Report No. 839

Gravimetric
Proficiency Testing Program

Round Three

January 2014

ACKNOWLEDGMENTS

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Ms L Apthorpe, Pickford & Rhyder Consulting Pty Ltd. This assistance included the design of the program, technical advice and discussion in the final report. PTA also wishes to gratefully acknowledge the calibration of the two 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 Three.

The exercise was conducted during the period March to December 2013 by Proficiency Testing Australia. The Program Coordinator was Dr M Li. The Program Technical Adviser was Ms L Apthorpe, Pickford & Rhyder Consulting Pty Ltd. This report was authorised by Dr M Bunt, Statistician, PTA. 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 22 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 22 participants. Four sets of weights were used, to reduce the time required to cover all 22 laboratories. 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. That is, the E_n number indicates whether laboratories are within their particular uncertainty of measurement of the reference value. The E_n ratio is defined as:

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

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 22 laboratories received samples. The set of PTA 1 was distributed to participants with laboratory codes 1, 5, 9, 13, 16 and 20, the set of PTA 2 was distributed to participants with laboratory codes 2, 6, 10, 14, 19 and 22, the set of PTA 3 was distributed to participants with laboratory codes 3, 8, 11, 15 and 17,

the set of PTA 4 was distributed to participants with laboratory codes 4, 7, 12, 18 and 21.

(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 participating laboratories' results and the reference value in 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. $|E_n| \leq 1.0$). Laboratory code 9 did not report uncertainties, and its E_n numbers are not able to be calculated.

6. PTA AND TECHNICAL ADVISER'S COMMENTS

Background Information

Australian Standards AS2985-2009[2], AS3640-2009[3] and AS3853.1-2006[4] were used as test methods, therefore, results were analysed without method groups.

This inter-laboratory 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: AS2985-2009[2], AS3640-2009[3] and AS3853.1-2006[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.

Laboratory code 9 did not report uncertainties, and its E_n numbers are not able to be calculated. A number of laboratories did not report the range of microbalance 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.

Future Rounds

Stainless steel reference weights may be used in future rounds of this program.

7. REFERENCES

[1] Guide to Proficiency Testing Australia (2012). (This document is located on the PTA website at www.pta.asn.au).

[2] AS2985-2009: *'Workplace atmospheres - Method for sampling and gravimetric determination of respirable dust'.*

[3] AS3640-2009: *'Workplace atmospheres – Method for sampling and gravimetric determination of inhalable dust'.*

[4] AS3853.1-2006: *'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 Code	Nominated 5/6 place	Nominal 5 mg (PTA 1-05)					Nominal 20 mg (PTA 1-20)				
		Sample No.	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n	Sample No.	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n
1A	**5**	PTA 1	5.19	0.01	0.000	-0.04	PTA 1	19.77	0.01	0.012	0.85
1B	**5**	PTA 1	5.19	0.01	0.000	-0.04	PTA 1	19.77	0.01	0.012	0.85
5	**5**	PTA 1	5.20	0.03	0.010	0.31	PTA 1	19.77	0.04	0.012	0.28
9	**5**	PTA 1	5.22		n/a	n/a	PTA 1	19.74		n/a	n/a
13	**5**	PTA 1	5.2	0.01	0.010	0.81	PTA 1	19.77	0.01	0.012	0.85
16	**7**	PTA 1	5.1916	0.0032	0.001	0.16	PTA 1	19.7621	0.0032	0.004	0.38
20	**6**	PTA 1	5.189	0.017	-0.002	-0.08	PTA 1	19.778	0.017	0.020	1.01
	Reference Lab Initial:		5.190	0.007				19.759	0.009		
	Reference Lab Final:		5.191	0.005				19.758	0.009		
	Average:		***5.191***	***0.006***				***19.759***	***0.009***		

