Report No. 1179

Gravimetric

Proficiency Testing Program

Round Six

March 2020

ACKNOWLEDGMENTS

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Ms L Apthorpe, Hibbs & Associates Pty Ltd. PTA also wishes to gratefully acknowledge the calibration of the aluminium weights by National Measurement Institute, Australia.

PO Box 7507, Silverwater NSW 2128, Australia

CONTENTS

1. FOREWORD

This report summarises the results of the interlaboratory comparison program Gravimetric Round Six.

The exercise was conducted during the period January 2019 to January 2020 by Proficiency Testing Australia (PTA). The Program Coordinator was Dr M Li. The Program Technical Adviser was Ms L Apthorpe, Hibbs & Associates Pty Ltd. This report was authorised by Mrs K Cividin, PTA Quality Manager. The main aim of the program was to assess laboratories' ability to competently determine the weight of two aluminium weights (nominally 5 mg and 20 mg respectively).

2. STATISTICAL DESIGN OF THE PROGRAM

Four sets of weights (PTA 1, PTA 2, PTA 3 and PTA 4) were distributed amongst the 26 participating laboratories. Each set consisted of two aluminium weights, one nominally 5mg, and the other nominally 20mg. All laboratories except one returned results for inclusion in the report.

The program was based on a measurement comparison design, where the two aluminium weights were sequentially distributed around the 26 participants. Four sets of weights were used, to reduce the time required to cover all 26 laboratories. The National Measurement Institute acted as the reference laboratory, deriving the reference values at the beginning and the end of the program.

A summary of results returned by the participating laboratories, compared to the reference values, appears in Appendix A1 to A4. Measurement performance is judged on the basis of an E_n number for each measurement. The E_n number is an internationally accepted method for determining the agreement of individual results with the reference values in relation to the uncertainties of measurement of each. uncertainty of measurement of the reference value. The E_n ratio is defined as:

$$E_n = \frac{Lab\,Result - Ref\,Value}{\sqrt{(U_{95}Lab)^2 + (U_{95}Ref)^2}}$$

That is, the E_n number indicates whether laboratories are within their particular where U_{95} is the reported uncertainty of measurement at a 95% confidence level. For the results to be acceptable, values of $|E_n| \leq 1.0$ are required.

3. FEATURES OF THE PROGRAM

(a) A total of 26 laboratories received samples. The set of PTA 1 was distributed to participants with laboratory codes 1, 3, 8, 12 and 22; the set of PTA 2 was distributed to participants with laboratory codes 2, 7, 11, 14, 21 and 22; the set of PTA 3 was distributed to participants with laboratory codes 5, 9, 13, 16, 17, 19 and

24; and the set of PTA 4 was distributed to participants with laboratory codes 4, 6, 10, 15, 18, 20, 25 and 26.

(b) Participants were supplied two aluminium weights, one nominally 5 mg, the other nominally 20 mg.

(c) Prior to sample distribution, and again at the conclusion of the round, all weights were calibrated by the National Measurement Institute.

(d) The results for each sample set as reported by participants are presented in Appendix A, together with calculated E_n numbers (A1-A4) and graphical presentations of the data (A5).

(e) Participating laboratories were requested to perform the tests according to the "Instructions to Participants", and to record their results on the accompanying "Results Sheet", all of which were distributed to laboratories with the weights. Copies of the "Instructions to Participants" and "Results Sheet" are given in Appendix B of this report.

(f) Each laboratory was randomly allocated a unique code number for the program to ensure confidentiality of results. Reference to each laboratory in this report is by its code number. Please note that some laboratories reported more than one set of results, therefore, one code number (with letter) could appear several times in the same date set.

4. FORMAT OF APPENDICES

Appendix A

For each sample set, the following information is provided for each sample.

(i) The weight determination (in mg) as reported by participating laboratories, together with their reported uncertainties of measurement at the 95% confidence level (U_{95}).

(ii) The reference value is mg.

(iii) The calculated E_n number for each participant's weight determinations.

(iv) A graph for each sample, displaying each participant's value, represented by a black diamond, together with their reported uncertainty of measurement, represented by bars extending above and below its value (see Appendix A5).

(v) Table A6 contains additional information provided by participants in relation to the microbalances used.

