to report their MU by repeatability (r) and / or reproducibility (R). Two laboratories reported MU for Ash by repeatability, and three by reproducibility. Three laboratories reported both repeatability and reproducibility MU for Ash. Two laboratories did not provide an estimate of the MU for their Ash results.

6.7 Dietary Fibre

Of the nine laboratories that tested the samples for Dietary Fibre, three used AOAC 985.29, one used AOAC 991.42, one used AACCI Method 32.21.01, one used AOAC 962.09, one used an in-house method, one indicated that they used another method, however did not specify the method used, while one laboratory did not specify any method used for testing. All methods were pooled to analyse the Dietary Fibre results.

The robust CVs for this round are 14.0% and 27.5%. As the robust CV for sample PTA 2 is considerably higher than the robust CV for sample PTA 1, a target CV was used to calculate the robust z-scores for sample PTA 2. The target value of the CV used was 14.0%, the same as the robust CV obtained for sample PTA 1.

Laboratories 2 and 10 reported outliers for both samples.

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 Dietary Fibre testing, the median and associated standard error (se) for each sample (expressed in g/100g) was as follows:

	PTA 1	PTA 2
Dietary Fibre, all methods pooled	3.090 ± 0.181	2.925 ± 0.336

Figure TA-5, below, shows the distribution of results from the methods used for Dietary Fibre testing in this round.

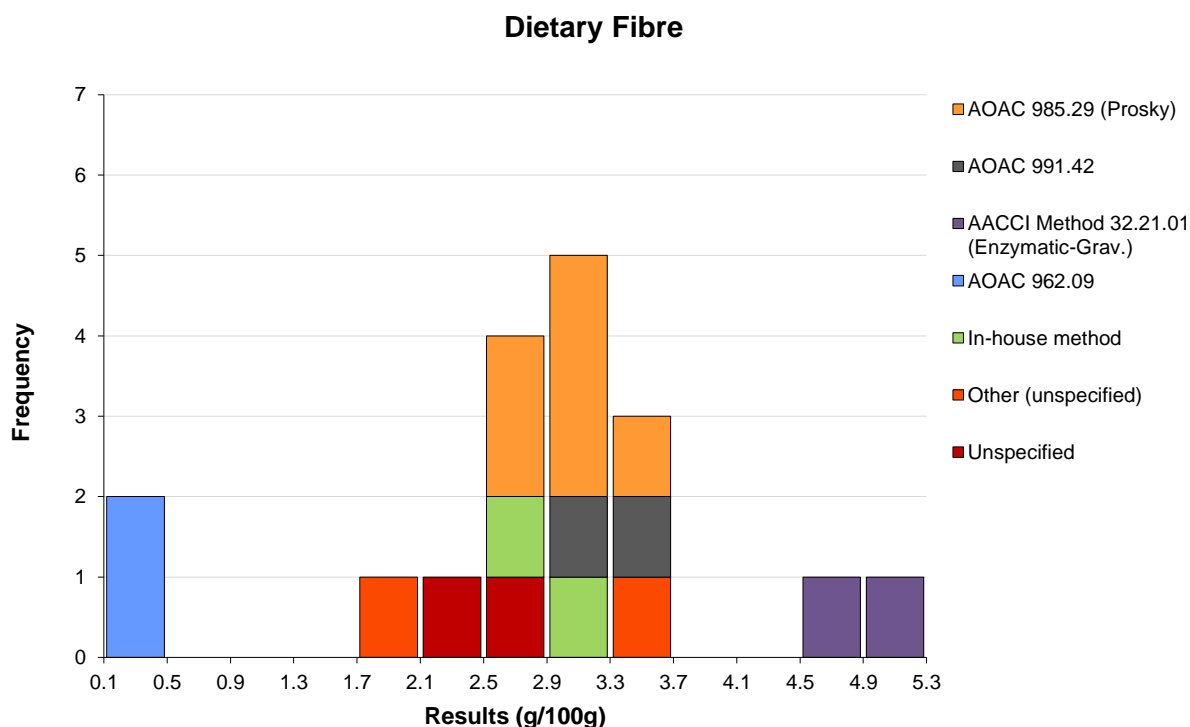


Figure TA-5. Spread of all results for Dietary Fibre testing of duplicate wheat flour samples PTA 1 and PTA 2.

For this proficiency round, laboratories were requested to report their MU by repeatability (r) and / or reproducibility (R). One laboratory reported MU for Dietary Fibre by repeatability, and one by reproducibility. Four laboratories reported both repeatability and reproducibility MU for Dietary Fibre. Three laboratories did not provide an estimate of the MU for their Dietary Fibre results.

6.8 Carbohydrate

Since November 2001, the Australia New Zealand Food Standards Code (FSC 1.2.8, 1) has defined Carbohydrate as the difference from 100 of Protein, Total Fat, Moisture, Ash and Dietary Fibre (and alcohol and any other unavailable carbohydrates). That is, Dietary Fibre is included in the difference calculation. So the equation for Carbohydrate value calculation for food labelling should be:

$$100 - \text{Protein} - \text{Total Fat} - \text{Moisture} - \text{Ash} - \text{Dietary Fibre}.$$

Eight laboratories reported results for Carbohydrate. Laboratory 10 tested the samples for Protein, Total Fat, Moisture, Ash and Dietary Fibre, but did not calculate Carbohydrate.

There were no outliers reported for either 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 Carbohydrate testing, the median and associated standard error (se) for each sample (expressed in g/100g) was as follows:

	PTA 1	PTA 2
Carbohydrate	70.25 ± 0.33	70.52 ± 0.27

Figure TA-6, below, shows the distribution of results for Carbohydrate testing in this round.

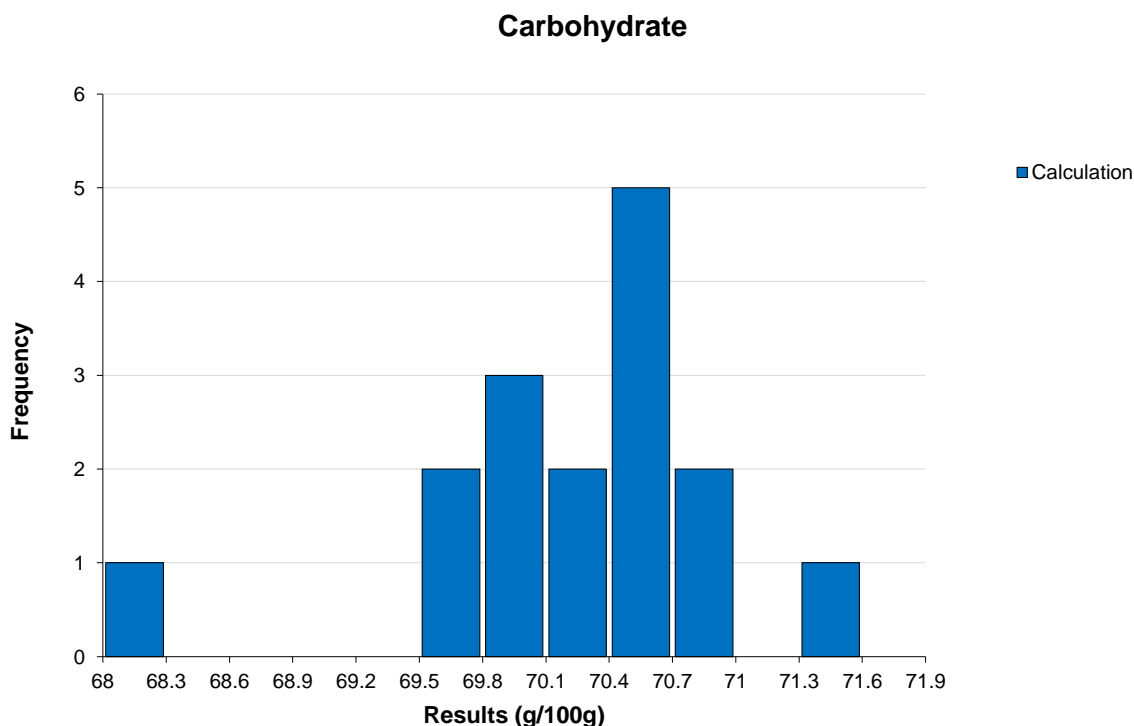


Figure TA-6. Spread of all results for Carbohydrate testing of duplicate wheat flour samples PTA 1 and PTA 2.

The robust CVs of 1.1% and 0.9% for the two samples were higher than the values of 0.4% and 0.4% obtained in Round 29 of the Food program (see Report No. 598).

For this proficiency round, laboratories were requested to report their MU by repeatability (r) and / or reproducibility (R). One laboratory reported MU for Carbohydrate by repeatability. Three laboratories reported both repeatability and reproducibility MU for Carbohydrate. Four laboratories did not provide an estimate of the MU for their Carbohydrate results.

6.9 Energy

The November 2001 changes to the FSC ascribed an average energy value to Dietary Fibre of 8 kJ/g. So the equation for Energy value calculation for food labelling (FSC 1.2.8, 2(2)) should be:

$$\text{Protein} \times 17 + \text{Total Fat} \times 37 + \text{Carbohydrate} \times 17 + \text{Dietary Fibre} \times 8.$$

Eight laboratories reported results for Energy. Laboratory 10 did not report results for Energy, but could have performed Energy calculations.