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 $|E_n| \leq 1.0$).
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 Code	Nominated 5/6 place	Nominal 5 mg (PTA 2-05)					Nominal 20 mg (PTA 2-20)				
		Sample No.	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n	Sample No.	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n
2	**6**	PTA 2	5.246	0.0023	0.001	0.22	PTA 2	19.519	0.0023	0.000	0.06
6	**5**	PTA 2	5.23	0.05	-0.015	-0.30	PTA 2	19.53	0.05	0.012	0.24
10	**6**	PTA 2	5.2510	0.015	0.006	0.37	PTA 2	19.52	0.015	0.002	0.12
14A	**6**	PTA 2	5.244	0.05	-0.001	-0.02	PTA 2	19.515	0.05	-0.003	-0.06
14B	**6**	PTA 2	5.243	0.05	-0.002	-0.04	PTA 2	19.515	0.05	-0.003	-0.06
19	**5/6**	PTA 2	5.244	0.02	-0.001	-0.05	PTA 2	19.518	0.04	0.000	0.00
22	**5**	PTA 2	5.25	0.22000	0.005	0.02	PTA 2	19.48	0.22000	-0.038	-0.17
	Reference Lab Initial:		5.246	0.007				19.518	0.009		
	Reference Lab Final:		5.244	0.005				19.518	0.007		
	Average:		***5.245***	***0.006***				***19.518***	***0.008***		

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 $|E_n| \leq 1.0$).
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)				
		Sample No.	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n	Sample No.	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n
3	**5**	PTA 3	5.20	0.03	-0.004	-0.13	PTA 3	19.88	0.04	0.000	-0.01
8	**5**	PTA 3	5.21	0.06	0.006	0.10	PTA 3	19.89	0.08	0.010	0.12
11	**6**	PTA 3	5.203	0.009	-0.001	-0.09	PTA 3	19.880	0.009	0.000	-0.04
15	**5**	PTA 3	5.21	0.03	0.006	0.20	PTA 3	19.88	0.05	0.000	-0.01
	Reference Lab Initial:		5.207	0.007				19.881	0.009		
	Reference Lab Final:		5.201	0.005				19.880	0.007		
	Average:		***5.204***	***0.006***				***19.881***	***0.008***		

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 $|E_n| \leq 1.0$).
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

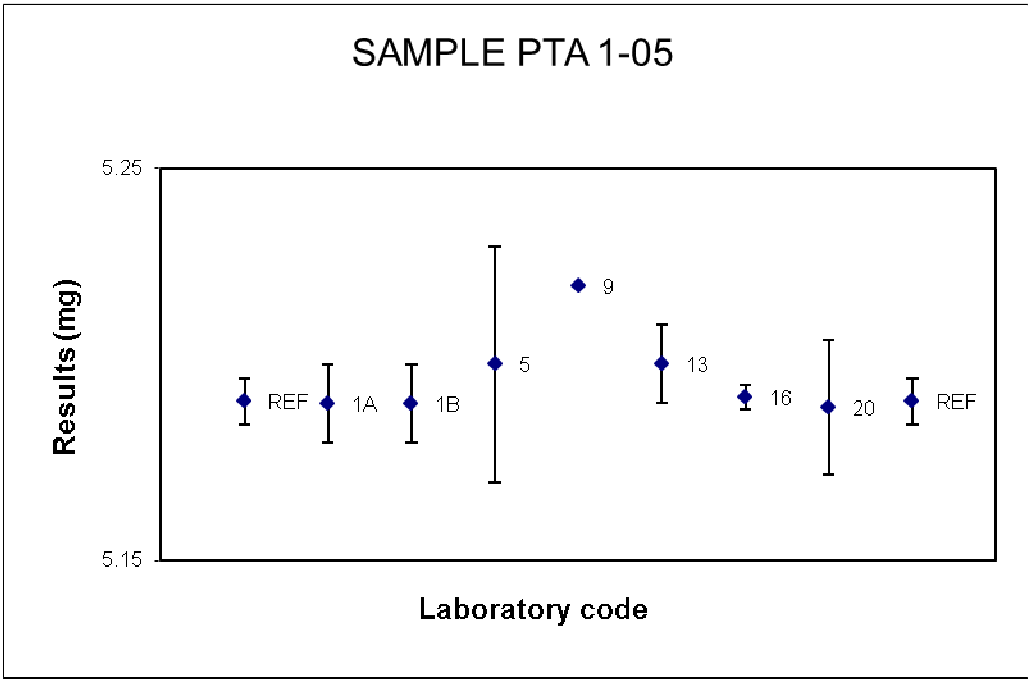
Lab	Nominated	Nominal 5 mg (PTA 4-05)					Nominal 20 mg (PTA 4-20)				
Code	5/6 place	Sample No.	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n	Sample No.	Weight (mg)	U_{95} (mg)	LAB - REF (mg)	E_n
4	**5**	PTA 4	5.2411	0.0009	-0.001	-0.15	PTA 4	19.5399	0.0009	-0.001	-0.07
7	**5**	PTA 4	5.24	0.02	-0.002	-0.10	PTA 4	19.54	0.02	-0.001	-0.02
12	**5**	PTA 4	5.24	0.009	-0.002	-0.18	PTA 4	19.54	0.009	-0.001	-0.04
18	**6**	PTA 4	5.240	0.003	-0.002	-0.30	PTA 4	19.540	0.004	-0.001	-0.06
21A	**6**	PTA 4	5.240	0.002	-0.002	-0.32	PTA 4	19.539	0.002	-0.002	-0.18
21B	**6**	PTA 4	5.239	0.002	-0.003	-0.47	PTA 4	19.539	0.002	-0.002	-0.18
	Reference Lab Initial:		5.245	0.007				19.540	0.009		
	Reference Lab Final:		5.239	0.005				19.541	0.007		
	Average:		***5.242***	***0.006***				***19.541***	***0.008***		