Appendix B

(i) Instructions to Participants

(ii) Results Sheet

5. OUTLIER RESULTS

In order to achieve the program aim of assessing laboratory testing performance, E_n numbers have been calculated for each participant's weight determination. The E_n number indicates whether laboratories are within their particular uncertainty of measurement of the reference value. For the result to be considered acceptable, the E_n number must lie between -1.0 and +1.0 (i.e. $|E_n| \leq 1.0$).

6. PTA AND TECHNICAL ADVISER'S COMMENTS

Background Information

Australian Standards AS 2985-2009[2], AS 3640-2009[3] and AS 3853.1-2006[4] were used as test methods, therefore, results were analysed without being separated into method groups.

This interlaboratory comparison program was designed to assist laboratories that perform the determination of inhalable/respirable dust, welding fumes and gases according to the following Australian Standards: AS 2985-2009[2], AS 3640-2009[3] and AS 3853.1-2006[4].

It is acknowledged that humidity and electrostatic charges are a major source of uncertainty in these analyses, and considerable thought was put into trying to include these factors in the scheme. These factors, however, could not be included in such a program, and can only be addressed by appropriate analytical methodology, correct laboratory practice and subsequent laboratory assessments by accreditation bodies.

Informal round robins conducted in Australia and UK some years ago indicated that some laboratories using calibrated weights and calibrated balances obtained grossly incorrect answers. Additionally, laboratory technique can provide further random and/or systematic errors. The present program is the formal attempt to use a simple test for the above errors, and then to assist laboratories reporting outliers to overcome their problems.

Uncertainties of Measurement (U_{95})

The uncertainties of measurement reported by some laboratories appear too small. Conversely, the U_{95} values reported by some laboratories appear too large. These laboratories should review their uncertainty estimates to provide realistic values.

A number of laboratories did not report the range of the microbalance used as requested by PTA. The microbalance range should be provided to allow a comprehensive review to occur.

It should be noted that some laboratories who reported large uncertainties have been classed as satisfactory in terms of their E_n number, when in fact they may have been identified as an outlier had they reported a more appropriate uncertainty. Alternatively, participants reporting small uncertainties may have falsely excluded themselves from being classed as satisfactory. This should be taken into account when assessing individual laboratory performance in this program.

Possible Sources of Error

The use of five place balances is a major source of error in this program, due to the problem of conforming with the current Australian standard balance requirements. Weight calibration certificates may also be another source of error, as some are issued with very large and inappropriate uncertainties that should not be applied to microbalances.

7. REFERENCES

[1] *Guide to Proficiency Testing Australia (2019).* (This document is located on the PTA website at www.pta.asn.au).

[2] AS 2985-2009: '*Workplace atmospheres - Method for sampling and gravimetric determination of respirable dust'.*

[3] AS 3640-2009: *'Workplace atmospheres – Method for sampling and gravimetric determination of inhalable dust'.*

[4] AS 3853.1-2006: *'Health and safety in welding and allied processes - Sampling of airborne particles and gases in the operator's breathing zone - Sampling of airborne particles'.*

APPENDIX A

Summary of Results

Appendix A1

SAMPLES PTA 1-05 and PTA 1-20

Lab	Nominated	Nominal 5 mg (PTA 1-05)					Nominal 20 mg (PTA 1-20)				
Code	5/6 place	Test Items	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n	Test Items	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n
1A	**6**	PTA 1	5.187	0.021	0.001	0.04	PTA 1	19.752	0.020	-0.001	-0.06
1B	**6**	PTA 1	5.187	0.000015	0.001	0.15	PTA 1	19.751	0.000015	-0.002	-0.34
1C	**6**	PTA 1	5.187	0.048	0.001	0.02	PTA 1	19.751	0.075	-0.002	-0.03
1D	**6**	PTA 1	5.187	0.021	0.001	0.04	PTA 1	19.751	0.020	-0.002	-0.10
1E	**6**	PTA 1	5.188	0.021	0.002	0.09	PTA 1	19.753	0.020	-0.0002	-0.01
1F	**6**	PTA 1	5.187	0.021	0.001	0.04	PTA 1	19.751	0.020	-0.002	-0.10
3	**5**	PTA 1	5.19	0.029	0.004	0.13	PTA 1	19.75	0.029	-0.003	-0.11
8	**6**	PTA 1	5.188	0.010	0.002	0.16	PTA 1	19.751	0.010	-0.002	-0.18
12	**6**	PTA 1	5.19	0.012	0.004	0.29	PTA 1	19.76	0.012	0.007	0.50
22	**6**	PTA 1	5.186	0.006	0.000	-0.01	PTA 1	19.751	0.006	-0.002	-0.25
	Reference Lab Initial:		5.187	0.010				19.755	0.010		
	Reference Lab Final:		5.185	0.003				19.751	0.003		
	Average:		***5.186***	***0.006***				***19.753***	***0.007***		

NOTES:

1 The current Australian Standards now accept 5 and 6 place balances which give very different uncertainties. These different uncertainties must be taken into account in terms of the significance of results generated by each class of balance.

