

**REPORT NO. 533**

**Food Proficiency Testing Program  
Round 25 – Wheat Flour**

**February 2007**

**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 twenty fifth round of an ongoing series of programs involving chemical analysis of foodstuffs.

Proficiency Testing Australia conducted the testing program in November 2006. The aim of the program was to assess laboratories' ability to competently perform the nominated tests.

## 2. FEATURES OF THE PROGRAM

- (a) A total of 19 laboratories participated in the program, all of which returned results for inclusion in the final report. Laboratories from the following states and territories participated:

6	NSW
4	QLD
7	VIC
2	WA

To ensure confidential treatment of results, each laboratory was allocated a unique code number. All reference to participants is by allocated code numbers. Please note that some laboratories reported more than one set of results and therefore one 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 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 for which they hold NATA accreditation and were invited to report results for any of the other tests.

- (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 uncertainty of measurement for each result, on the accompanying *Results Sheet*, which was distributed with the samples. An additional sheet was included with the *Results Sheet* for participants to describe the method they used to determine the measurement uncertainty for each reported measurement result. Copies of these documents appear in Appendix C.

- (f) Prior to sample distribution, ten randomly selected samples were analysed for homogeneity by AgriQuality New Zealand Ltd. Based on the results of this testing, the homogeneity of the samples was established (see Appendix B).

### 3. FORMAT OF THE APPENDICES

- (a) Appendix A is divided into 7 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:

- i) a table of results reported by laboratories, along with an estimate of their uncertainty of measurement;
  - ii) a table of calculated z-scores;
  - iii) a listing of the summary statistics;
  - iv) ordered z-score charts (between-laboratories and within-laboratory);
  - v) a Youden diagram of laboratories' results for the sample pair.
- (b) Appendix B contains details of the homogeneity testing.
  - (c) Appendix C contains copies of the *Instructions to Participants* and *Results Sheets*.

### 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. EXTREME RESULTS

Robust z-scores have been used to assess each laboratory's testing performance. When calculated from pairs of results, z-scores are used to detect excessively large between-laboratories or within-laboratory variations.

Each result reported by a laboratory has a between-laboratories z-score and a within-laboratory z-score calculated. Any pair of results which has an absolute z-score greater than three (ie  $<-3$  or  $>3$ ) 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* (2006).

**Table A: Summary Statistics for All Tests**

The following summary statistics were sent to participants shortly after the return of results to provide them with “early information” about the results for the program.

Test	Summary Statistics*	PTA 1	PTA 2
Protein (N × 5.7) (g/100g)	No. of Results	20	20
	Median	13.085	13.065
	Normalised IQR	0.239	0.343
Total Fat (acid hydrolysis) (g/100g)	No. of Results	12	12
	Median	1.720	1.830
	Normalised IQR	0.169	0.167
Moisture (g/100g)	No. of Results	19	19
	Median	13.240	13.240
	Normalised IQR	0.311	0.382
Ash (g/100g)	No. of Results	18	18
	Median	0.700	0.705
	Normalised IQR	0.062	0.041
Dietary Fibre (g/100g)	No. of Results	11	11
	Median	4.400	4.230
	Normalised IQR	0.691	1.428
Carbohydrate (with dietary fibre in calculations) (g/100g)	No. of Results	9	9
	Median	79.864	79.830
	Normalised IQR	0.630	1.275
Energy (with dietary fibre in calculations) (kJ/100g)	No. of Results	10	10
	Median	1683.331	1681.025
	Normalised IQR	4.935	18.241

\* With the exception of moisture, statistics reported for each test are based on the dry basis (corrected) result.

**Notes:**

1. The results for fat have been analysed for acid hydrolysis only.
2. The results for carbohydrate and energy have been analysed only for those laboratories that included dietary fibre in their calculations.

**Table B: Summary of Statistical Outliers**

Test	Sample Pair PTA 1 & PTA 2	
	Between Laboratories	Within Laboratory
Protein	4, 16, 22	8, 15, 16, 18
Total Fat (acid hydrolysis)	10	7, 8, 10
Moisture	12, 19	19
Ash	15, 16	3, 15
Dietary Fibre	-	-
Carbohydrate (including dietary fibre)	12	17A, 19
Energy (including dietary fibre)	5, 12	-

## 6. PTA AND TECHNICAL ADVISER'S COMMENTS

The summary statistics and outliers identified 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 of the 19 laboratories that participated in the program returned results. Eleven of these 19 laboratories (58%) provided results for all seven of the tests.

The return rate for all tests is as follows:

- Protein                      19 out of 19      100%
- Total Fat                    17 out of 19      89%
- Moisture                    19 out of 19      100%
- Ash                            18 out of 19      95%
- Dietary Fibre              11 out of 19      58%
- Carbohydrate              15 out of 19      79%
- Energy                        15 out of 19      79%

## 6.2 Performance summary

One or more statistical outliers were reported by 13 of the 19 laboratories (68%) that returned results in this round of the Food program. The last wheat flour round of the Food program was round 17, conducted in February 2003 (see PTAC Report No 411). For comparison, 42% of the participants in round 17 of the Food program reported statistical outliers.

A total of 198 results were analysed in this round of the program. Of these results, 23 (12%) were outlier results. In round 17 of the Food program 8% of the total results reported were outlier results.

## 6.3 Protein

The majority of laboratories used methodology based on Kjeldahl digestion.

The robust CVs of 1.8% and 2.6% for the two samples are higher than the values of 1.4% and 1.8% obtained in round 17 of the Food program (see PTAC report 411).

Laboratories 4, 16 and 22 reported between-laboratories outliers. Laboratory 19 obtained an  $|z|$  score  $> 2$  for between-laboratories precision. Laboratories 8, 15, 16 and 18 reported within-laboratory outliers. Laboratory 12 obtained an  $|z|$  score  $> 2$  for within-laboratory precision.

Four of the laboratories that reported results for protein did not provide an estimate of the measurement uncertainty for their results.

## 6.4 Total Fat

The majority of laboratories used an acid hydrolysis procedure for total fat. Simple solvent extractions (without acid hydrolysis) cannot extract bound fats from materials such as wheat flour. This results in differences between results for the two types of methods. These differences become insignificant in fat measurements on materials with higher fat contents. The results for total fat were, therefore, statistically evaluated by method used. Unfortunately, there were not enough results reported using Soxhlet extraction only, or any other methods (or combination of methods) to calculate z-scores. Therefore, z-scores have only been calculated for those laboratories that used acid hydrolysis.

For acid hydrolysis, the robust CVs were 9.8% and 9.1%. These are lower than the values of 14.1% and 14.5%, obtained in round 17 of the Food program (see PTAC report 411).

Laboratory 10 reported a between-laboratories outlier using acid hydrolysis. Laboratory 9 obtained an  $|z|$  score  $> 2$  for between-laboratories precision. Laboratories 7, 8 and 10 reported within-laboratory outliers.

Three of the laboratories that reported results for total fat did not provide an estimate of the measurement uncertainty for their results.

## 6.5 Moisture

The majority of laboratories used an AOAC method for moisture. A diverse range of temperatures and times were reported for moisture determination, but the results were all comparable. Temperature and heating times are shown in Appendix A (A3.1).

The robust CVs of 2.4% and 2.9% for the two samples are lower than the values of 3.2% and 3.5% obtained in round 17 of the Food program (see PTAC report 411).

Laboratories 12 and 19 reported between-laboratories outliers. Laboratory 19 reported a within-laboratory outlier.

Four of the laboratories that reported results for moisture did not provide an estimate of the measurement uncertainty for their results.

## 6.6 Ash

The majority of laboratories reported results employing AOAC-based methods. A diverse range of temperatures and times for ashing were reported. Temperatures and times are shown in Appendix A (A4.1).

The robust CVs of 8.8% and 5.8% for the two samples are slightly lower than the values of 9.3% and 6.6% obtained in round 17 of the Food program (see PTAC report 411).

Laboratories 15 and 16 reported between-laboratories outliers. Laboratory 10 obtained an  $|z|$  score  $> 2$  for between-laboratories precision. Laboratories 3 and 15 reported within-laboratory outliers. Laboratories 4 and 16 obtained  $|z|$  scores  $> 2$  for within-laboratory precision.

Four of the laboratories that reported results for ash did not provide an estimate of the measurement uncertainty for their results.

## 6.7 Dietary Fibre

The Australasian Food Standards Code (FSC) allows the use of either Prosky or Lee methods (AOAC 985.29 or 991.43). None of the participants used the Lee method for dietary fibre.

The robust CVs of 15.7% and 33.8% for the two samples are much higher than the values of 13.1% and 10.2% obtained in round 17 of the Food program (see PTAC report 411).

There were no between-laboratories outliers or within-laboratory outliers reported for dietary fibre. Laboratories 5 and 17 obtained  $|z|$  scores  $> 2$  for within-laboratory precision.

Three of the laboratories that reported results for dietary fibre did not provide an estimate of the measurement uncertainty for their 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 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:

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

All laboratories calculated carbohydrate “by difference”, but the calculation formulae were quite varied. Five of the results did not include dietary fibre in the calculations. Nine results did include dietary fibre in the calculations. One laboratory (15) included crude fibre in their calculations for carbohydrate. The carbohydrate results for this laboratory, as well as the results for the laboratories that did not include dietary fibre in the calculations, were not included in the statistical evaluation, due to the small number of results using these formulae. Therefore, only the laboratories that included dietary fibre in the calculations were statistically evaluated.

For the laboratories that included dietary fibre in their calculations, the robust CVs were 0.8% and 1.6%. These are similar to the values of 1.2% and 1.2%, obtained in round 17 of the Food program (see PTAC report 411).

Of the laboratories that did include dietary fibre in their calculations, one laboratory (12) reported a between-laboratories outlier. Two other laboratories (17 and 19) reported within-laboratory outliers.

Eight of the laboratories that reported results for carbohydrate did not provide an estimate of the measurement uncertainty for their 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:

(protein  $\times$  17 + total fat  $\times$  37 + carbohydrate  $\times$  17 + dietary fibre  $\times$  8).