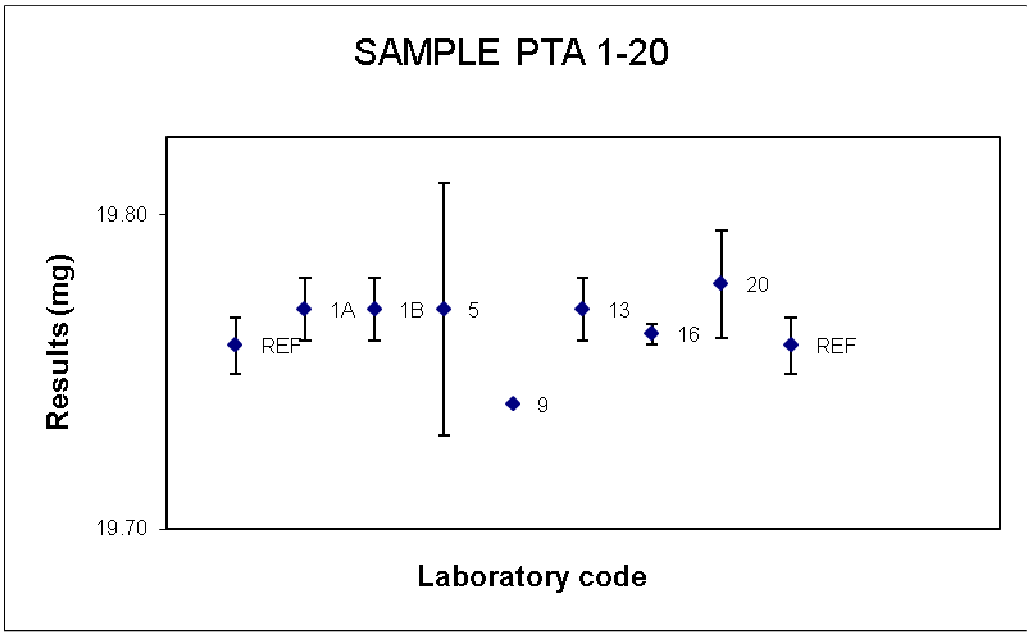
NOTES:

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 $|E_n| \leq 1.0$).
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 Participant's Results





NOTE: Laboratory codes 1a, 1b, 5, 9 and 13 nominated 5 place microbalances, laboratory code 20 nominated 6 place microbalances, and laboratory code 20 nominated 7 place microbalances. Laboratory code 9 did not reported uncertainties.

