

REPORT NO. 565

**Food Proficiency Testing Program
Round 27 – Meat Paste**

February 2008

ACKNOWLEDGMENTS

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Dr Pieter Scheelings, Queensland Health Scientific Services, and Mr Mervyn Withers, AsureQuality Australia Pty Limited, who arranged for the supply of samples.

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1.	FOREWORD	1
2.	FEATURES OF THE PROGRAM	1
3.	FORMAT OF THE APPENDICES	2
4.	STATISTICAL DESIGN OF THE PROGRAM	2
5.	EXTREME RESULTS	2
	Table A: Summary Statistics for All Tests	3
	Table B: Summary of Statistical Outliers	4
6.	PTA AND TECHNICAL ADVISER'S COMMENTS	4
	Table C: Method of Measurement Uncertainty Estimation	9
7.	REFERENCES	11
APPENDICES		
APPENDIX A		
	Summary of Results	
	Protein	A1
	Fat	A2
	Moisture	A3
	Ash	A4
	Phosphorus	A5
	Salt	A6
	Carbohydrate	A7
	Energy	A8
APPENDIX B		
	Homogeneity Testing	B1
APPENDIX C		
	Instructions to Participants	C1
	Results Sheets	C2

1. FOREWORD

This report summarises the results of a proficiency testing program involving the analysis of meat paste samples. It constitutes the twenty seventh round of an ongoing series of programs involving chemical analysis of foodstuffs.

Proficiency Testing Australia conducted the testing program in December 2007. The program coordinator was Mark Bunt. 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 14 laboratories participated in the program. One laboratory did not return results for inclusion in the final report. Laboratories from the following states and countries participated in the program:

4	NSW
3	QLD
4	VIC
2	WA
1	THAILAND

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 meat paste labelled PTA 1 and PTA 2.
- (d) Participants were asked to perform tests for:
- protein
 - fat
 - moisture
 - ash
 - phosphorus
 - salt
 - carbohydrate
 - energy

Laboratories were required to perform all tests for which they hold 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. Copies of these documents appear in Appendix C.

- (f) Prior to sample distribution, ten randomly selected samples were analysed for homogeneity byASUREQuality New Zealand Pty Limited. Based on the results of this testing, the homogeneity of the samples was established.

3. FORMAT OF THE APPENDICES

- (a) Appendix A is divided into 8 sections (A1-A8). These sections contain the analysis of results reported by laboratories for protein, fat, moisture, ash, phosphorus, salt, carbohydrate and energy.

Each of these sections contains:

- i) a table of results reported by laboratories for each test with calculated z-scores and method codes;
 - ii) a listing of the summary statistics;
 - iii) ordered z-score charts;
 - iv) 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, fat, moisture, ash, phosphorus, salt, carbohydrate and energy.

5. EXTREME 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 high or excessively low results in comparison to the consensus value (the median). Any result with an absolute z-score greater than three (i.e. <-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 table summaries the results submitted by participants for the program.

Test	Summary Statistics	PTA 1	PTA 2
Protein (N x 6.25) (g/100g)	No. of Results	13	13
	Median	16.80	16.60
	Normalised IQR	0.15	0.37
Fat (g/100g)	No. of Results	13	13
	Median	10.10	10.10
	Normalised IQR	0.62	0.37
Moisture (g/100g)	No. of Results	13	13
	Median	63.70	63.80
	Normalised IQR	0.24	0.30
Ash (g/100g)	No. of Results	13	13
	Median	2.530	2.540
	Normalised IQR	0.052	0.074
Salt (g/100g)	No. of Results	11	11
	Median	1.180	1.140
	Normalised IQR	0.072	0.074
Carbohydrate (g/100g)	No. of Results	13	13
	Median	6.920	6.930
	Normalised IQR	0.252	0.326
Energy (kJ/100g)	No. of Results	13	13
	Median	777.0	776.0
	Normalised IQR	14.1	14.1

Note:

Summary statistics were not calculated for phosphorus, as the results submitted were insufficient to perform a meaningful statistical evaluation.

Table B: Summary of Statistical Outliers

Test	Sample PTA 1	Sample PTA 2
Protein	-	-
Fat	-	-
Moisture	-	1, 12
Ash	4, 10, 12	4, 10
Phosphorus		
Salt	1	1, 6
Carbohydrate	-	1
Energy	-	1

Note:

Robust z-scores were not calculated for phosphorus, as the results submitted were insufficient to perform a meaningful statistical evaluation.

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

Of the 14 laboratories that participated in the program, 13 laboratories (93%) submitted results. Eight of these 13 laboratories (62%) provided results for all of the tests.

The return rate for all tests is as follows:

- Protein 13 out of 13 100%
- Fat 13 out of 13 100%
- Moisture 13 out of 13 100%
- Ash 13 out of 13 100%
- Phosphorus 8 out of 13 62%
- Salt 11 out of 13 85%
- Carbohydrate 13 out of 13 100%
- Energy 13 out of 13 100%

6.2 Performance summary

One or more statistical outliers were reported by 5 of the 13 laboratories (38%) that returned results in this round of the Food program. The last meat paste round of the Food program was round 19, conducted in 2004 (see Report No 439). For comparison, 38% of the participants in round 19 of the Food program reported statistical outliers.

A total of 178 results were analysed in this round of the program. Of these results, 12 (7%) were outlier results. In round 19 of the Food program 9% of the total results reported were outlier results (see Report No 439).

6.3 Protein

The majority of laboratories used methodology based on Kjeldahl digestion. One laboratory did not specify the method used.

The robust CV values for protein for this round are 0.9% for sample PTA 1 and 2.2% for sample PTA 2. For comparison, the robust CV values for the last meat paste round of this program (round 19) were 1.7% and 2.0% (see Report No 439). As the robust CV for sample PTA 1 is considerably lower than the robust CV for sample PTA 2, and is not consistent with the CVs of previous meat paste rounds of the Food program, a target CV was used to calculate the robust z-scores for sample PTA 1. The target value of the CV used was the same as the robust CV obtained for sample PTA 2. This was done to avoid unfairly identifying laboratories as having reported outlier results for sample PTA 1.

There were no outliers reported for either sample for protein. For more information on the calculation of the z-scores for sample PTA 1, see Appendix A (A1.1).

6.4 Fat

The majority of laboratories used a soxhlet extraction procedure for fat. One laboratory did not specify the method used.

The robust CV values for fat for this round are 6.1% for sample PTA 1 and 3.7% for sample PTA 2. For comparison, the robust CV values for round 19 of this program were 3.0% and 3.8% (see Report No 439).

There were no outliers reported for either sample for fat.

6.5 Moisture

The majority of laboratories used an air oven for moisture. One laboratory did not specify the method used. 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 CV values for moisture for this round are 0.4% for sample PTA 1 and 0.5% for sample PTA 2. For comparison, the robust CV values for round 19 of this program were 0.6% and 0.8% (see Report No 439).

There were no outliers reported for sample PTA 1. Two laboratories (1 and 12) reported outliers for sample PTA 2.

6.6 Ash

The majority of laboratories used a muffle furnace for ash. One laboratory did not specify the method used. Temperatures used for ashing ranged between 510 °C and 600 °C. Times of ashing varied between 2 hours and 18 hours. Temperatures and times are shown in Appendix A (A4.1).

The robust CV values for ash for this round are 2.1% for sample PTA 1 and 2.9% for sample PTA 2. For comparison, the robust CV values for round 19 of this program were 1.6% and 1.7% (see Report No 439).

Two laboratories (4 and 10) reported outlier results for both samples. Laboratory 12 reported an outlier for sample PTA 1.

6.7 Phosphorus

The majority of laboratories used the ICP method.

There were only 8 laboratories that submitted results for phosphorus. The results submitted were insufficient to perform a meaningful statistical evaluation. Therefore, robust z-scores and summary statistics have not been calculated for the phosphorus results.

Five of the laboratories (3, 8,13,14 and 15) reported results that are fairly comparable. Laboratory 9 probably made a calculation or transcription error (in both the reported value and the MU). Laboratory 1 (sample PTA 1) and laboratory 2 (both samples) have, most likely, reported outlier results, if the results of laboratories 3, 8,13,14 and 15 are to be considered accurate. It is, therefore, recommended that laboratories 1, 2 and 9 revisit their data and, in particular, look at their recoveries of any CRMs or spikes they have used in their analysis batch.