2 E_n refers to Error Normalised. An E_n number between -1.0 and +1.0 is considered acceptable (i.e $|E_n| < 1.0$).

3 U_{95} refers to uncertainty of measurement at the 95% confidence level.

4 Weight and U_{95} in the above table are shown 'as reported' by participants.

5 LAB - REF refers to Participating Laboratory's result minus the Reference Laboratory's result.

6 Where a laboratory submitted results for more than one analyst, an 'A', 'B' etc has been added to the numerical code to denote the different analyst.

7 The expanded uncertainty of measurement for the Reference Laboratory takes into account the variation between the initial and final calibration, and represents the true uncertainty of measurement at the 95% confidence level.

Appendix A2

SAMPLES PTA 2-05 and PTA 2-20

Lab	Nominated	Nominal 5 mg (PTA 2-05)					Nominal 20 mg (PTA 2-20)				
Code	5/6 place	Test Items	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n	Test Items	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n
2	**5**	PTA 2	5.24	0.014	-0.001	-0.07	PTA 2	19.51	0.015	-0.002	-0.12
7	**6**	PTA 2	5.241	0.010	0.000	-0.01	PTA 2	19.513	0.010	0.001	0.09
11	**5**	PTA 2	5.25	0.05	0.009	0.18	PTA 2	19.53	0.03	0.018	0.59
14	**6**	PTA 2	5.240	0.009	-0.001	-0.10	PTA 2	19.514	0.004	0.002	0.28
21	**5**	PTA 2	5.25	0.01	0.009	0.75	PTA 2	19.52	0.01	0.008	0.68
23	**5**	PTA 2	5.24	0.03	-0.001	-0.04	PTA 2	19.50	0.03	-0.012	-0.39
	Reference Lab Initial:		5.240	0.010				19.512	0.010		
	Reference Lab Final:		5.242	0.003				19.512	0.003		
	Average:		***5.241***	***0.006***				***19.512***	***0.007***		

NOTES:

1 The current Australian Standards now accept 5 and 6 place balances which give very different uncertainties. These different uncertainties must be taken into account in terms of the significance of results generated by each class of balance.

2 E_n refers to Error Normalised. An E_n number between -1.0 and +1.0 is considered acceptable (i.e $|E_n| < 1.0$).

3 U_{95} refers to uncertainty of measurement at the 95% confidence level.

4 Weight and U_{95} in the above table are shown 'as reported' by participants.

5 LAB - REF refers to Participating Laboratory's result minus the Reference Laboratory's result.

6 Where a laboratory submitted results for more than one analyst, an 'A', 'B' etc has been added to the numerical code to denote the different analyst.

7 The expanded uncertainty of measurement for the Reference Laboratory takes into account the variation between the initial and final calibration, and represents the true uncertainty of measurement at the 95% confidence level.

Appendix A3

SAMPLES PTA 3-05 and PTA 3-20

Lab Code	Nominated 5/6 place	Nominal 5 mg (PTA 3-05)					Nominal 20 mg (PTA 3-20)				
		Test Items	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n	Test Items	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n
5	**6**	PTA 3	5.200	0.05	0.0009	0.02	PTA 3	19.881	0.05	0.0012	0.02
9	**6**	PTA 3	5.201	0.010	0.0019	0.16	PTA 3	19.880	0.010	0.0002	0.02
13	**6**	PTA 3	5.201	0.004	0.0019	0.25	PTA 3	19.880	0.003	0.0002	0.03
16	**6**	PTA 3	5.201	0.008	0.0019	0.18	PTA 3	19.880	0.008	0.0002	0.02
17	**6**	PTA 3	5.1999	0.0022	0.0008	0.11	PTA 3	19.8789	0.0023	-0.0009	-0.13
19	**5**	PTA 3	5.19	0.26	-0.0091	-0.04	PTA 3	19.88	0.99	0.0002	0.00
24	**5**	PTA 3	5.21	0.015	0.0109	0.67	PTA 3	19.92	0.003	0.0402	5.58
	Reference Lab Initial:		5.199	0.010				19.881	0.010		
	Reference Lab Final:		5.199	0.003				19.879	0.003		
	Average:		***5.199***	***0.006***				***19.880***	***0.007***		

NOTES:

1 The current Australian Standards now accept 5 and 6 place balances which give very different uncertainties. These different uncertainties must be taken into account in terms of the significance of results generated by each class of balance.