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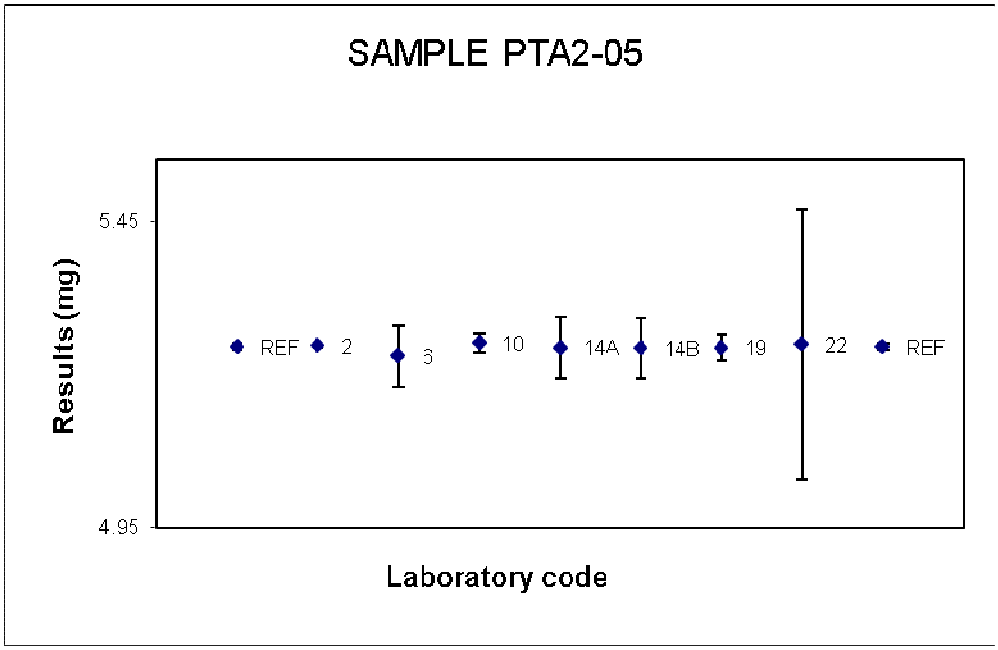