6.8 Salt

Volhard titration was the most commonly used method amongst the laboratories that returned results for salt. One laboratory did not specify the method used.

The robust CV values for salt for this round are 6.1% for sample PTA 1 and 6.5% for sample PTA 2. For comparison, the robust CV values for round 19 of this program were 7.7% and 7.3% (see Report No 439).

Laboratory 1 reported outlier results for both samples. Laboratory 6 reported an outlier for sample PTA 2.

6.9 Carbohydrate

The robust CV values for carbohydrate for this round are 3.6% for sample PTA 1 and 4.7% for sample PTA 2. For comparison, the robust CV values for round 19 of this program were 9.4% and 12.1% (see Report No 439).

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

6.10 Energy

The robust CV values for energy for this round are 1.8% for sample PTA 1 and 1.8% for sample PTA 2. For comparison, the robust CV values for round 19 of this program were 1.8% and 1.5% (see Report No 439).

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

6.11 Measurement Uncertainty

For this round of the program, laboratories were requested to report an estimate of their measurement uncertainty (MU) for each test result. The proportion of MU estimates returned for each individual test is as follows:

• Protein	12 out of 13	92%
• Fat	12 out of 13	92%
• Moisture	12 out of 13	92%
• Ash	12 out of 13	92%
• Phosphorus	7 out of 8	88%
• Salt	10 out of 11	91%
• Carbohydrate	8 out of 13	62%
• Energy	8 out of 13	62%

It is clear from this study, as well as other inter-laboratory studies, that the estimation of measurement uncertainty is far from an exact science.

The range of uncertainties provided by participating laboratories for each determination is summarised in the following table, together with the expanded MU calculated from the Horwitz relationship between analyte concentration and the reproducibility RSD.

Analyte	Concentration (g/100g)	MU range provided by participating labs	Expanded MU based on Horwitz equation for RSD (σ_H)* at analyte concentration
protein	16.8	0.2 - 1.6	0.9
fat	10.1	0.001 - 1.2	0.6
moisture	63.7	0.004 - 6.4	2.8
ash	2.5	0.004 - 0.25	0.17
phosphorus	0.23	0.0032 - 0.35	0.02
salt	1.1	0.012 - 1.063	0.1
carbohydrate	6.9	0.7 - 2.9	0.4
energy (kJ/100g)	777	26 - 137	By calculation

* $\sigma_H = 0.02 C^{0.85}$ (refer to NATA Technical Note 33 – June 2006).

In all measurements, other than for protein, the estimated uncertainty covered at least a 100 fold range from the smallest to the largest value and, for fat and moisture, a 1000 fold range. In one case the uncertainty, or error associated with the reported result, was of the same magnitude as the actual analytical value reported, which is more consistent with measurements at the sub-ppb level than at the % composition level.

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

Given that all laboratories reported using some variation of the ‘top down’ approach, involving in-house precision data, one might expect much closer correlation of the MU values reported by the laboratories.

It is recommended that laboratories ensure that greater attention be given to the manner in which they estimate MU and that they ensure a consistent and defensible approach to reporting MU.

Table C: Method of Measurement Uncertainty Estimation

Lab Code	Method
1	In-house precision data.
3	Top down.
4	Proficiency trial data.
6	In-house precision data.
8	Running standard deviation from control charts and sample homogeneity variant from long term duplicate data.
9	Protein, fat, moisture, ash, phosphorus: in-house precision data. Salt: via ash. Carbohydrate, energy: by addition.
12	In-house.
13	In-house precision data (excluding phosphorus). Phosphorus: 2 x RPD of duplicates.
15	In-house precision data.

The measurement uncertainty reported by participating laboratories can be bench-marked against the 'expected' uncertainty derived from the Horwitz equation, based on reported values of collaborative studies. Where estimated uncertainties vary considerably from the Horwitz values, laboratories are encouraged to review their MU estimates. Measurement uncertainty estimates based on in-house precision data are likely to be smaller than those determined from the Horwitz equation, which is based on inter-laboratory precision data.

An attempt to estimate MU from the study precision data was not practical. In several of the determinations, the normalised IQR values were significantly different from that predicted by the Horwitz equation and fell outside the accepted HORRAT range of 0.5 to 2.0, as summarised in the table on the next page.

Analyte	σ_H	IQR – PTA 1 ($\sim S_R$)	Horrat value S_R/σ_H	Acceptable result based on HORRAT range of 0.5 to 2.0?	IQR – PTA 2 ($\sim S_R$)	Horrat value S_R/σ_H	Acceptable result based on HORRAT range of 0.5 to 2.0?
protein	0.44	0.15	0.34	no	0.37	0.84	yes
fat	0.29	0.62	2.1	marginal	0.37	1.3	yes
moisture	1.4	0.24	0.17	no	0.3	0.21	no
ash	0.087	0.05	0.57	yes	0.074	0.85	yes
phosphorus	0.011	0.044	4.0	no	0.041	3.7	no
salt	0.043	0.072	1.7	yes	0.074	1.7	yes
carbohydrate	0.2	0.252	1.3	yes	0.326	1.6	yes

Note:

- S_R values are suspect when the HORRAT value exceeds by more than a factor of 2 what is expected from the Horwitz equation.
- When S_R is much smaller than σ_H , it is likely that the collaborative method (or study) is not performed correctly and that it gives values of S_R that are too optimistic.
- When S_R is larger than σ_H by a factor of 2, the method is performing worse than could be hoped.

6.12 Overall comments

Overall, the performance of the participating laboratories, based on the reported results, seems reasonable. Given that the samples were true duplicates, most laboratories obtained good agreement between each sample for each analyte. Laboratories should re-examine their data where result correlation between samples was poor.

In terms of the different methods used by laboratories, there did not seem to be any significant bias in any of the methods.

In reporting data, laboratories should take care in reporting only the number of significant figures in which they can demonstrate confidence, particularly as laboratories are now developing an increasing understanding of errors associated with measurements.

7. REFERENCES

[1] *Guide to Proficiency Testing Australia* (2006).

This document is located on the PTA website at www.pta.asn.au, under "Documents".

[2] NATA Technical Note 33 – June 2006.

[3] Massart et al., LC-GC Europe - *Benchmarking for Analytical Methods: The Horwitz Curve* (www.lcgceurope.com)

APPENDIX A

Summary of Results

Section A1

Protein

A1.1

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

Lab Code	PTA 1		PTA 2		Z-Scores		Method Code
	Average	MU (±)	Average	MU (±)	PTA 1	PTA 2	
1	17.4	1	17.1	1	1.60	1.35	2
2	16.70	0.12	16.65	0.12	-0.27	0.13	1
3	17.2	0.516	16.6	0.511	1.07	0.00	2
4	16.2	0.4	16.3	0.4	-1.60	-0.81	1
6	16.735	0.167	16.585	0.166	-0.17	-0.04	1
8	16.8	1.6	16.1	1.6	0.00	-1.35	2
9	16.8	0.9	16.9	0.9	0.00	0.81	1
10	15.8	-	15.7	-	-2.67	-2.43	1
11	16.7	0.5	16.5	0.5	-0.27	-0.27	1
12	16.9	0.9	16.4	0.9	0.27	-0.54	-
13	16.7	0.63	16.6	0.63	-0.27	0.00	1
14	17.3	0.2	17.3	0.2	1.33	1.89	1
15	16.8	0.2	16.9	0.2	0.00	0.81	2

Statistic	PTA 1	PTA 2
No of Results	13	13
Median	16.80	16.60
Norm IQR	0.15	0.37
Robust CV	0.88%	2.23%
Target IQR	0.38	0.37
Target CV	2.23%	2.23%
Minimum	15.8	15.7
Maximum	17.4	17.3
Range	1.6	1.6

Method Codes

Number

1 = Kjeldahl digestion	8
2 = Combustion (Dumas, Leco)	4
3 = Other	0
Unknown	1

Notes:

1. A target CV was used to calculate the robust z-scores for sample PTA 1. The target value of the CV used was the same as the robust CV obtained for sample PTA 2. Robust z-scores, using a target CV, are calculated by the formula:

$$Z = (\text{result} - \text{median}) \div (\text{target IQR}),$$

where

$$\text{target IQR} = \text{target CV} \times \text{median}$$

and the target CV is expressed as a decimal.