Laboratory 7 reported an outlier result for sample PTA 1. There were no outliers reported 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 Energy testing, the median and associated standard error (se) for each sample (expressed in kJ/100g) was as follows:

	PTA 1	PTA 2
Energy	1466.3 ± 10.5	1466.0 ± 8.4

Figure TA-7, below, shows the distribution of results for Energy testing in this round.

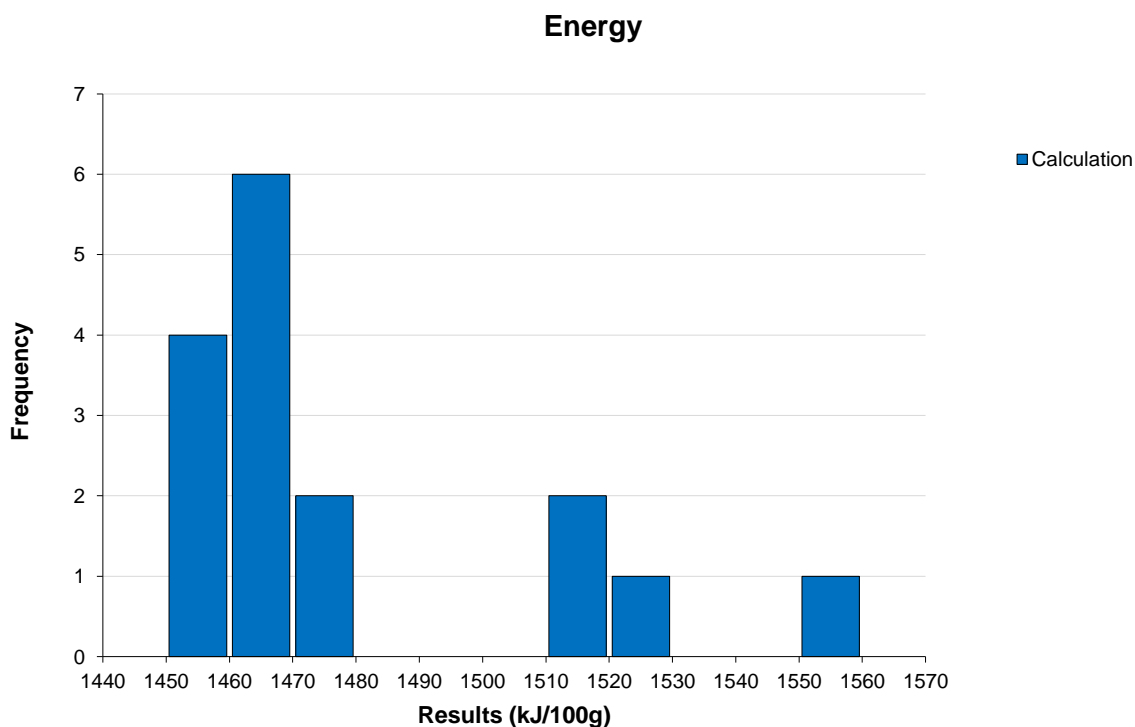


Figure TA-7. Spread of all results for Energy testing of duplicate wheat flour samples PTA 1 and PTA 2.

The robust CVs of 1.6% and 1.3% for the two samples are higher than the values of 0.4% and 0.4% obtained in Round 29 of the Food program (see Report No. 598).

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

6.10 Measurement Uncertainty

For this proficiency round, 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>
Protein	6 out of 10 60%	6 out of 10 60%
Total Fat	6 out of 9 67%	5 out of 9 56%
Moisture	6 out of 10 60%	6 out of 10 60%
Ash	5 out of 10 50%	6 out of 10 60%
Dietary Fibre	5 out of 9 56%	5 out of 9 56%
Carbohydrate	4 out of 8 50%	3 out of 8 38%
Energy	4 out of 8 50%	3 out of 8 38%

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

Table C: Method of Measurement Uncertainty Estimation

Lab Code	Method
1	In-house precision data.
2	PT data and in-house precision.
3	In-house precision data.
5	GUM.
7	Eurachem Citac / Guide.
9	GUM (bottom up).
10	Eurachem / Citac Guide Quantifying Uncertainty in Analytical Measurement.

7. REFERENCES

1. *Guide to Proficiency Testing Australia (2012)*.
(This document is located on the PTA website at www.pta.asn.au under Programs / Documents).
2. AOAC 2001.11. *Protein (Crude) in Animal Feed, Forage (Plant Tissue), Grain, and Oilseeds. Block Digestion Method Using Copper Catalyst and Steam Distillation into Boric Acid.*
3. AOAC 992.23. *Crude Protein in Cereal Grains and Oilseeds. Generic Combustion Method.*
4. AOAC 990.03. *Protein (Crude) in Animal Feed. Combustion Method.*
5. ISO 20483: 2013. *Cereals and pulses – Determination of the nitrogen content and calculation of the crude protein content – Kjeldahl method.*
6. AACCI Method 30-10.1. *Crude Fat in Flour, Bread, and Baked Cereal Products Not Containing Fruit.*
7. AOAC 925.10. *Solids (Total) and Loss on Drying (Moisture) in Flour. Air Oven Method.*
8. AOAC 930.15. *Loss on Drying (Moisture) for Feeds (at 135 °C for 2 hours).*
9. ISO 712: 2009. *Cereals and cereal products – Determination of moisture content – Reference method.*
10. AOAC 923.03. *Ash of Flour. Direct Method.*
11. AACCI Method 08-01.01. *Ash – Basic Method.*
12. ISO 2171: 2007. *Cereals, pulses and by-products – Determination of ash yield by incineration.*
13. AOAC 985.29. *Total Dietary Fibre in Foods. Enzymatic-Gravimetric Method.*
14. AOAC 991.42. *Insoluble Dietary Fibre in Foods and Food Products. Enzymatic-Gravimetric Method.*
15. AACCI Method 32.21.01. *Insoluble and Soluble Dietary Fibre in Oat Products – Enzymatic-Gravimetric Method.*
16. AOAC 962.09. *Fibre (Crude) in Animal Feed and Pet Food. Ceramic Fibre Filter Method.*

APPENDIX A

Summary of Results

Section A1

Protein

A1.1

Wheat Flour – Protein (g/100g) 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	11.3	11.5	-	0.03	11.3	11.5	-	0.03
2	10.7	10.8	0.2	-	11.0	10.9	0.2	-
3	11.3	11.1	0.153	0.306	11.2	11.1	0.151	0.303
4	11.2	11.1	-	0.03	11.1	11.1	-	0.03
5	13.9	13.2	1.6	-	12.5	13.1	0.3	-
6	11.28	11.28	-	-	11.35	11.28	-	-
7	10.84	10.94	0.05	0.47	10.82	10.90	0.04	0.39
8	12.6	12.5	-	-	12.6	12.5	-	-
9	11.54	11.43	0.2	0.2	11.47	11.53	0.2	0.2
10	11.38	11.34	0.78	0.78	11.38	11.35	0.78	0.78

Wheat Flour – Protein (g/100g) Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code	Dumas Calibrating Material
	Average	Z-Score	Average	Z-Score		
1	11.4	0.31	11.4	0.19	1	-
2	10.8	-2.22	11.0	-1.26	2	EDTA
3	11.2	-0.47	11.2	-0.61	2	ETDA
4	11.2	-0.66	11.1	-0.78	1	-
5	13.6	8.68 §	12.8	4.72 §	6	-
6	11.3	-0.16	11.3	-0.08	1	Tryptophan Recovery
7	10.9	-1.67	10.9	-1.55	7	O in HCl
8	12.6	4.79 §	12.6	3.91 §	-	-
9	11.5	0.64	11.5	0.52	2	EDTA
10	11.4	0.16	11.4	0.08	1	-

Method Codes

1 = AOAC 2001.11 (Kjeldahl digestion)	4
2 = AOAC 992.23, 990.03 (Dumas combustion)	3
3 = AACCI Method 46-15.01 (5-Minute Biuret)	0
4 = AACCI Method 46-19.01 (Calc %Total-N)	0
5 = ICC 105/I	0
6 = ISO 20483:2006 (Kjeldahl method)	1
7 = Other	1
- = Unspecified	1

A1.2

Summary Statistics

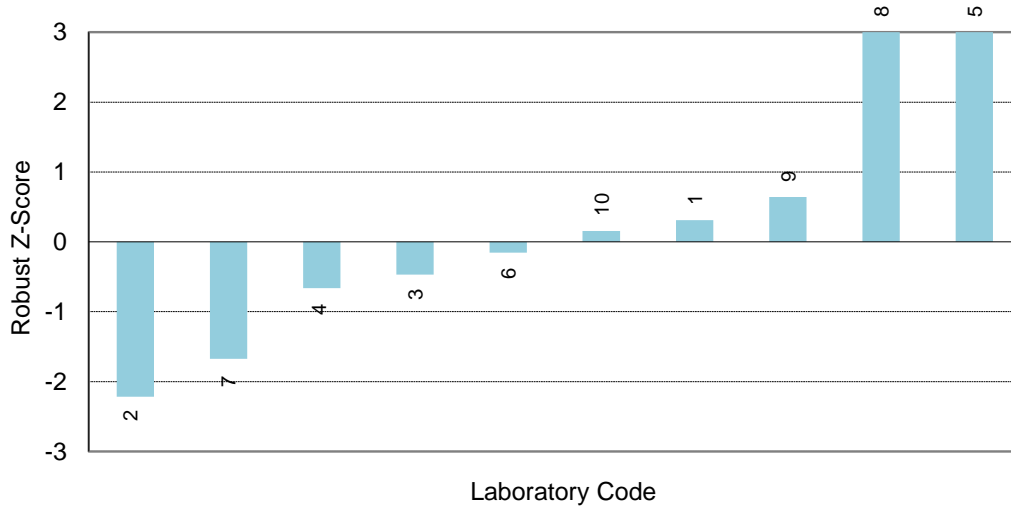
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	10	10
Median	11.32	11.34
Norm IQR	0.26	0.31
Uncertainty (Median)	0.10	0.12
Robust CV	2.3%	2.7%
Minimum	10.8	10.9
Maximum	13.6	12.8
Range	2.8	1.9