The formulae reported for calculating energy results were also quite varied. Four of these results did not include dietary fibre in the calculations. Ten results did include dietary fibre. There was also laboratory 15, which included crude fibre in their calculations. As with the carbohydrate results, only the laboratories that included dietary fibre in their energy calculations were statistically evaluated.

For the laboratories that included dietary fibre in their calculations, the robust CVs were 0.3% and 1.1%. These are similar to the values of 0.6% and 0.5%, obtained in round 17 of the Food program (see PTAC report 411).

Of the laboratories that did include dietary fibre in their calculations, two laboratories (5 and 12) reported between-laboratories outliers. There were no within-laboratory outliers reported.

Eight of the laboratories that reported results for energy did not provide an estimate of the measurement uncertainty for their results.

## 6.10 Measurement Uncertainty

For this program, laboratories were requested to report an estimate of measurement uncertainty (MU) for each test result and to describe the method used for this estimation. Fifteen of the 19 laboratories (79%) that submitted results for this program provided estimates of their MU for some or all of their results. Of these 15 laboratories, 11 supplied a description of the method(s) used to estimate their MU.

The proportion of laboratories that returned MU estimates for each individual test is as follows:

• Protein	15 out of 19	79%
• Total Fat	14 out of 17	82%
• Moisture	15 out of 19	79%
• Ash	14 out of 18	78%
• Dietary Fibre	8 out of 11	73%
• Carbohydrate	7 out of 15	47%
• Energy	7 out of 15	47%

The number of laboratories that quoted measurement uncertainties for their analytical data in this program is encouraging. From the results reported, there were a variety of methods used and a wide range of uncertainties reported, as shown in the tables in Appendix A.

## **7. REFERENCES**

*Guide to Proficiency Testing Australia* (2006).

This document is located on the PTA website at [www.proficiencytesting.com.au](http://www.proficiencytesting.com.au), under "Documents".

# **APPENDIX A**

## **Summary of Results**

# **Section A1**

## **Protein**

## A1.1

### Protein (g/100g) – Samples PTA 1 & PTA 2 – All Results

Lab Code	PTA 1			PTA 2			Method Code
	Average	Corrected	MU (±)	Average	Corrected	MU (±)	
1	11.46	13.21	0.18	11.48	13.25	0.18	2
3	11.40	13.22	3% RE	11.42	13.24	3% RE	2
4	12.07	13.92		12.24	14.12		1
5	11.35	13.07	0.81	11.44	13.18	0.82	1
6	11.11	12.82	0.3	11.14	12.86	0.3	1
7	11.16	12.93	0.2	11.10	12.86	0.2	1
8	11.3	13.1	0.6	11.0	12.8	0.6	1
9	11.20	12.96	0.39	11.20	12.96	0.39	1
10	11.7	13.5	0.3	11.7	13.5	0.3	1
11	11.44	13.26	0.06	11.42	13.24	0.06	1
12	11.42	13.02	0.8	11.66	13.29	0.8	1
13	11.56	13.24	1.0	11.54	13.21	1.0	2
15	11.45	13.17	0.4	10.87	12.50	0.4	2
16	8.32	9.62		7.68	8.88		3
17A	11.40	13.12	1.7%	11.44	13.17	1.7%	1
17B	11.14	12.82	1.5%	11.14	12.84	1.5%	2
18	11.34	13.05		11.07	12.72		1
19	11.34	12.46		11.36	12.33		3
21	11.63	13.40	0.26	11.71	13.50	0.26	2
22	9.77	11.21	0.52	9.82	11.28	0.52	1

### Method Codes

1 = Kjeldahl	12
2 = Dumas	6
3 = Other	2

#### Notes:

1. RE denotes relative error.
2. Laboratories 19 and 22 did not provide a dry basis (corrected) result for protein. These results were calculated using the formula:

$$\text{moisture free result} = (100 \times \text{result}) \div (100 - \text{moisture result}).$$

## A1.2

### Protein (g/100g) – Samples PTA 1 & PTA 2 Z-Scores on Corrected Results

Lab Code	PTA 1	PTA 2	Between Labs Z-Score	Within Lab Z-Score	Method Code
1	13.21	13.25	0.65	-0.30	2
3	13.22	13.24	0.65	-0.10	2
4	13.92	14.12	3.37 §	-1.88	1
5	13.07	13.18	0.28	-0.99	1
6	12.82	12.86	-0.70	-0.30	1
7	12.93	12.86	-0.51	0.79	1
8	13.10	12.80	-0.32	3.07 §	1
9	12.96	12.96	-0.28	0.10	1
10	13.50	13.50	1.58	0.10	1
11	13.26	13.24	0.72	0.30	1
12	13.02	13.29	0.39	-2.57	1
13	13.24	13.21	0.63	0.40	2
15	13.17	12.50	-0.72	6.73 §	2
16	9.62	8.88	-13.08 §	7.42 §	3
17A	13.12	13.17	0.35	-0.40	1
17B	12.82	12.84	-0.73	-0.10	2
18	13.05	12.72	-0.54	3.36 §	1
19	12.46	12.33	-2.23	1.32	3
21	13.40	13.50	1.41	-0.89	2
22	11.21	11.28	-6.20 §	-0.52	1

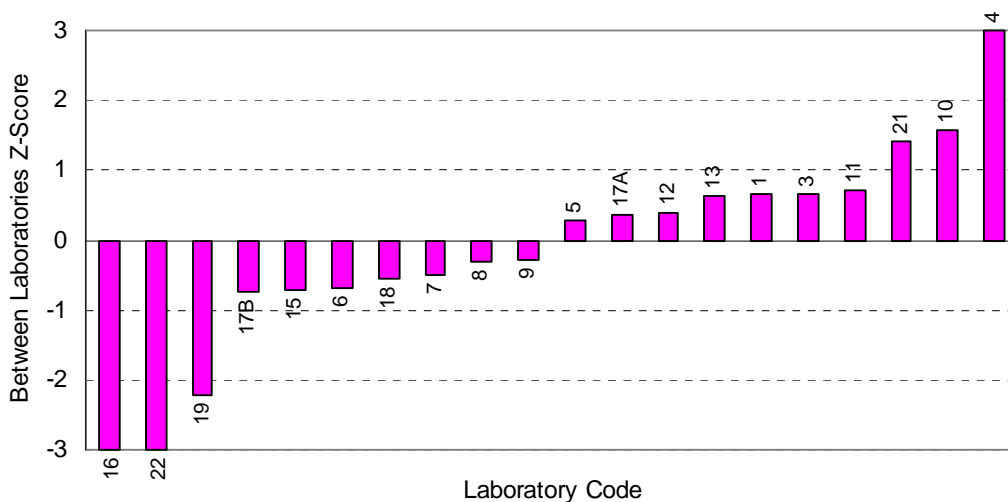
Statistic	PTA 1	PTA 2
No of Results	20	20
Median	13.085	13.065
Norm IQR	0.239	0.343
Robust CV	1.83%	2.62%
Minimum	9.62	8.88
Maximum	13.92	14.12
Range	4.30	5.24

**Notes:**

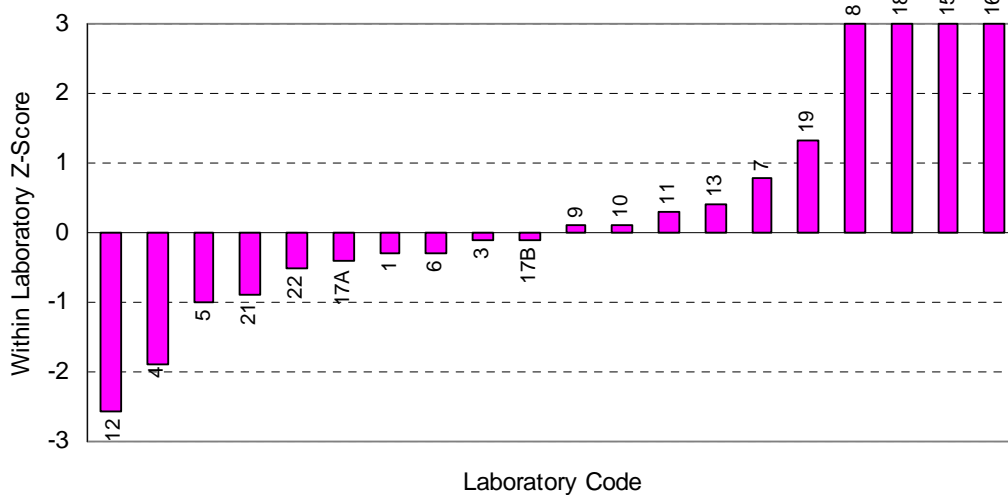
1. § denotes an outlier (i.e. |z-score| > 3).

