



**Report No. 1073**

**Gravimetric  
Proficiency Testing Program**

**Round Five**

**April 2018**

**ACKNOWLEDGMENTS**

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

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### APPENDIX A

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## 1. **FOREWORD**

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

The exercise was conducted during the period March 2017 to March 2018 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 F Watton, 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 19 participating laboratories. Each set consisted of two aluminium weights, one nominally 5 mg, and the other nominally 20 mg. 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 19 participants. Four sets of weights were used, to reduce the time required to cover all 19 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. 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:

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

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 19 laboratories received samples. The set 'PTA 1' was distributed to participants with laboratory codes 1, 6, 10 and 2B; the set 'PTA 2' was distributed to participants with laboratory codes 2A, 7, 11, 15 and 19; the set 'PTA 3' was distributed to participants with laboratory codes 3A, 3B, 3C, 5, 8, 16 and 17; and

the set 'PTA 4' was distributed to participants with laboratory codes 4, 9, 12, 13 and 18.

- (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 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.  $|E_n| \leq 1.0$ ).

## 6. PTA AND TECHNICAL ADVISER'S COMMENTS

### **Background Information**

Australian Standards AS 2985-2009<sup>[2]</sup>, AS 3640-2009<sup>[3]</sup> and AS 3853.1-2006<sup>[4]</sup> 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 gravimetric determination of inhalable/respirable dust, and welding fumes according to the following Australian Standards: AS 2985-2009<sup>[2]</sup>, AS 3640-2009<sup>[3]</sup> and AS 3853.1-2006<sup>[4]</sup>.

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 (2016)*. (This document is located on the PTA website at [www.pta.asn.au](http://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

Results for Set PTA 1	A1
Results for Set PTA 2	A2
Results for Set PTA 3	A3
Results for Set PTA 4	A4
Graphical Presentation of Participants' Results	A5
Additional Information Provided	A6

## 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)				
		Test Items	Weight (mg)	U <sub>95</sub> (mg)	LAB - REF (mg)	E <sub>n</sub>	Test Items	Weight (mg)	U <sub>95</sub> (mg)	LAB - REF (mg)	E <sub>n</sub>
<b>1</b>	<b>6</b>	PTA 1	5.187	0.008	0.000	0.00	PTA 1	19.755	0.008	0.000	0.04
<b>6</b>	<b>5</b>	PTA 1	5.189	0.05	0.002	0.04	PTA 2	19.752	0.05	-0.003	-0.05
<b>10</b>	<b>5</b>	PTA 1	5.18	0.014	-0.007	-0.43	PTA 1	19.74	0.015	-0.015	-0.84
<b>2B</b>	<b>5</b>	PTA 1	5.19	0.029	0.003	0.10	PTA 1	19.76	0.029	0.006	0.18

**Reference Lab Initial:** 5.187 0.007 19.754 0.007

**Reference Lab Final:** 5.187 0.010 19.755 0.010

**Average:** **5.187 0.009 19.755 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<sub>n</sub> refers to Error Normalised. An E<sub>n</sub> number between -1.0 and +1.0 is considered acceptable (i.e |E<sub>n</sub>| < 1.0).
- 3 U<sub>95</sub> refers to uncertainty of measurement at the 95% confidence level.
- 4 Weight and U<sub>95</sub> 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)				
		Test Items	Weight (mg)	U <sub>95</sub> (mg)	LAB - REF (mg)	E <sub>n</sub>	Test Items	Weight (mg)	U <sub>95</sub> (mg)	LAB - REF (mg)	E <sub>n</sub>
<b>2A</b>	<b>5</b>	PTA 2	5.25	0.012	0.01	0.65	PTA 2	19.51	0.012	-0.003	-0.17
<b>7</b>	<b>5</b>	PTA 2	5.23	0.26	-0.01	-0.04	PTA 2	19.52	0.98	0.007	0.01
<b>11</b>	<b>5</b>	PTA 2	5.24	0.014	0.00	-0.03	PTA 2	19.51	0.015	-0.003	-0.15
<b>15</b>	<b>5</b>	PTA 2	5.22	0.07	-0.02	-0.29	PTA 2	19.58	0.08	0.067	0.84
<b>19</b>	<b>6</b>	PTA 2	5.241	0.006	0.00	0.05	PTA 2	19.513	0.006	0.000	0.05