Notes:

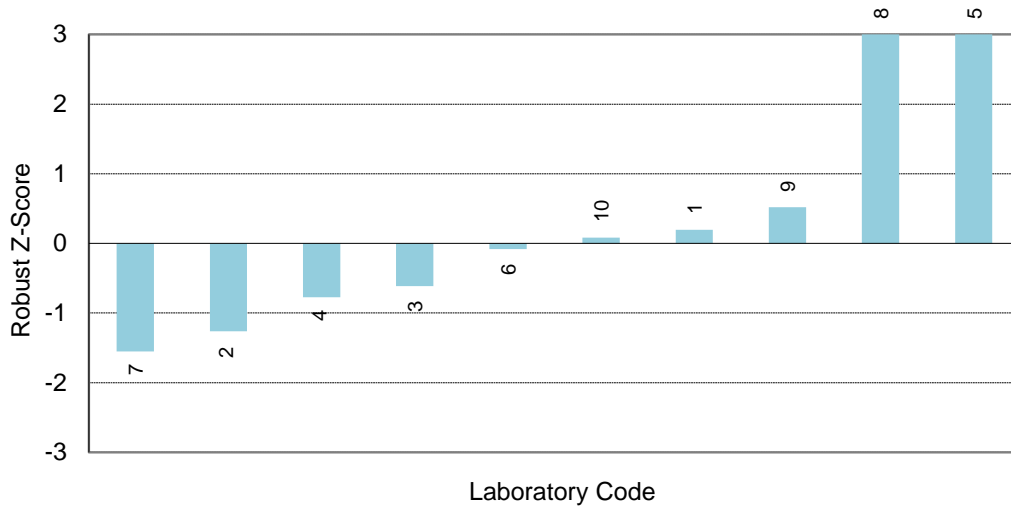
1. § denotes an outlier (i.e. $|z\text{-score}| \geq 3.0$).
2. There was a mistake in the Instructions to Participants for the Protein method codes. Method code 1 should have been listed as AOAC 2001.11, instead of AOAC 2000.11 (please see Appendix C).
3. The MU_r results submitted for laboratory 3 were their combined MU results.
4. The MU_R results submitted by laboratory 3 were their expanded MU results.
5. The Youden diagram on the following page is provided for information only.

A1.3

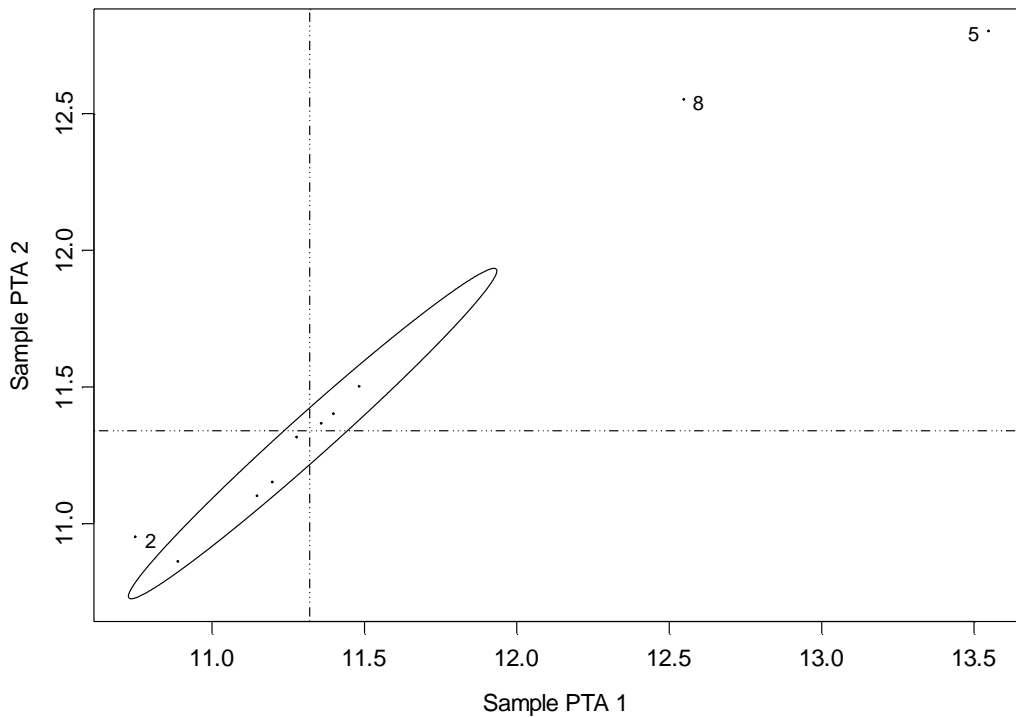
Protein (g/100g) - Sample 1



Protein (g/100g) - Sample 2



Protein (g/100g)



Section A2

Total Fat

A2.1

Wheat Flour – Total Fat (g/100g) 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	1.68	1.63	-	1.00	1.60	1.57	-	1.00
2	0.93	0.94	0.3	-	0.94	0.93	0.3	-
3	1.39	1.31	0.050	0.100	1.45	1.27	0.051	0.102
5	0.86	0.81	0.11	-	0.84	1.11	0.03	-
6	1.43	1.48	-	-	1.38	1.44	-	-
7	4.27	4.39	0.06	1.34	2.43	2.53	0.05	1.96
8	1.0	1.1	-	-	1.0	1.2	-	-
9	1.26	1.28	0.2	0.5	1.12	1.24	0.2	0.5
10	0.86	0.85	0.02	0.02	0.86	0.85	0.02	0.02

Wheat Flour – Total Fat (g/100g) – Methods

Lab Code	Sample PTA 1	Sample PTA 2	Method Code
	Average	Average	
1	1.66	1.59	1
2	0.94	0.94	2
3	1.35	1.36	1
5	0.84	0.98	2
6	1.46	1.41	1
7	4.33	2.48	6
8	1.05	1.10	-
9	1.27	1.18	1
10	0.86	0.86	2

Method Codes

1 = * Acid-hydrolysis	4
2 = ** Soxhlet extraction	3
3 = AACC 30-25	0
4 = AOAC 996.01	0
5 = ISO 11085:2008 (Randall extraction method)	0
6 = Other	1
- = Unspecified	1

Notes:

1. Z-scores and summary statistics were not calculated for the Total Fat results.
2. * “Acid hydrolysis” includes all methods employing acid hydrolysis of test material prior to ethers extraction.
3. ** “Soxhlet extraction” includes all methods which employ ethers extraction without any prior hydrolysis.
4. The MU_r results submitted for laboratory 3 were their combined MU results.
5. The MU_R results submitted by laboratory 3 were their expanded MU results.

Section A3

Moisture

A3.1

Wheat Flour – Moisture (g/100g) 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	13.0	13.0	-	0.65	13.4	13.3	-	0.65
2	12.2	12.2	0.1	-	12.3	12.4	0.1	-
3	13.3	13.2	0.104	0.207	13.2	13.2	0.104	0.207
4	13.4	13.4	-	0.2	13.6	13.4	-	0.2
5	13.2	13.2	0.1	-	13.1	13.2	0.2	-
6	13.4	13.5	-	-	13.4	13.4	-	-
7	11.41	11.62	0.11	0.92	12.06	11.90	0.08	0.67
8	13.5	13.6	-	-	13.6	13.5	-	-
9	13.51	13.44	0.2	0.4	13.43	13.44	0.2	0.4
10	13.15	13.12	0.36	0.36	13.14	13.12	0.36	0.36

Wheat Flour – Moisture (g/100g) Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code	Temp (°C)	Time (hours)
	Average	Z-Score	Average	Z-Score			
1	13.0	-0.65	13.4	0.30	1	70	15
2	12.2	-2.98	12.4	-3.72 §	1	105	8
3	13.3	0.07	13.2	-0.30	1	130	2.5
4	13.4	0.51	13.5	0.91	1	130	2
5	13.2	-0.07	13.2	-0.50	13	131	1.30
6	13.5	0.65	13.4	0.50	1	130	1
7	11.5	-4.96 §	12.0	-5.21 §	13	105	5
8	13.6	0.94	13.6	1.11	-	-	-
9	13.5	0.73	13.4	0.64	1	130	1
10	13.1	-0.26	13.1	-0.58	3	135	2.0