### A1.3

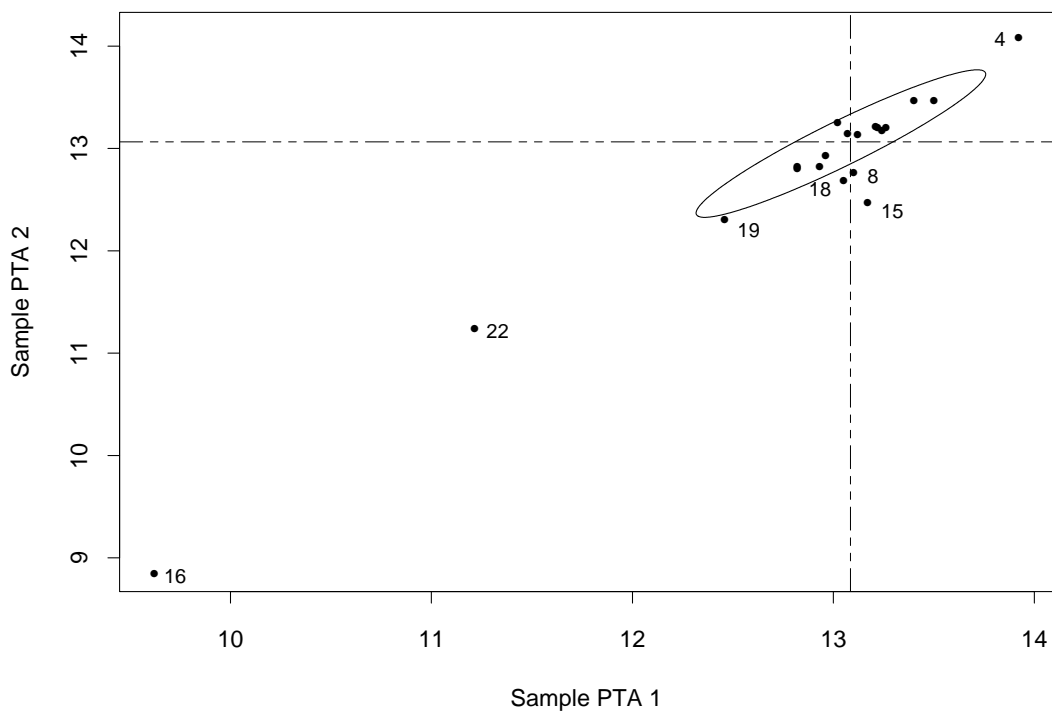
Protein (g/100g)



Protein (g/100g)



Protein (g/100g)



## **Section A2**

### **Total Fat**

## A2.1

### Total Fat (g/100g) – Samples PTA 1 & PTA 2 – All Results

Lab Code	PTA 1			PTA 2			Method Code
	Average	Corrected	MU (±)	Average	Corrected	MU (±)	
1	1.38	1.59	0.36	1.37	1.58	0.36	1
3	1.28	1.48	10%RE	1.29	1.50	10% RE	2
4	1.64	1.89		1.62	1.87		1
5	1.45	1.67	0.17	1.47	1.69	0.17	1
6	1.50	1.73	0.2	1.48	1.70	0.2	1
7	1.42	1.64	0.6	1.72	1.99	0.6	1
8	1.5	1.7	0.1	1.6	1.9	0.1	1
9	1.85	2.14	0.57	1.80	2.08	0.57	1
10	1.3	1.5	0.3	1.1	1.3	0.3	1
11	1.79	2.07		1.74	2.02		1
12	1.09	1.24	0.03	1.03	1.20	0.03	2
13	1.19	1.36	0.1	1.18	1.35	0.1	2
15	1.14	1.31	0.1	1.15	1.32	0.1	2
17A	1.64	1.89	1.0%	1.63	1.88	1.0%	1
19	1.78	1.96		1.53	1.66		3
21	1.64	1.89	0.36	1.55	1.79	0.36	1
22	1.49	1.71	0.10	1.54	1.77	0.10	1

### Method Codes

1 = Acid hydrolysis	12
2 = Soxhlet extraction	4
3 = Other	1

#### Notes:

1. RE denotes relative error.
2. There were not enough total fat results submitted using Soxhlet extraction, or other methods, to analyse these results. Therefore, only the acid hydrolysis results have been analysed.
3. Laboratories 19 and 22 did not provide a dry basis (corrected) result for total fat. These results were calculated using the formula:

$$\text{moisture free result} = (100 \times \text{result}) \div (100 - \text{moisture result}).$$

## A2.2

### Total Fat (acid hydrolysis) (g/100g) – Samples PTA 1 & PTA 2 Z-Scores on Corrected Results

Lab Code	PTA 1	PTA 2	Between Labs Z-Score	Within Lab Z-Score	Method Code
1	1.59	1.58	-1.72	0.08	1
4	1.89	1.87	0.56	-0.08	1
5	1.67	1.69	-0.98	0.58	1
6	1.73	1.70	-0.71	-0.25	1
7	1.64	1.99	0.06	6.00 §	1
8	1.70	1.90	-0.06	3.53 §	1
9	2.14	2.08	2.33	-0.74	1
10	1.50	1.30	-3.14 §	-3.04 §	1
11	2.07	2.02	1.83	-0.58	1
17A	1.89	1.88	0.60	0.08	1
21	1.89	1.79	0.25	-1.40	1
22	1.71	1.77	-0.53	1.20	1

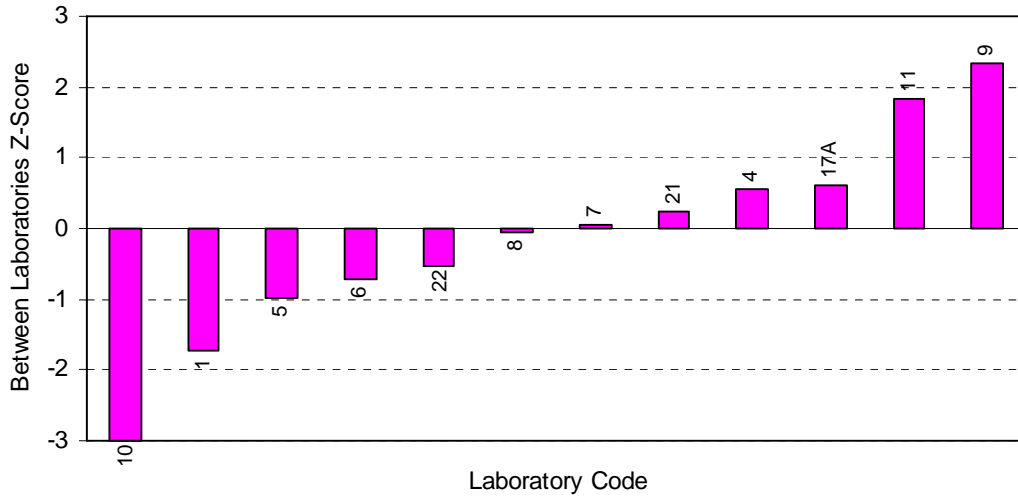
Statistic	PTA 1	PTA 2
No of Results	12	12
Median	1.720	1.830
Norm IQR	0.169	0.167
Robust CV	9.80%	9.11%
Minimum	1.50	1.30
Maximum	2.14	2.08
Range	0.64	0.78

**Notes:**

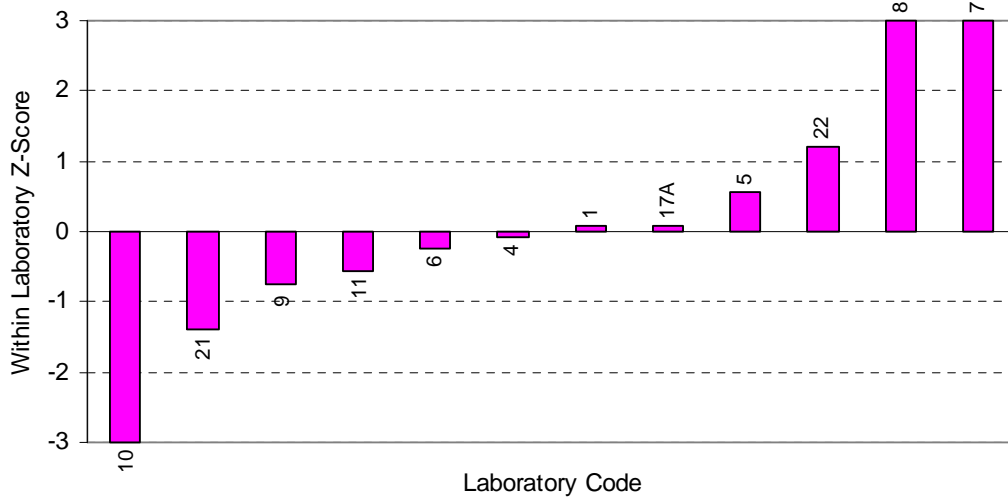
1. § denotes an outlier (i.e. |z-score| > 3).

### A2.3

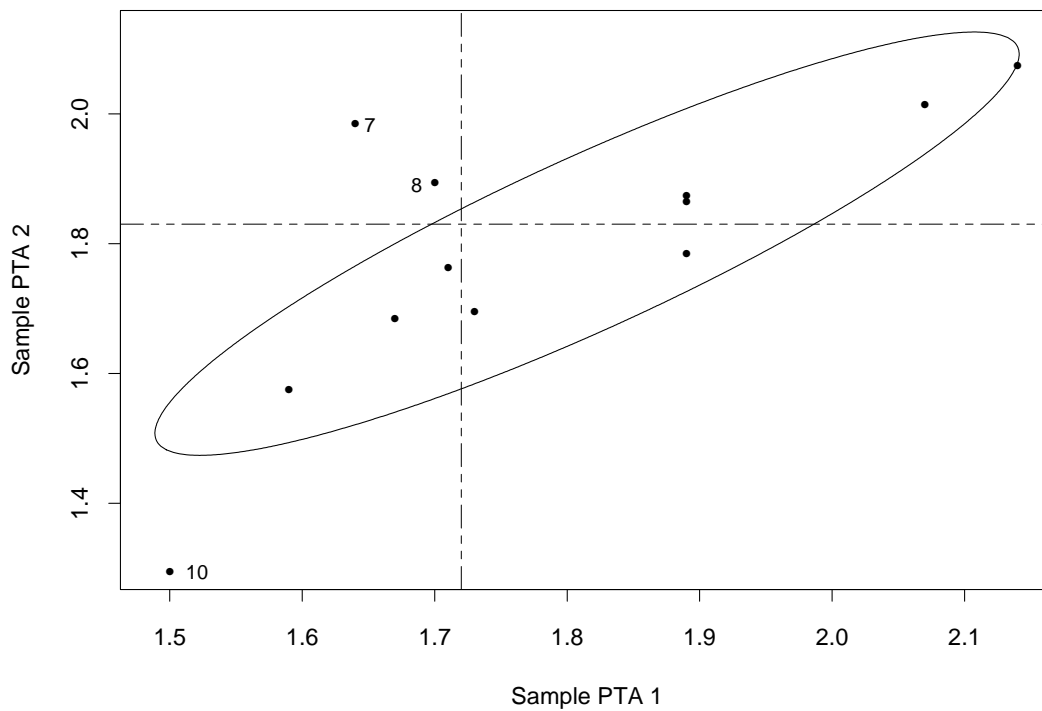
Total Fat (acid hydrolysis) (g/100g)