<b>Reference Lab Initial:</b>	5.241	0.007	19.513	0.007
<b>Reference Lab Final:</b>	5.240	0.010	19.512	0.010
<b>Average:</b>	<b>5.241</b>	<b>0.009</b>	<b>19.513</b>	<b>0.009</b>

#### 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<sub>n</sub> refers to Error Normalised. An E<sub>n</sub> number between -1.0 and +1.0 is considered acceptable (i.e |E<sub>n</sub>| < 1.0).
- 3 U<sub>95</sub> refers to uncertainty of measurement at the 95% confidence level.
- 4 Weight and U<sub>95</sub> 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 <sub>95</sub> (mg)	LAB - REF (mg)	E <sub>n</sub>	Test Items	Weight (mg)	U <sub>95</sub> (mg)	LAB - REF (mg)	E <sub>n</sub>
<b>3A</b>	<b>5</b>	PTA 3	5.20	0.000015	0.0005	0.06	PTA 3	19.88	0.000015	-0.0010	-0.12
<b>3B</b>	<b>5</b>	PTA 3	5.18	0.014	-0.0195	<b>-1.19</b>	PTA 3	19.88	0.014	-0.0010	-0.06
<b>3C</b>	<b>5</b>	PTA 3	5.22	0.015	0.0205	<b>1.19</b>	PTA 3	19.88	0.015	-0.0010	-0.06
<b>5</b>	<b>6</b>	PTA 3	5.202	0.006	0.0025	0.24	PTA 3	19.880	0.006	-0.0010	-0.10
<b>8</b>	<b>6</b>	PTA 3	5.201	0.004	0.0015	0.16	PTA 3	19.881	0.006	0.0000	0.00
<b>16</b>	<b>6</b>	PTA 3	5.2000	0.0012	0.0005	0.06	PTA 3	19.8793	0.0009	-0.0017	-0.20
<b>17</b>	<b>6</b>	PTA 3	5.200740	0.015	0.0012	0.07	PTA 3	19.882606	0.015	0.0016	0.09

<b>Reference Lab Initial:</b>	5.200	0.007	19.881	0.007
<b>Reference Lab Final:</b>	5.199	0.010	19.881	0.010
<b>Average:</b>	<b>5.200</b>	<b>0.009</b>	<b>19.881</b>	<b>0.009</b>

#### 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<sub>n</sub> refers to Error Normalised. An E<sub>n</sub> number between -1.0 and +1.0 is considered acceptable (i.e |E<sub>n</sub>| < 1.0).
- 3 U<sub>95</sub> refers to uncertainty of measurement at the 95% confidence level.
- 4 Weight and U<sub>95</sub> 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 <sub>95</sub> (mg)	LAB - REF (mg)	E <sub>n</sub>	Test Items	Weight (mg)	U <sub>95</sub> (mg)	LAB - REF (mg)	E <sub>n</sub>
<b>4</b>	<b>6</b>	PTA 4	5.239	0.007	0.0010	0.09	PTA 4	19.539	0.007	0.002	0.18
<b>9</b>	<b>6</b>	PTA 4	0.00522	0.02	-5.2328	<b>-240.79</b>	PTA 4	0.01955	0.02	-19.517	<b>-898.13</b>
<b>12</b>	<b>6</b>	PTA 4	5.241	0.006	0.0030	0.29	PTA 4	19.539	0.006	0.002	0.19
<b>13</b>	<b>5</b>	PTA 4	5.22	0.05	-0.0180	-0.35	PTA 4	19.52	0.05	-0.017	-0.34
<b>18</b>	<b>6</b>	PTA 4	5.237	0.015	-0.0010	-0.06	PTA 4	19.538	0.016	0.001	0.06

<b>Reference Lab Initial:</b>	5.238	0.007	19.539	0.007
<b>Reference Lab Final:</b>	5.238	0.010	19.535	0.010
<b>Average:</b>	<b>5.238</b>	<b>0.009</b>	<b>19.537</b>	<b>0.009</b>

