REPORT NO. 1155

Food Proficiency Testing Program

Round 43 – Chicken Liver Pâté

September 2019

ACKNOWLEDGMENTS

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Mrs S Mott and Dr R Hutchinson, Global Proficiency Ltd (New Zealand). Thanks also to Ms S Giannoulidis, Global Proficiency Pty Ltd (Australia), who arranged for the supply of the samples.

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CONTENTS

1. FOREWORD

This report summarises the results of a proficiency testing program involving the analysis of chicken liver pâté samples. It constitutes the forty-third round of an ongoing series of programs involving chemical analysis of foodstuffs. This program is accredited to ISO/IEC 17043: 2010 "*Conformity assessment - General requirements for proficiency testing"* by International Accreditation New Zealand (IANZ).

Proficiency Testing Australia (PTA) conducted the testing program in July / August 2019. 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 Mrs S Mott, Global Proficiency Ltd (New Zealand). This report was authorised by Mrs K Cividin, PTA Quality Manager.

2. FEATURES OF THE PROGRAM

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

5	AUSTRALIA
1	INDONESIA
1	NEW ZEALAND
1	SULTANATE OF OMAN

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

(b) The results reported by participants are presented in Appendix A.

(c) Laboratories were provided with two samples of approximately 110 g of chicken liver pâté, labelled PTA 1 and PTA 2.

(d) Participants were requested to determine the levels of:

- Protein
- Fat
- Moisture
- Ash
- Salt
- 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, eight samples were analysed for homogeneity by Dairy Technical Services Ltd, Melbourne VIC. Additional samples were analysed for stability by Dairy Technical Services Ltd, Melbourne VIC. 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, Fat, Moisture, Ash, Salt, 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

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

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

Summary Statistics

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

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

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

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

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

Z-Scores

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

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

Ordered Z-Score Charts

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

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

Youden Diagrams

Youden two-sample diagrams are presented to highlight laboratory systematic differences. They are based on a plot of each laboratory's pair of results (*i.e.* sample PTA 2 versus sample PTA 1) and represented by a black spot.

These diagrams also feature an approximate 95% confidence ellipse for the bivariate analysis of the results, and dashed lines which mark the median value for each of the samples.

All points which lie outside the ellipse are labelled with the laboratory's code number. Note, however, that these points may not correspond with those identified as outliers. This is because the outlier criteria ($|z\text{-score}| \geq 3.0$) has a confidence level of approximately 99%, whereas the ellipse is an approximate 95% confidence region.

The points outside the ellipse on the Youden diagram roughly correspond to those with z-scores greater than 2.0 or less than -2.0. Laboratories which are outside the ellipse but have not been identified as outliers (*i.e.* have $2.0 < |z\text{-score}| < 3.0$) are encouraged to review their results.

As a guide to the interpretation of these diagrams:

(i) Laboratories with significant systematic error components (*i.e.* between-laboratory variation) will usually have results outside the ellipse in either the upper right hand quadrant (as formed by the median lines) or the lower left hand quadrant (*i.e.* unusually high or low results for both samples); and

(ii) Laboratories with significant random error components (*i.e.* within-laboratory variation) will have returned results that are substantially more variable than other participants, and these results will usually lie outside the ellipse in either the upper left or lower right hand quadrants (*i.e.* an unusually high result for one sample and low for the other).

For further details on the calculation and interpretation of robust z-scores and ordered z-score charts and the construction and interpretation of Youden diagrams, please see the *Guide to Proficiency Testing Australia (2019)*.

5. OUTLIER RESULTS

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

Table A: Summary Statistics for All Tests

Test	Summary Statistics	PTA 1	PTA 2
Protein (N x 6.25) (g/100g)	Number of Results	6	6
	Median	9.63	9.70
	Normalised IQR	0.33	0.11
	Uncertainty (Median)	0.17	0.05
Fat (g/100g)	Number of Results	10	10
	Median	31.35	31.25
	Normalised IQR	0.93	0.76
	Uncertainty (Median)	0.37	0.30
Moisture (g/100g)	Number of Results	10	10
	Median	53.35	53.33
	Normalised IQR	0.70	0.55
	Uncertainty (Median)	0.28	0.22
Ash (g/100g)	Number of Results	7	7
	Median	1.225	1.250
	Normalised IQR	0.045	0.018
	Uncertainty (Median)	0.021	0.009
Salt (g/100g)	Number of Results	5	5
	Median	n/a	n/a
	Normalised IQR	n/a	n/a
	Uncertainty (Median)	n/a	n/a
Carbohydrate (g/100g)	Number of Results	6	6
	Median	4.55	3.65
	Normalised IQR	1.53	1.13
	Uncertainty (Median)	0.78	0.58
Energy (kJ/100g)	Number of Results	6	6
	Median	1406.0	1405.0
	Normalised IQR	46.2	47.3
	Uncertainty (Median)	23.6	24.2

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 Salt.

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	4	4
Fat	6, 7	4, 7
Moisture	6	3, 6
Ash	-	-
Salt		
Carbohydrate	6	3
Energy	6	-

Notes:

1. A target CV was used to calculate the z-scores for sample PTA 2 for Ash.
2. Z-scores were not calculated for Salt.

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 eight laboratories that participated in the program submitted results. Five of these eight laboratories (63%) provided results for all of the tests.

The return rate for all tests is as follows:

• Protein	6 out of 8	75%
• Fat	8 out of 8	100%
• Moisture	8 out of 8	100%
• Ash	7 out of 8	88%
• Salt	5 out of 8	63%
• Carbohydrate	6 out of 8	75%
• Energy	6 out of 8	75%

6.2 Performance Summary

One or more statistical outliers were reported by four of the eight laboratories (50%) that returned results in this round of the Food program. The last round of the Food program where chicken liver pâté samples were used was Round 40 (see Report No. 995). For comparison, 22% of the participants in Round 40 of the Food program reported statistical outliers.

A total of 100 results were analysed in this proficiency round. Of these results, twelve (12%) were outlier results. In Round 40 of the Food program, 6% of the total results reported were outlier results (see Report No. 995).

6.3 Protein

Of the six laboratories that tested the samples for Protein, four laboratories tested using methodology based on Kjeldahl digestion. Two laboratories used a combustion method. All methods were pooled to analyse the Protein results.

Laboratory code 4 reported outlier results 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 and / or method within a 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	9.63 ± 0.17	9.70 ± 0.05

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

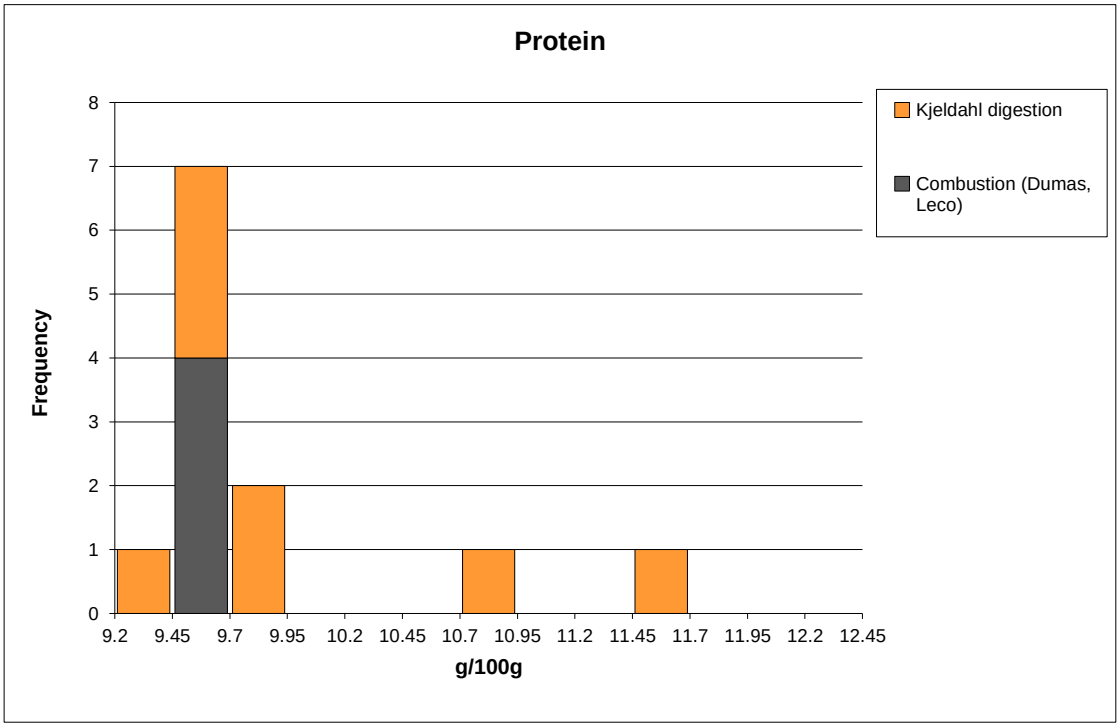


Figure TA-1. Spread of all results for Protein testing of duplicate Pâté samples PTA 1 & PTA 2.

The robust CVs of 3.4% and 1.1% for the two samples compare well with the values of 3.0% and 2.3% obtained in Round 40 of the Food program (see Report No. 995).

For this proficiency round, laboratories were requested to report their MU by repeatability (r) and / or reproducibility (R). One laboratory reported MU for Protein by repeatability, and one by reproducibility. Four laboratories reported both repeatability and reproducibility MU for Protein. These values are included in section A1 for comparison.

6.4 Fat

Eight laboratories tested the samples for Fat. Of these laboratories, six tested using Soxhlet extraction, including one laboratory that submitted three sets of results. One laboratory tested using Foss-Lett. One laboratory tested using other methods. All methods were pooled to analyse the Fat results.

Laboratory code 7 reported outlier results for both samples, which were over twice the median values for both samples. Laboratory code 6 reported an outlier result for sample PTA 1, which was sitting at about 20% of the median value for the sample, however, compared well to the median for sample PTA 2. Laboratory code 4 reported an outlier result for sample PTA 2. As samples PTA 1 and PTA 2 were duplicate samples, results for each would be expected to be very close to each other.

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 and / or method within a test. For the Fat testing, the median and associated standard error (se) for each sample (expressed in g/100g) was as follows:

	PTA 1	PTA 2
Fat, all methods pooled	31.35 ± 0.37	31.25 ± 0.30

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

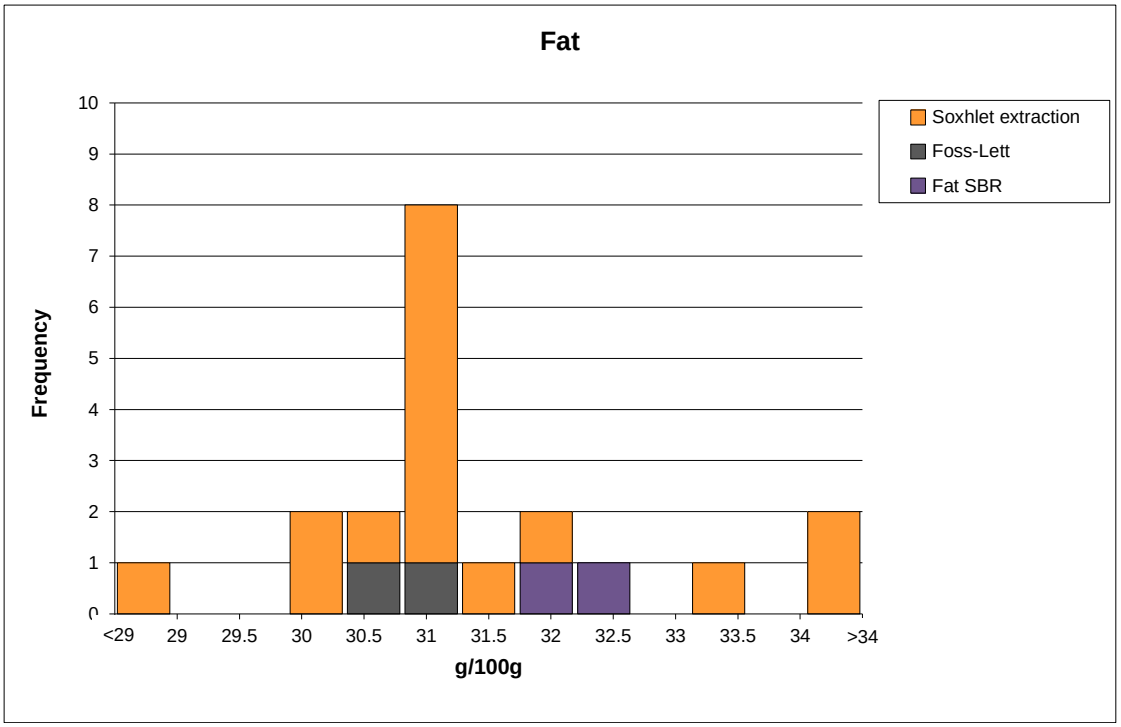


Figure TA-2. Spread of all results for Fat testing of duplicate Pâté samples PTA 1 & PTA 2.