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

Section A2

Fat

A2.1

Fat (g/100g) – Samples PTA 1 & PTA 2

Lab Code	PTA 1		PTA 2		Z-Scores		Method Code
	Average	MU (\pm)	Average	MU (\pm)	PTA 1	PTA 2	
1	9.67	0.5	10.8	0.5	-0.70	1.89	1
2	10.58	0.06	10.53	0.13	0.78	1.16	3
3	9.90	0.740	10.0	0.748	-0.33	-0.27	1
4	10.2	1.2	10.1	1.2	0.16	0.00	1
6	9.650	0.001	9.690	0.001	-0.73	-1.11	3
8	9.53	0.95	9.63	0.95	-0.93	-1.27	1
9	10.1	0.4	10.2	0.4	0.00	0.27	1
10	10.5	-	10.6	-	0.65	1.35	1
11	10.5	0.9	10.4	0.9	0.65	0.81	3
12	10.5	0.6	9.9	0.5	0.65	-0.54	-
13	10.3	1.03	10.3	1.03	0.33	0.54	1*
14	9.52	0.20	9.55	0.20	-0.94	-1.48	1
15	10.0	0.3	10.0	0.3	-0.16	-0.27	1

Statistic	PTA 1	PTA 2
No of Results	13	13
Median	10.10	10.10
Norm IQR	0.62	0.37
Robust CV	6.09%	3.67%
Minimum	9.52	9.55
Maximum	10.58	10.80
Range	1.06	1.25

Method Codes

1 = Soxhlet extraction
 2 = Foss-Lett
 3 = Other
 Unknown

Number

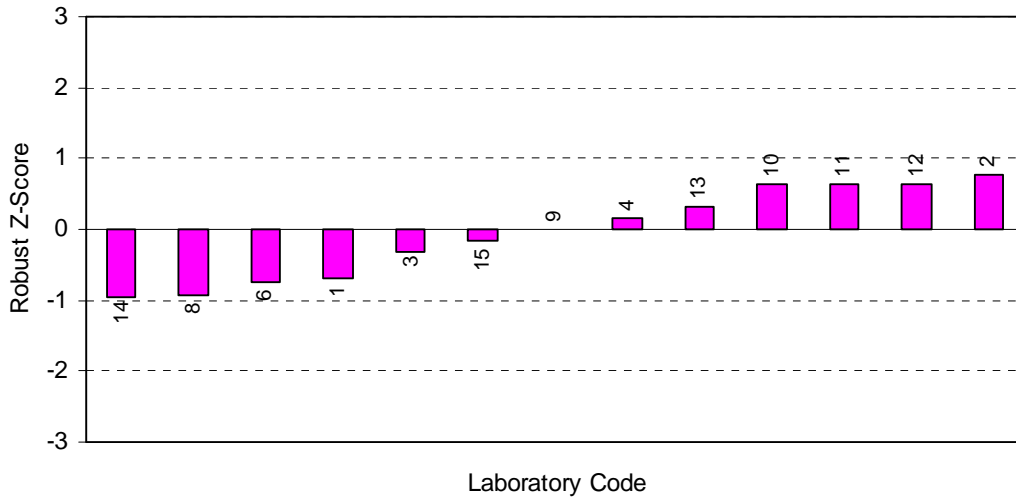
9
 0
 3
 1

Notes:

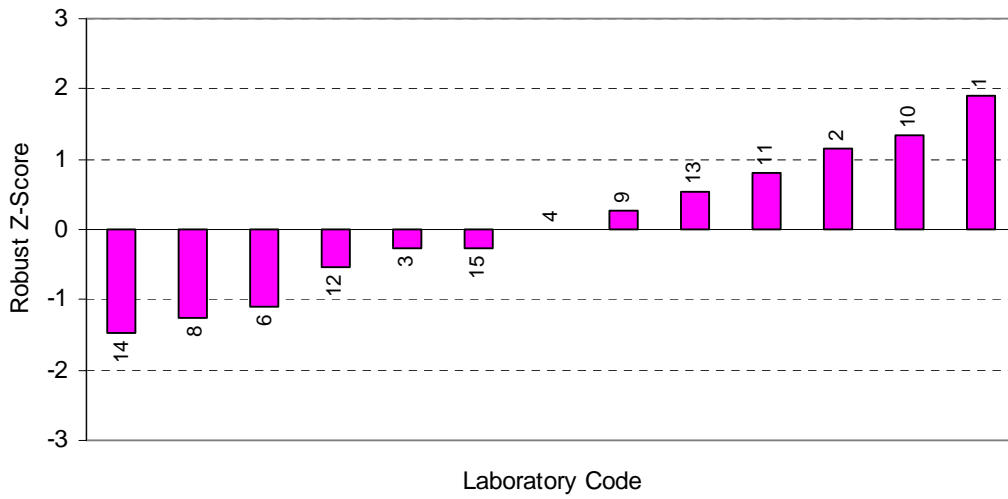
1. Laboratory 13 used an acid hydrolysis pre-treatment for fat.
2. The Youden diagram on the following page is provided for information only.

A2.2

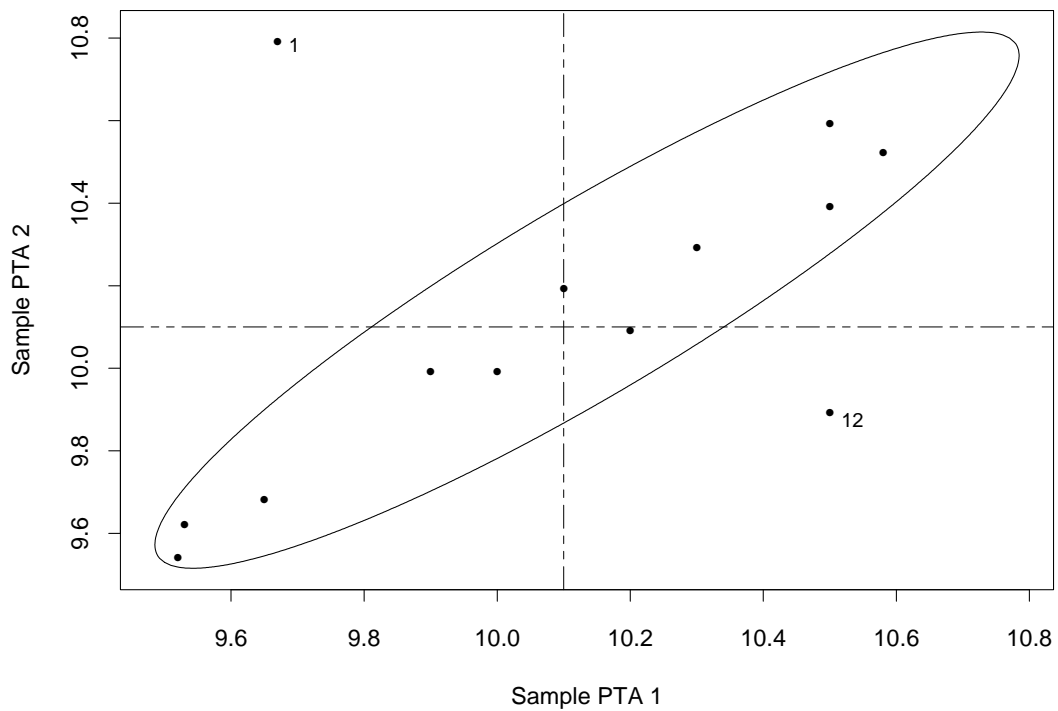
Fat (g/100g) - Sample 1



Fat (g/100g) - Sample 2



Fat (g/100g)



Section A3

Moisture

A3.1

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

Lab Code	PTA 1		PTA 2		Z-Scores		Temp. (°C)	Time (hrs)	Method Code
	Average	MU (±)	Average	MU (±)	PTA 1	PTA 2			
1	63.6	3	61.4	3	-0.41	-8.09 §	105	16	1
2	63.57	0.04	63.57	0.03	-0.53	-0.78	100	16	1
3	63.4	0.461	63.7	0.463	-1.23	-0.34	90	12	1
4	64.2	0.6	64.0	0.6	2.04	0.67	102	o/n	1
6	64.000	0.004	64.000	0.004	1.23	0.67	125	3	1
8	63.9	6.4	64.0	6.4	0.82	0.67	104	16	1
9	63.6	1.4	63.5	1.4	-0.41	-1.01	102	16	1
10	63.5	-	63.8	-	-0.82	0.00	104	16	1
11	63.7	0.5	64.1	0.5	0.00	1.01	101	16	1
12	63.2	1.3	64.9	1.4	-2.04	3.71 §	105	2	-
13	63.7	0.64	63.8	0.64	0.00	0.00	100	16	1
14	63.9	0.2	64.0	0.2	0.82	0.67	102 ± 2	16	1
15	63.8	1.3	63.6	1.3	0.41	-0.67	105	8	1