2 E_n refers to Error Normalised. An E_n number between -1.0 and +1.0 is considered acceptable (i.e $|E_n| < 1.0$).

3 U_{95} refers to uncertainty of measurement at the 95% confidence level.

4 Weight and U_{95} in the above table are shown 'as reported' by participants.

5 LAB - REF refers to Participating Laboratory's result minus the Reference Laboratory's result.

6 Where a laboratory submitted results for more than one analyst, an 'A', 'B' etc has been added to the numerical code to denote the different analyst.

7 The expanded uncertainty of measurement for the Reference Laboratory takes into account the variation between the initial and final calibration, and represents the true uncertainty of measurement at the 95% confidence level.

Appendix A4

SAMPLES PTA 4-05 and PTA 4-20

Lab Code	Nominated 5/6 place	Nominal 5 mg (PTA 4-05)					Nominal 20 mg (PTA 4-20)				
		Test Items	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n	Test Items	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n
4	**5**	PTA 4	5.23	0.020	-0.0079	-0.35	PTA 4	19.54	0.020	0.004	0.19
6	**6**	PTA 4	5.239	0.010	0.0011	0.08	PTA 4	19.540	0.010	0.004	0.34
10	**6**	PTA 4	5.238	0.005	0.0001	0.01	PTA 4	19.539	0.009	0.003	0.27
15A	**6**	PTA 4	5.239	0.0006	0.0011	0.11	PTA 4	19.541	0.0006	0.005	0.77
15B	**6**	PTA 4	5.239	0.0004	0.0011	0.11	PTA 4	19.540	0.0014	0.004	0.60
18	**6**	PTA 4	5.25	0.02	0.0121	0.54	PTA 4	19.55	0.02	0.014	0.67
20	**5**	PTA 4	5.26	0.05	0.0221	0.43	PTA 4	19.51	0.04	-0.026	-0.64
25A	**5**	PTA 4	5.25	0.018	0.0121	0.59	PTA 4	19.55	0.018	0.014	0.73
25B	**5**	PTA 4	5.24	0.018	0.0021	0.10	PTA 4	19.55	0.018	0.014	0.73
26	**5**	PTA 4	5.25	0.11	0.0121	0.11	PTA 4	19.54	0.11	0.004	0.04
	Reference Lab Initial:		5.238	0.010				19.535	0.010		
	Reference Lab Final:		5.238	0.010				19.537	0.003		
	Average:		***5.238***	***0.010***				***19.536***	***0.007***		

NOTES:

1 The current Australian Standards now accept 5 and 6 place balances which give very different uncertainties. These different uncertainties must be taken into account in terms of the significance of results generated by each class of balance.