The robust CVs of 3.0% and 2.4% for the two samples are lower than the values of 5.0% and 3.3% obtained in Round 40 of the Food program (see Report No. 995).

Some important factors to consider for improving accuracy and / or repeatability (USDA, CLG-FAT.03; Young et.al., 2012) where higher than expected results were obtained include:

- Excessive drying may oxidize the fat and give high results.
- Meat products containing water-soluble carbohydrates should be washed with water prior to drying and solvent extraction because the presence of carbohydrates may lead to erroneously high fat values due to extraction of sugar.

For this proficiency round, laboratories were requested to report their MU by repeatability (r) and / or reproducibility (R). Two laboratories reported MU for Fat by repeatability, and one by reproducibility. Three laboratories reported both repeatability and reproducibility MU for Fat. Two laboratories did not provide an estimate of the MU for their Fat results. These values are included in section A2 for comparison.

6.5 Moisture

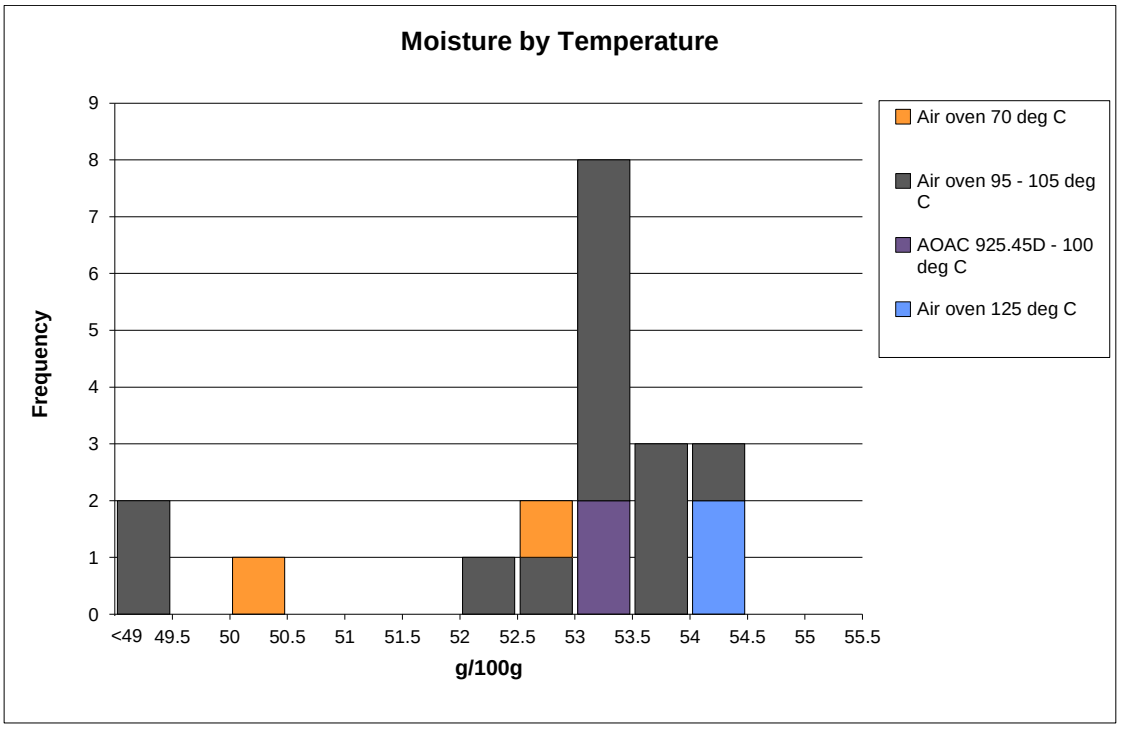
Seven of the laboratories that tested the samples for Moisture used an air oven, including one laboratory that submitted three sets of results. One laboratory tested using other methods. All methods were pooled to analyse the Moisture results.

Laboratory code 6 reported outlier results for both samples, with results significantly lower than the medians for each sample. Laboratory code 3 reported an outlier result for sample PTA 2, also lower than the median.

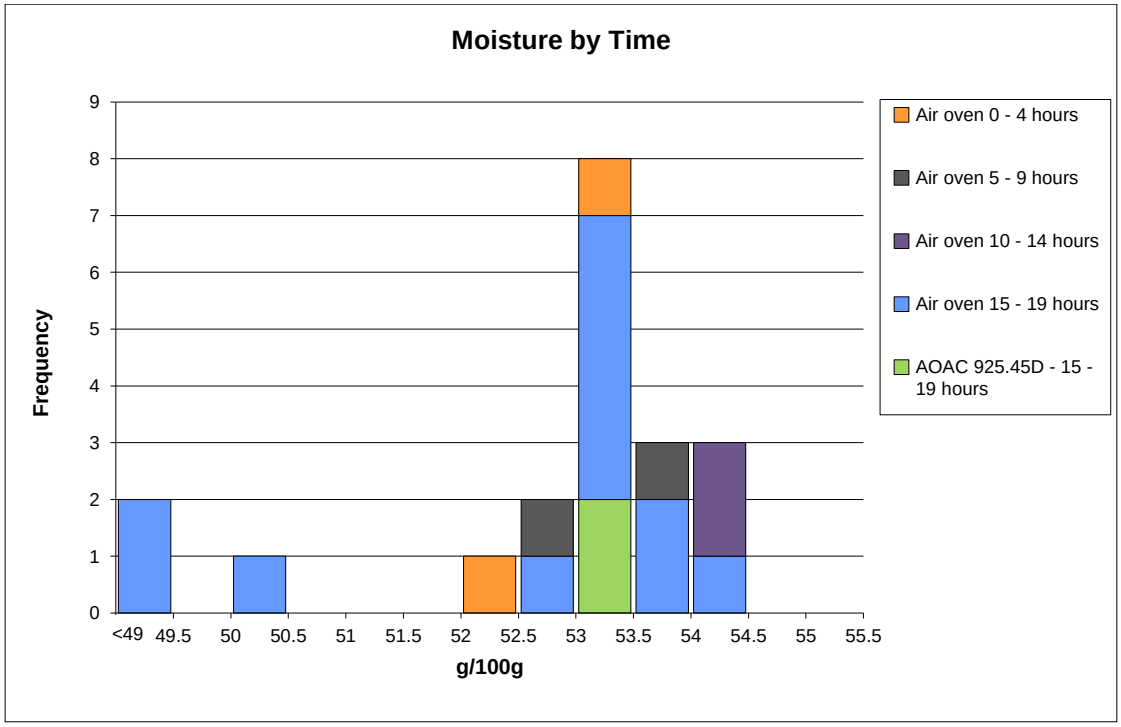
Confidence in the medians can be expressed as the uncertainty of the median (as defined in page 3 of this report), which was calculated for each test and / or method within a test. For the Moisture testing, the median and associated standard error (se) for each sample (expressed in g/100g) was as follows:

	PTA 1	PTA 2
Moisture, all methods pooled	53.35 ± 0.28	53.33 ± 0.22

Figures TA-3 (a) and (b), below, show the distribution of results from the methods used for Moisture testing in this study.



(a)



(b)

Figure TA-3. Spread of all results for Moisture testing of duplicate Pâté samples PTA 1 & PTA 2, grouped by (a) temperature and (b) time.

The robust CVs of 1.3% and 1.0% for the two samples compare well with the values of 1.1% and 1.0% obtained in Round 40 of the Food program (see Report No. 995).

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 one by reproducibility. Three laboratories reported both repeatability and reproducibility MU for Moisture. Two laboratories did not provide an estimate of their MU for this test. These values are included in section A3 for comparison.

The temperatures used for Moisture determination ranged between 16 hours at 70 °C to 12 hours at 125 °C. The maximum time for Moisture determination was 18 hours at 102 °C, with the minimum time being 2.5 hours at 105 °C. The majority of laboratories performed their drying at 100 – 105 °C for between 2.5 – 18 hours.

Some important factors to consider for improving accuracy and / or repeatability include (USDA, CLG-MOI.03; Young et.al, 2012):

- The importance of using drying dishes of suitable dimensions to get good moisture transfer from the surface of a sample.
- Quick and effective moisture removal can be aided by carefully mixing samples with sand to increase the surface area of the sample and prevent clumping of the meat that might trap moisture during the drying step.
- Do not overload the drying oven, as this can lead to the sample being insufficiently dried, leading to low results. No dishes should be touching and dishes should not be placed on a solid tray. Proper air circulation is necessary.
- Drying time should start when the original temperature has been reached (< 10 minutes). Use the oven's booster heater (if it has one), to minimise this recovery time.
- If the laboratory is not air-conditioned, and humidity may present a problem, it is advisable to desiccate dishes prior to the initial and final weighing.
- It is also important to remember that the dried sample residue is not satisfactory for subsequent fat determination when drying at higher temperatures (≈ 125 °C).

6.6 Ash

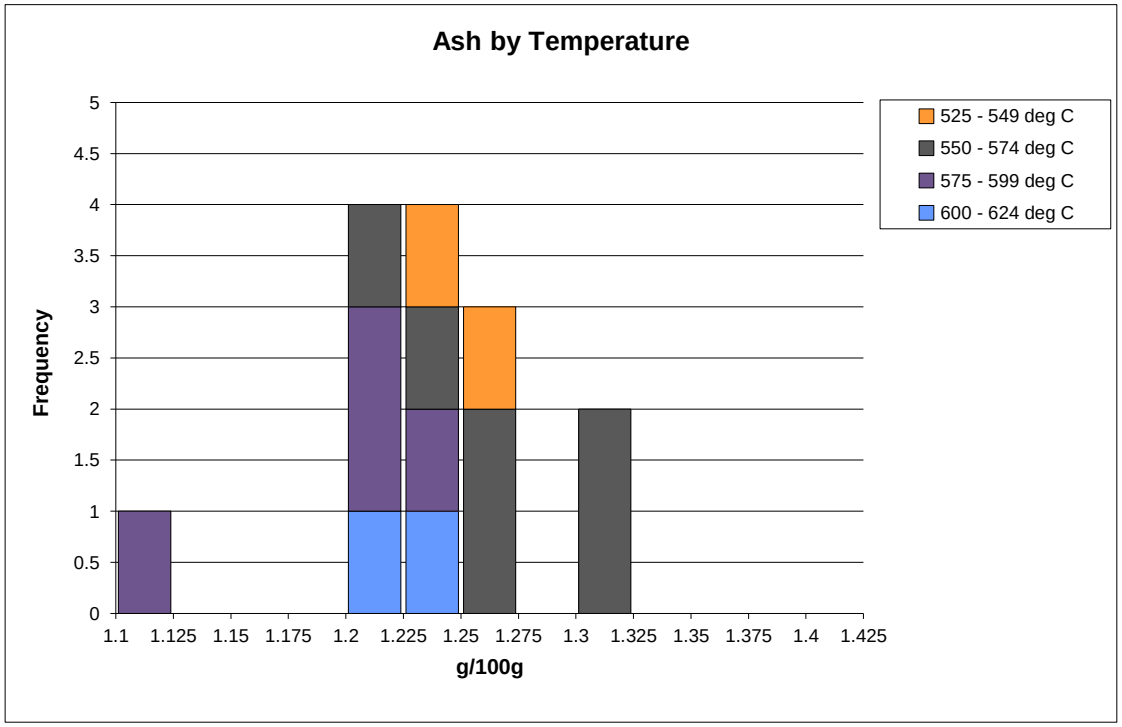
All of the seven laboratories that tested the samples for Ash used a muffle furnace.