Total Fat (acid hydrolysis) (g/100g)



Total Fat (acid hydrolysis) (g/100g)



## **Section A3**

### **Moisture**

### A3.1

#### Moisture (g/100g) – Samples PTA 1 & PTA 2 – All Results

Lab Code	PTA 1		PTA 2		Method Code	Temp. (°C)	Time (hrs)
	Average	MU (±)	Average	MU (±)			
1	13.28	0.22	13.34	0.22	1	103	3
3	13.72	10% RE	13.70	10% RE	2	131	1.5
4	13.32		13.24		1	130	1
5	13.18	1.18	13.23	1.19	1	130	2
6	13.33	0.3	13.41		1	130	1
7	13.66	0.4	13.69	0.4	1	130	1
8	14.0	3.3	13.9	3.2	1	105	4
9	13.55	0.48	13.60	0.48	1	130	1
10	13.1	0.2	13.2	0.2	1	102±2	16
11	13.71	0.04	13.75	0.04	2	130	1
12	12.1	2.1	12.0	2.1	1	130	1.5
13	12.66	1.0	12.62	1.0	1	105	4
15	13.09	0.04	13.07	0.04	2	130	1
16	13.48		13.48		2	130	1.5
17A	13.13	0.2%	13.13	0.2%	1	130	1
18	13.10		12.98		1	130	1
19	8.97		7.9		2		
21	13.24	0.23	13.28	0.23	1	130	1
22	12.87	2.95	12.91	2.95	1	103	16

#### Method Codes

1 = AOAC

14

2 = Other

5

#### Notes:

1. RE denotes relative error.

A3.2

Moisture (g/100g) – Samples PTA 1 & PTA 2  
Z-Scores on Average Results

Lab Code	PTA 1	PTA 2	Between Labs Z-Score	Within Lab Z-Score	Method Code
1	13.28	13.34	0.14	-0.77	1
3	13.72	13.70	1.30	0.26	2
4	13.32	13.24	0.06	1.03	1
5	13.18	13.23	-0.16	-0.64	1
6	13.33	13.41	0.32	-1.03	1
7	13.66	13.69	1.20	-0.39	1
8	14.00	13.90	1.99	1.28	1
9	13.55	13.60	0.91	-0.64	1
10	13.10	13.20	-0.32	-1.28	1
11	13.71	13.75	1.36	-0.51	2
12	12.10	12.00	-3.49 §	1.28	1
13	12.66	12.62	-1.79	0.51	1
15	13.09	13.07	-0.52	0.26	2
16	13.48	13.48	0.63	0.00	2
17A	13.13	13.13	-0.38	0.00	1
18	13.10	12.98	-0.63	1.54	1
19	8.97	7.90	-13.92 §	13.75 §	2
21	13.24	13.28	0.00	-0.51	1
22	12.87	12.91	-1.07	-0.51	1

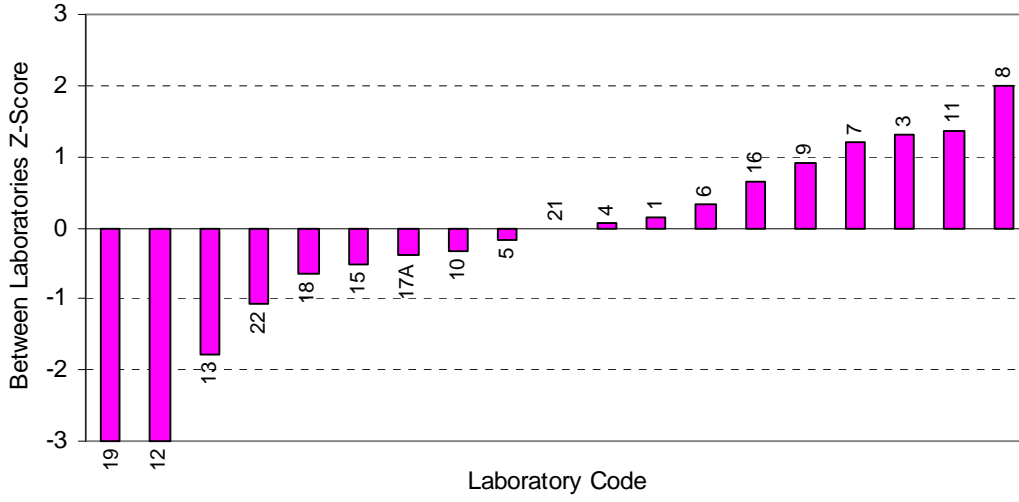
Statistic	PTA 1	PTA 2
No of Results	19	19
Median	13.240	13.240
Norm IQR	0.311	0.382
Robust CV	2.35%	2.88%
Minimum	8.97	7.90
Maximum	14.00	13.90
Range	5.03	6.00

**Notes:**

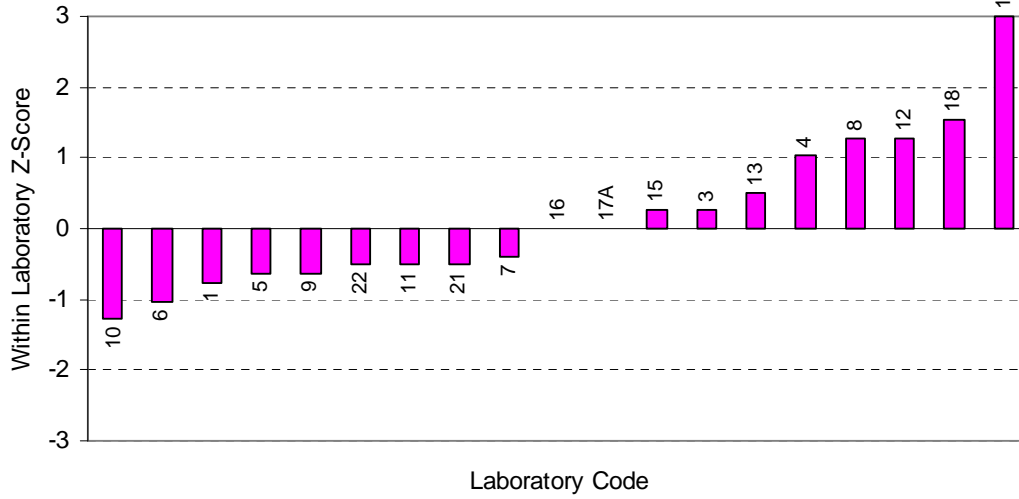
1. § denotes an outlier (i.e. |z-score| > 3).

### A3.3

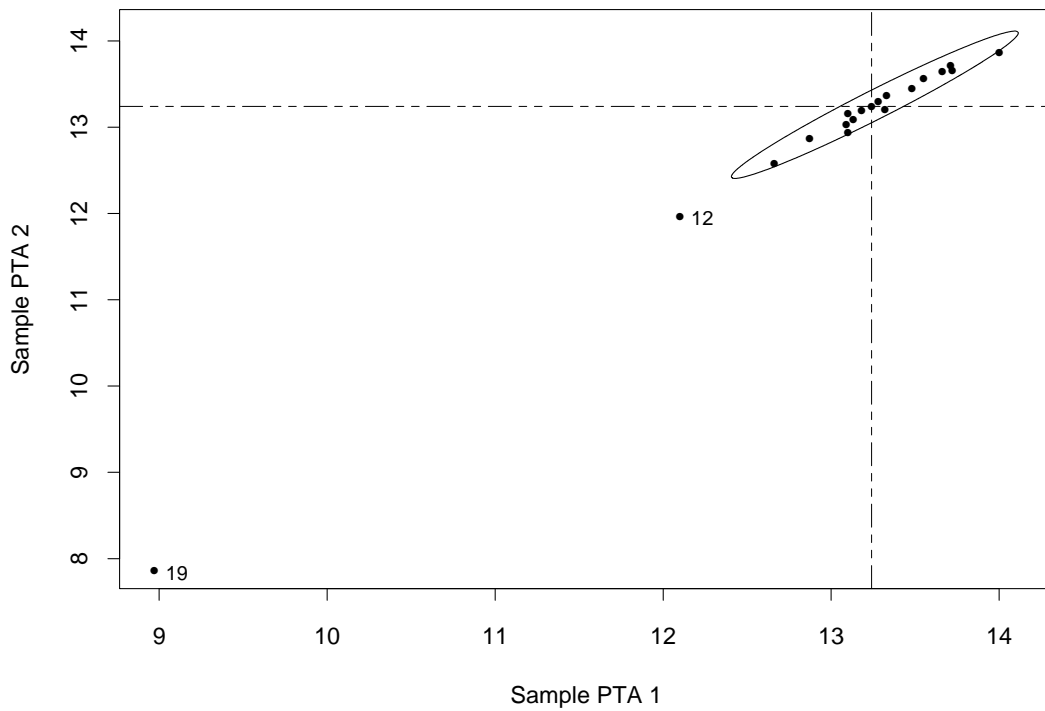
Moisture (g/100g)



Moisture (g/100g)



Moisture (g/100g)



## **Section A4**

### **Ash**

## A4.1

### Ash (g/100g) – Samples PTA 1 & PTA 2 – All Results

Lab Code	PTA 1			PTA 2			Method Code	Temp. (°C)	Time (hrs)
	Average	Corrected	MU (±)	Average	Corrected	MU (±)			
1	0.61	0.70	0.14	0.63	0.73	0.14	2	560	16
3	0.46	0.53	10% RE	0.61	0.71	10% RE	1	600	5
4	0.52	0.60		0.58	0.67		1	550	15.5
5	0.65	0.75	0.02	0.63	0.73	0.02	1	600	2
6	0.67	0.77	0.04	0.67	0.77	0.04	1	550	16
7	0.64	0.74	0.04	0.62	0.72	0.04	3	580	16
8	0.6	0.7	0.1	0.6	0.7	0.1	1	550	o/n
9	0.60	0.69	0.08	0.61	0.71	0.08	3	590	16
10	0.70	0.81	0.3	0.70	0.81	0.3	1	525	16
11	0.56	0.65		0.58	0.67		1	525	16
12	0.61	0.70	0.02	0.59	0.67	0.02	1	550	18
13	0.62	0.71	0.1	0.60	0.69	0.1	1	550	16
15	0.67	0.77	0.2	1.00	1.15	0.2	1	600	2
16	0.42	0.49		0.47	0.54		3	600±25	1.0
17A	0.61	0.70	0.5%	0.60	0.69	0.5%	3	550	8
19	0.58	0.64		0.59	0.64		3		
21	0.64	0.74	0.04	0.64	0.74	0.04	1	550	3
22	0.59	0.68	0.11	0.61	0.70	0.11	1	600	2