Statistic	PTA 1	PTA 2
No of Results	13	13
Median	63.70	63.80
Norm IQR	0.24	0.30
Robust CV	0.38%	0.46%
Minimum	63.2	61.4
Maximum	64.2	64.9
Range	1.0	3.5

Method Codes

1 = Air Oven
 2 = Vacuum Oven
 3 = Rapid Microwave
 4 = Other
 Unknown

Number

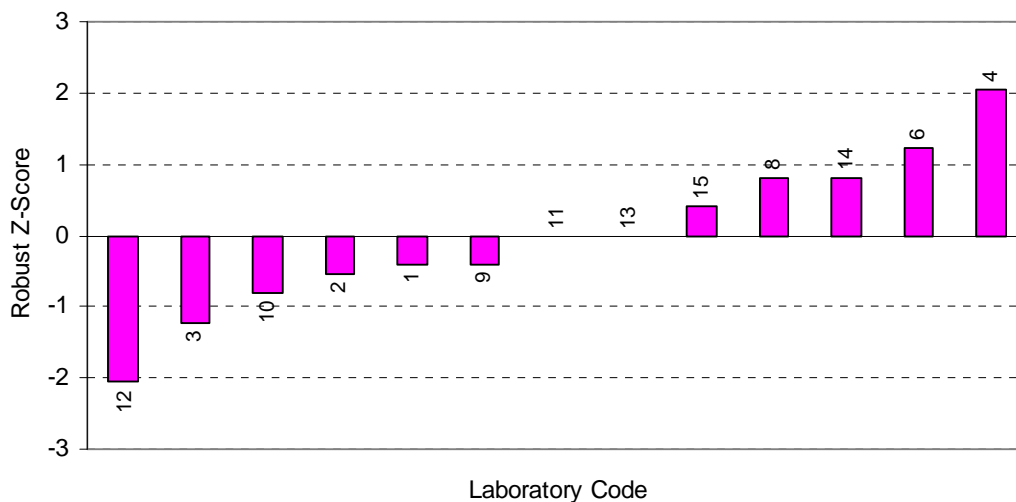
12
 0
 0
 0
 1

Notes:

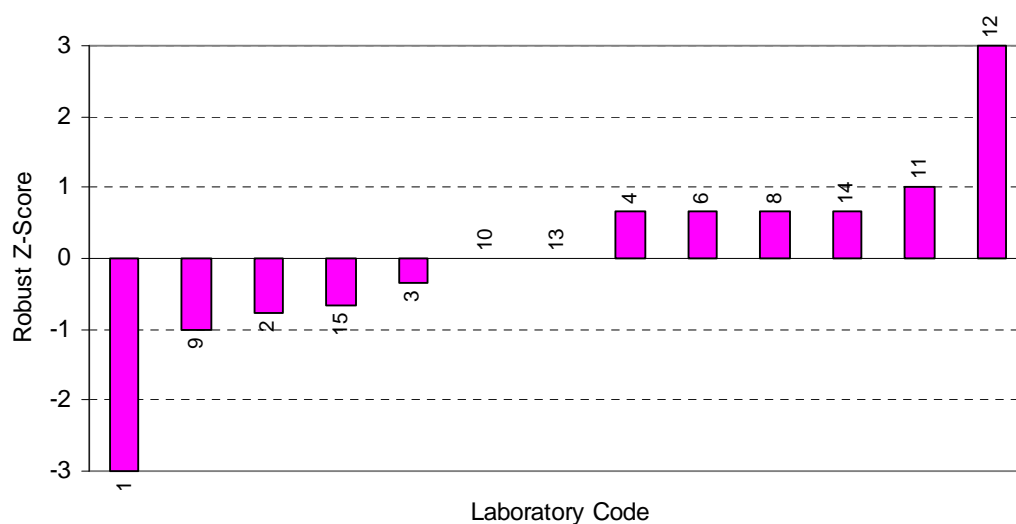
- § denotes an outlier (i.e. |z-score| > 3).
- The Youden diagram on the following page is provided for information only.

A3.2

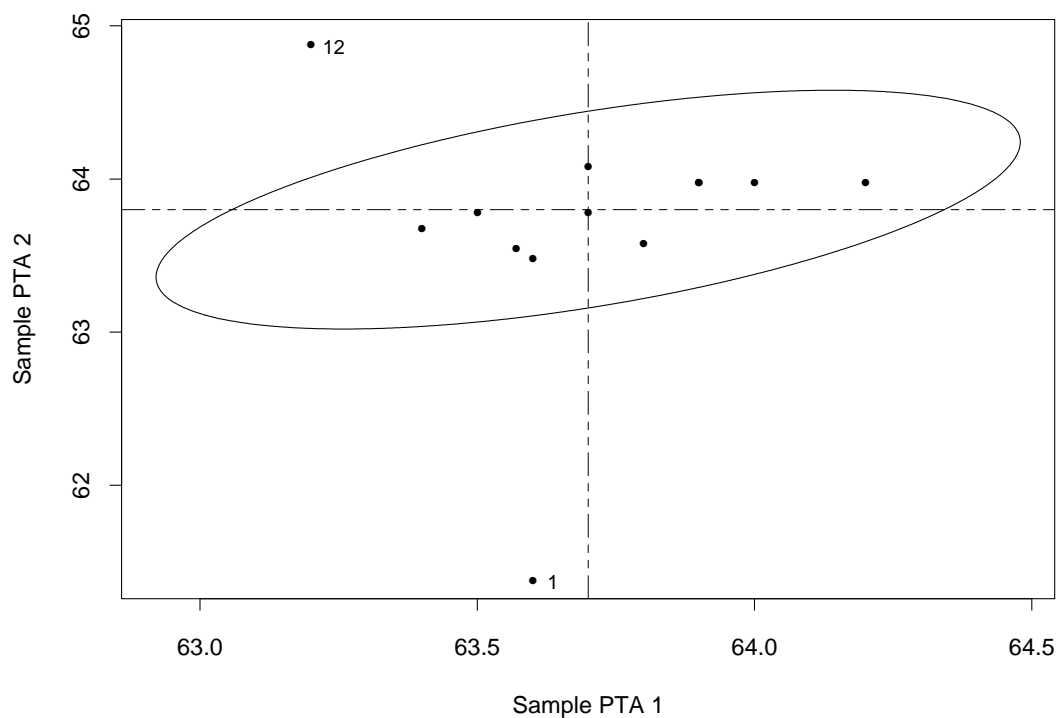
Moisture (g/100g) - Sample 1



Moisture (g/100g) - Sample 2



Moisture (g/100g)



Section A4

Ash

A4.1

Ash (g/100g) – Samples PTA 1 & PTA 2

Lab Code	PTA 1		PTA 2		Z-Scores		Temp. (°C)	Time (hrs)	Method Code
	Average	MU (±)	Average	MU (±)	PTA 1	PTA 2			
1	2.41	0.2	2.43	0.2	-2.31	-1.48	600	2	1
2	2.64	0.01	2.64	0.01	2.12	1.35	550	6	1
3	2.50	0.092	2.50	0.092	-0.58	-0.54	600	4	1
4	2.25	0.12	2.24	0.12	-5.40 §	-4.05 §	580	16	1
6	2.545	0.004	2.560	0.004	0.29	0.27	550	18	1
8	2.52	0.25	2.53	0.25	-0.19	-0.13	550	16	1
9	2.52	0.16	2.46	0.16	-0.19	-1.08	525	16	1
10	2.7	-	2.8	-	3.28 §	3.51 §	550	16	1
11	2.53	0.07	2.55	0.07	0.00	0.13	525	16	1
12	2.7	0.2	2.6	0.2	3.28 §	0.81	-	-	-
13	2.57	0.11	2.54	0.11	0.77	0.00	600	2	1
14	2.46	0.20	2.44	0.20	-1.35	-1.35	525	16	1
15	2.55	0.13	2.55	0.13	0.39	0.13	510	7	1