2 E_n refers to Error Normalised. An E_n number between -1.0 and +1.0 is considered acceptable (i.e $|E_n| < 1.0$).

3 U_{95} refers to uncertainty of measurement at the 95% confidence level.

4 Weight and U_{95} in the above table are shown 'as reported' by participants.

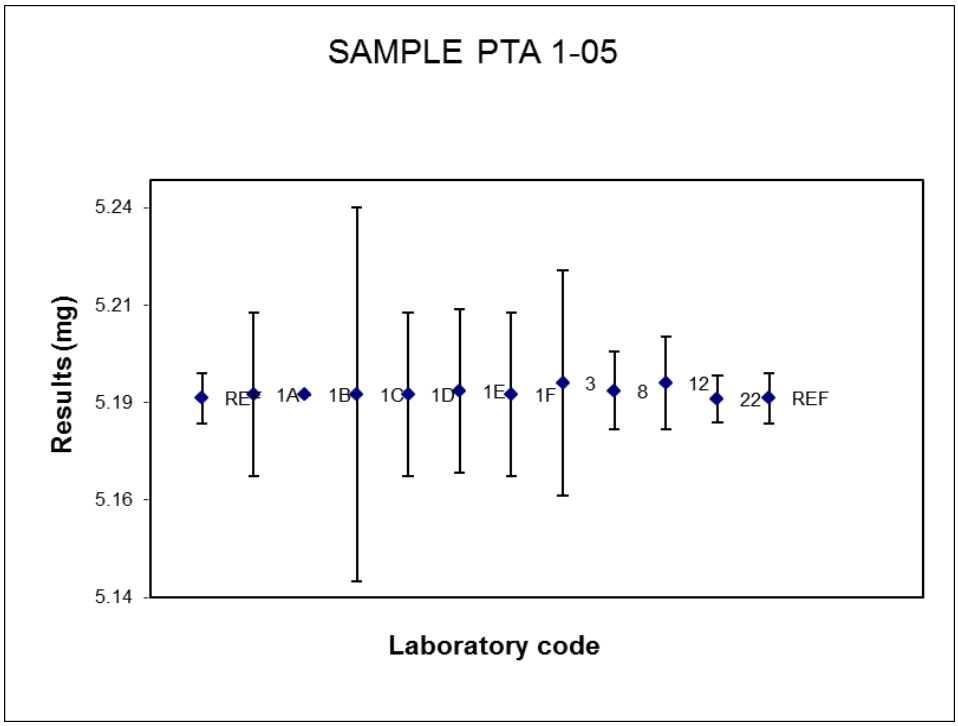
5 LAB - REF refers to Participating Laboratory's result minus the Reference Laboratory's result.

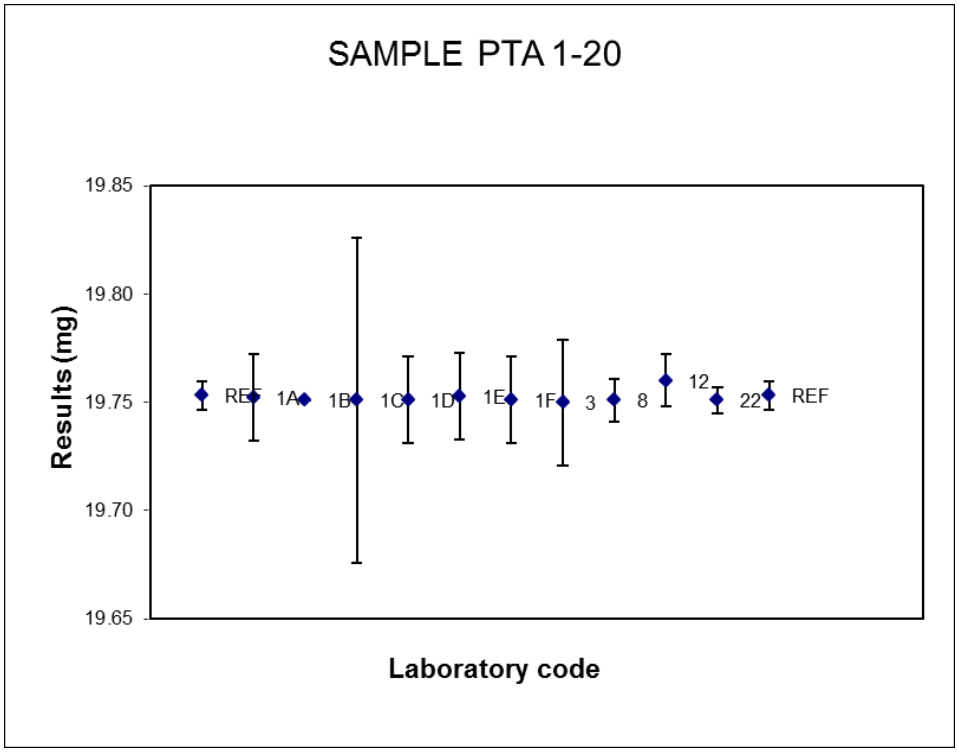
6 Where a laboratory submitted results for more than one analyst, an 'A', 'B' etc has been added to the numerical code to denote the different analyst.

7 The expanded uncertainty of measurement for the Reference Laboratory takes into account the variation between the initial and final calibration, and represents the true uncertainty of measurement at the 95% confidence level.