### Method Codes

1 = AOAC	12
2 = Pearson	1
3 = Other	5

#### Notes:

1. RE denotes relative error.
2. Laboratories 16, 19 and 22 did not provide a dry basis (corrected) result for ash. These results were calculated using the formula:

$$\text{moisture free result} = (100 \times \text{result}) \div (100 - \text{moisture result}).$$

## A4.2

### Ash (g/100g) – Samples PTA 1 & PTA 2 Z-Scores on Corrected Results

Lab Code	PTA 1	PTA 2	Between Labs Z-Score	Within Lab Z-Score	Method Code
1	0.70	0.73	0.28	1.06	2
3	0.53	0.71	-1.51	6.71 §	1
4	0.60	0.67	-1.23	2.57	1
5	0.75	0.73	0.76	-0.82	1
6	0.77	0.77	1.33	-0.07	1
7	0.74	0.72	0.57	-0.82	3
8	0.70	0.70	0.00	-0.07	1
9	0.69	0.71	0.00	0.69	3
10	0.81	0.81	2.08	-0.07	1
11	0.65	0.67	-0.76	0.69	1
12	0.70	0.67	-0.28	-1.19	1
13	0.71	0.69	0.00	-0.82	1
15	0.77	1.15	4.92 §	14.25 §	1
16	0.49	0.54	-3.52 §	2.11	3
17A	0.70	0.69	-0.09	-0.44	3
19	0.64	0.64	-1.16	0.07	3
21	0.74	0.74	0.76	-0.07	1
22	0.68	0.70	-0.21	0.81	1

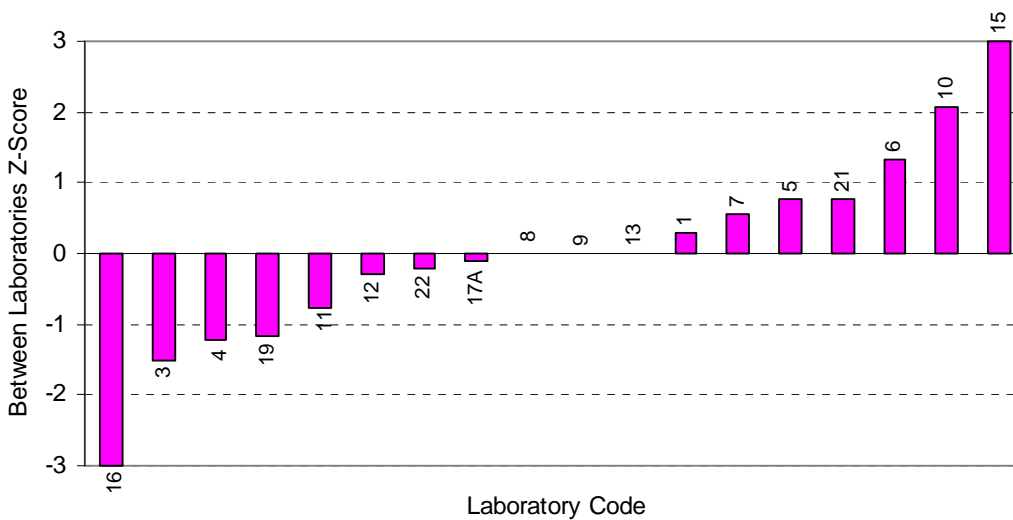
Statistic	PTA 1	PTA 2
No of Results	18	18
Median	0.700	0.705
Norm IQR	0.062	0.041
Robust CV	8.81%	5.78%
Minimum	0.49	0.54
Maximum	0.81	1.15
Range	0.32	0.61

**Notes:**

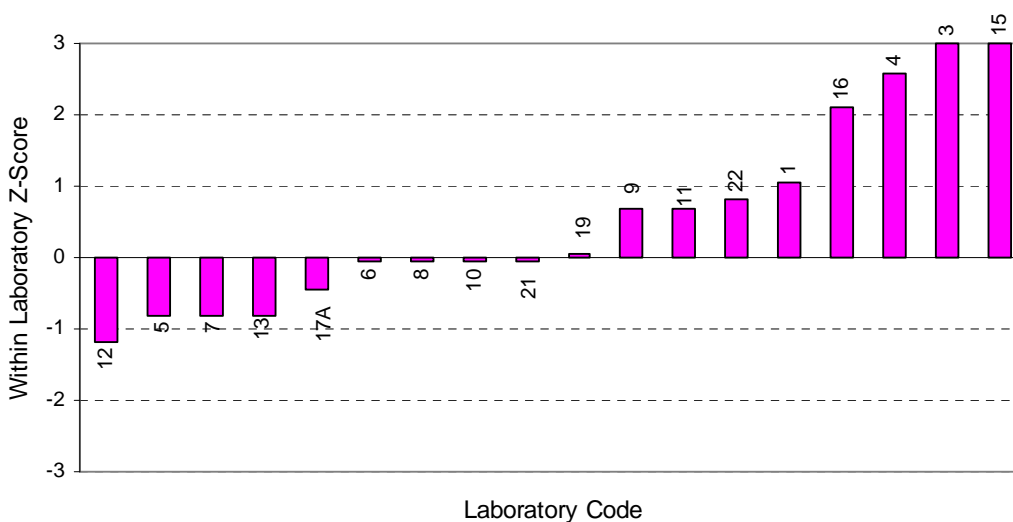
1. § denotes an outlier (i.e. |z-score| > 3).

### A4.3

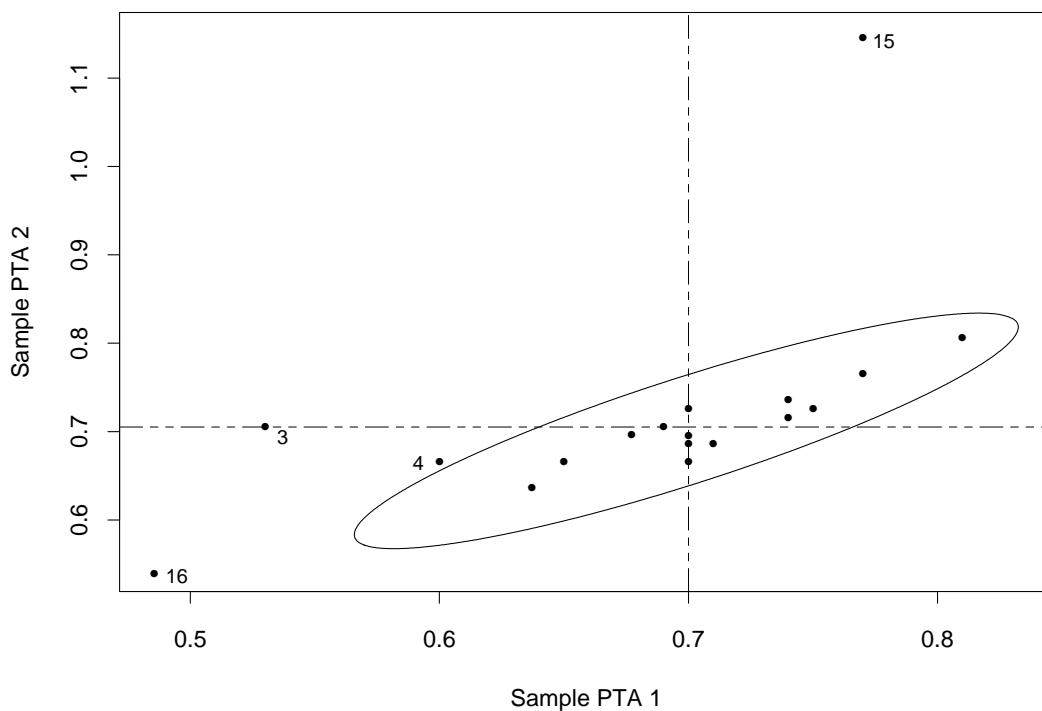
Ash (g/100g)



Ash (g/100g)



Ash (g/100g)



**Section A5**

**Dietary Fibre**

## A5.1

### Dietary Fibre (g/100g) – Samples PTA 1 & PTA 2 – All Results

Lab Code	PTA 1			PTA 2			Method Code
	Average	Corrected	MU ( $\pm$ )	Average	Corrected	MU ( $\pm$ )	
1	4.08	4.70	0.94	4.00	4.62	0.92	1
3	3.60	4.17	10% RE	3.40	3.94	10% RE	1
5	4.6	5.30		6.2	7.32		3
6	3.49	4.02	0.7	3.25	3.75	0.7	1
7	3.51	4.06	0.9	3.54	4.10	0.9	1
9	3.80	4.40		3.60	4.17		1
12	4.45	5.07	0.8	4.53	5.16	0.8	1
13	2.93	3.35	0.3	3.01	3.44	0.3	1
17A	4.25	4.89	20%	5.92	6.81	20%	1
19	4.6	5.05		6.2	6.73		3
21	3.49	4.02	0.34	3.67	4.23	0.34	1

### Method Codes

1 = AOAC 985.29 (Prosky)	9
2 = AOAC 991.43 (Lee)	0
3 = Other	2

#### Notes:

1. RE denotes relative error.
2. Laboratory 19 did not provide a dry basis (corrected) result for dietary fibre. This result was calculated using the formula:

$$\text{moisture free result} = (100 \times \text{result}) \div (100 - \text{moisture result}).$$