Method Codes

1 = AOAC 925.10	6
2 = AOAC 925.40	0
3 = AOAC other (please specify)	1
4 = AACCI Method 44-01.01	0
5 = AACCI Method 44-11.01 (Dielectric Meter)	0
6 = AACCI Method 44-15.02 (Air-Oven)	0
7 = AACCI Method 44-40.01 (Mod. Vac-Oven)	0
8 ISO 712-1985 (reference method)	0
9 ISO 712-1986 (routine method)	0
10 ISO 712:1998 (routine reference method)	0
11 ISO 7700-1:1984 (moisture meter calibration)	0
12 Moisture Meter	0
13 Other	2
- = Unspecified	1

A3.2

Summary Statistics

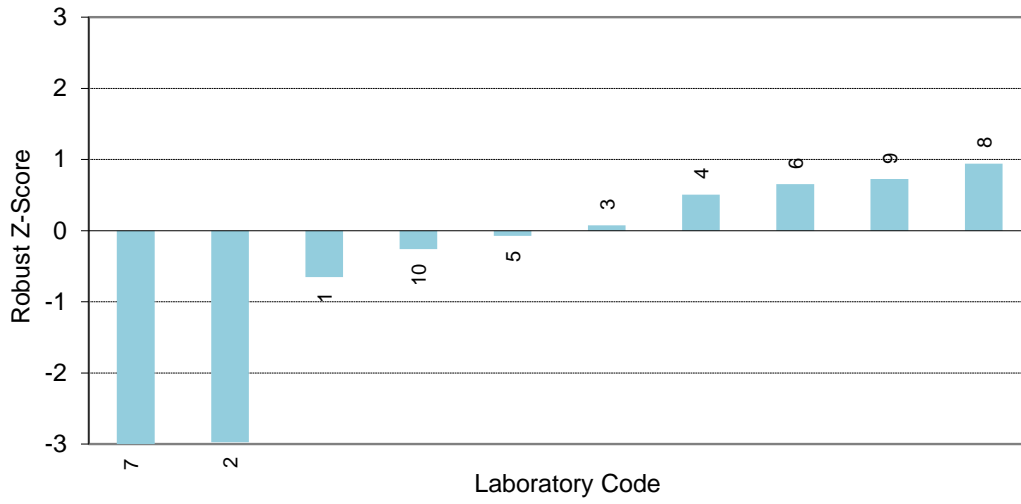
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	10	10
Median	13.23	13.28
Norm IQR	0.34	0.25
Uncertainty (Median)	0.14	0.10
Robust CV	2.6%	1.9%
Minimum	11.5	12.0
Maximum	13.6	13.6
Range	2.0	1.6

Notes:

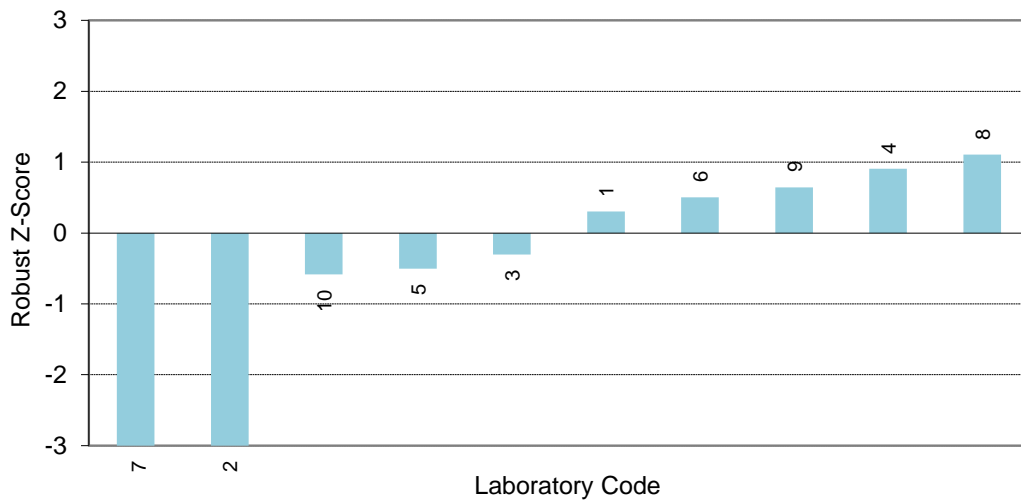
1. § denotes an outlier (i.e. $|z\text{-score}| \geq 3.0$).
2. The MU_r results submitted for laboratory 3 were their combined MU results.
3. The MU_R results submitted by laboratory 3 were their expanded MU results.
4. The Youden diagram on the following page is provided for information only.

A3.3

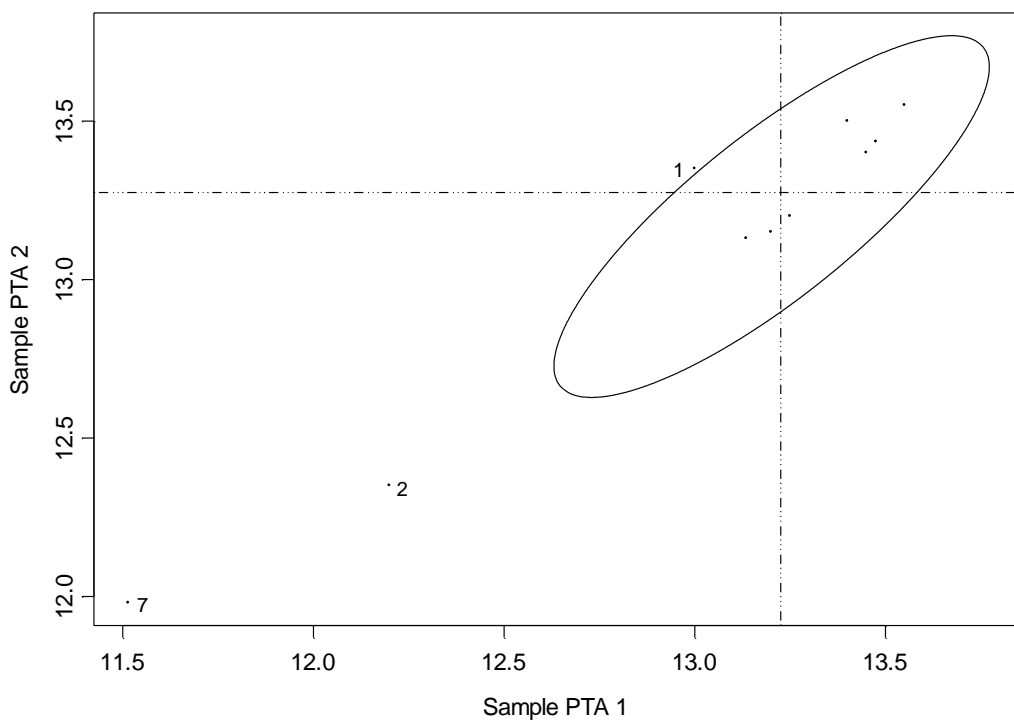
Moisture (g/100g) - Sample 1



Moisture (g/100g) - Sample 2



Moisture (g/100g)



Section A4

Ash

A4.1

Wheat Flour – Ash (g/100g) 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	0.47	0.43	-	0.1	0.43	0.41	-	0.1
2	0.40	0.43	0.05	-	0.50	0.49	0.05	-
3	0.47	0.47	-	0.03	0.48	0.48	-	0.03
4	0.46	0.46	-	0.01	0.46	0.46	-	0.01
5	0.51	0.50	0.03	-	0.48	0.46	0.05	-
6	0.50	0.48	-	-	0.50	0.48	-	-
7	0.44	0.47	0.02	3.65	0.49	0.45	0.02	4.98
8	0.5	0.5	-	-	0.5	0.5	-	-
9	0.49	0.49	0.04	0.17	0.47	0.47	0.04	0.17
10	0.48	0.48	0.01	0.01	0.47	0.47	0.01	0.01

Wheat Flour – Ash (g/100g) Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code	Temp (°C)	Time (hours)
	Average	Z-Score	Average	Z-Score			
1	0.45	-0.87	0.42	-3.35 §	1	590	15
2	0.42	-2.08	0.50	1.67	1	525	8
3	0.47	-0.17	0.48	0.67	1	600	20
4	0.46	-0.52	0.46	-0.67	1	550	12
5	0.51	1.04	0.47	0.00	8	550	4
6	0.49	0.52	0.49	1.34	1	550	6
7	0.46	-0.69	0.47	0.00	11	550	8
8	0.50	0.87	0.50	2.01	-	-	-
9	0.49	0.52	0.47	0.00	3	580	16
10	0.48	0.17	0.47	0.00	11	555	3.0

Method Codes

1 = AOAC 923.03	5
2 = AOAC other (please specify)	0
3 = AACCI Method 08-01.01	1
4 = AACCI Method 08-02.01 (Rapid Mg Acetate)	0
5 = AACCI Method 08-03.01 (Rapid 2-Hour, 600°)	0
6 = AACCI Method 08-21.01 (NIR)	0
7 = ISO 2171:1993	0
8 ISO 2171:2007	1
9 ICC Method No. 104/1 (1990)	0
10 Pearson	0
11 Other	2
- = Unspecified	1