There were no outliers reported for either sample.

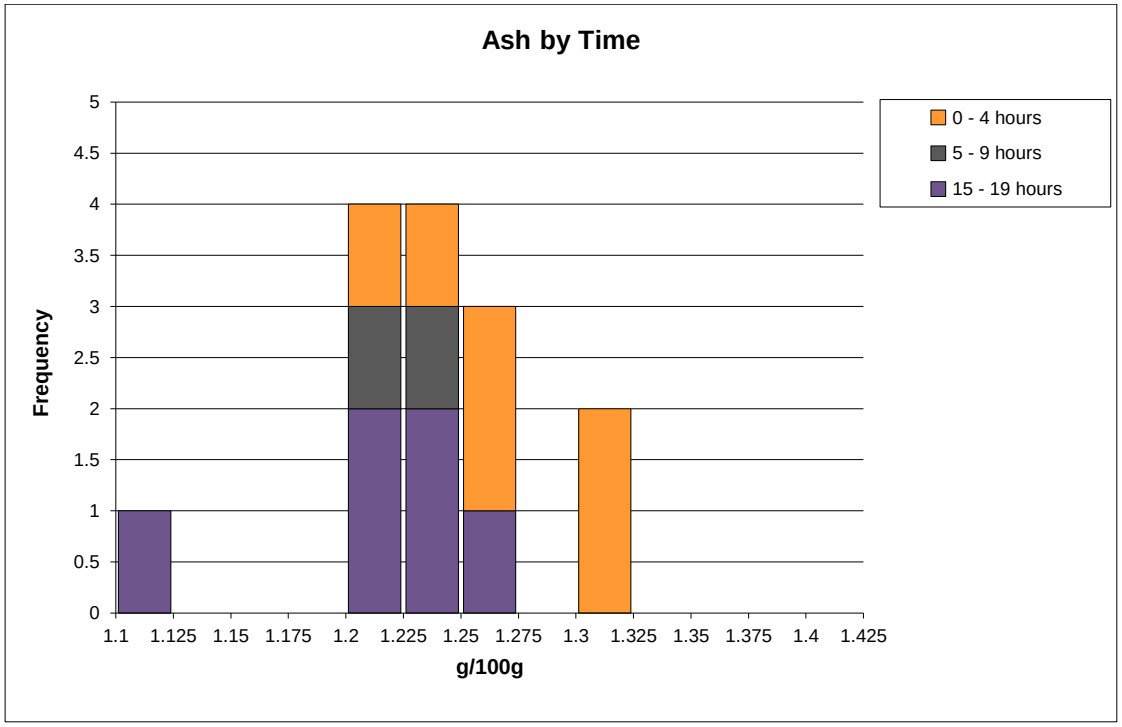
Confidence in the medians can be expressed as the uncertainty of the median (as defined in page 3 of this report), which was calculated for each test and / or method within a test. For the Ash testing, the median and associated standard error (se) for each sample (expressed in g/100g) was as follows:

	PTA 1	PTA 2
Ash, muffle furnace method	1.225 ± 0.021	1.250 ± 0.009

Figures TA-4 (a) and (b), below, show the distribution of results from the methods used for Ash testing in this round, grouped by ashing time and temperature.



(a)



(b)

Figure TA-4. Spread of all results for Ash testing of duplicate Pâté samples PTA 1 & PTA 2, grouped by ashing (a) temperature and (b) time.

The robust CVs for the results for this round were 3.7% and 1.4%. The robust CV of 1.4% for sample PTA 2 was considered inappropriate to evaluate the performance of the participants in this round, so a target CV was used to calculate the z-scores for sample PTA 2. The target CV chosen was 3.7%. This value was chosen because it was the same CV as that obtained for sample PTA 1.

The temperatures used for ashing ranged between 525 °C and 600 °C. The time for ashing ranged between 1.5 to 16 hours.

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 one by reproducibility. Three laboratories reported both repeatability and reproducibility MU for Ash. One laboratory did not provide an estimate of the MU for their Ash results. These values are included in section A4 for comparison.

6.7 Salt

Only five laboratories tested the samples for Salt. Two of these laboratories used Volhard titration and three used other methods.

Of the other methods used by the participants for testing Salt, one laboratory reported using method AACC 43-31.01. Method AACC 43-31.01 could not be found. It is instead thought that this is most likely to be method AACC **40**-31.01 – Chlorides in Ash as Sodium Chloride – Volumetric Method. If this was the method used in this round, it should be noted it was designed for cereal based food products. The low bias of this method, compared to the Volhard titration method, has been noted in previous rounds (refer to Report 825 - Round 37), although data is limited in this round.

There were not enough results submitted for Salt to calculate z-scores or summary statistics.

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

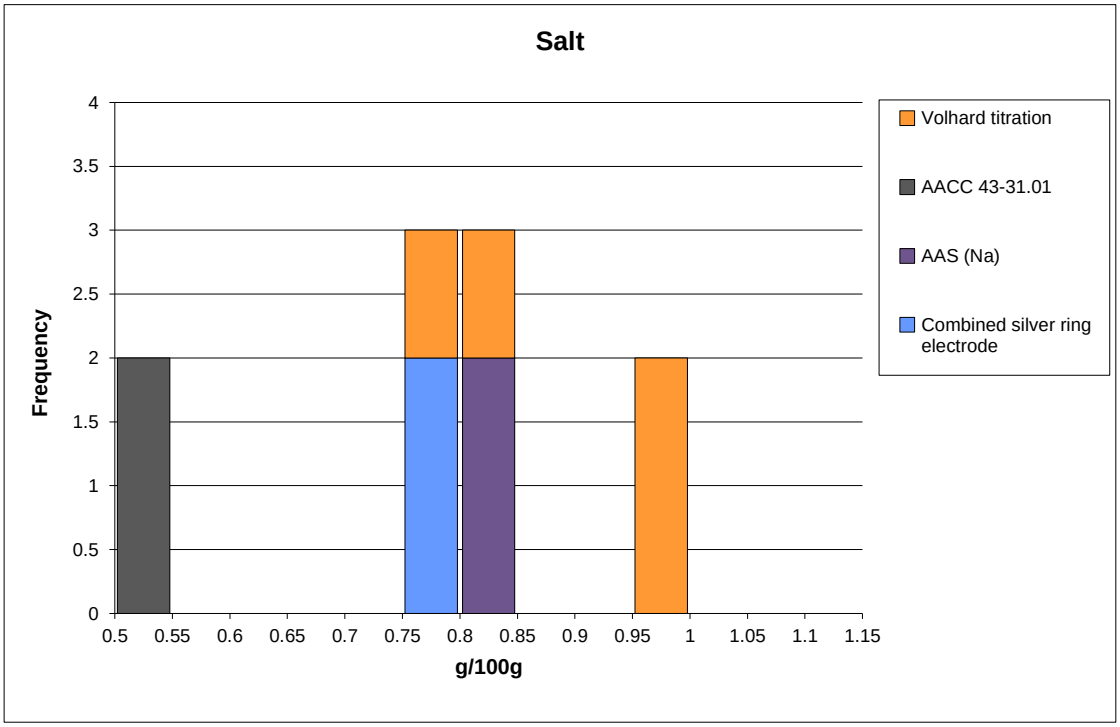


Figure TA-5. Spread of all results for Salt testing of duplicate Pâté samples PTA 1 & PTA 2.

For this proficiency round, laboratories were requested to report their MU by repeatability (r) and / or reproducibility (R). One laboratory reported MU for Salt by repeatability, and one by reproducibility. Two laboratories reported both repeatability and reproducibility MU for Salt. One laboratory did not provide an estimate of the MU for their Salt results. These values are included in section A5 for comparison.

6.8 Carbohydrate

Six laboratories reported results for Carbohydrate.

Laboratory code 6 reported an outlier result for sample PTA 1, which is attributable to the very low Fat result obtained for this sample. Laboratory code 3 reported an outlier result for sample PTA 2, which is attributable to the low Moisture result obtained for this sample.

Confidence in the medians can be expressed as the uncertainty of the median (as defined in page 3 of this report), which was calculated for each test. For the Carbohydrate testing, the median and associated standard error (se) for each sample (expressed in g/100g) was as follows:

	PTA 1	PTA 2
Carbohydrate	4.55 ± 0.78	3.65 ± 0.58

Samples PTA 1 and PTA 2 were duplicate samples, yet there was quite some variation between carbohydrate medians produced by these two datasets, as well as high CVs, indicating variation within the data set for each sample. This is not unexpected, given the test is a calculation by difference (Carbohydrate = 100 – (%Protein + %Fat + %Moisture + %Ash)), and the levels of Protein, Fat, Moisture and Ash are high, thereby resulting in low Carbohydrate results.

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

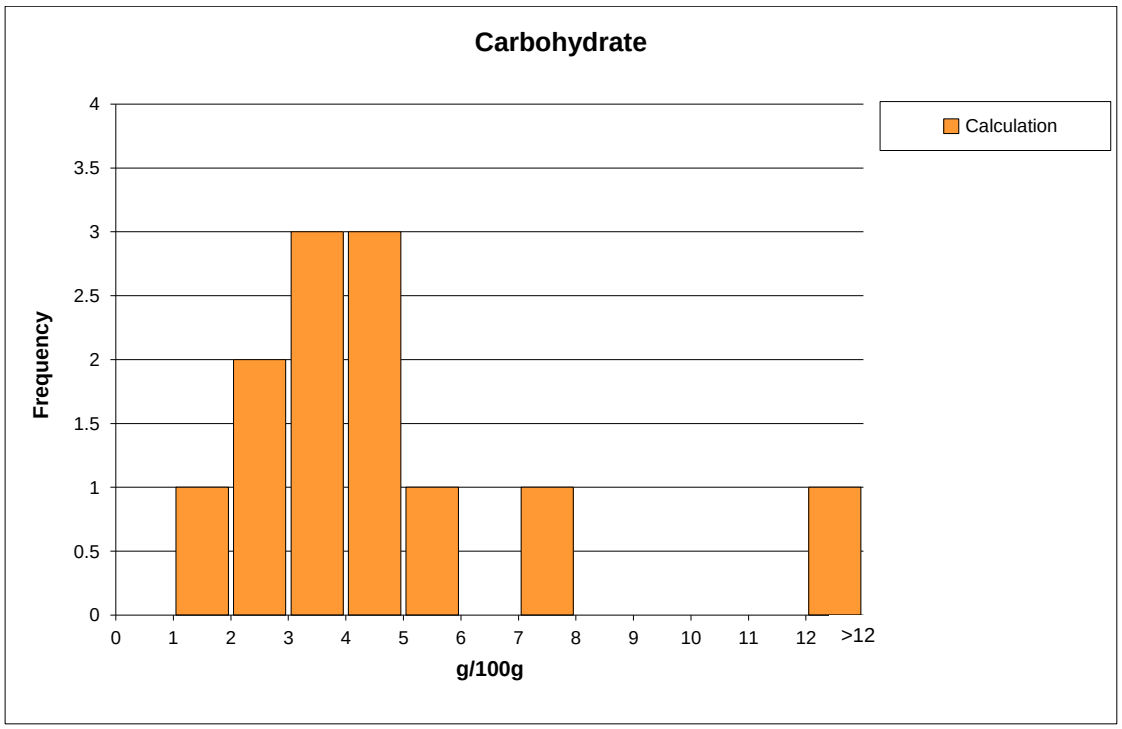


Figure TA-6. Spread of all results for the Carbohydrate test on duplicate Pâté samples PTA 1 & PTA 2.