**A5.2**

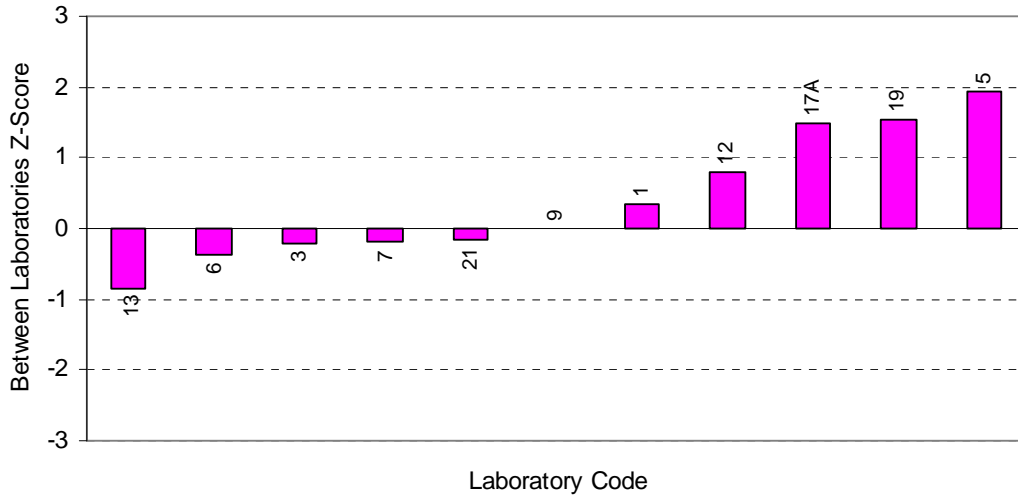
**Dietary Fibre (g/100g) – Samples PTA 1 & PTA 2  
Z-Scores on Corrected Results**

Lab Code	PTA 1	PTA 2	Between Labs Z-Score	Within Lab Z-Score	Method Code
1	4.70	4.62	0.36	0.21	1
3	4.17	3.94	-0.22	0.39	1
5	5.30	7.32	1.93	-2.37	3
6	4.02	3.75	-0.38	0.44	1
7	4.06	4.10	-0.20	0.06	1
9	4.40	4.17	0.00	0.39	1
12	5.07	5.16	0.79	0.00	1
13	3.35	3.44	-0.85	0.00	1
17A	4.89	6.81	1.49	-2.25	1
19	5.05	6.73	1.53	-1.95	3
21	4.02	4.23	-0.15	-0.15	1

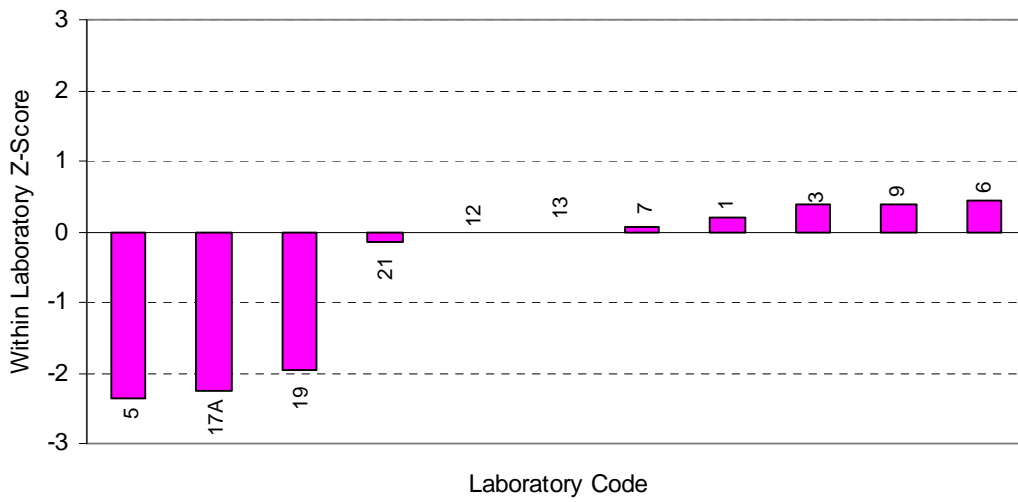
Statistic	PTA 1	PTA 2
No of Results	11	11
Median	4.400	4.230
Norm IQR	0.691	1.428
Robust CV	15.70%	33.75%
Minimum	3.35	3.44
Maximum	5.30	7.32
Range	1.95	3.88

### A5.3

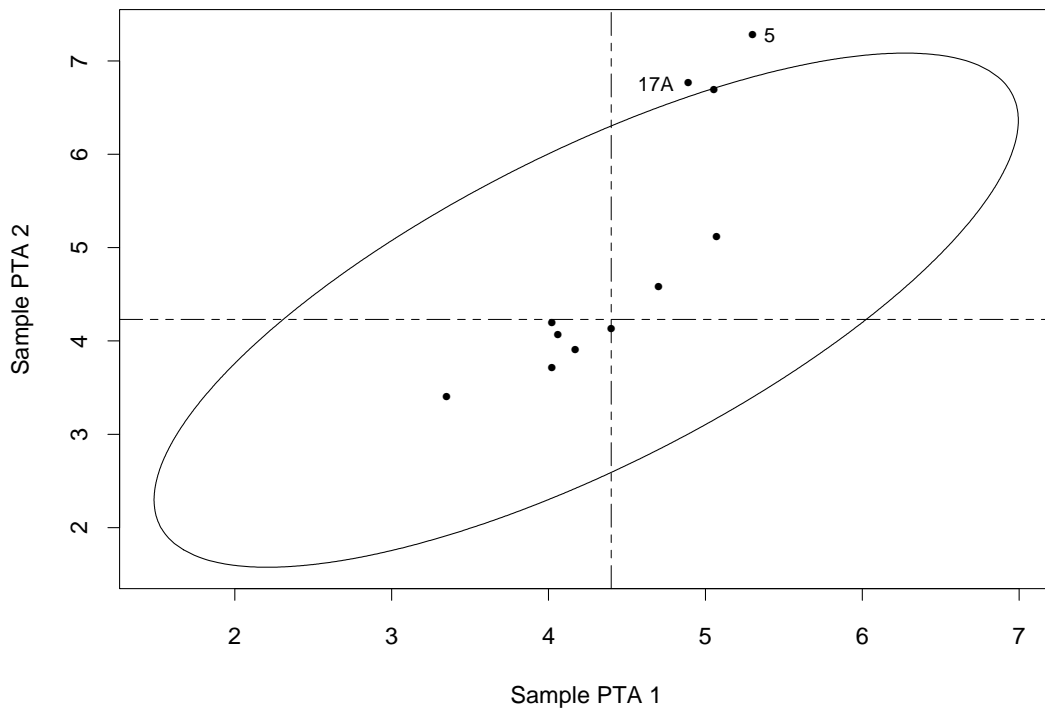
Dietary Fibre (g/100g)



Dietary Fibre (g/100g)



Dietary Fibre (g/100g)



**Section A6**  
**Carbohydrate**

## A6.1

### Carbohydrate (g/100g) – Samples PTA 1 & PTA 2 – All Results

Lab Code	PTA 1			PTA 2			Formula Code
	Average	Corrected	MU (±)	Average	Corrected	MU (±)	
1	69.19	79.78		69.18	79.83		2
3	73.14	84.78		72.98	84.57		1
4	72.45	70.27		72.32	70.10		1
5	68.77	64.03		67.03	61.83		1
6	69.90	80.66	2	70.05	80.92	2	2
7	69.61	80.63	1.6	69.33	80.33	1.6	2
8	72.6	84.4	4.1	72.9	84.7	4.0	1
9	69.00	79.81		69.19	80.08		2
10		71			71		1
12	70.33	66.17	10.8	70.19	65.98	10.8	2
13	71.04	81.34	7	71.05	81.31	7	2
15	73.6	84.69	0.4	73.8	84.90	0.4	3
17A	68.97	79.39		67.28	77.45		2
19	72.7	79.86		72.4	78.61		2
21	69.36	79.94	1.22	69.15	79.74	1.22	2

### Formula Codes

1 = Did not include dietary fibre in calculation	5
2 = Included dietary fibre in calculation	9
3 = Included crude fibre in calculation	1

### Notes:

1. Only the carbohydrate results that included dietary fibre in the calculation have been analysed.
2. Laboratory 17 used their protein results using Kjeldahl digestion to calculate their carbohydrate results.
3. Laboratory 19 did not provide a dry basis (corrected) result for carbohydrate. This result was calculated using the formula:

$$\text{moisture free result} = (100 \times \text{result}) \div (100 - \text{moisture result}).$$