A4.2

Summary Statistics

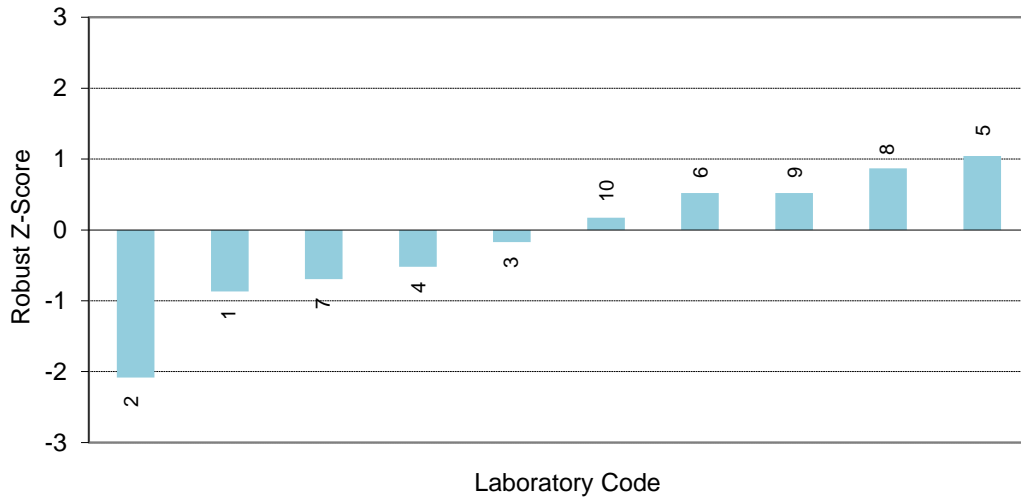
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	10	10
Median	0.475	0.470
Norm IQR	0.029	0.015
Uncertainty (Median)	0.011	0.006
Robust CV	6.1%	3.2%
Minimum	0.42	0.42
Maximum	0.51	0.50
Range	0.09	0.08

Notes:

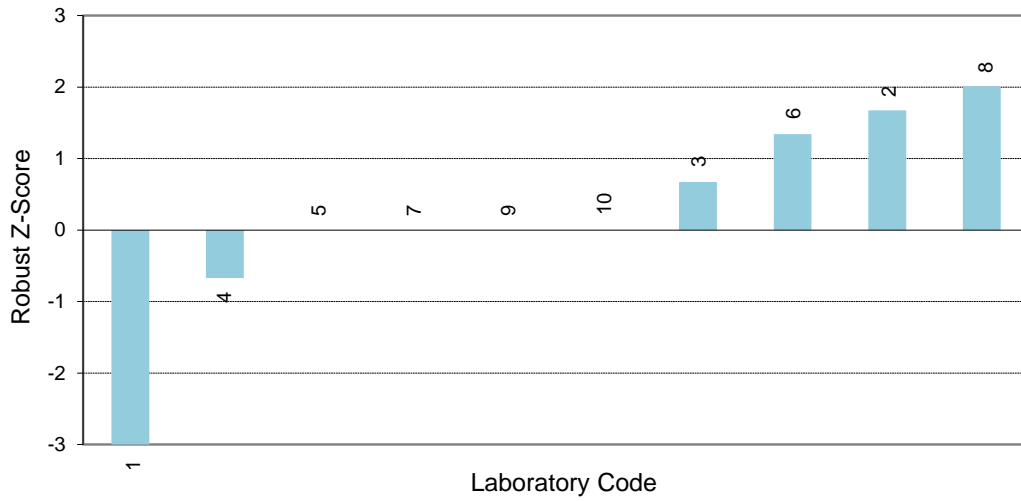
1. § denotes an outlier (i.e. $|z\text{-score}| \geq 3.0$).
2. The MU_r results submitted for laboratory 3 were their combined MU results.
3. The MU_R results submitted by laboratory 3 were their expanded MU results.
4. The Youden diagram on the following page is provided for information only.

A4.3

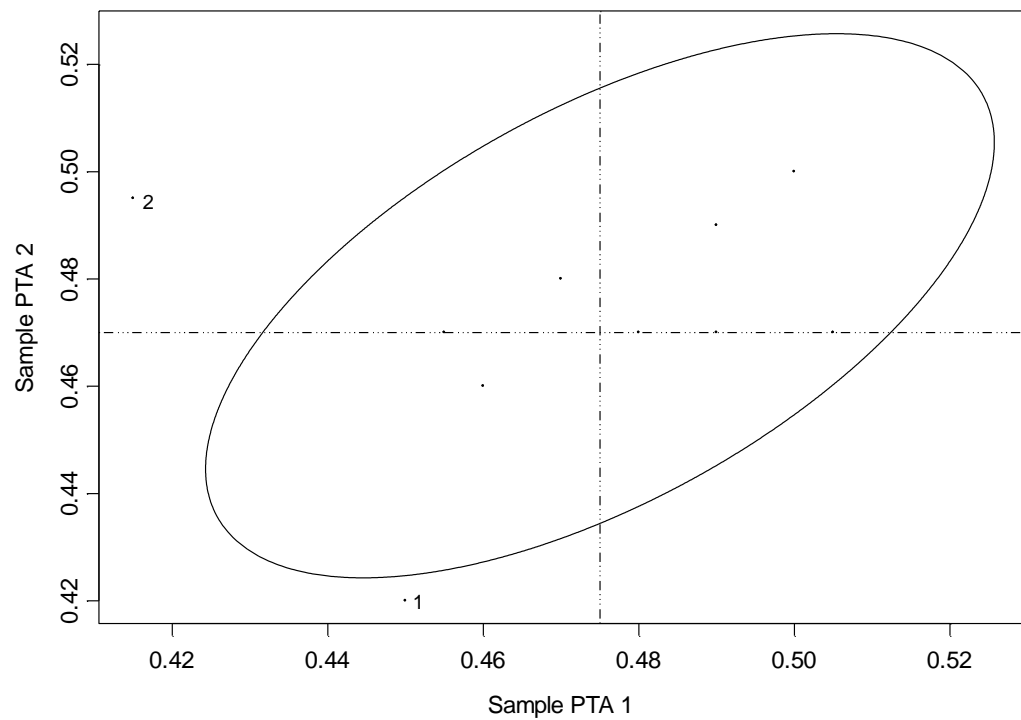
Ash (g/100g) - Sample 1



Ash (g/100g) - Sample 2



Ash (g/100g)



Section A5
Dietary Fibre

A5.1

Wheat Flour – Dietary Fibre (g/100g) 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	3.16	3.29	-	1.00	3.38	3.32	-	1.00
2	5.1	5.0	0.1	-	4.6	4.7	0.1	-
3	2.60	3.00	0.097	0.194	3.30	3.20	0.139	0.278
5	3.35	3.95	-	-	1.81	1.80	-	-
6	3.19	2.99	-	-	3.26	3.38	-	-
7	3.16	3.09	0.04	1.12	2.61	2.72	0.06	2.06
8	2.7	2.7	-	-	2.4	2.4	-	-
9	2.78	2.68	0.9	1.2	2.98	2.87	0.9	1.2
10	0.16	0.20	0.07	0.07	0.15	0.13	0.05	0.05

Wheat Flour – Dietary Fibre (g/100g) Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code
	Average	Z-Score	Average	Z-Score	
1	3.23	0.31	3.35	1.04	2
2	5.05	4.53 §	4.65	4.21 §	8
3	2.80	-0.67	3.25	0.79	1
5	3.65	1.29	1.81	-2.73	9
6	3.09	0.00	3.32	0.96	1
7	3.13	0.08	2.67	-0.63	9
8	2.70	-0.90	2.40	-1.28	-
9	2.73	-0.83	2.93	0.00	1
10	0.18	-6.73 §	0.14	-6.80 §	9

Method Codes

1 = AOAC 985.29 (Prosky)	3
2 = AOAC 991.42	1
3 = AOAC 991.43 (Lee)	0
4 = AOAC 993.19	0
5 = AOAC 994.13 (Theander)	0
6 = AACCI Method 32.05.01	0
7 = AACCI Method 32.07.01	0
8 = AACCI Method 32.21.01 (Enzymatic-Grav.)	1
9 = Other	3
- = Unspecified	1

A5.2

Summary Statistics

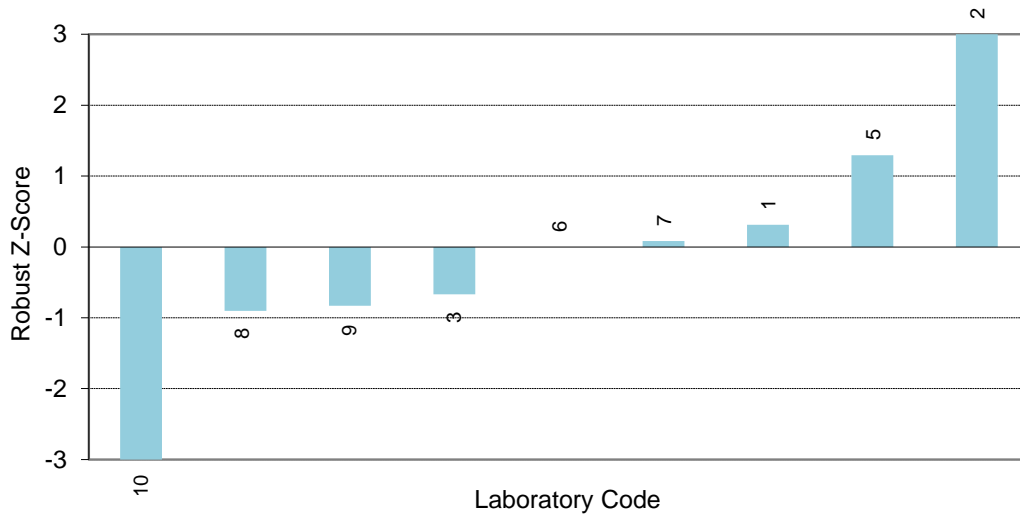
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	9	9
Median	3.090	2.925
Norm IQR	0.433	0.804
Uncertainty (Median)	0.181	0.336
Robust CV	14.0%	27.5%
Target CV	14.0%	14.0%
Minimum	0.18	0.14
Maximum	5.05	4.65
Range	4.87	4.51