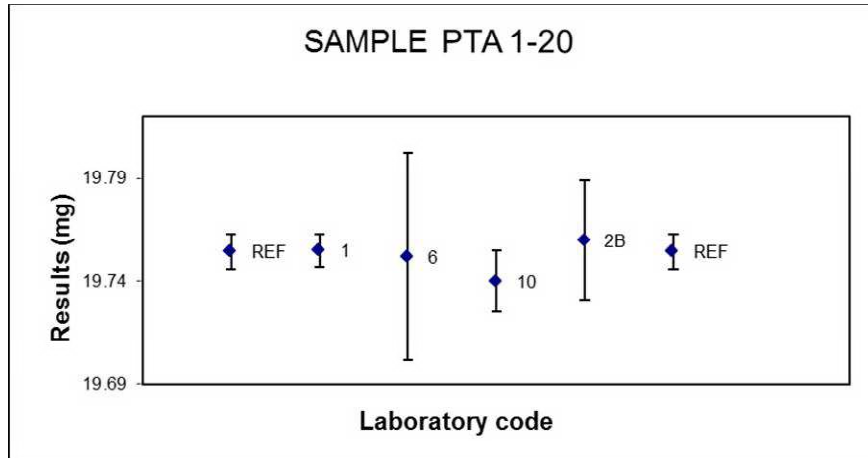
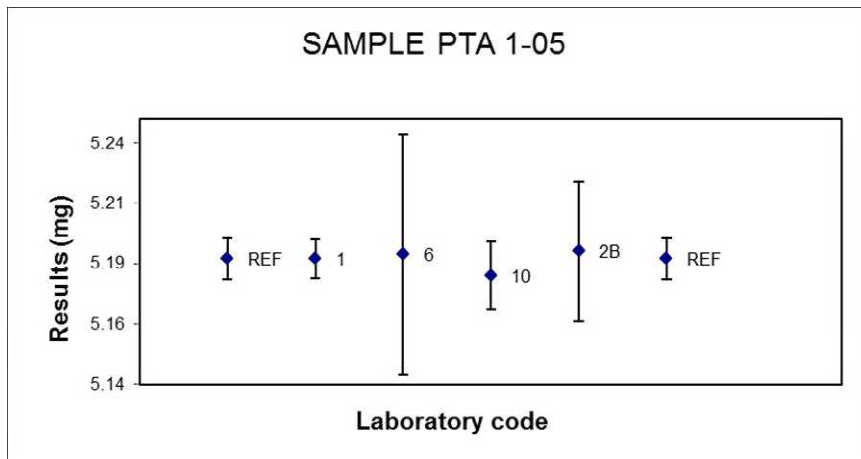
#### NOTES:

- 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<sub>n</sub> refers to Error Normalised. An E<sub>n</sub> number between -1.0 and +1.0 is considered acceptable (i.e |E<sub>n</sub>| < 1.0).
- 3 U<sub>95</sub> refers to uncertainty of measurement at the 95% confidence level.
- 4 Weight and U<sub>95</sub> 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.

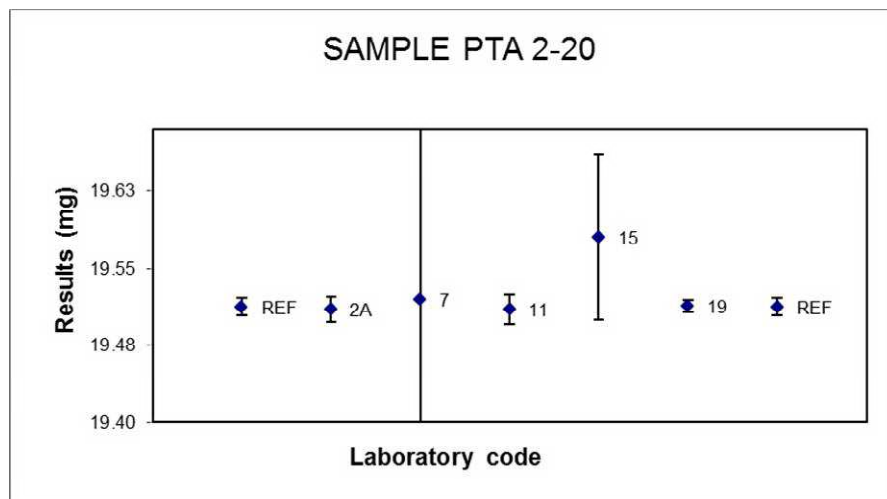
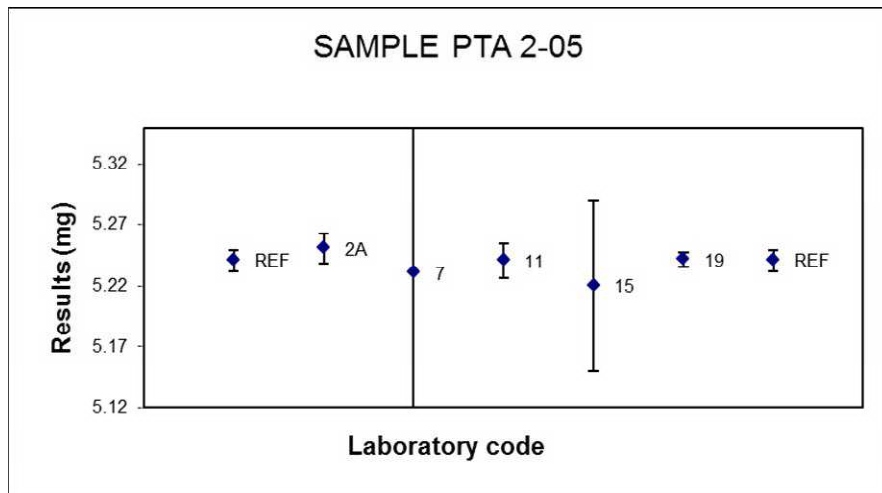