The robust CVs of 33.6% and 30.9% for the two samples are lower than the values of 69.9% and 63.8% obtained in Round 40 of the Food program (see Report No. 995).

For this proficiency round, laboratories were requested to report their MU by repeatability (r) and / or reproducibility (R). Two laboratories reported MU for Carbohydrate by repeatability. Two laboratories reported both repeatability and reproducibility MU for Carbohydrate. Two laboratories did not provide an estimate of the MU for their Carbohydrate results. These values are included in section A6 for comparison.

6.9 Energy

Six laboratories reported results for Energy.

Laboratory code 6 reported an outlier result for sample PTA 1, which is attributable to the very low Fat result obtained for this sample.

Confidence in the medians can be expressed as the uncertainty of the median (as defined in page 3 of this report), which was calculated for each test. For the Energy testing, the median and associated standard error (se) for each sample (expressed in kJ/100g) was as follows:

	PTA 1	PTA 2
Energy	1406.0 ± 23.6	1405.0 ± 24.2

Based on median results for all the parameters incorporated in the Energy calculation; Energy = (%Protein x 17) + (%Carbohydrate x 17) + (%Fat x 37), the theoretical result for these samples should have been in the vicinity of 1392 kJ/100g, which compared well to the medians obtained in the round.

The robust CVs of 3.3% and 3.4% for the two samples are higher than the values of 2.3% and 2.8% obtained in Round 40 of the Food program (see Report No. 995).

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

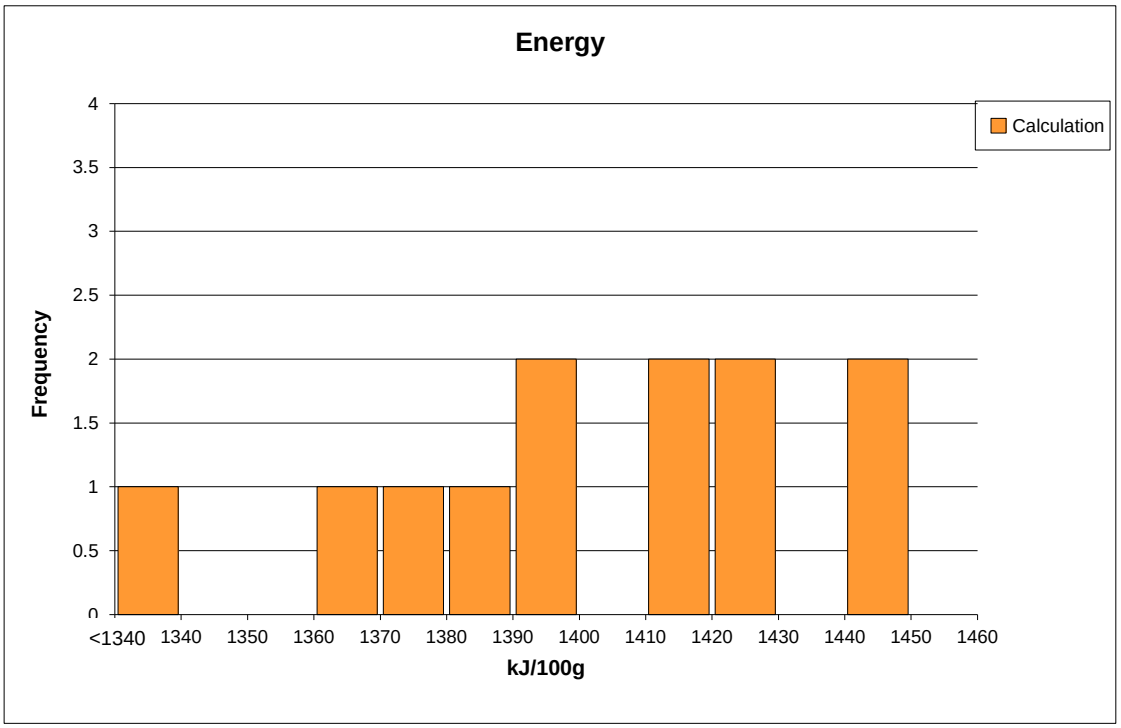


Figure TA-7. Spread of all results for the Energy test on duplicate Pâté samples PTA 1 & PTA 2.

For this proficiency round, laboratories were requested to report their MU by repeatability r and / or reproducibility R. Two laboratories reported MU for Energy by repeatability. Two laboratories reported MU for Energy by both repeatability and reproducibility. Two laboratories did not provide an estimate of the MU for their Energy results. These values are included in section A7 for comparison.

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 by each laboratory for each individual test is as follows:

Test	Repeatability (r)	Reproducibility (R)
Protein	5 out of 6 83%	5 out of 6 83%
Fat	5 out of 8 63%	4 out of 8 50%
Moisture	5 out of 8 63%	4 out of 8 50%
Ash	5 out of 7 71%	4 out of 7 57%
Salt	4 out of 5 80%	2 out of 5 40%
Carbohydrate	4 out of 6 67%	2 out of 6 33%
Energy	4 out of 6 67%	2 out of 6 33%

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

Table C: Method of Measurement Uncertainty Estimation

Lab Code	Method
3	Top down.
5	GUM.
8	GUM (bottom up).

Laboratories are encouraged to regularly review their MU estimates, keeping in mind that MUs based on in-house precision data are likely to be smaller than those determined by the Horwitz equation or inter-laboratory precision data.

7. REFERENCES

1. Guide to Proficiency Testing Australia (2019). (This document is located on the PTA website at www.pta.asn.au under Programs / Documents).

2. ISO/IEC 17043: 2010 *Conformity assessment – General requirements for proficiency testing*.

3. AOAC 925.45-1925, Loss on drying (moisture) in sugars. Association of Official Analytical Chemists; Washington D.C, USA.

4. AACCI Method 40-31.01 - Chlorides in Ash as Sodium Chloride – Volumetric Method (AACC Approved Methods of Analysis, 11th Edition). American Association of Cereal Chemists; Minnesota, USA.

5. USDA, CLG-FAT.03 (2009) Determination of Fat, United States Department of Agriculture Food Safety and Inspection Service, Office of Public Health Science.
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   http://www.fsis.usda.gov/wps/wcm/connect/4dab93c9-cdf3-427d-ab00-c9928eeb89f7/CLG_MOI_03.pdf?MOD=AJPERES

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   http://books.google.co.nz/books?id=1ZRCMRXbbwoC&pg=PA144&dq=AOAC+991.36&hl=en&sa=X&ei=9sJAUpC0OsnFkwW50oHIDw&ved=0CC4Q6AEwAA#v=onepage&q=AOAC%20991.36&f=false

APPENDIX A

Summary of Results

Section A1

Protein

Chicken Liver Pâté – 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	9.6	9.6	0.3	-	9.6	9.6	0.3	-
3	9.5	9.5	-	0.3	9.7	9.4	-	0.3
4	11.5	11.5	0	0	10.9	10.9	0	0
5	9.4	9.4	1.9	1.9	10.0	9.4	2.0	1.9
6	9.6	9.7	0.5	0.5	9.7	9.7	0.5	0.5
8	10.0	9.9	0.4	0.6	9.8	9.7	0.4	0.6

Chicken Liver Pâté – Protein (g/100g)
Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code
	Average	Z-Score	Average	Z-Score	
1	9.60	-0.08	9.60	-0.95	2
3	9.50	-0.38	9.55	-1.42	1
4	11.50	5.70 **§**	10.90	11.35 **§**	1
5	9.40	-0.68	9.70	0.00	1
6	9.65	0.08	9.70	0.00	2
8	9.95	0.99	9.75	0.47	1

Method Codes

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

Summary Statistics

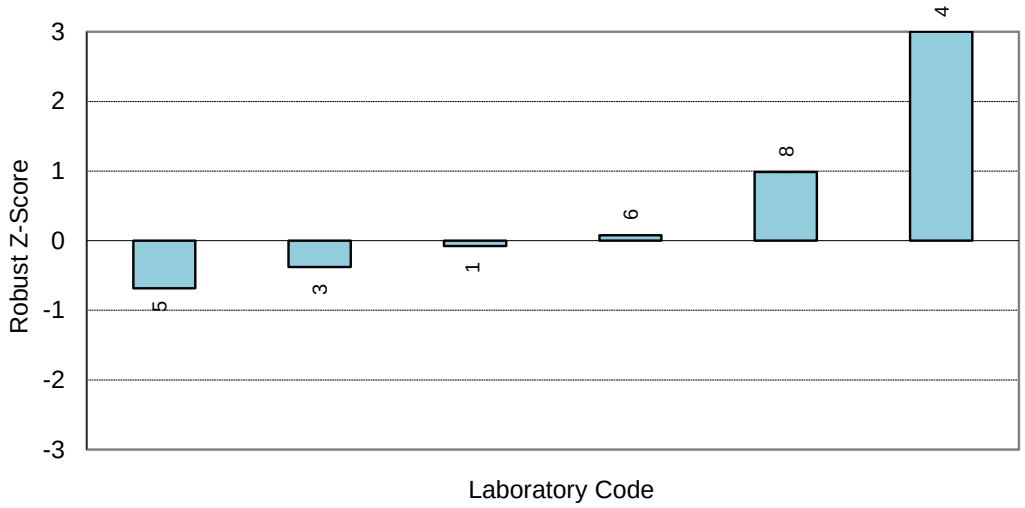
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	6	6
Median	9.63	9.70
Norm IQR	0.33	0.11
Uncertainty (Median)	0.17	0.05
Robust CV	3.4%	1.1%
Minimum	9.40	9.55
Maximum	11.50	10.90
Range	2.10	1.35

Notes:

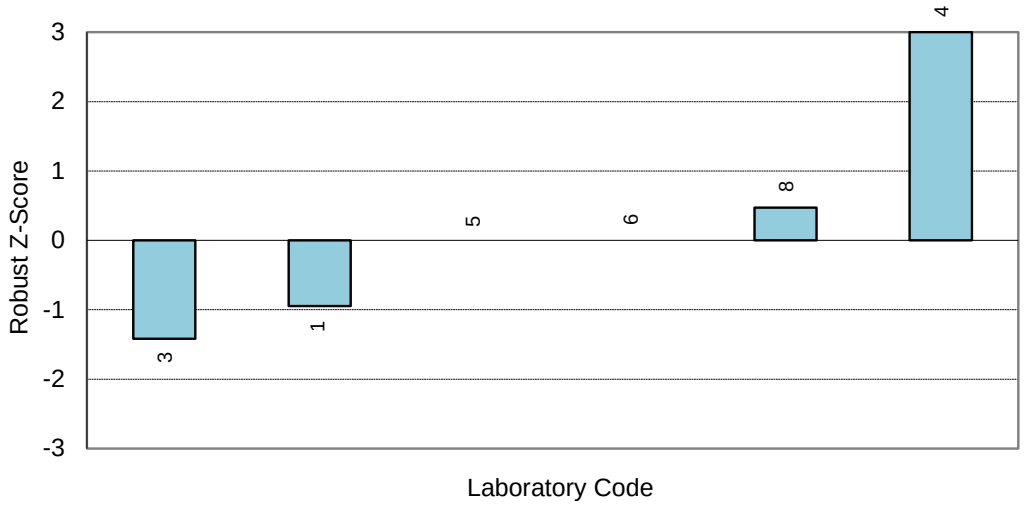
1. **§** denotes an outlier (i.e. |z-score| ≥ 3.0).
2. Z-scores were calculated for the average result for each sample.
3. The Youden diagram on the following page is provided for information only.