Notes:

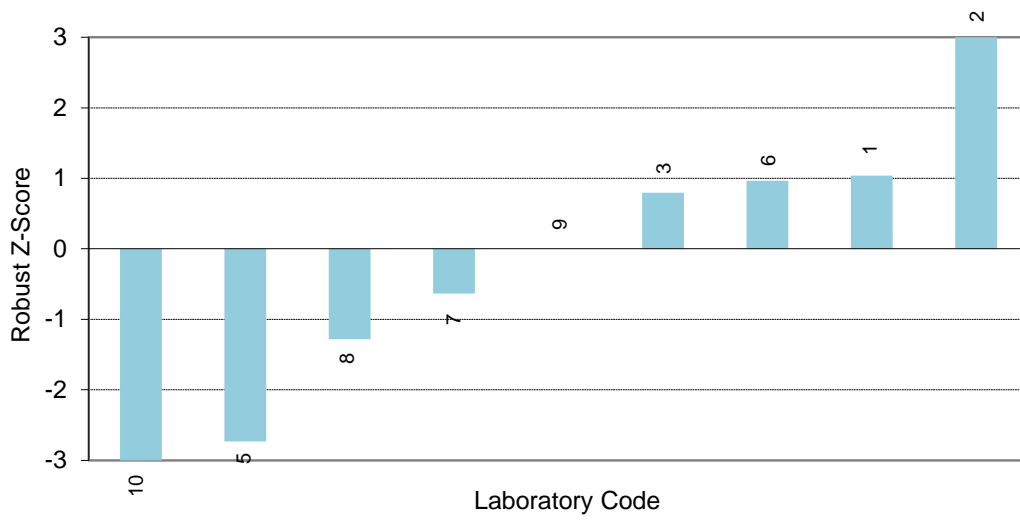
1. § denotes an outlier (i.e. $|z\text{-score}| \geq 3.0$).
2. A target CV was used to calculate the robust z-scores for sample PTA 2. The target value of the CV used was the same as the robust CV obtained for sample PTA 1.
3. The MU_r results submitted for laboratory 3 were their combined MU results.
4. The MU_R results submitted by laboratory 3 were their expanded MU results.
5. The Youden diagram on the following page is provided for information only.

A5.3

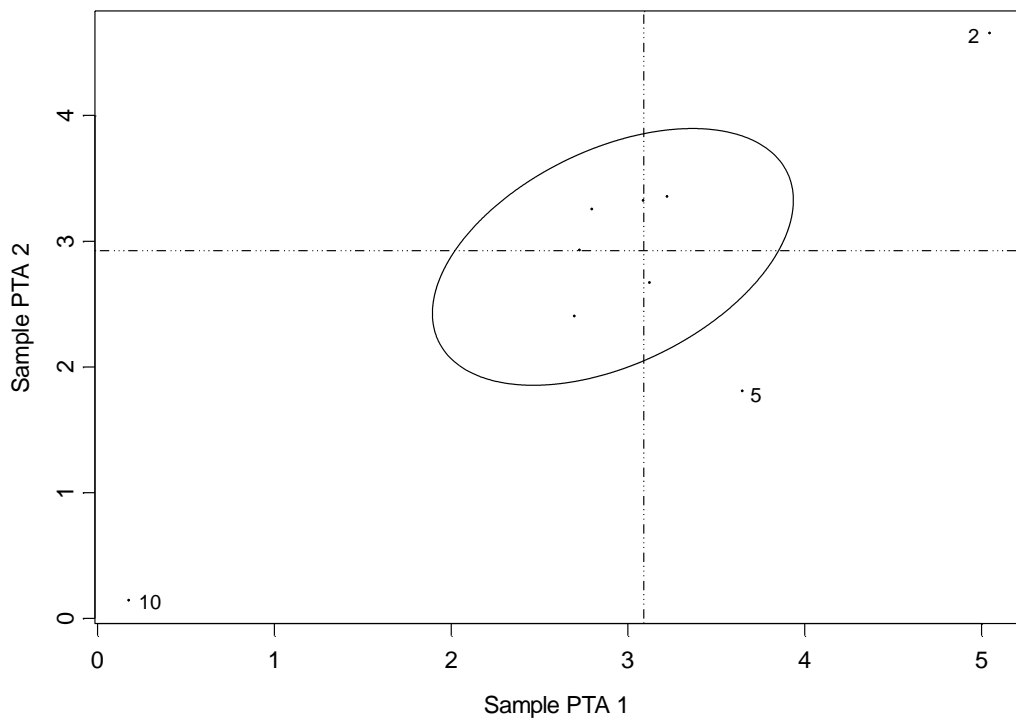
Dietary Fibre (g/100g) - Sample 1



Dietary Fibre (g/100g) - Sample 2



Dietary Fibre (g/100g)



Section A6
Carbohydrate

A6.1

Wheat Flour – Carbohydrate (g/100g) Results and Measurement Uncertainty

Lab Code	Sample PTA 1				Sample PTA 2			
	Result 1	Result 2	MU r (\pm)	MU R (\pm)	Result 1	Result 2	MU r (\pm)	MU R (\pm)
1	70.4	70.2	-	-	69.9	69.9	-	-
2	70.7	-	0.5	-	70.7	-	0.5	-
3	70.9	70.8	1.171	2.342	70.4	70.7	1.186	2.372
5	68.1	68.3	-	-	71.2	70.3	-	-
6	70.1	70.3	-	-	70.1	70.0	-	-
7	69.92	69.49	0.22	0.31	71.59	71.50	0.05	0.06
8	69.7	69.8	-	-	69.9	69.9	-	-
9	70.42	70.68	0.6	1.6	70.53	70.45	0.6	1.6

Wheat Flour – Carbohydrate (g/100g) Z-Scores

Lab Code	Sample PTA 1		Sample PTA 2	
	Average	Z-Score	Average	Z-Score
1	70.3	0.07	69.9	-1.01
2	70.7	0.60	70.7	0.29
3	70.9	0.80	70.6	0.05
5	68.2	-2.74	70.8	0.37
6	70.2	-0.07	70.1	-0.76
7	69.7	-0.73	71.5	1.66
8	69.8	-0.67	69.9	-1.01
9	70.6	0.40	70.5	-0.05

Summary Statistics

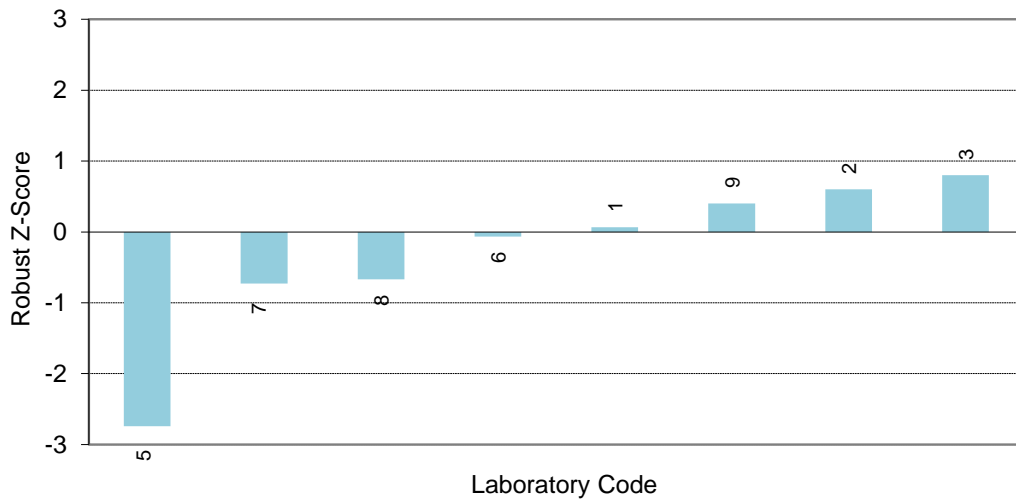
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	8	8
Median	70.25	70.52
Norm IQR	0.75	0.62
Uncertainty (Median)	0.33	0.27
Robust CV	1.1%	0.9%
Minimum	68.2	69.9
Maximum	70.9	71.5
Range	2.7	1.6

Note:

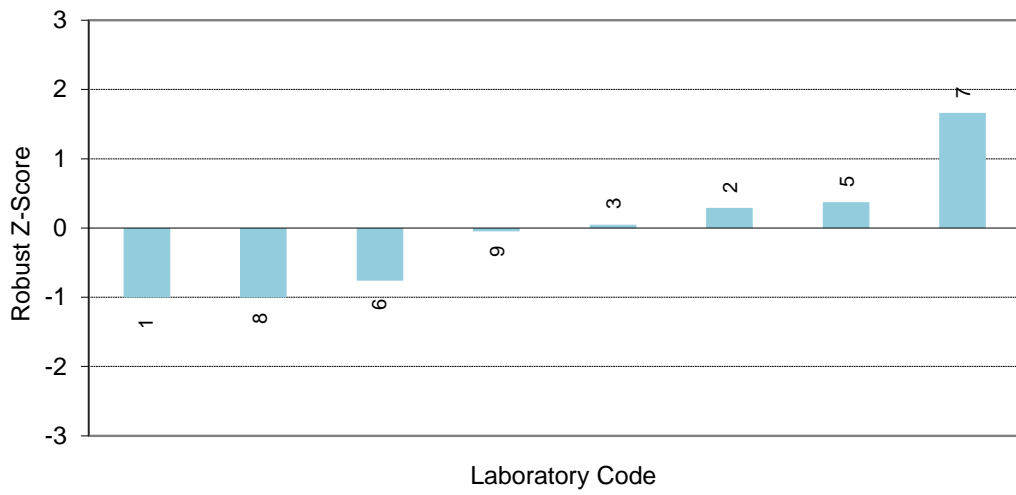
1. The Youden diagram on the following page is provided for information only.