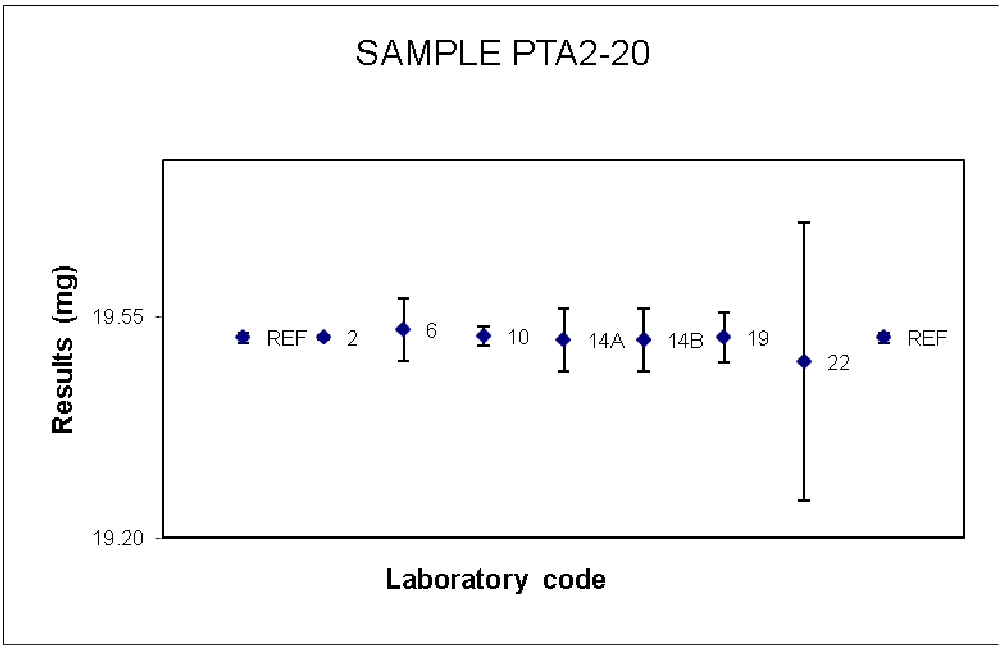
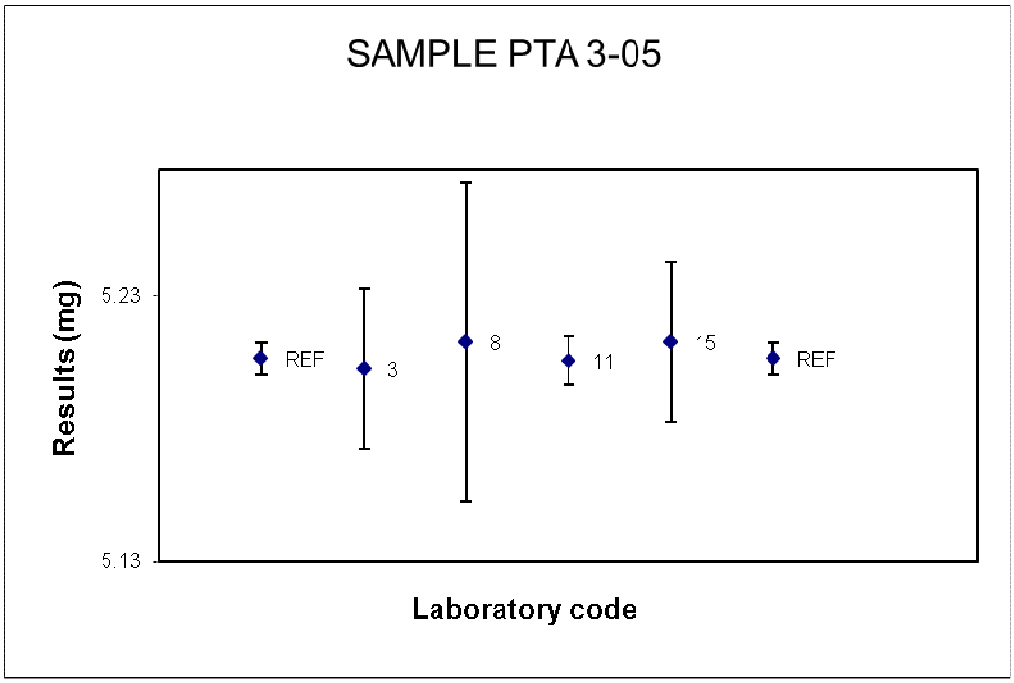
Graphical Presentation of Participant's Results