Appendix A5

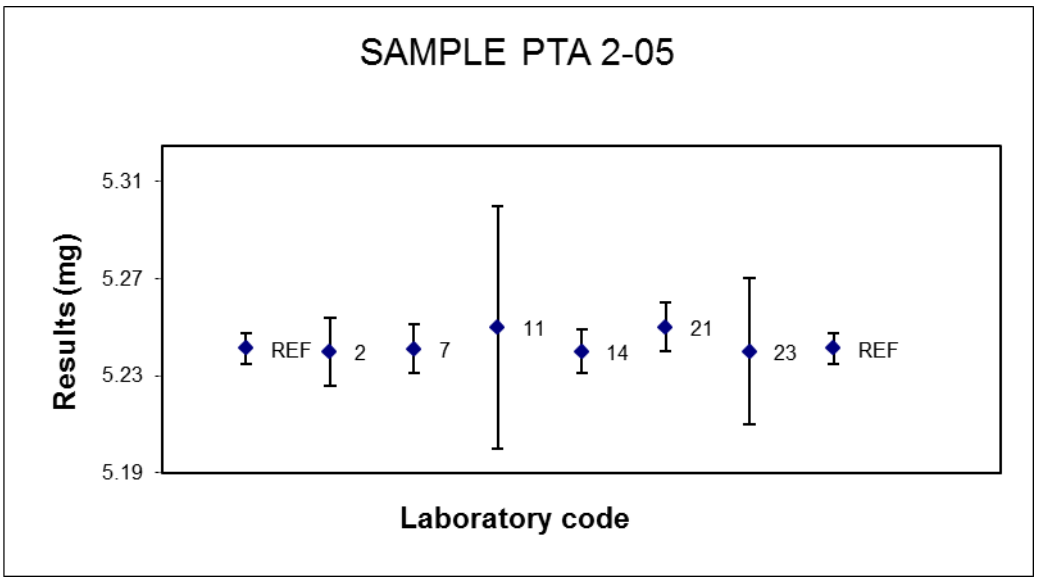
Graphical Presentation of Participants' Results