A6.2

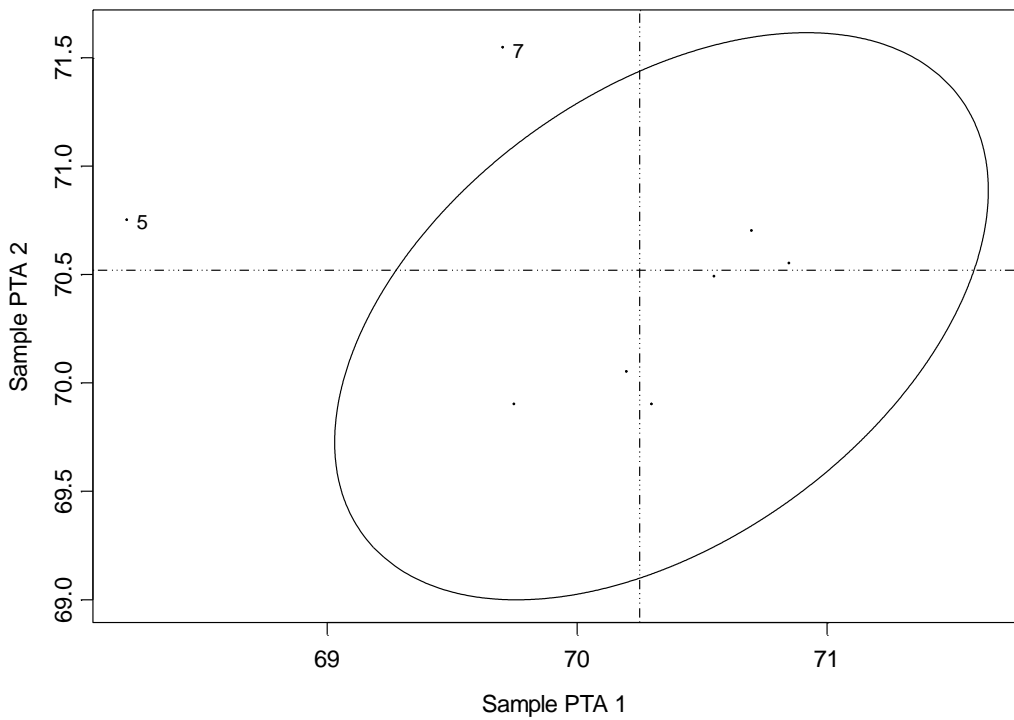
Carbohydrate (g/100g) - Sample 1



Carbohydrate (g/100g) - Sample 2



Carbohydrate (g/100g)



Section A7

Energy

A7.1

Wheat Flour – Energy (kJ/100g) 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	1476	1476	-	-	1467	1468	-	-
2	1530	-	10	-	1520	-	10	-
3	1470	1465	5.766	11.533	1467	1462	5.899	11.798
5	1453.98	1447.75	-	-	1469.65	1473.78	-	-
6	1460	1470	-	-	1460	1460	-	-
7	1556.19	1554.46	0.87	0.06	1511.76	1516.17	2.21	0.15
8	1460	1460	-	-	1460	1460	-	-
9	1462.18	1464.67	10	30	1459.28	1462.5	10	30

Wheat Flour – Energy (kJ/100g) Z-Scores

Lab Code	Sample PTA 1		Sample PTA 2	
	Average	Z-Score	Average	Z-Score
1	1476	0.41	1468	0.08
2	1530	2.69	1520	2.84
3	1468	0.05	1465	-0.08
5	1451	-0.65	1472	0.30
6	1465	-0.05	1460	-0.32
7	1555	3.76 §	1514	2.52
8	1460	-0.26	1460	-0.32
9	1463	-0.12	1461	-0.27

Summary Statistics

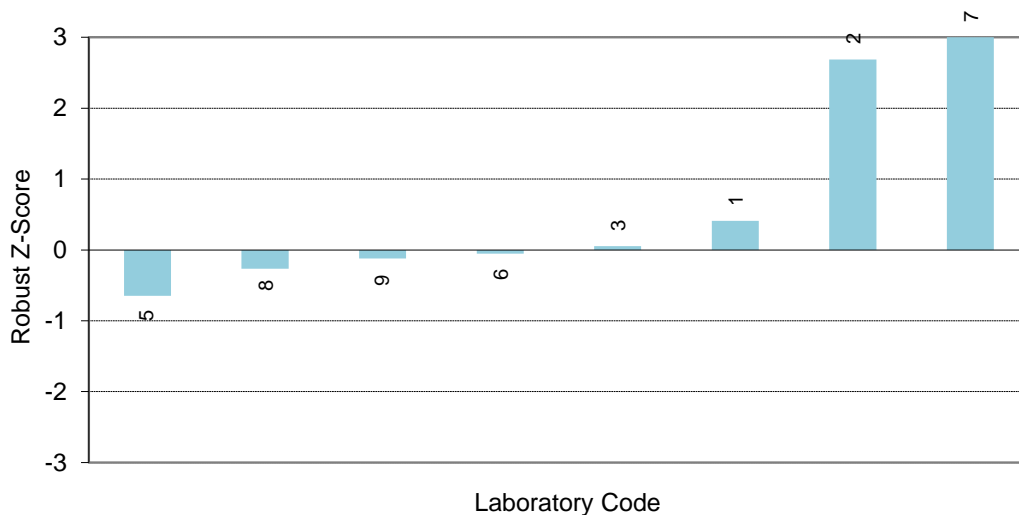
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	8	8
Median	1466.3	1466.0
Norm IQR	23.7	19.0
Uncertainty (Median)	10.5	8.4
Robust CV	1.6%	1.3%
Minimum	1451	1460
Maximum	1555	1520
Range	104	60

Notes:

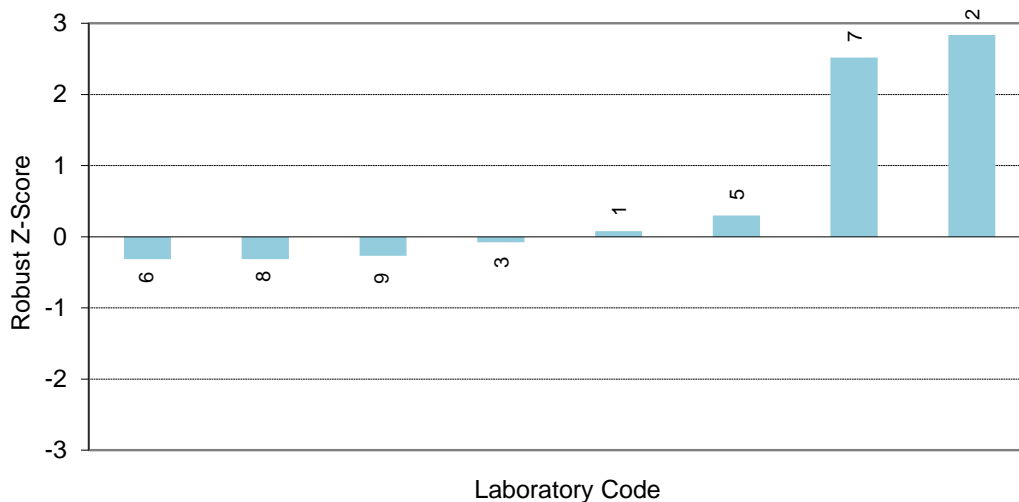
- § denotes an outlier (i.e. |z-score| ≥ 3.0).
- The Youden diagram on the following page is provided for information only.