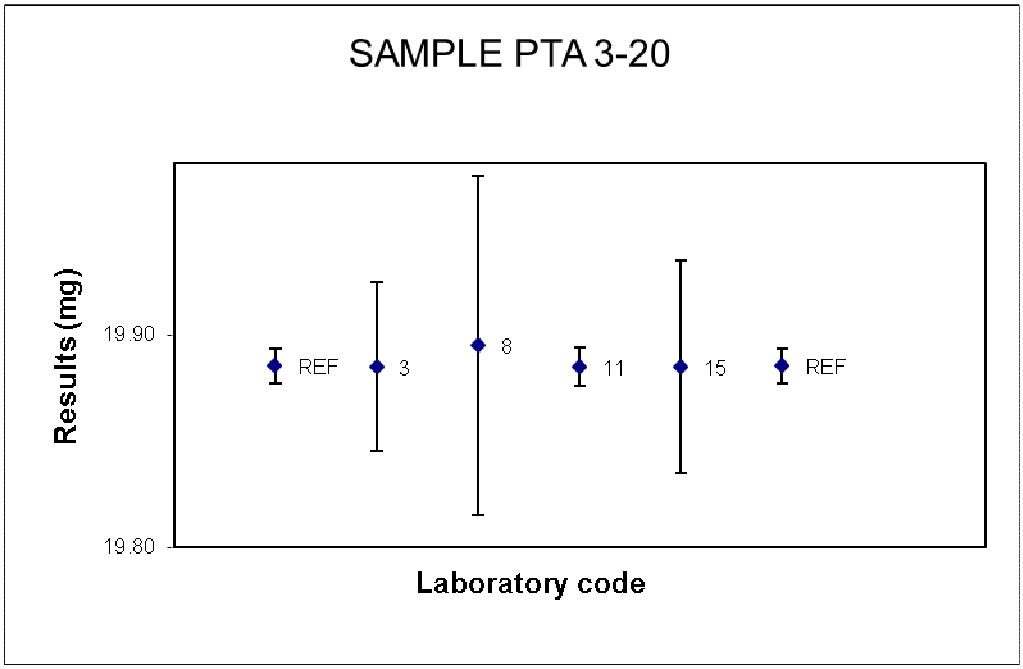




NOTE: Laboratory codes 6 and 22 nominated 5 place microbalances, laboratory code 19 nominated both 5 and 6 place microbalances, and laboratory codes 2, 10, 14A and 14B nominated 6 place microbalances.

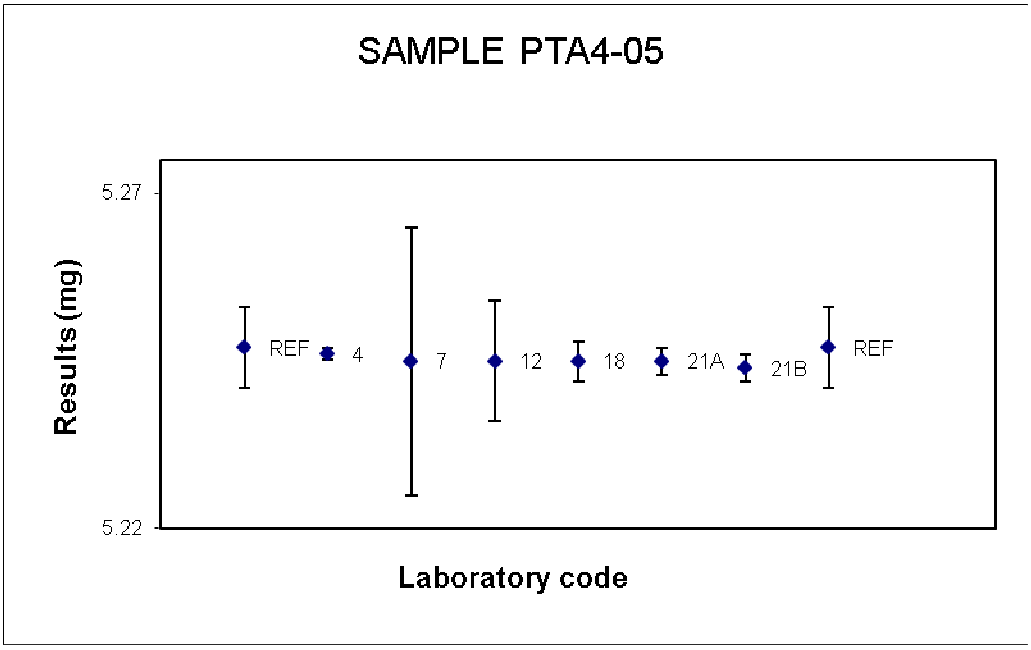
Graphical Presentation of Participant's Results