Statistic	PTA 1	PTA 2
No of Results	13	13
Median	2.530	2.540
Norm IQR	0.052	0.074
Robust CV	2.05%	2.92%
Minimum	2.25	2.24
Maximum	2.70	2.80
Range	0.45	0.56

Method Codes

1 = Muffle furnace
 2 = Other
 Unknown

Number

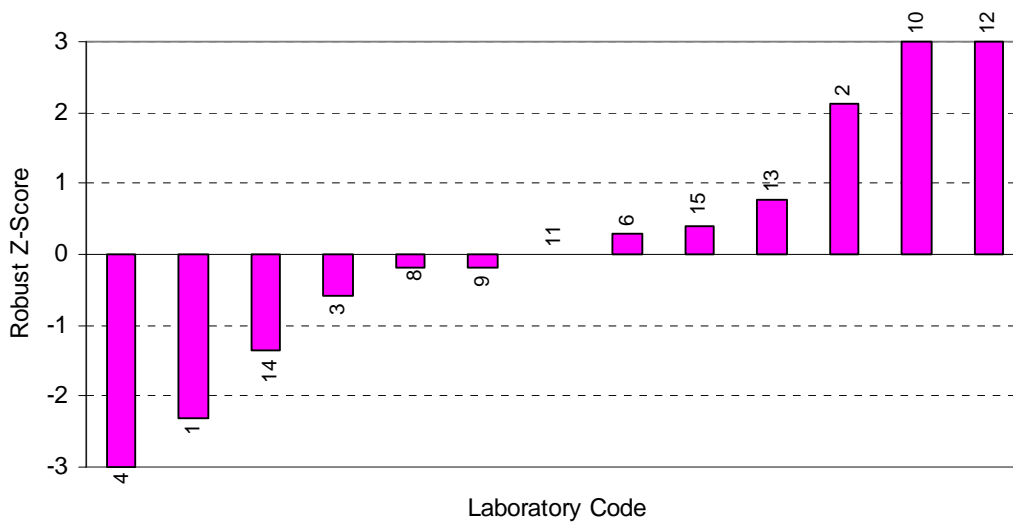
12
 0
 1

Notes:

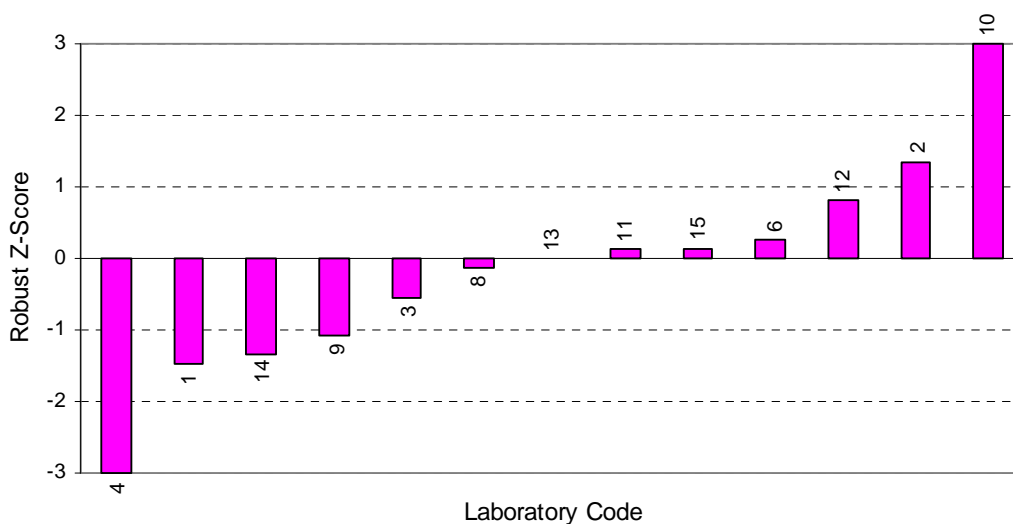
- § denotes an outlier (i.e. |z-score| > 3).
- The Youden diagram on the following page is provided for information only.

A4.2

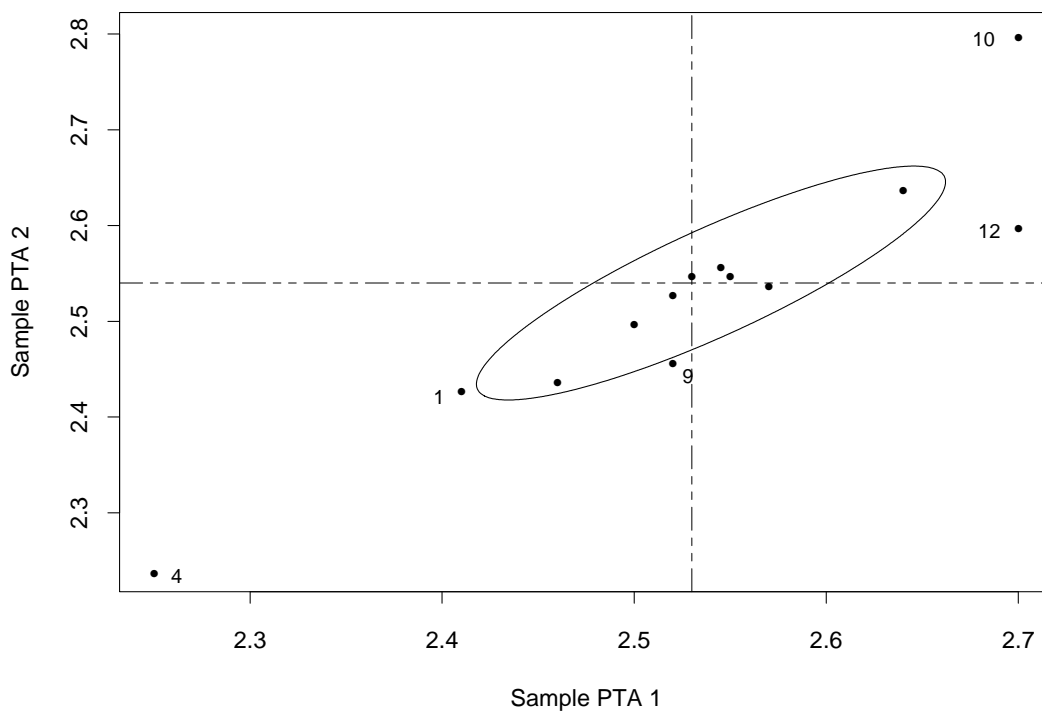
Ash (g/100g) - Sample 1



Ash (g/100g) - Sample 2



Ash (g/100g)



Section A5

Phosphorus

A5.1

Phosphorus (g/100g) – Samples PTA 1 & PTA 2

Lab Code	PTA 1		PTA 2		Method Code
	Average	MU (\pm)	Average	MU (\pm)	
1	0.162	0.02	0.221	0.02	2
2	0.33	-	0.33	-	1
3	0.266	0.0153	0.264	0.0153	2
8	0.238	0.025	0.237	0.025	2
9	1.73	0.35	1.88	0.35	2
13	0.223	0.0032	0.226	0.0072	1
14	0.221	0.051	0.221	0.051	2
15	0.231	0.005	0.233	0.005	2

Method Codes

1 = Spectrophotometry
 2 = ICP
 3 = Other

Number

2
 6
 0

Notes:

1. Robust z-scores and summary statistics were not calculated for phosphorus, as the results submitted were insufficient to perform a meaningful statistical evaluation.