A7.2

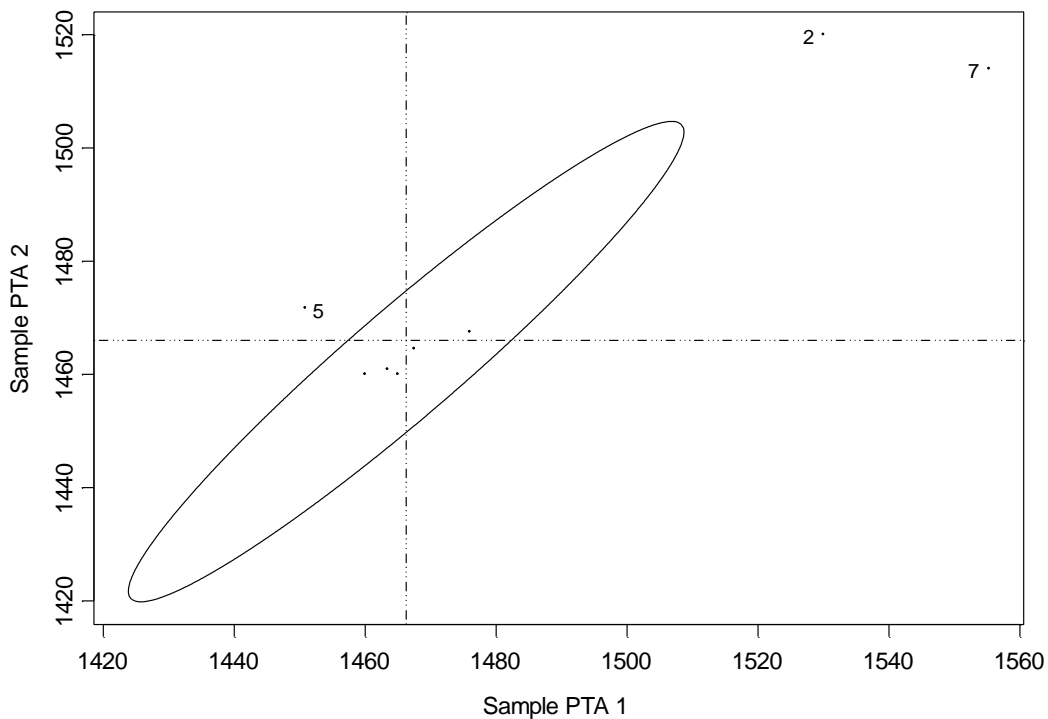
Energy (kJ/100g) - Sample 1



Energy (kJ/100g) - Sample 2



Energy (kJ/100g)



APPENDIX B

Homogeneity and Stability Testing

B1.1

Homogeneity Testing

Prior to distribution, seven samples of wheat flour were selected at random and tested for homogeneity by Global Proficiency Ltd (New Zealand). Each sample was tested in duplicate for Moisture and Protein. The results of the homogeneity testing appear in the following tables.

Wheat Flour – Moisture (g/100g)	
Result A	Result B
12.47	12.60
12.45	12.33
12.61	12.92
12.65	12.67
12.59	12.56
12.62	12.68
12.74	12.52

Wheat Flour – Protein (g/100g)	
Result A	Result B
12.40	12.18
12.01	11.99
12.11	11.98
12.12	12.12
12.12	12.03
12.10	12.00
12.07	12.27

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.

B2.1

Stability Testing

Three samples were selected at random and tested for stability by Global Proficiency Ltd (New Zealand). The results, below, indicated that the samples were sufficiently stable for use in this program.

Wheat Flour – Moisture (g/100g)	
Result A	Result B
12.48	12.44
12.54	12.70
12.44	12.48

Wheat Flour – Protein (g/100g)	
Result A	Result B
12.11	11.97
12.05	11.96
11.95	12.09

APPENDIX C

Instructions to Participants

and

Results Sheet

C1.1

PROFICIENCY TESTING AUSTRALIA
FOOD PROFICIENCY TESTING PROGRAM
ROUND 38, JULY 2014
INSTRUCTIONS TO PARTICIPANTS



To ensure that results obtained in this program can be analysed properly, participants are asked to adhere carefully to the following instructions.

1. Each participant will be supplied with two 100 g samples of wheat flour labelled PTA 1 and PTA 2.
2. Store your samples in the original packaging in a cool, dry place until testing commences.
3. The tests to be performed in this program are:
 - Protein
 - Total Fat
 - Moisture
 - Ash
 - Dietary Fibre
 - Carbohydrate[†]
 - Energy^{††}

Notes: [†] determined by difference.
 ^{††} determined by calculation.

4. 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 $N \times 5.7$.
5. Tests are to be performed on each sample in duplicate and the results reported on the Results Sheet.
6. Report results on the attached Results Sheet to the specified number of decimal places (d.p.). Results should not be reported as “greater than ...” or “less than ...”, as such data cannot be statistically analysed.
7. 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.
8. Laboratories are also requested to calculate and report an estimate of uncertainty of measurement for each reported measurement result. All estimates of uncertainty of measurement must be given as a 95% confidence interval (coverage factor $k \approx 2$).
9. Return the Results Sheet, 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 **Thursday 7 August 2014**. Results reported later than this date may not be analysed in the final report.

C1.2

PROFICIENCY TESTING AUSTRALIA
FOOD PROFICIENCY TESTING PROGRAM
ROUND 38, JULY 2014



INSTRUCTIONS TO PARTICIPANTS

METHOD CODES

Analysis	Method	Code
Protein (g/100g)	AOAC 2000.11 (Kjeldahl digestion)	1
	AOAC 992.23, 990.03 (Dumas combustion)	2
	AACCI Method 46-15.01 (5-Minute Biuret)	3
	AACCI Method 46-19.01 (Calc %Total-N)	4
	ICC 105/l	5
	ISO 20483:2006 (Kjeldahl method)	6
	Other (please specify)	7
Total Fat (g/100g)	* Acid-hydrolysis	1
	** Soxhlet extraction	2
	AACC 30-25	3
	AOAC 996.01	4
	ISO 11085:2008 (Randall extraction method)	5
	Other (please specify)	6
Moisture (g/100g)	AOAC 925.10	1
	AOAC 925.40	2
	AOAC other (please specify)	3
	AACCI Method 44-01.01	4
	AACCI Method 44-11.01 (Dielectric Meter)	5
	AACCI Method 44-15.02 (Air-Oven)	6
	AACCI Method 44-40.01 (Mod. Vac-Oven)	7
	ISO 712-1985 (reference method)	8
	ISO 712-1986 (routine method)	9
	ISO 712:1998 (routine reference method)	10
	ISO 7700-1:1984 (moisture meter calibration)	11
	Moisture Meter	12
	Other (please specify)	13
Ash (g/100g)	AOAC 923.03	1
	AOAC other (please specify)	2
	AACCI Method 08-01.01	3
	AACCI Method 08-02.01 (Rapid Mg Acetate)	4
	AACCI Method 08-03.01 (Rapid 2-Hour, 600°)	5
	AACCI Method 08-21.01 (NIR)	6
	ISO 2171:1993	7
	ISO 2171:2007	8
	ICC Method No. 104/1 (1990)	9
	Pearson	10
	Other (please specify)	11

Continued over page

C1.3

FOOD PROFICIENCY TESTING PROGRAM ROUND 38, JULY 2014



INSTRUCTIONS TO PARTICIPANTS

METHOD CODES AND CARBOHYDRATE / ENERGY CALCULATIONS

Analysis	Method	Code
Dietary Fibre (g/100g)	AOAC 985.29 (Prosky)	1
	AOAC 991.42	2
	AOAC 991.43 (Lee)	3
	AOAC 993.19	4
	AOAC 994.13 (Theander)	5
	AACCI Method 32.05.01	6
	AACCI Method 32.07.01	7
	AACCI Method 32.21.01 (Enzymatic-Grav.)	8
	Other (please specify)	9

* "Acid hydrolysis" includes all methods employing acid hydrolysis of test material prior to ethers extraction.

** "Soxhlet extraction" includes all methods which employ ethers extraction without any prior hydrolysis.

Since November 2001, the Australia New Zealand Food Standards Code (FSC 1.2.8, 1) has defined carbohydrate as the difference from 100 of moisture, protein, total fat, ash and dietary fibre (and alcohol and any other unavailable carbohydrates). That is, dietary fibre is included in the difference calculation. So the equation for carbohydrate value calculation for food labelling should be:

$$100 - \text{moisture} - \text{protein} - \text{total fat} - \text{ash} - \text{dietary fibre}.$$

The November 2001 changes to the FSC ascribed an average energy value to dietary fibre of 8 kJ/g. So the equation for energy value calculation for food labelling (FSC 1.2.8, 2(2)) should be:

$$\text{protein} \times 17 + \text{total fat} \times 37 + \text{carbohydrate} \times 17 + \text{dietary fibre} \times 8.$$

C2.1

PROFICIENCY TESTING AUSTRALIA
FOOD PROFICIENCY TESTING PROGRAM
ROUND 38, JULY 2014



RESULTS SHEET

Laboratory Code:

Date Samples Received: _____ Temperature on Arrival: _____

Test	Sample PTA 1				Sample PTA 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		
Protein (N x 5.7) (g/100g 1 d.p.)										
Total Fat (g/100g 2 d.p.)										
Moisture (g/100g 1 d.p.)										
Ash (g/100g 2 d.p.)										
Dietary Fibre (g/100g 2 d.p.)										
Carbohydrate (g/100g 1 d.p.)										
Energy (kJ/100g)										

Please specify the calibrating material for Dumas nitrogen determination:- _____.
(e.g. pure chemical (EDTA etc.) OR Kjeldahl reference material)

Please specify the temperature/time of moisture determination: _____ °C/ _____ hours.

Please specify the temperature/time of ashing: _____ °C/ _____ hours.

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

Date: _____ Signature: _____

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