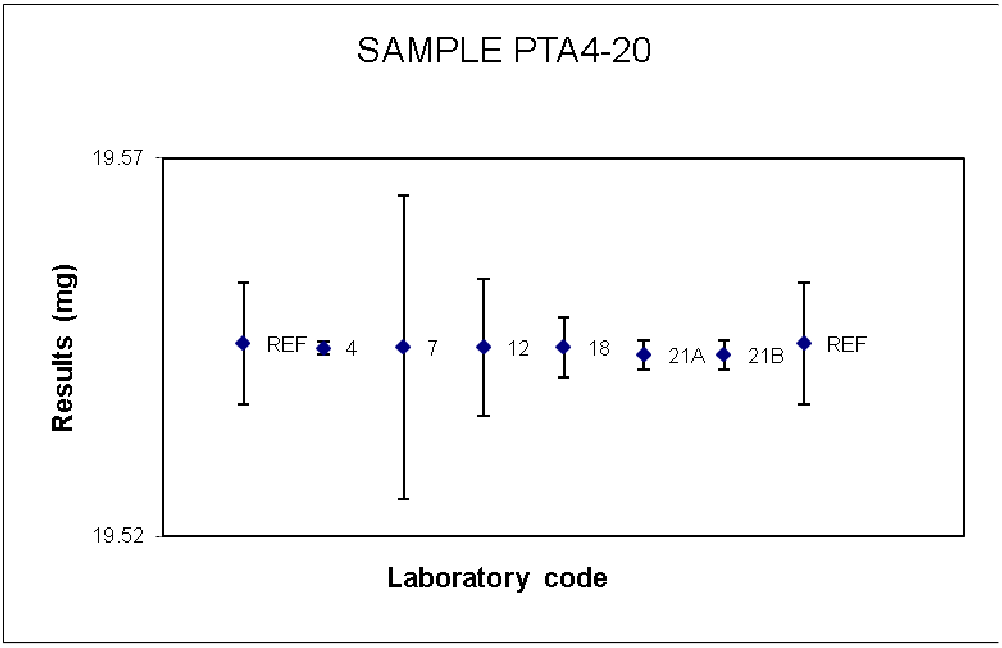




NOTES: Laboratory codes 3, 8 and 15 nominated 5 place microbalances, and laboratory code 11 nominated 6 place microbalances.

Graphical Presentation of Participant's Results





NOTE: Laboratory codes 4, 7 and 12 nominated 5 place microbalances, and laboratory codes 18, 21A and 21B nominated 6 place microbalances.

Appendix A6

ADDITIONAL INFORMATION
(As Reported by Participants)

Lab Code	Microbalance		
	Brand	Model	Range
1A	AND	GR-202	Fine
1B	AND	GR-202	Fine
2	Mettler Toledo	XP2U	0-1g
3	Sartorius	CP225D	0.80g
4	Mettler Toledo	XP2U	2g
5	Mettler Toledo	AG 285	0.01mg-41g
6	AND	GR-202	0-40g
7	Ohaus	DV215CD	0-210g
8	Mettler Toledo	AX 205	0.00001g-81g
9	AND	GH-252	
10	Mettler Toledo	MT5	
11	Mettler	MT5	0-5g
12	Mettler Toledo	XS 105 Dual Range	max 41g
13	Mettler Toledo	XS 105 DU	41g
14A	Mettler Toledo	MX5	0.000-5,000mg
14B	Mettler Toledo	MX5	0.000-5,000mg
15	Ohaus	AP250D	0-52 g
16	Mettler	UMX2	0-500mg
18	Mettler	XP6	0-6g
19	Mettler Toledo	MX-5	6 dec place
20	Mettler Toledo	MX5	500mg
21A	Mettler Toledo	XP6	0-6.1g
21B	Mettler Toledo	XP6	0-6.1g
22	Sartorious	MSA 225S	0-220000mg

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 3

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).

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

(iii) The Uncertainty of Measurement (at 95% confidence level) *must* also be reported for each specimen.

(iv) If there is more than one analyst in your laboratory, each analyst is encouraged to participate (without comparison with others), and to submit results on separate copies of the attached Result Sheet.

3 IMPORTANT NOTES

(i) Please 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 result sheets, the weights and tweezers to PTA promptly **(please no later than << >>)**, properly packaged 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 3

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 **<<** **>>** 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 ----