## A6.2

### Carbohydrate (including dietary fibre in calculation) (g/100g) Samples PTA 1 & PTA 2 – Z-Scores on Corrected Results

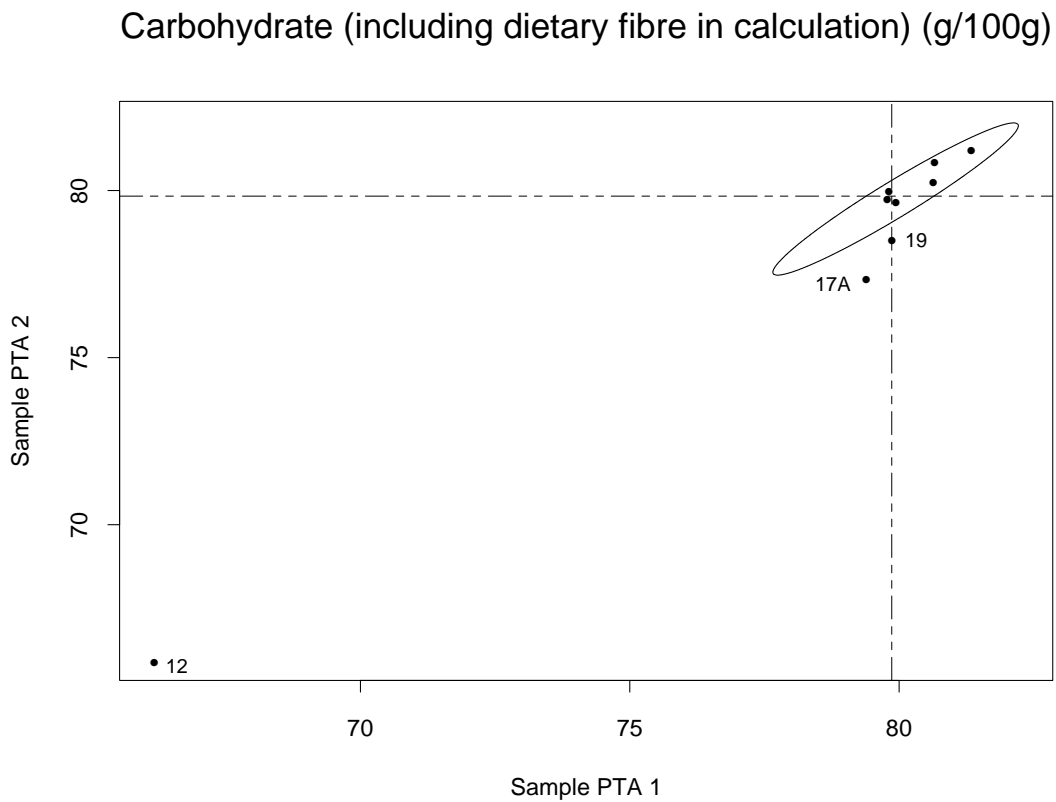
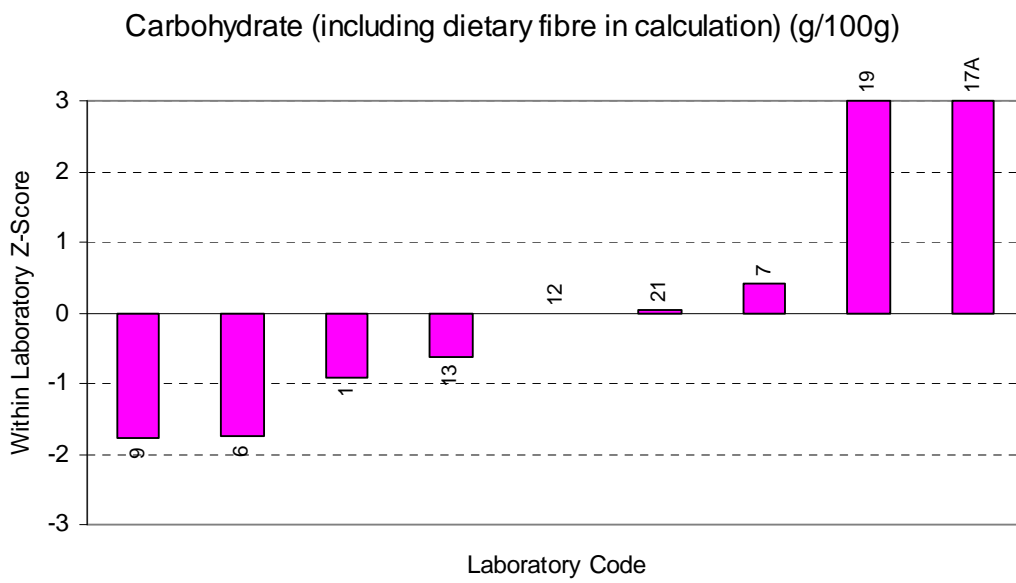
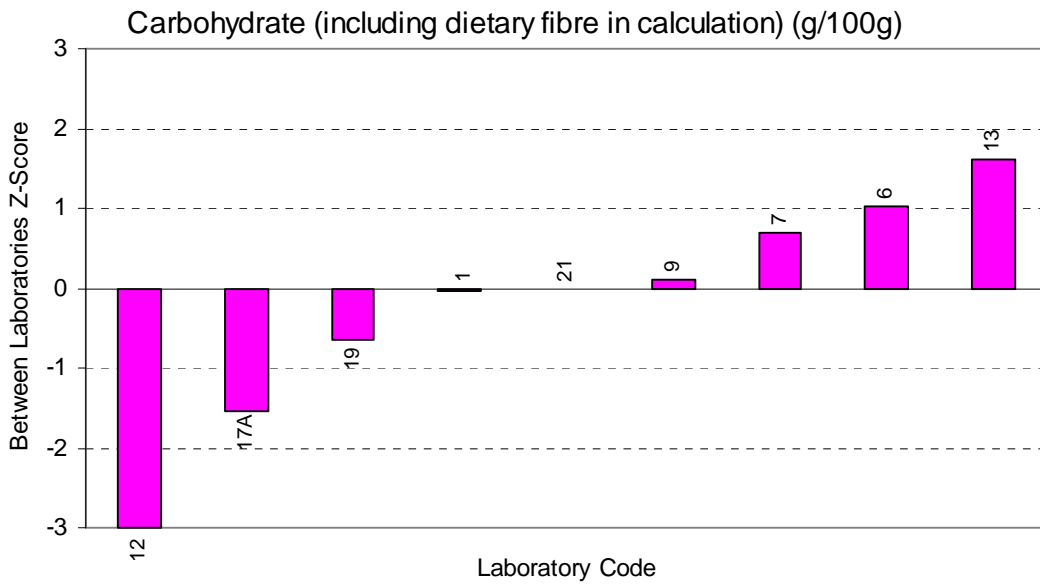
Lab Code	PTA 1	PTA 2	Between Labs Z-Score	Within Lab Z-Score	Formula Code
1	79.78	79.83	-0.04	-0.93	2
6	80.66	80.92	1.03	-1.73	2
7	80.63	80.33	0.69	0.42	2
9	79.81	80.08	0.11	-1.77	2
12	66.17	65.98	-14.94 §	0.00	2
13	81.34	81.31	1.61	-0.62	2
17A	79.39	77.45	-1.54	6.74 §	2
19	79.86	78.61	-0.65	4.10 §	2
21	79.94	79.74	0.00	0.04	2

Statistic	PTA 1	PTA 2
No of Results	9	9
Median	79.864	79.830
Norm IQR	0.630	1.275
Robust CV	0.79%	1.60%
Minimum	66.17	65.98
Maximum	81.34	81.31
Range	15.17	15.33

**Notes:**

1. § denotes an outlier (i.e. |z-score| > 3).

### A6.3



## **Section A7**

### **Energy**

## A7.1

### Energy (kJ/100g) – Samples PTA 1 & PTA 2 – All Results

Lab Code	PTA 1			PTA 2			Formula Code
	Average	Corrected	MU (±)	Average	Corrected	MU (±)	
1	1454.75	1677.52		1453.91	1677.72		2
3	1484.54	1720.69		1482.53	1717.93		1
4	1497.53	1501.16		1484.13	1500.93		1
5	1452	1415		1438	1396		2
6	1460	1685.33	52	1461	1687.16	52	2
7	1453.71	1683.70	30	1459.27	1690.70	30	2
8	1452	1723	84	1486	1726	82	1
9	1462.25	1691.44		1462.03	1692.16		2
10		1490			1490		1
12	1470	1438	294	1474	1439	294	2
13	1471.67	1684.99	140	1471.77	1684.33	140	2
15	1488.03	1712.15	0.4	1481.94	1704.75	0.4	3
17A	1460.97	1681.79		1445.91	1664.45		2
19	1532	1682.96		1530	1661.24		2
21	1465.43	1689.06	46.00	1461.33	1685.11	46.00	2

### Formula Codes

1 = Did not include dietary fibre in calculation	4
2 = Included dietary fibre in calculation	10
3 = Included crude fibre in calculation	1

### Notes:

1. Only the energy results that included dietary fibre in the calculation have been analysed.
2. Laboratory 17 used their protein results using Kjeldahl digestion to calculate their energy results.
3. Laboratory 19 did not provide a dry basis (corrected) result for energy. This result was calculated using the formula:

$$\text{moisture free result} = (100 \times \text{result}) \div (100 - \text{moisture result}).$$

## A7.2

### Energy (including dietary fibre in calculation) (kJ/100g) Samples PTA 1 & PTA 2 – Z-Scores on Corrected Results

Lab Code	PTA 1	PTA 2	Between Labs Z-Score	Within Lab Z-Score	Formula Code
1	1677.52	1677.72	-0.33	-0.04	2
5	1415.00	1396.00	-25.61 §	1.70	2
6	1685.33	1687.16	0.47	-0.19	2
7	1683.70	1690.70	0.56	-0.65	2
9	1691.44	1692.16	0.99	-0.09	2
12	1438.00	1439.00	-22.54 §	-0.11	2
13	1684.99	1684.33	0.33	0.04	2
17A	1681.79	1664.45	-0.75	1.55	2
19	1682.96	1661.24	-0.84	1.94	2
21	1689.06	1685.11	0.55	0.34	2

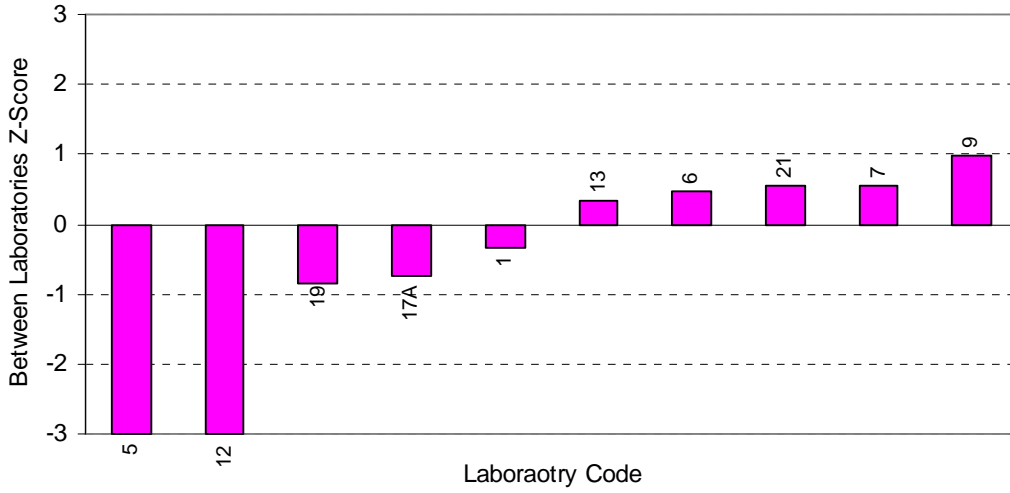
Statistic	PTA 1	PTA 2
No of Results	10	10
Median	1683.331	1681.025
Norm IQR	4.935	18.241
Robust CV	0.29%	1.09%
Minimum	1415.00	1396.00
Maximum	1691.44	1692.16
Range	276.44	296.16