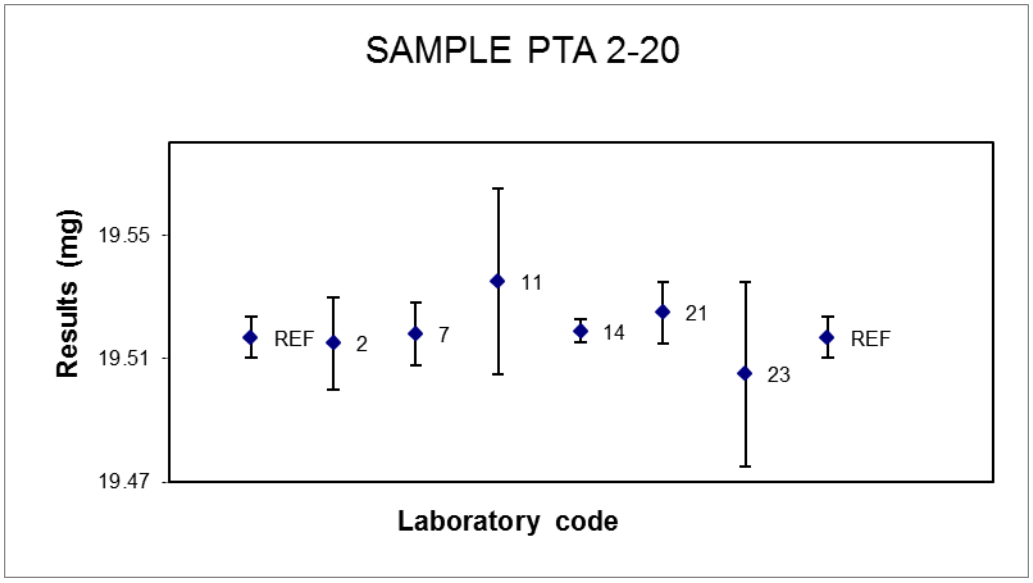




Appendix A5

Graphical Presentation of Participants' Results

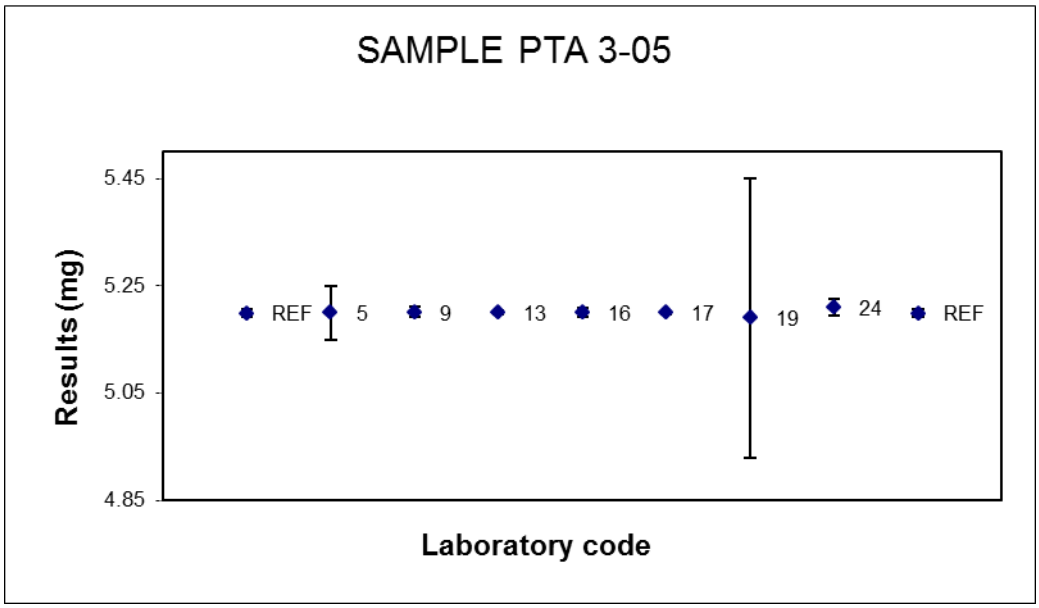




A5.3

Appendix A5

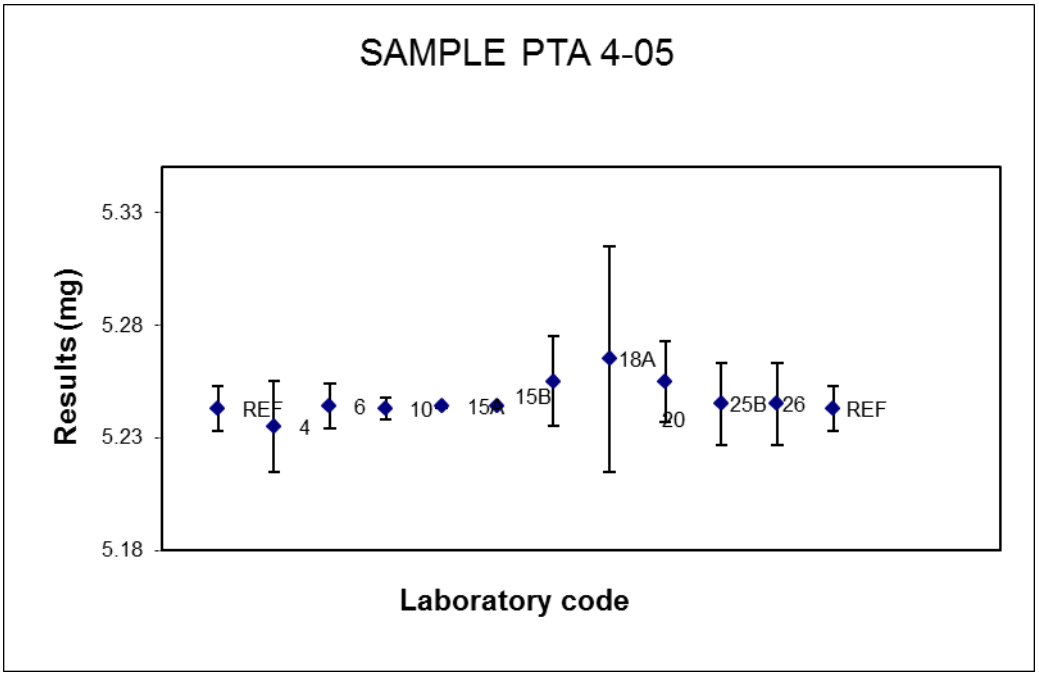
Graphical Presentation of Participants' Results



A5.4

Appendix A5

Graphical Presentation of Participants' Results



Appendix A6

ADDITIONAL INFORMATION
(Microbalances used by Participants)