A1.2

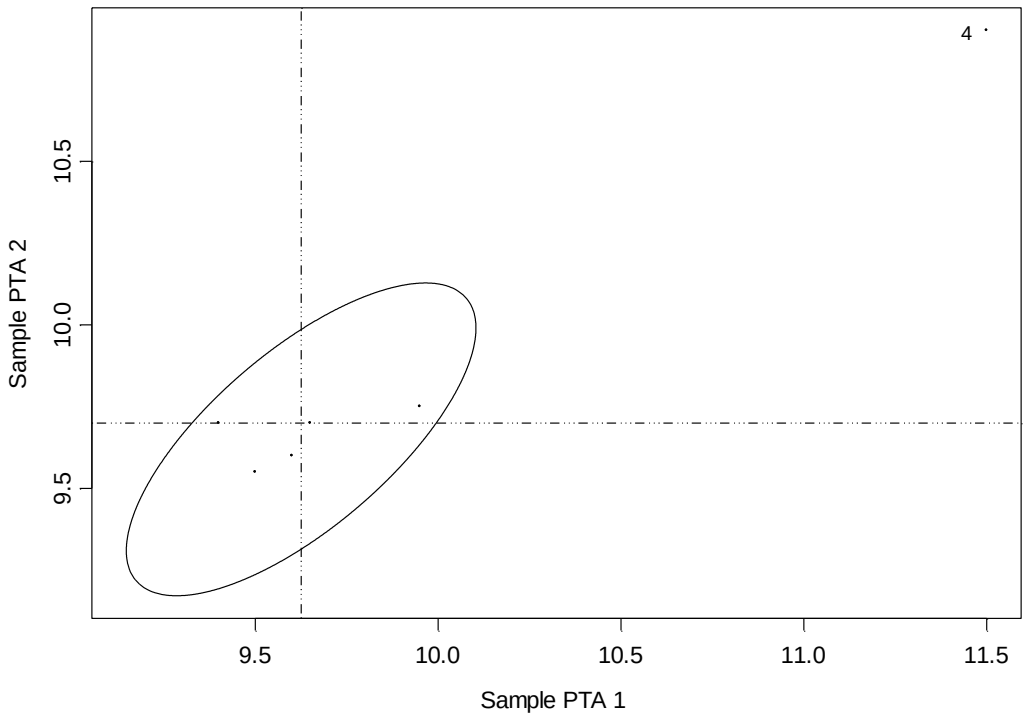
Protein (g/100g) - Sample 1



Protein (g/100g) - Sample 2



Protein (g/100g)



Section A2

Fat

A2.1

Chicken Liver Pâté – 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	31.3	31.4	0.3	-	31.0	31.0	0.3	-
2A	31.2	31.5	-	-	31.5	31.8	-	-
2B	31.0	31.1	-	-	30.4	30.1	-	-
2C	30.5	31.0	-	-	31.5	31.0	-	-
3	31.8	31.2	-	0.7	31.3	31.2	-	0.7
4	32.2	31.9	0.58	-	33.3	33.9	1.1	-
5	30.3	30.5	1.2	1.3	31.2	31.1	1.3	1.3
6	6.5	6.6	2.3	2.3	31.7	30.8	2.3	2.3
7	67.76	68.41	-	-	65.93	64.54	-	-
8	32.7	32.4	0.4	0.5	32.2	32.2	0.4	0.5

Chicken Liver Pâté – Fat (g/100g)
Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code
	Average	Z-Score	Average	Z-Score	
1	31.35	0.00	31.00	-0.33	2
2A	31.35	0.00	31.65	0.53	1
2B	31.05	-0.32	30.25	-1.32	1
2C	30.75	-0.65	31.25	0.00	1
3	31.50	0.16	31.25	0.00	1
4	32.05	0.75	33.60	3.10 **§**	1
5	30.40	-1.02	31.15	-0.13	1
6	6.55	-26.73 **§**	31.25	0.00	1
7	68.09	39.59 **§**	65.24	44.88 **§**	1
8	32.55	1.29	32.20	1.25	3

Method Codes

1	=	Soxhlet extraction	8
2	=	Foss-Lett	1
3	=	Other	1

Summary Statistics

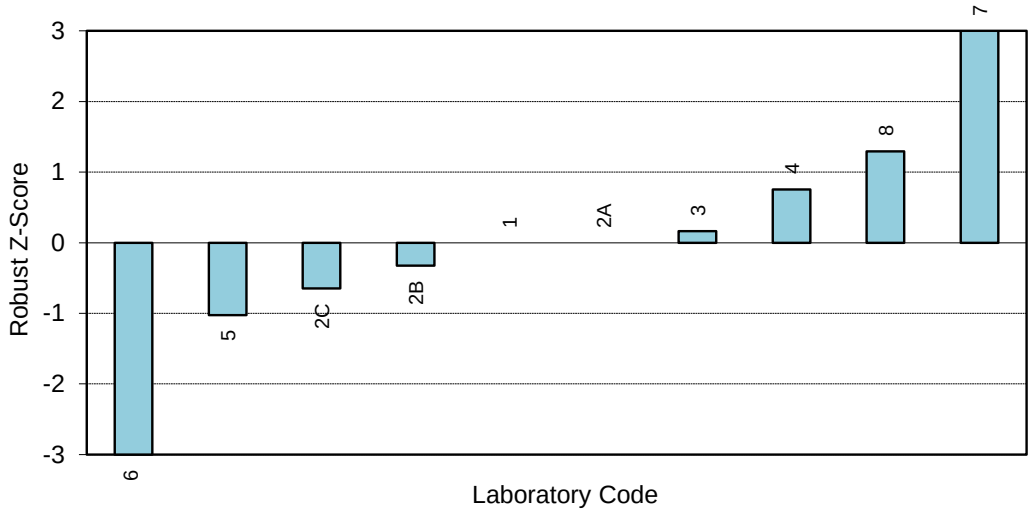
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	10	10
Median	31.35	31.25
Norm IQR	0.93	0.76
Uncertainty (Median)	0.37	0.30
Robust CV	3.0%	2.4%
Minimum	6.55	30.25
Maximum	68.09	65.24
Range	61.54	34.99

Notes:

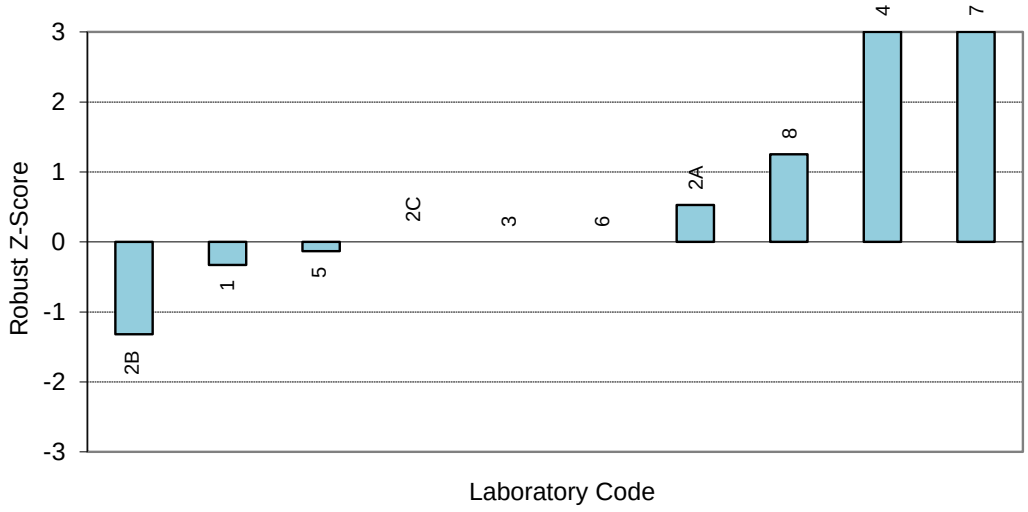
1. **§** denotes an outlier (i.e. |z-score| ≥ 3.0).
2. Z-scores were calculated for the average result for each sample.
3. The method used by laboratory code 8 was Fat SBR.
4. The Youden diagram on the following page is provided for information only.
5. Laboratory code 7 is not included on the Youden diagram on the following page.

A2.3

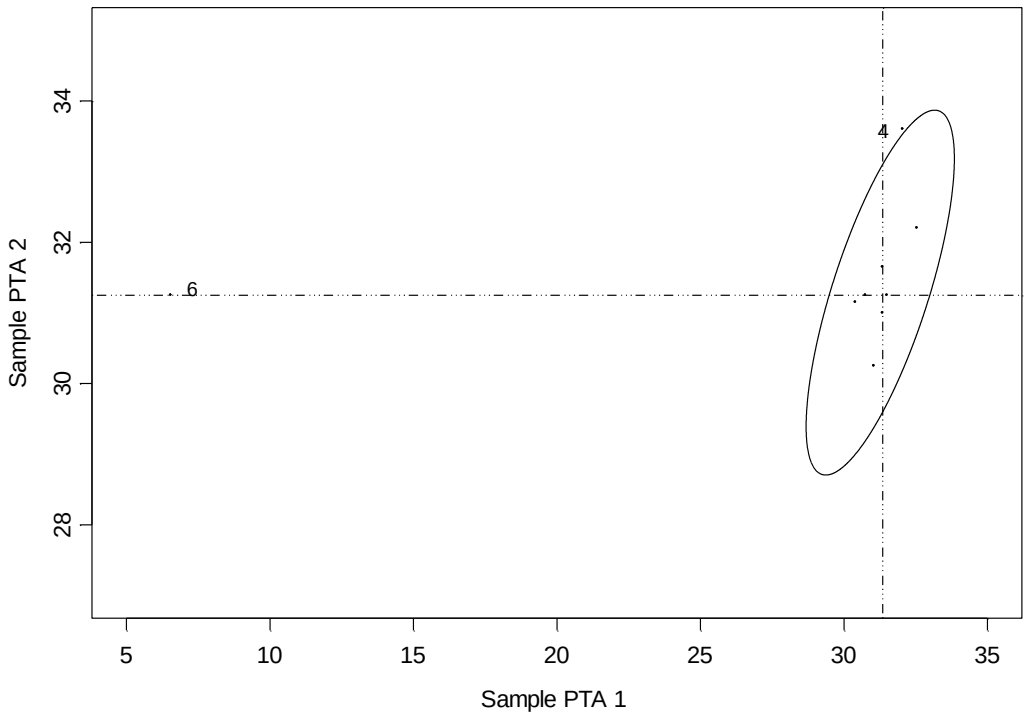
Fat (g/100g) - Sample 1



Fat (g/100g) - Sample 2



Fat (g/100g)



Section A3

Moisture

A3.1

Chicken Liver Pâté – 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	53.4	53.4	0.6	-	53.2	53.4	0.6	-
2A	53.6	53.5	-	-	54.6	54.2	-	-
2B	53.3	53.7	-	-	54.4	53.5	-	-
2C	53.7	53.2	-	-	53.4	53.0	-	-
3	52.6	53.0	-	1.9	50.2	50.4	-	1.8
4	52.5	52.3	0.4	-	53.2	53.2	0	-
5	54.3	54.2	1.1	1.1	54.3	54.6	1.1	1.1
6	46.1	46.3	0.9	0.9	46.4	46.4	0.9	0.9
7	52.78	52.46	-	-	53.71	53.36	-	-
8	53.2	53.4	1.6	2.7	53.5	53.2	1.6	2.7