**Notes:**

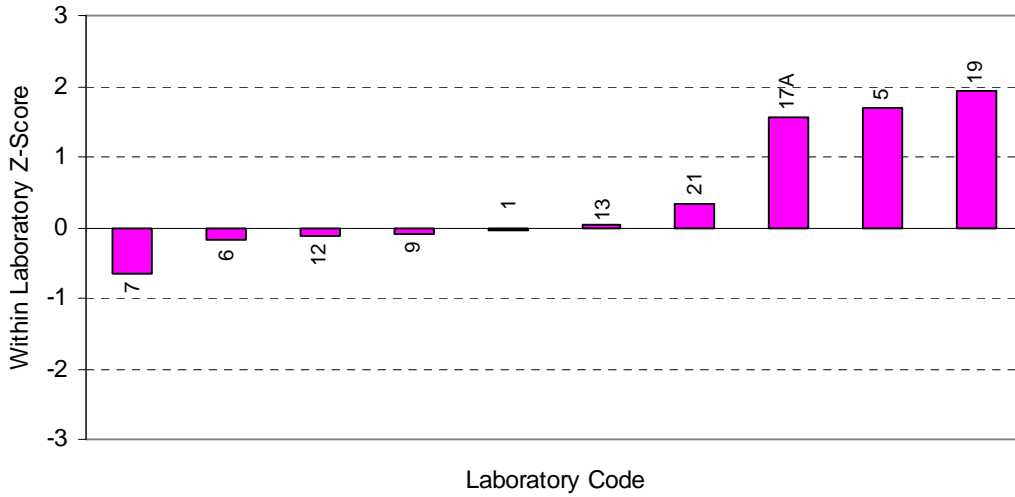
- § denotes an outlier (i.e. |z-score| > 3).

### A7.3

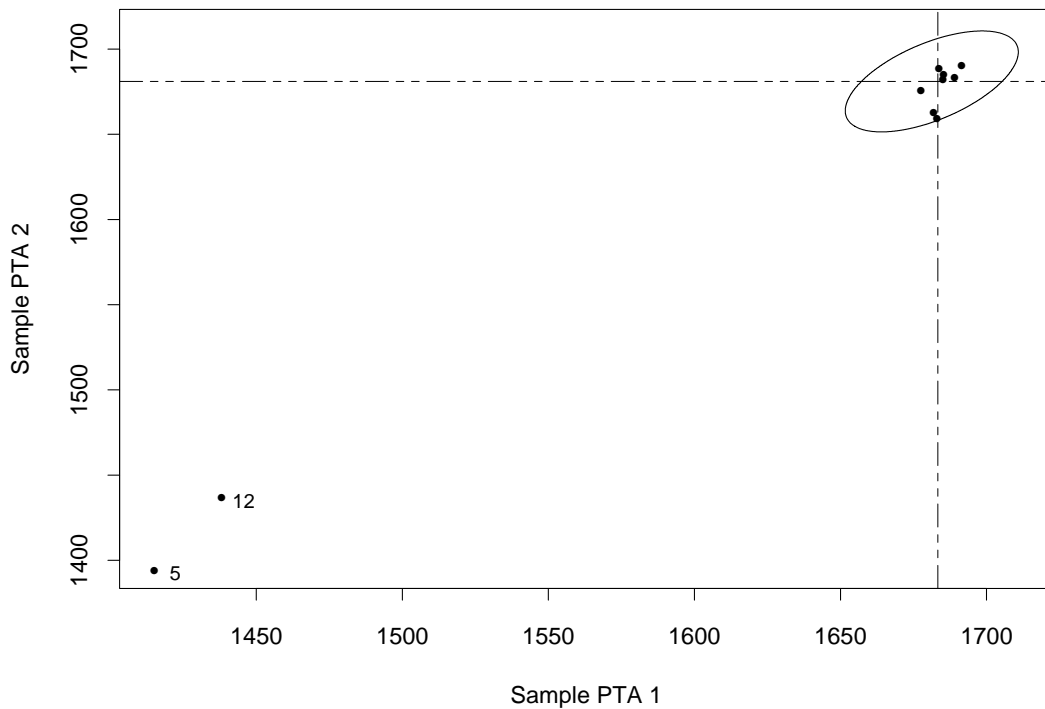
Energy (including dietary fibre in calculation) (kJ/100g)



Energy (including dietary fibre in calculation) (kJ/100g)



Energy (including dietary fibre in calculation) (kJ/100g)



# **APPENDIX B**

## **Homogeneity Testing**

## B1.1

### HOMOGENEITY TESTING

Prior to distribution, ten samples of wheat flour were selected at random and set aside for homogeneity testing by AgriQuality New Zealand Limited. Each sample was tested in duplicate for protein and moisture. The results of this testing appear in the following table.

#### Homogeneity Testing Results

Wheat Flour (Job Number 652985)

Lab Ref	Protein (%m/m)		Moisture (%m/m)	
	Result 1	Result 2	Result 1	Result 2
652985-1	11.452	11.451	13.354	13.445
652985-2	11.388	11.420	13.651	13.428
652985-3	11.436	11.462	13.486	13.301
652985-4	11.454	11.432	13.540	13.602
652985-5	11.442	11.499	13.447	13.329
652985-6	11.497	11.451	13.457	13.597
652985-7	11.428	11.456	13.489	13.420
652985-8	11.474	11.496	13.459	13.529
652985-9	11.367	11.405	13.484	13.424
652985-10	11.382	11.343	13.613	13.524

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

# **APPENDIX C**

**Instructions to Participants**

**and**

**Results Sheets**

**PROFICIENCY TESTING AUSTRALIA  
FOOD PROFICIENCY TESTING PROGRAM  
ROUND 25, NOVEMBER 2006  
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 175g samples of wheat flour labelled PTA 1 and PTA 2.
2. Testing may commence as soon as samples are received. Store your samples in the original packaging in a cool, dry place until testing commences.
3. The following tests are to be performed on each sample in **duplicate** and the **average result reported**:
  - Protein
  - Moisture
  - Dietary Fibre
  - Energy<sup>††</sup>
  - Total Fat
  - Ash
  - Carbohydrate<sup>†</sup>

Notes:   <sup>†</sup> determined by difference  
          <sup>††</sup> determined by calculation

Analysts should be aware of analyte stability and perform tests in an appropriate order. The conversion factor to be used for reporting protein is  $N \times 5.7$ . Results are to be reported on a moisture free (dry solids) basis.

4. Participants are requested to perform all tests listed above for which NATA accreditation is held. Laboratories should use the routine test methods which would normally be used to test customer supplied samples.
5. Please identify the methods on the Results Sheet, using the Method Codes listed on Page 2 of these instructions.
6. Participants are welcome to report results for any other tests for which NATA accreditation is not held, however, please note this on the Results Sheet.
7. Results are to be reported to 2 decimal places.
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. **All laboratories must return results no later than Friday 10 November 2006 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
--------------------------------------------------------------------------------------------------------------------------------------------------------------------------

**PROFICIENCY TESTING AUSTRALIA  
FOOD PROFICIENCY TESTING PROGRAM  
ROUND 25, NOVEMBER 2006**



**INSTRUCTIONS TO PARTICIPANTS**

**METHOD CODES**

Analysis	Method	Code
Protein (g/100g)	Kjeldahl digestion	1
	Dumas (combustion)	2
	Other (please specify)	3
Total Fat (g/100g)	* Acid-hydrolysis	1
	** Soxhlet extraction	2
	Other (please specify)	3
Moisture (g/100g)	AOAC (please specify)	1
	Other (please specify)	2
Dietary Fibre (g/100g)	AOAC 985.29 (Prosky)	1
	AOAC 991.43 (Lee)	2
	Other (please specify)	3
Ash (g/100g)	AOAC (please specify)	1
	Pearson	2
	Other (please specify)	3

**NOTE:**

Please also specify the calibrant for the Dumas method, temperatures and drying times used for determining ash and moisture.

\* "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.

**PROFICIENCY TESTING AUSTRALIA  
FOOD PROFICIENCY TESTING PROGRAM  
ROUND 25, NOVEMBER 2006**



**RESULTS SHEET**

Laboratory Code:

Test	SAMPLE 1			SAMPLE 2			Method Code
	Average Result	Corrected Result (Dry Basis)	MU ( $\pm$ )	Average Result	Corrected Result (Dry Basis)	MU ( $\pm$ )	
Protein (Nx5.7) (g/100g)							
Total Fat (g/100g)							
Moisture (g/100g)							
Ash (g/100g)							
Dietary Fibre (g/100g)							
Carbohydrate (g/100g)							
Energy (kJ/100g)							

Please specify the calibrating material for Dumas nitrogen determination:- \_\_\_\_\_  
(eg pure chemical (EDTA etc.) OR Kjeldahl reference material)

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

Please specify the temperature/time of ashing: \_\_\_\_\_ $^{\circ}$ C/ \_\_\_\_\_ hours.

Please describe the equation used to calculate Carbohydrate:-

Please describe the equation used to calculate Energy:-

**NOTE:** PTA recognises that the precision requested (required for statistical purposes) may exceed the usual number of decimal places reported by laboratories.

Date: \_\_\_\_\_

Signature: \_\_\_\_\_

C2.2

**PROFICIENCY TESTING AUSTRALIA  
FOOD PROFICIENCY TESTING PROGRAM  
ROUND 25, NOVEMBER 2006**



**MEASUREMENT UNCERTAINTY COMMENTS**

**Laboratory Code:**

Please use the space below to briefly describe the method used to determine the measurement uncertainty for each reported measurement result.

**Please return results no later than Friday 10 November 2006 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