Lab Code	Microbalance		
	Brand	Model	Range
1A	Mettler Toledo	MX5	0.5-5.1
1B	Mettler Toledo	MX5	5
1C	Mettler Toledo	MX5	0.5-5.1
1D	Mettler Toledo	MX5	5.1
1E	Mettler Toledo	MX5	5.1g
1F	Mettler Toledo	MX5	5
2	Mettler Toledo	XS105DU	2
3	Mettler Toledo	XSE105	Range 2 (max) 41g
4	Mettler Toledo	X5 105 Dual Range	MAX 41g
5	Mettler Toledo	MX5	0.000-5.000 mg
6	Mettler Toledo	MX5	0
7	Mettler Toledo	XP6	0-6.1
8	Mettler Toledo	XP6	0-500mg
9	Mettler Toledo	XP6	0-6.1g
10	Mettler Toledo	XP6	
11	AND	GH-252	100g, resolution 0.00001g
13	Mettler Toledo	MX5	0-5g
14	Mettler Toledo	XP6	0-6g
15A	Rdw ag In, Micro Balance	MYA 5.4 Y.F.B	0.001mg-5.1g accurancy: 1x10-6mg
15B	Rdw ag In, Micro Balance	MYA 5.4 Y.F.B	0.001mg-5.1g accurancy: 1x10-6mg
16	Mettler Toledo	MX5	6 dec place
17	Mettler Toledo	XP2U	0-2.1g
18	AND	GR 202	1-210g
19	Mettler Toledo	AG135	0
20	Shimadzu	AUW120D	Range A
21	OHAVS Analytical Plus	AP250D-0	0
22	Mettler Toledo	XPR6UD5	0-100mg
23	AND	GH-252	100g
24	Mettler Toledo	XS205	5 places
25A	Mettler Toledo	X5205DU	0.0005g
25B	Mettler Toledo	X5205DU	0.0005g
26	Mettler Toledo	XSP5DU	41g-120g

NOTE: Where a laboratory submitted results for more than one analyst, an 'A', 'B' etc has been added to the numerical code to denote the different analyst.

APPENDIX B

Instructions to Participants

and

Results Sheet

PROFICIENCY TESTING AUSTRALIA
Proficiency Testing Program

Gravimetric (Round 6)

INSTRUCTIONS TO PARTICIPANTS

Please read instructions carefully **BEFORE** commencing testing. To ensure that the results of this program can be analysed properly, participants are asked to carefully note the following:

1 Enclosed are two aluminium weights in separate labelled containers. They are nominally 5mg and 20 mg respectively. Plastic tweezers are also enclosed.

2 Each participant is requested to do the following:

(i) Determine the weight of each specimen using your laboratory's routine procedures (omit those parts of the procedures that relate to the use of blank filters) to your best measurement capability.

(ii) If using a five place balance, report to five places. If using a six place balance, report to six places.

(iii) Record your observations into your own laboratory system, and also on the attached Results Sheet.

(iv) The Uncertainty of Measurement (at 95% confidence level, coverage factor k≈2.) *must* also be reported for each specimen.

(v) If there is more than one analyst in your laboratory registered to participate, each analyst is asked to submit results on the separate copies of the Results Sheet.

3 IMPORTANT NOTES

(i) Use **only** the supplied plastic tweezers to handle the weights.

(ii) Under **no** circumstances handle the weights with metal tweezers, or by hand.

(iii) Do **not** use the weights in a 'dirty' environment.

(iv) Only have one weight out of its container at any one time (to avoid mix-up).

(v) Return results sheet, the weights and tweezers to PTA properly packaged **by <date >** to:

Dr Michael Li, Proficiency Testing Australia.
PO Box 7507, Silverwater NSW 2128 Australia.
Phone: +61 2 9736 8397 Fax: +61 2 9743 6664
Email: michael.li@pta.asn.au

PROFICIENCY TESTING AUSTRALIA

Gravimetric Round 6

Results Sheet

Lab Code:

Sample Identification	Weight (mg)	Uncertainty (U_{95})* (mg)
Nominal 5 mg		
Nominal 20 mg		

*Note: U_{95} refers to your laboratory's uncertainty of measurement at the 95% confidence level.

Analyst's Name: _____

Date analysed: _____

Microbalance Brand: _____

Microbalance Model: _____

Microbalance Range used: _____

Signed: _____ **Date:** _____

Return no later than **<date>** to:

Dr Michael Li, Proficiency Testing Australia.
P O Box 7507, Silverwater NSW 2128 Australia.
Phone:+61 2 9736 8397 Fax:+61 2 9743 6664
Email: michael.li@pta.asn.au

---- End of Report ----