Chicken Liver Pâté – Moisture (g/100g)
Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code	Temp (^{o}C)	Time (hrs)
	Average	Z-Score	Average	Z-Score			
1	53.40	0.07	53.30	-0.05	4	100	16
2A	53.55	0.28	54.40	1.95	1	103 ± 2	16
2B	53.50	0.21	53.95	1.13	1	103 ± 2	16
2C	53.45	0.14	53.20	-0.23	1	103 ± 2	16
3	52.80	-0.78	50.30	-5.49 **§**	1	70	16
4	52.40	-1.35	53.20	-0.23	1	105	2.5
5	54.25	1.28	54.45	2.04	1	125	12
6	46.20	-10.19 **§**	46.40	-12.56 **§**	1	105	16
7	52.62	-1.04	53.54	0.38	1	95 - 100	5
8	53.30	-0.07	53.35	0.05	1	102	18

Method Codes

1	=	Air Oven	9
2	=	Vacuum Oven	0
3	=	Rapid Microwave	0
4	=	Other	1

A3.2

Summary Statistics

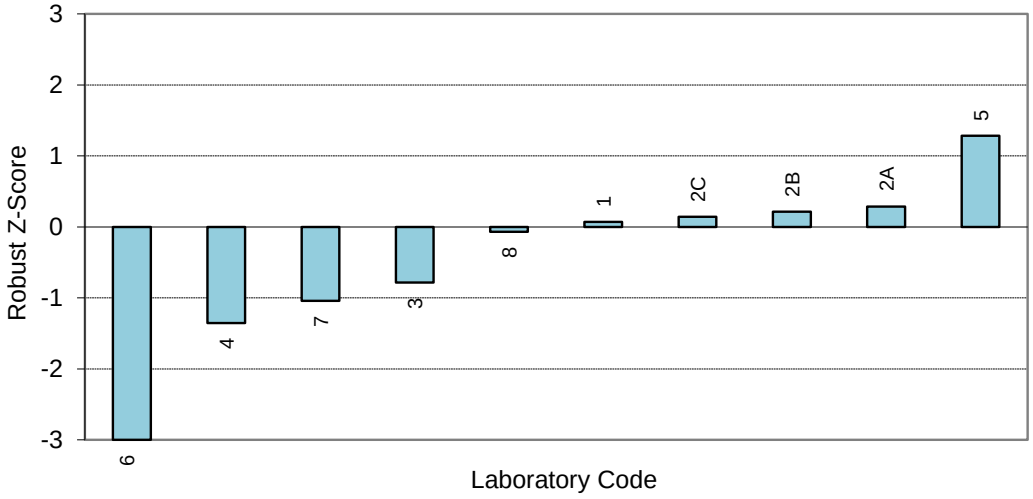
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	10	10
Median	53.35	53.33
Norm IQR	0.70	0.55
Uncertainty (Median)	0.28	0.22
Robust CV	1.3%	1.0%
Minimum	46.20	46.40
Maximum	54.25	54.45
Range	8.05	8.05

Notes:

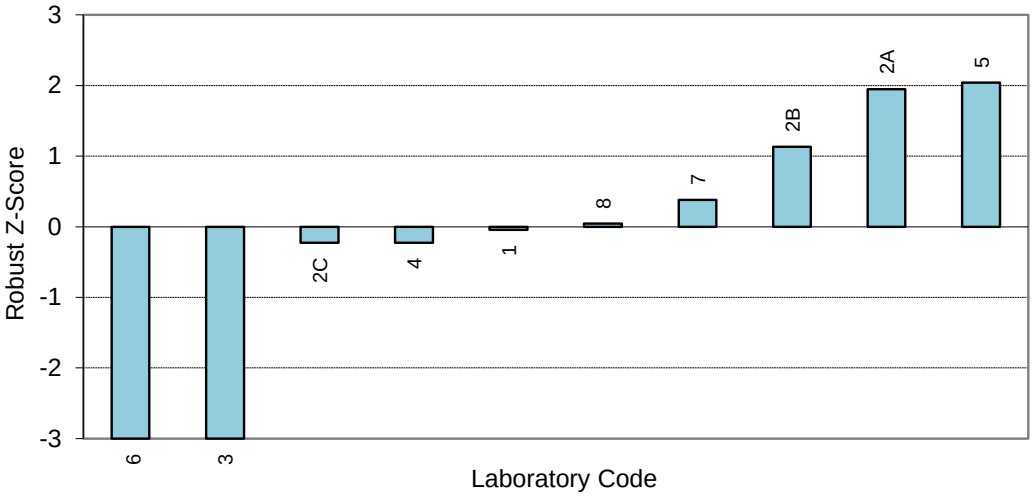
1. **§** denotes an outlier (i.e. |z-score| ≥ 3.0).
2. Z-scores were calculated for the average result for each sample.
3. The method used by laboratory code 1 was AOAC 925.45D.
4. The Youden diagram on the following page is provided for information only.

A3.3

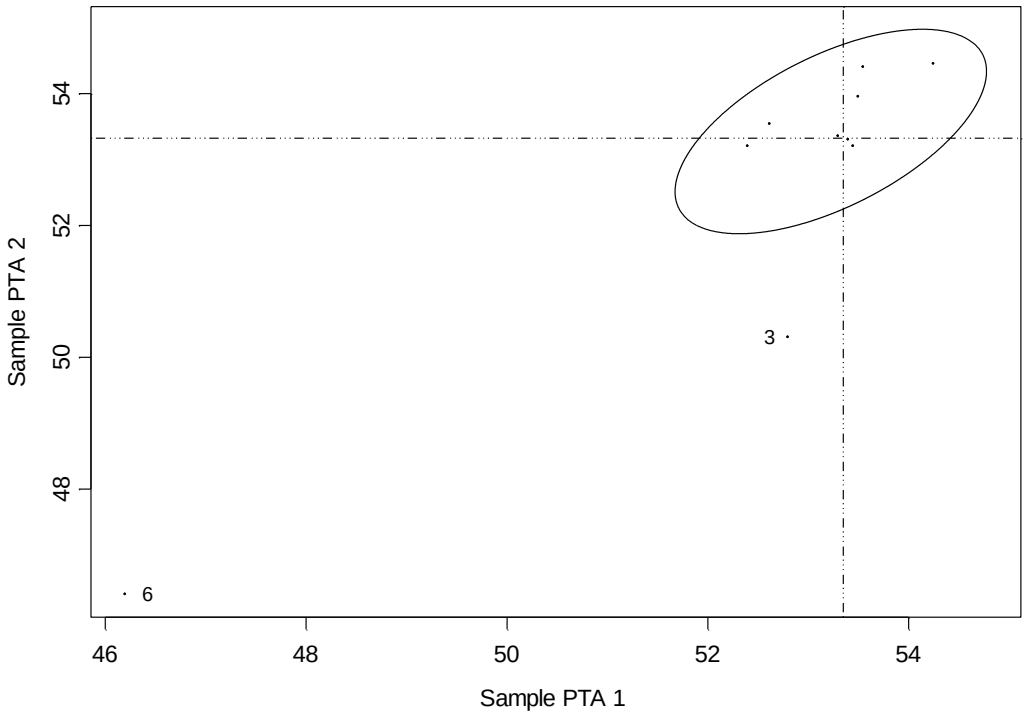
Moisture (g/100g) - Sample 1



Moisture (g/100g) - Sample 2



Moisture (g/100g)



Section A4

Ash

A4.1

Chicken Liver Pâté – 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	1.24	1.21	0.2	-	1.22	1.23	0.2	-
3	1.07	1.14	-	0.1	1.13	1.34	-	0.1
4	1.15	1.28	0.26	-	1.25	1.25	0	-
5	1.30	1.32	0.2	0.2	1.30	1.35	0.2	0.2
6	1.28	1.26	0.1	0.1	1.25	1.25	0.1	0.1
7	1.26	1.26	-	-	1.26	1.27	-	-
8	1.25	1.18	0.20	0.30	1.23	1.25	0.2	0.3

Chicken Liver Pâté – Ash (g/100g)
Z-Scores and Methods

Lab Code	Sample PTA 1		Sample PTA 2		Method Code	Temp (^{o}C)	Time (hrs)
	Average	Z-Score	Average	Z-Score			
1	1.225	0.00	1.225	-0.54	1	580	16
3	1.105	-2.66	1.235	-0.33	1	590	16
4	1.215	-0.22	1.250	0.00	1	600	1.5
5	1.310	1.89	1.325	1.63	1	550	2
6	1.270	1.00	1.250	0.00	1	525	16
7	1.260	0.78	1.265	0.33	1	550 ± 25	2
8	1.215	-0.22	1.240	-0.22	1	550	6

Method Codes

1	=	Muffle furnace	7
2	=	Other	0

Summary Statistics

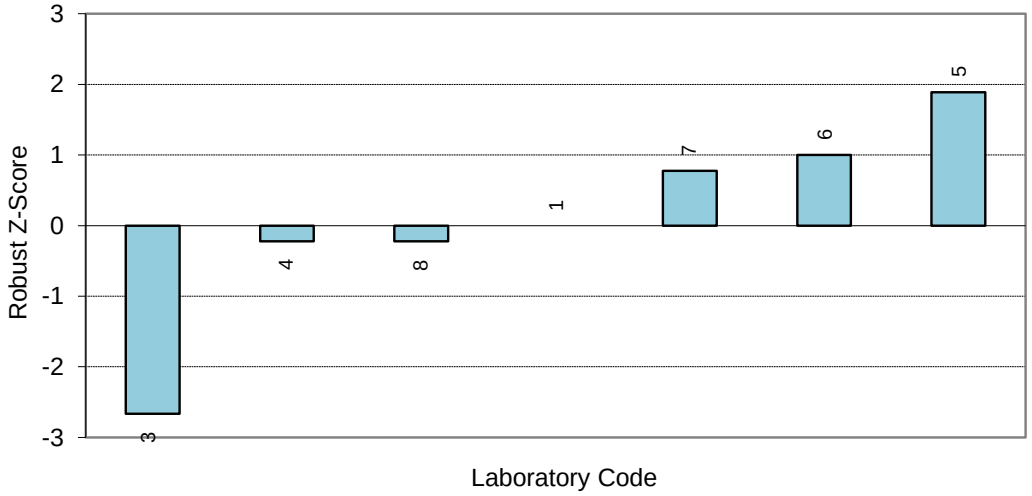
Statistic	Sample PTA 1	Sample PTA 2
No of Results	7	7
Median	1.225	1.250
Norm IQR	0.045	0.018
Uncertainty (Median)	0.021	0.009
Robust CV	3.7%	1.4%
Target SD	-	0.046
Target CV	-	3.7%
Minimum	1.105	1.225
Maximum	1.310	1.325
Range	0.205	0.100

Notes:

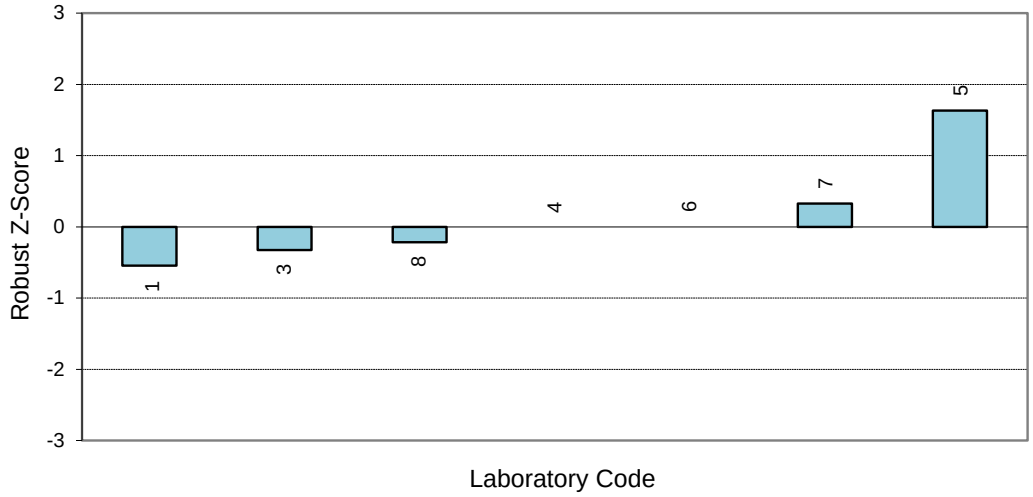
1. Z-scores were calculated for the average result for each sample.
2. A target CV was used to calculate the robust z-scores for sample PTA 2. The target CV chosen was 3.7%.
3. The target SD was obtained for sample PTA 2 by multiplying the target CV by the median. This value was used to calculate the z-scores for sample PTA 2. For more information on the use of target CVs to calculate z-scores, please see the Guide to Proficiency Testing Australia (2019).
4. The Youden diagram on the following page is provided for information only.