Section A6

Salt

A6.1

Salt (g/100g) – Samples PTA 1 & PTA 2

Lab Code	PTA 1		PTA 2		Z-Scores		Method Code
	Average	MU (\pm)	Average	MU (\pm)	PTA 1	PTA 2	
1	1.86	0.2	1.75	0.2	9.41 §	8.23 §	3
2	1.02	-	1.04	-	-2.21	-1.35	1
3	1.20	1.063	1.10	0.975	0.28	-0.54	2
6	1.215	0.012	1.755	0.018	0.48	8.30 §	3
8	1.18	0.1	1.18	0.1	0.00	0.54	1
9	1.20	0.08	1.18	0.08	0.28	0.54	3
11	1.03	0.10	1.05	0.10	-2.08	-1.21	1
12	1.1	0.2	1.1	0.2	-1.11	-0.54	-
13	1.17	0.049	1.14	0.048	-0.14	0.00	1
14	1.12	0.1	1.12	0.1	-0.83	-0.27	1
15	1.22	0.02	1.22	0.02	0.55	1.08	1

Statistic	PTA 1	PTA 2
No of Results	11	11
Median	1.180	1.140
Norm IQR	0.072	0.074
Robust CV	6.13%	6.50%
Minimum	1.02	1.04
Maximum	1.86	1.76
Range	0.84	0.72

Method Codes

1 = Volhard titration
 2 = ISE
 3 = Other
 Unknown

Number

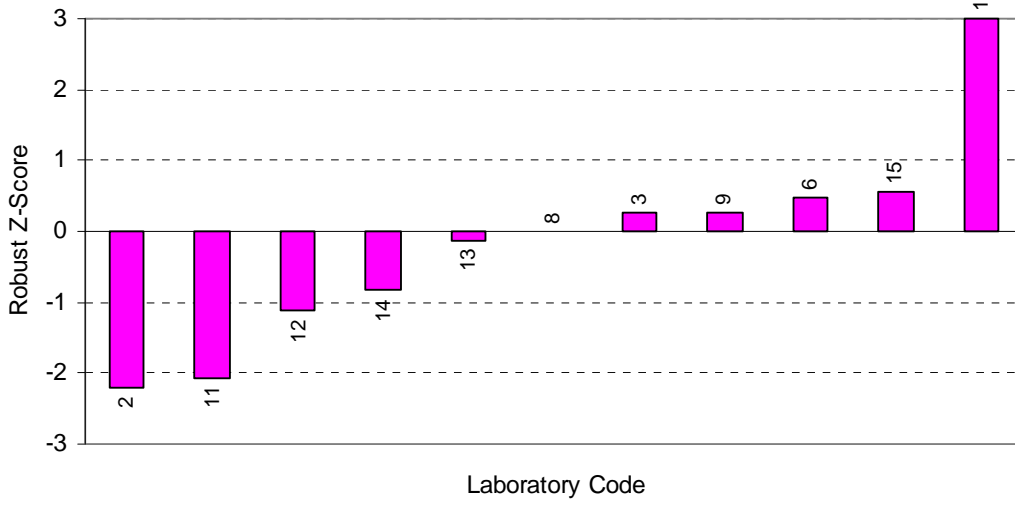
6
 1
 3
 1

Notes:

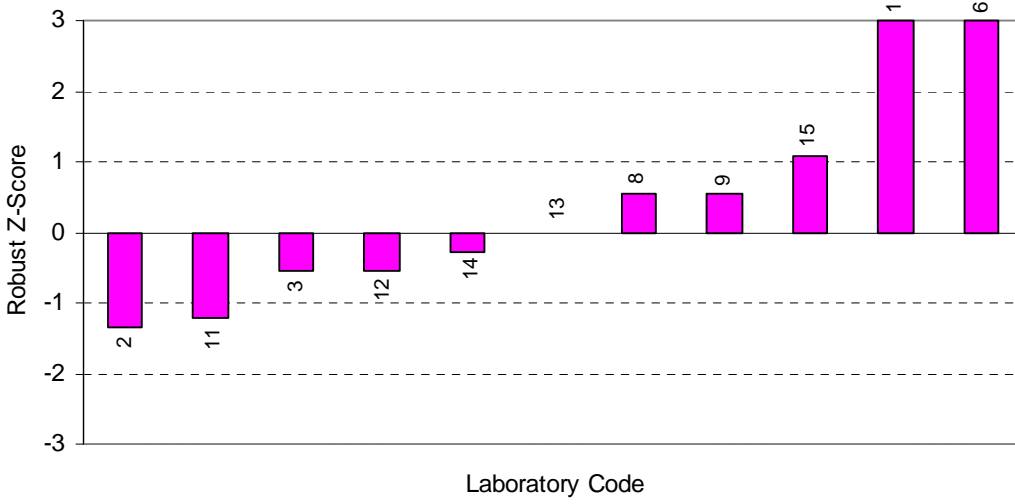
- § denotes an outlier (i.e. $|z\text{-score}| > 3$).
- The Youden diagram on the following page is provided for information only.

A6.2

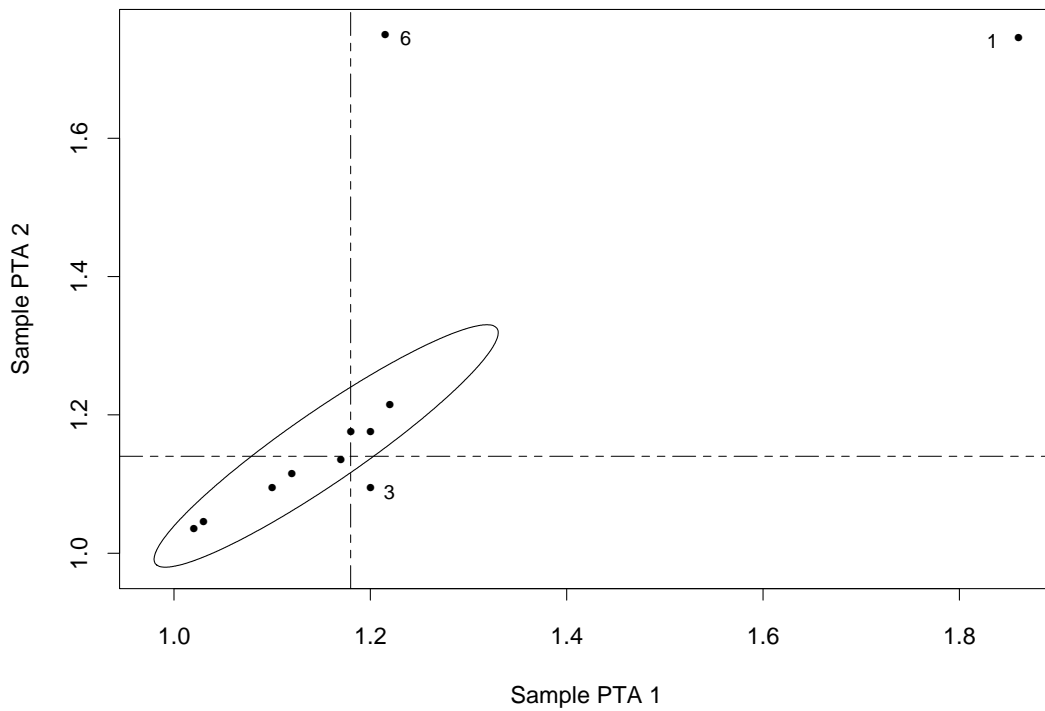
Salt (g/100g) - Sample 1



Salt (g/100g) - Sample 2



Salt (g/100g)



Section A7
Carbohydrate

A7.1

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

Lab Code	PTA 1		PTA 2		Z-Scores	
	Average	MU (\pm)	Average	MU (\pm)	PTA 1	PTA 2
1	6.92	-	8.32	-	0.00	4.26 §
2	6.51	-	6.61	-	-1.63	-0.98
3	7.00	1.18	7.20	1.20	0.32	0.83
4	7.15	1.0	7.36	1.0	0.91	1.32
6	7.070	-	7.165	-	0.60	0.72
8	7.25	0.7	7.74	0.7	1.31	2.48
9	6.97	2.9	6.93	2.9	0.20	0.00
10	7.5	-	7.1	-	2.30	0.52
11	6.61	0.62	6.50	0.62	-1.23	-1.32
12	6.7	3.0	6.2	3.0	-0.87	-2.24
13	6.73	-	6.76	-	-0.75	-0.52
14	6.8	0.9	6.8	0.9	-0.48	-0.40
15	6.88	0.80	6.89	0.80	-0.16	-0.12

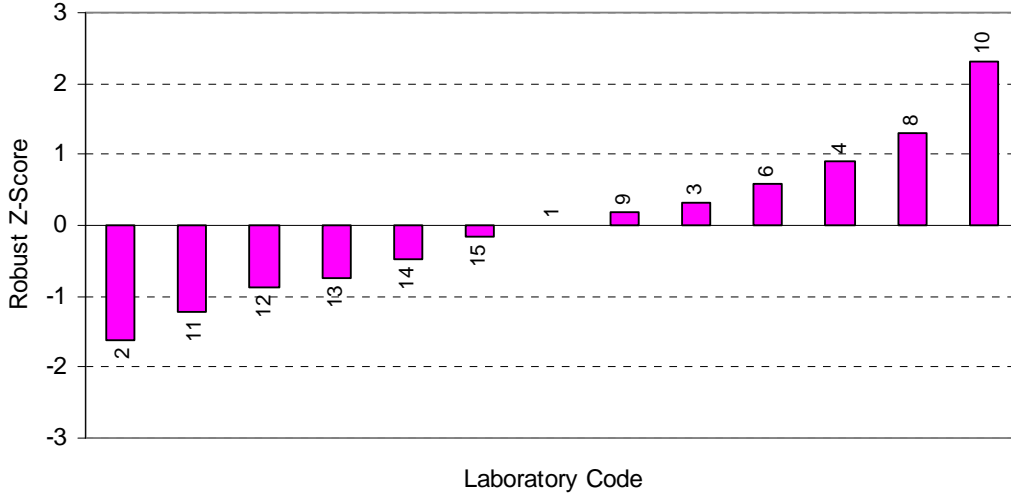
Statistic	PTA 1	PTA 2
No of Results	13	13
Median	6.920	6.930
Norm IQR	0.252	0.326
Robust CV	3.64%	4.71%
Minimum	6.51	6.20
Maximum	7.50	8.32
Range	0.99	2.12

Notes:

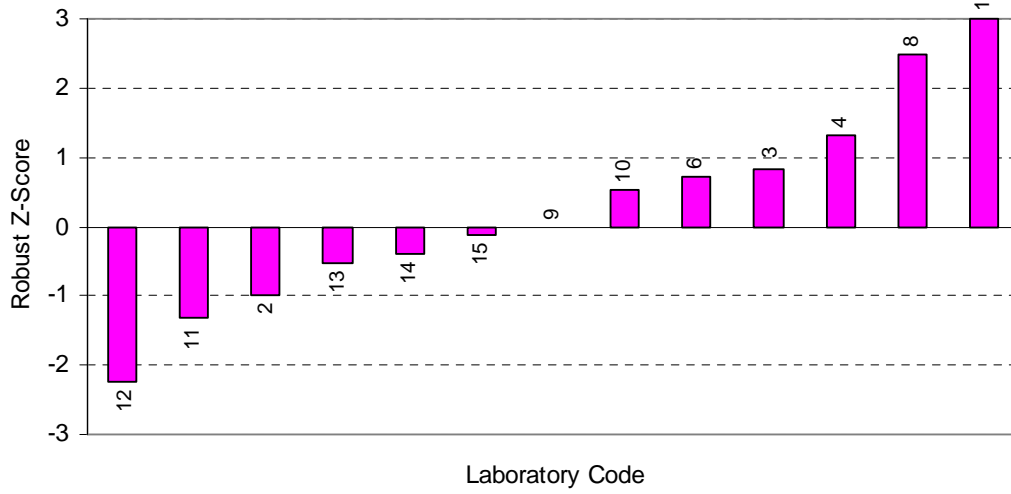
1. § denotes an outlier (i.e. |z-score| > 3).
2. The Youden diagram on the following page is provided for information only.

A7.2

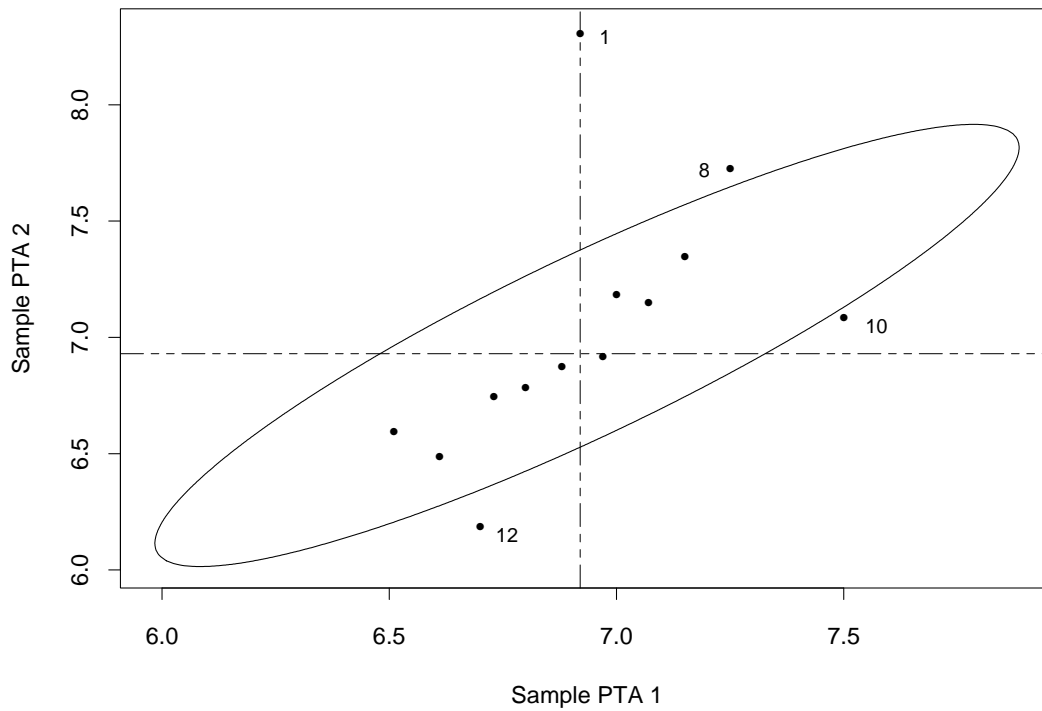
Carbohydrate (g/100g) - Sample 1



Carbohydrate (g/100g) - Sample 2



Carbohydrate (g/100g)



Section A8

Energy

A8.1

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

Lab Code	PTA 1		PTA 2		Z-Scores	
	Average	MU (±)	Average	MU (±)	PTA 1	PTA 2
1	765	-	823	-	-0.85	3.34 §
2	789.85	-	788.80	-	0.91	0.91
3	777	14.4	776	14.3	0.00	0.00
4	774	30	776	30	-0.21	0.00
6	754.665	-	755.115	-	-1.59	-1.48
8	762	76	761	76	-1.06	-1.06
9	778	79	783	79	0.07	0.50
10	785	-	780	-	0.57	0.28
11	784	137	776	136	0.50	0.00
12	790	89	751	85	0.92	-1.77
13	779	-	778	-	0.14	0.14
14	760	26	760	26	-1.21	-1.14
15	774	124	775	124	-0.21	-0.07

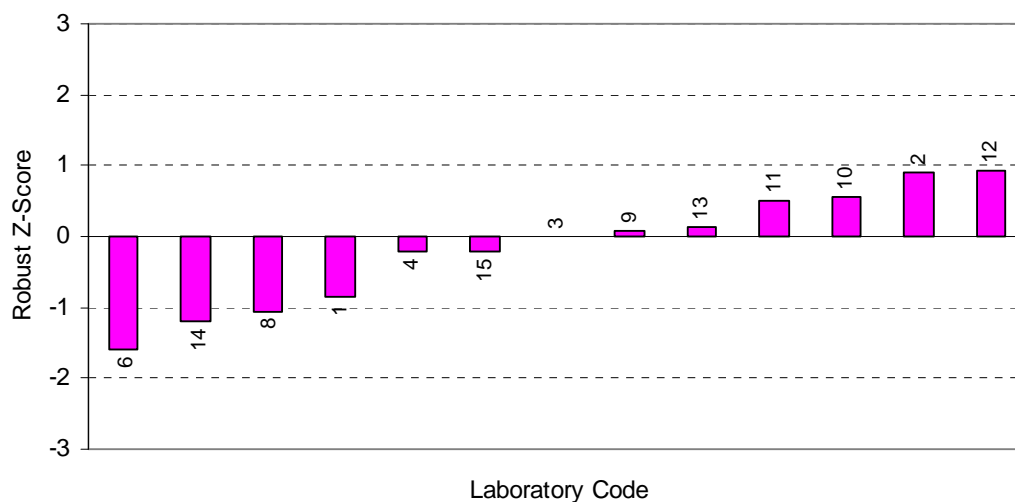
Statistic	PTA 1	PTA 2
No of Results	13	13
Median	777.0	776.0
Norm IQR	14.1	14.1
Robust CV	1.81%	1.82%
Minimum	755	751
Maximum	790	823
Range	35	72