A4.3

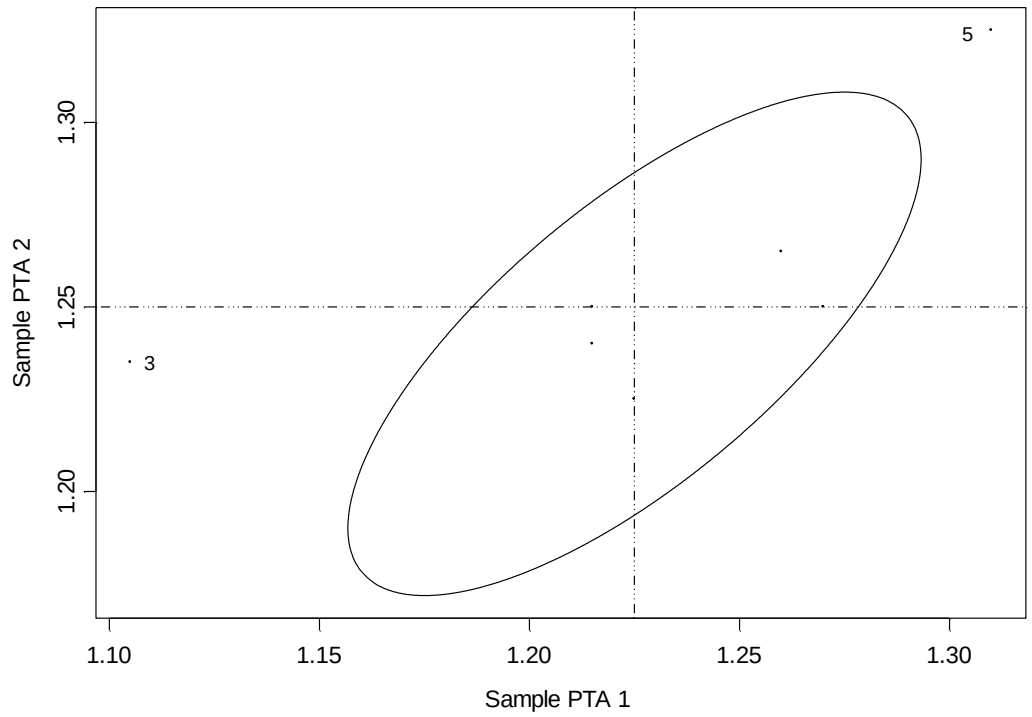
Ash (g/100g) - Sample 1



Ash (g/100g) - Sample 2



Ash (g/100g)



Section A5

Salt

A5.1

Chicken Liver Pâté – Salt (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	0.53	0.53	0.4	-	0.52	0.52	0.4	-
3	0.86	0.73	-	-	0.87	0.78	-	-
4	0.99	0.99	0	-	0.98	1.01	0.05	-
5	0.82	0.82	0.05	0.05	0.78	0.83	0.05	0.05
8	0.79	0.81	0.06	0.07	0.80	0.79	0.06	0.07

Chicken Liver Pâté – Salt (g/100g)
Average Results and Methods

Lab Code	Sample PTA 1	Sample PTA 2	Method Code
	Average	Average	
1	0.530	0.520	3
3	0.795	0.825	1
4	0.990	0.995	1
5	0.820	0.805	3
8	0.800	0.795	3

Method Codes

1	=	Volhard titration	2
2	=	ISE	0
3	=	Other	3

Notes:

1. There were not enough Salt results reported to calculate z-scores or summary statistics.
2. The method used by laboratory code 1 was AACC 43-31.01.
3. The method used by laboratory code 5 was AAS (Na).
4. The method used by laboratory code 8 was combined silver ring electrode.

Section A6

Carbohydrate

Chicken Liver Pâté – Carbohydrate (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	4.5	4.4	2	-	5.0	4.8	2	-
3	5.0	5.2	-	-	7.7	7.7	-	-
4	2.7	3.0	0.6	-	1.4	0.8	0.8	-
5	4.7	4.6	-	-	3.2	3.6	-	-
6	28.7	28.8	1.7	1.7	4.6	3.0	1.7	1.7
8	2.9	3.1	1.7	2.8	3.3	3.7	1.7	2.8

Chicken Liver Pâté – Carbohydrate (g/100g)
Z-Scores

Lab Code	Sample PTA 1		Sample PTA 2	
	Average	Z-Score	Average	Z-Score
1	4.45	-0.07	4.90	1.11
3	5.10	0.36	7.70	3.59 **§**
4	2.85	-1.11	1.10	-2.26
5	4.65	0.07	3.40	-0.22
6	28.75	15.85 **§**	3.80	0.13
8	3.00	-1.01	3.50	-0.13

Summary Statistics

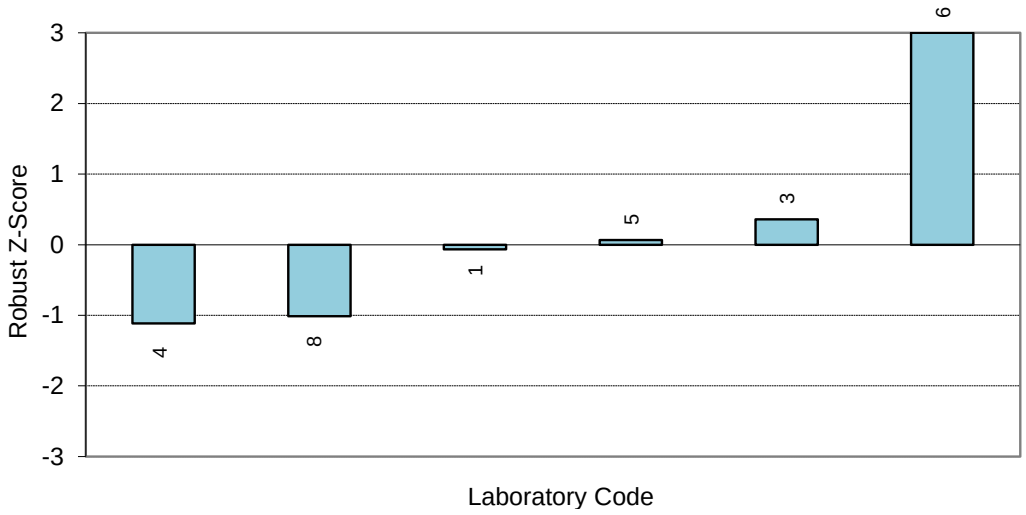
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	6	6
Median	4.55	3.65
Norm IQR	1.53	1.13
Uncertainty (Median)	0.78	0.58
Robust CV	33.6%	30.9%
Minimum	2.85	1.10
Maximum	28.75	7.70
Range	25.90	6.60

Notes:

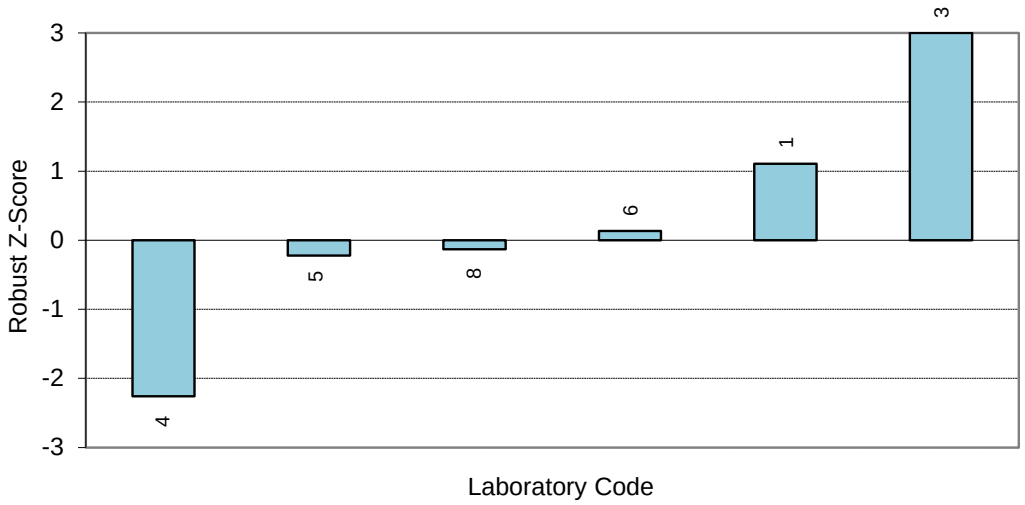
1. **§** denotes an outlier (i.e. |z-score| ≥ 3.0).
2. Z-scores were calculated for the average result for each sample.
3. The Youden diagram on the following page is provided for information only.

A6.2

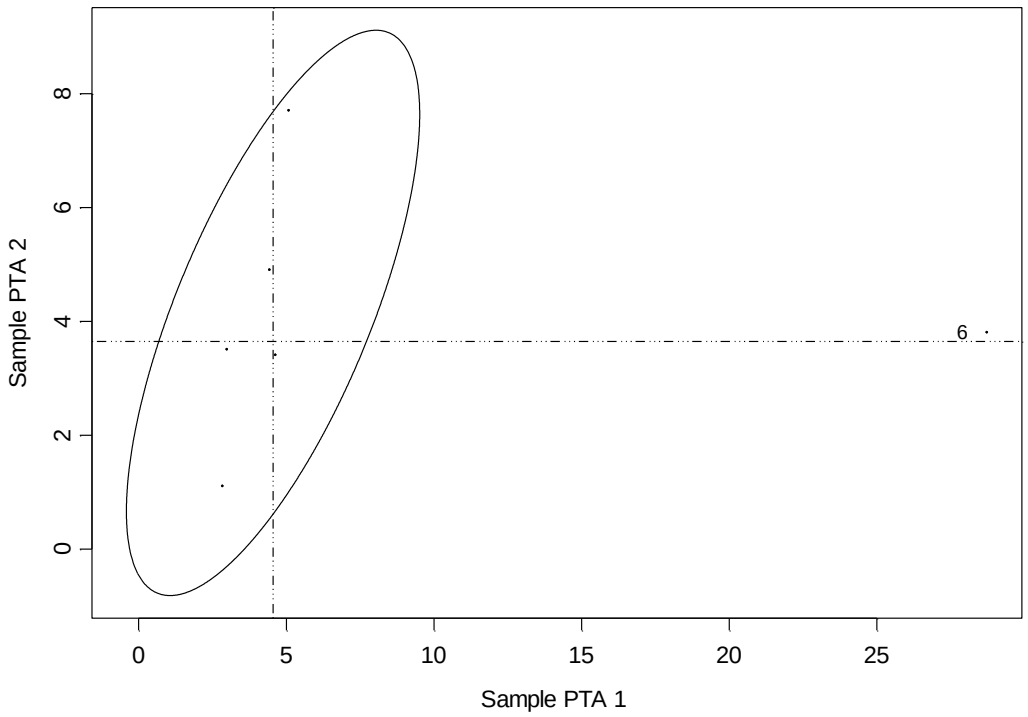
Carbohydrate (g/100g) - Sample 1



Carbohydrate (g/100g) - Sample 2



Carbohydrate (g/100g)