Notes:

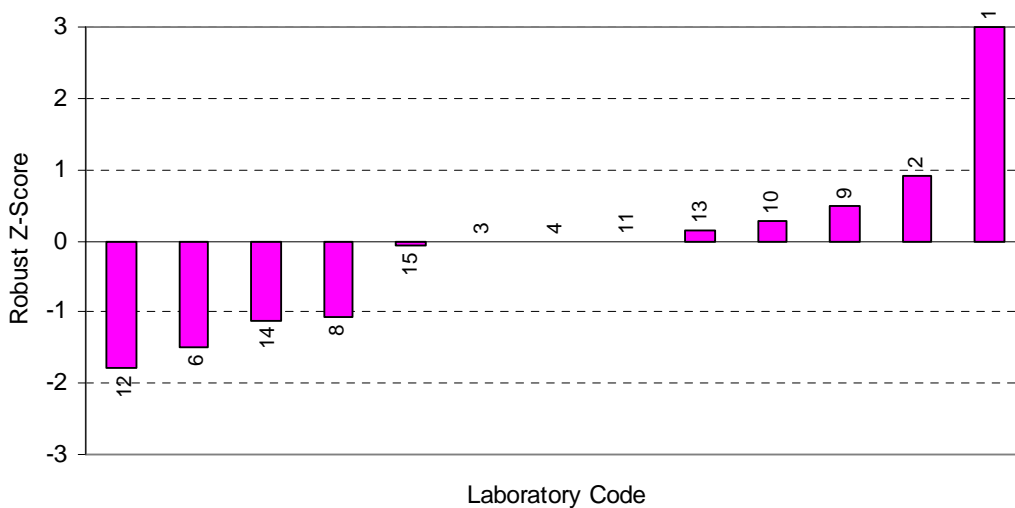
1. § denotes an outlier (i.e. |z-score| > 3).
2. The Youden diagram on the following page is provided for information only.

A8.2

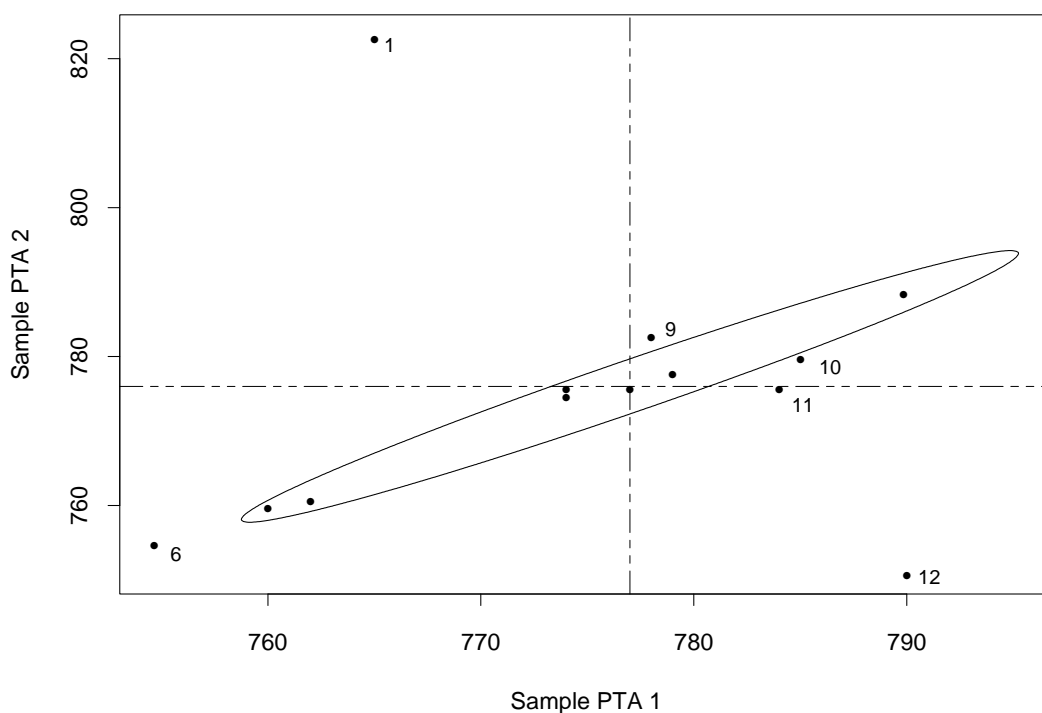
Energy (kJ/100g) - Sample 1



Energy (kJ/100g) - Sample 2



Energy (kJ/100g)



APPENDIX B

Homogeneity Testing

B1.1

HOMOGENEITY TESTING

Prior to distribution, ten samples of meat paste were selected at random and tested for homogeneity byASUREQuality New Zealand Pty Limited. Each sample was tested in duplicate for protein, fat and phosphorus. The results of this testing appear in the following table.

Homogeneity Testing Results

Meat Paste

Sample	Protein (%m/m)		Fat (g/100g)		Phosphorus (%m/m)	
	Result 1	Result 2	Result 1	Result 2	Result 1	Result 2
1	16.7	16.9	10.07	9.71	0.220	0.222
2	16.9	16.9	10.04	10.09	0.220	0.222
3	16.7	16.7	9.93	10.00	0.221	0.228
4	17.3	17.0	10.08	10.04	0.227	0.227
5	16.9	17.0	10.00	9.98	0.228	0.226
6	16.8	17.0	10.0	10.0	0.225	0.229
7	17.76	17.77	10.0	9.9	0.226	0.222
8	17.30	17.35	10.0	10.0	0.225	0.228
9	17.03	17.09	10.0	10.1	0.231	0.229
10	16.86	16.91	10.0	10.0	0.232	0.229

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 27, DECEMBER 2007
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 samples of approximately 110 g – 120 g of meat paste labelled PTA 1 and PTA 2.
2. Testing may commence as soon as samples are received. The samples may be stored at ambient temperature, provided that the storage areas are air-conditioned. Where air-conditioning is turned off at night, or over the weekend, it is recommended that the samples are refrigerated.
3. The following tests are to be performed on each sample in **duplicate** and the **average result reported**:
 - Protein
 - Fat
 - Moisture
 - Ash
 - Phosphorus
 - Salt (as Sodium Chloride)
 - Carbohydrate[†]
 - Energy^{††}

Notes: [†] determined by difference.
 ^{††} 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 $N \times 6.25$.

4. Participants are requested to perform all tests listed above for which 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 accreditation is not held, however, please note this on the Results Sheet.
7. Results are to be reported to 3 significant figures.
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 7 December 2007 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
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PROFICIENCY TESTING AUSTRALIA
FOOD PROFICIENCY TESTING PROGRAM
ROUND 27, DECEMBER 2007
INSTRUCTIONS TO PARTICIPANTS



METHOD CODES

Analysis	Method	Code
Protein (g/100g)	Kjeldahl digestion	1
	Combustion (Dumas, Leco)	2
	Other (please specify)	3
Fat (g/100g)	Soxhlet extraction	1
	Foss-Lett	2
	Other (please specify)	3
Moisture (g/100g)	Air Oven	1
	Vacuum Oven	2
	Rapid Microwave	3
	Other (please specify)	4
Ash (g/100g)	Muffle furnace	1
	Other (please specify)	2
Phosphorus (g/100g)	Spectrophotometry	1
	ICP	2
	Other (please specify)	3
Salt (g/100g)	Volhard titration	1
	ISE	2
	Other (please specify)	3

NOTE:

Salt expressed as sodium chloride.

PROFICIENCY TESTING AUSTRALIA
FOOD PROFICIENCY TESTING PROGRAM
ROUND 27, DECEMBER 2007



RESULTS SHEET

Laboratory Code:

Test	<u>SAMPLE 1</u>		<u>SAMPLE 2</u>		Date Tested	Method Code
	Average Result	Measurement Uncertainty (\pm)	Average Result	Measurement Uncertainty (\pm)		
Protein (Nx6.25) (g/100g)						
Fat (g/100g)						
Moisture (g/100g)						
Ash (g/100g)						
Phosphorus (g/100g)						
Salt (g/100g)						
Carbohydrate (g/100g)						
Energy (kJ/100g)						

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

Please specify the temperature/time of ashing: _____ $^{\circ}$ 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 -----