Section A7

Energy

Chicken Liver Pâté – 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	1398	1399	40	-	1395	1392	40	-
3	1423	1404	-	-	1454	1445	-	-
4	1432	1427	10	-	1440	1452	20	-
5	1361	1367	-	-	1378	1371	-	-
6	893	899	2.9	2.9	1384	1389	2.9	2.9
8	1429	1420	33	52	1414	1419	33	52

Chicken Liver Pâté – Energy (kJ/100g)
Z-Scores

Lab Code	Sample PTA 1		Sample PTA 2	
	Average	Z-Score	Average	Z-Score
1	1398.5	-0.16	1393.5	-0.24
3	1413.5	0.16	1449.5	0.94
4	1429.5	0.51	1446.0	0.87
5	1364.0	-0.91	1374.5	-0.64
6	896.0	-11.05 **§**	1386.5	-0.39
8	1424.5	0.40	1416.5	0.24

Summary Statistics

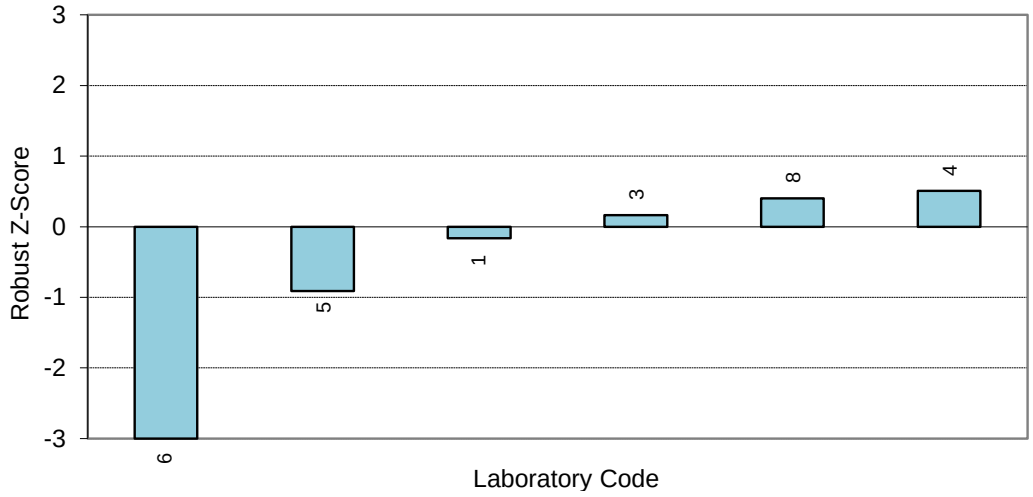
Statistic	Sample PTA 1	Sample PTA 2
Number of Results	6	6
Median	1406.0	1405.0
Norm IQR	46.2	47.3
Uncertainty (Median)	23.6	24.2
Robust CV	3.3%	3.4%
Minimum	896.0	1374.5
Maximum	1429.5	1449.5
Range	533.5	75.0

Notes:

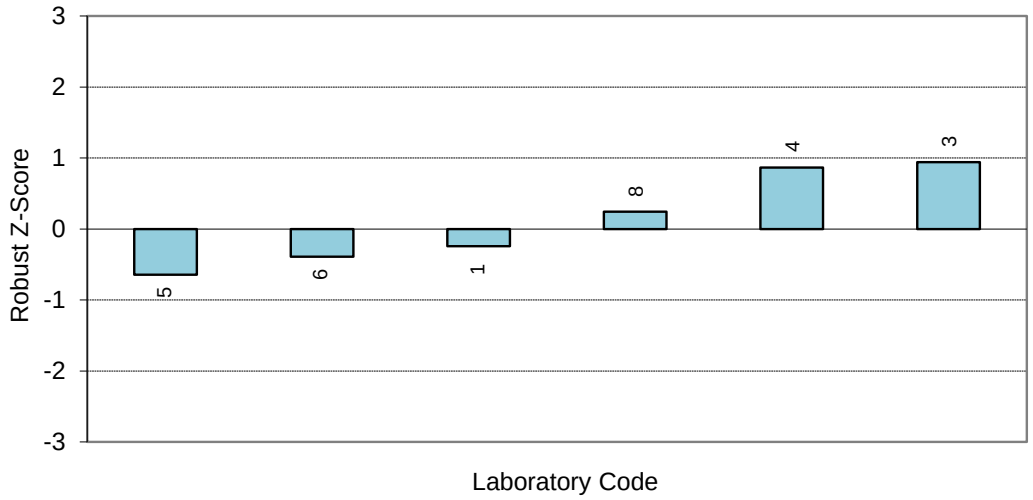
1. **§** denotes an outlier (i.e. |z-score| ≥ 3.0).
2. Z-scores were calculated for the average result for each sample.
3. The Youden diagram on the following page is provided for information only.

A7.2

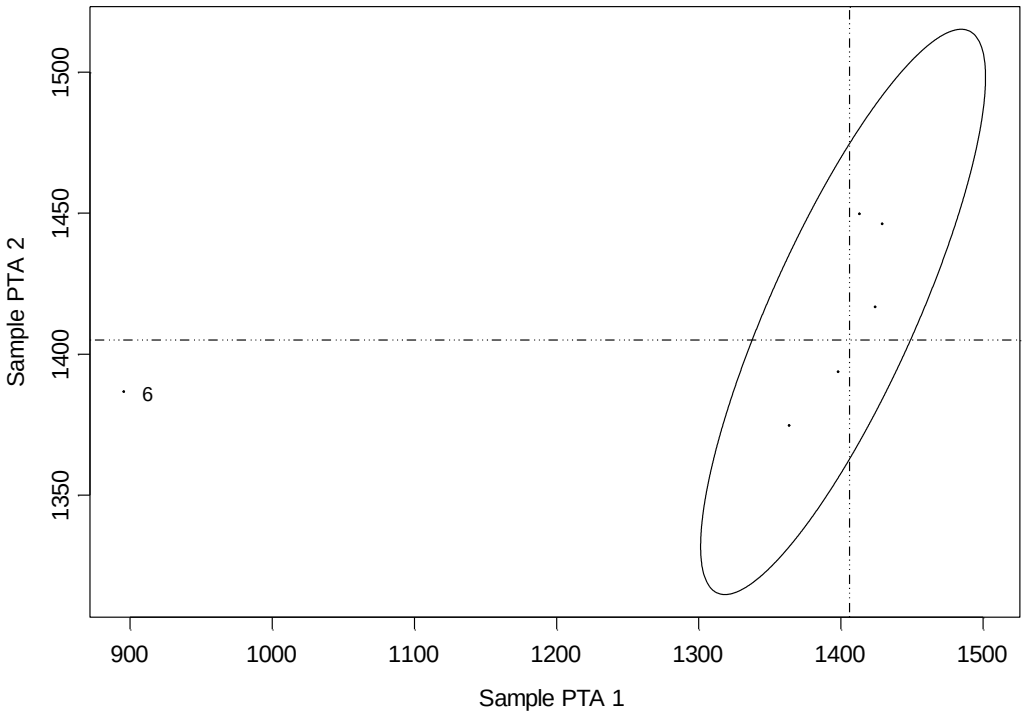
Energy (kJ/100g) - Sample 1



Energy (kJ/100g) - Sample 2



Energy (kJ/100g)



APPENDIX B

Homogeneity and Stability Testing

Homogeneity Testing

Prior to distribution, eight samples of chicken liver pâté were selected at random and tested for homogeneity by Dairy Technical Services Ltd, Melbourne VIC. Each sample was tested in duplicate for Fat. The results of the homogeneity testing appear in the following table.

Chicken Liver Pâté – Fat (g/100g)		
Sample No.	**Result A**	**Result B**
13	31.6	31.5
21	32.0	32.3
37	31.9	31.9
61	32.2	32.5
68	31.6	31.9
70	31.5	31.6
74	32.0	32.2
79	32.5	32.6

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.

Stability Testing

Three samples were selected at random and were exposed to ambient temperatures for a total of four days. At the end of this period, the samples were tested for stability by Dairy Technical Services Ltd, Melbourne VIC. Results of the stability testing appear in the following table.

Chicken Liver Pâté – Fat (g/100g)		
Sample No.	**Result A**	**Result B**
20	31.2	31.3
38	30.6	30.7
42	32.0	32.2

Although these results indicate there may have been an issue with stability, it was felt that the exposure of the pâté samples to ambient temperatures for four days may have been too severe, due to the fact that pâté is a refrigerated product transported using insulated packaging with chill packs, and participants are advised to refrigerate upon receipt. It was also noted that the samples were dispatched on 15 July, and all participants reported receiving samples on either 16 or 17 July, so none were exposed to ambient temperatures for four days. Given the overall performance of the round, it does not appear there were issues attributable to sample instability.

APPENDIX C

Instructions to Participants

and

Results Sheet

C1.1

PROFICIENCY TESTING AUSTRALIA

FOOD PROFICIENCY TESTING PROGRAM

ROUND 43, JULY 2019

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. Two samples of chicken liver paste (each approximately 110 g), labelled PTA 1 and PTA 2, have been provided for compositional analysis. These samples are provided in plastic vacuum sealed slices, wrapped in aluminium foil. Keep the samples refrigerated in the aluminium foil wrapping until ready to test. The samples may be tested for some, or all of the following tests, according to each laboratory's requirements.

2. The tests to be performed in this program are:

   - Protein
   - Fat
   - Moisture
   - Ash
   - Salt (as Sodium Chloride)
   - Carbohydrate†
   - Energy††

   Notes: † determined by difference.
   †† determined by calculation.

   PLEASE ENSURE A WELL-MIXED REPRESENTATIVE SUB-SAMPLE IS REMOVED FOR TESTING

3. The tests may commence as soon as samples are received. Analysts should be aware of analyte stability and perform tests in an appropriate order. The conversion factor to be used for reporting protein is N × 6.25.

4. Tests are to be performed on each sample in **duplicate** and the **results reported** on the ***Results Sheet***.

5. Report results on the attached ***Results Sheet*** to the specified number of decimal places. Results should not be reported as "greater than ...." or "less than ....", as such data cannot be statistically analysed.

6. 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.

7. 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 ≈ 2).

8. Return *Results Sheets*, either by mail, facsimile or email to:

Mark Bunt
Proficiency Testing Australia
PO Box 7507
Silverwater NSW 2128
AUSTRALIA

Telephone: +61 2 9736 8397 (1300 782 867)
Fax: +61 2 9743 6664
Email: mbunt@pta.asn.au

All results should arrive at the above address by no later than **Monday 5 August 2019**. Results reported later than this date may not be analysed in the final report.

C1.2

PROFICIENCY TESTING AUSTRALIA

FOOD PROFICIENCY TESTING PROGRAM

ROUND 43, JULY 2019

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
Salt (g/100g)	Volhard titration	1
	ISE	2
	Other (please specify)	3

NOTE:

Salt expressed as sodium chloride.

C2.1

PROFICIENCY TESTING AUSTRALIA

FOOD PROFICIENCY TESTING PROGRAM

ROUND 43, JULY 2019

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 (±) r	MU (±) R	Result 1	Result 2	MU (±) r	MU (±) R		
Protein (Nx6.25) (g/100g 1 d.p.)										
Fat (g/100g 1 d.p.)										
Moisture (g/100g 1 d.p.)										
Ash (g/100g 2 d.p.)										
Salt (g/100g 2 d.p.)										
Carbohydrate* (g/100g 1 d.p.)										
Energy** (kJ/100g)										

* Carbohydrate = 100 – (%Protein + %Fat + %Moisture + %Ash).

** Energy = (%Protein x 17) + (%Carbohydrate x 17) + (%Fat x 37).

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

Please specify the temperature/time of ashing: _____^{o}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 -----