## Appendix A5

### Graphical Presentation of Participants' Results



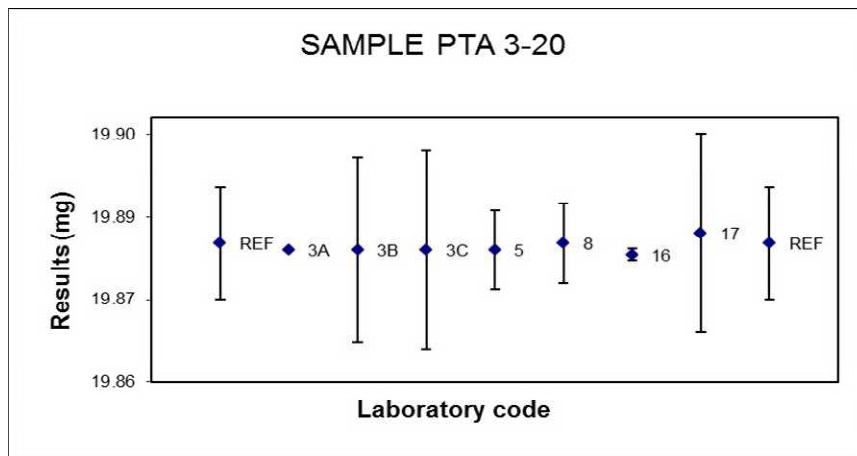
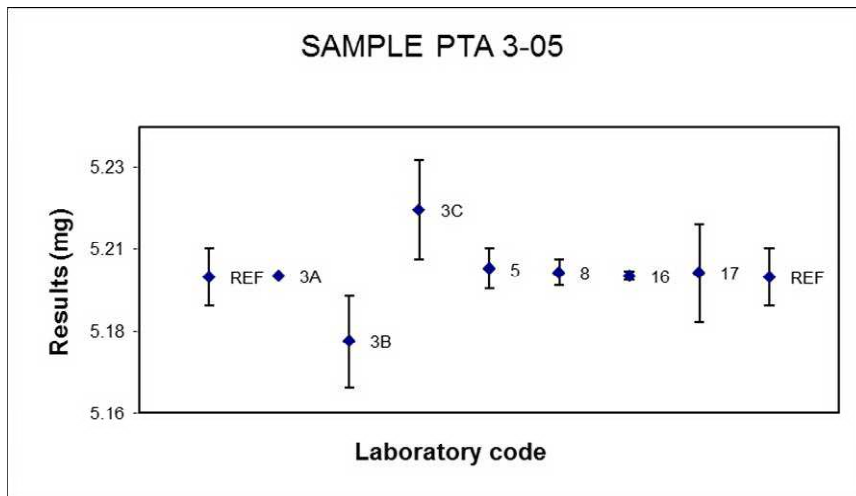
### Graphical Presentation of Participants' Results



Note: The uncertainty bars for Laboratory Code 7 extend beyond the chart limits.

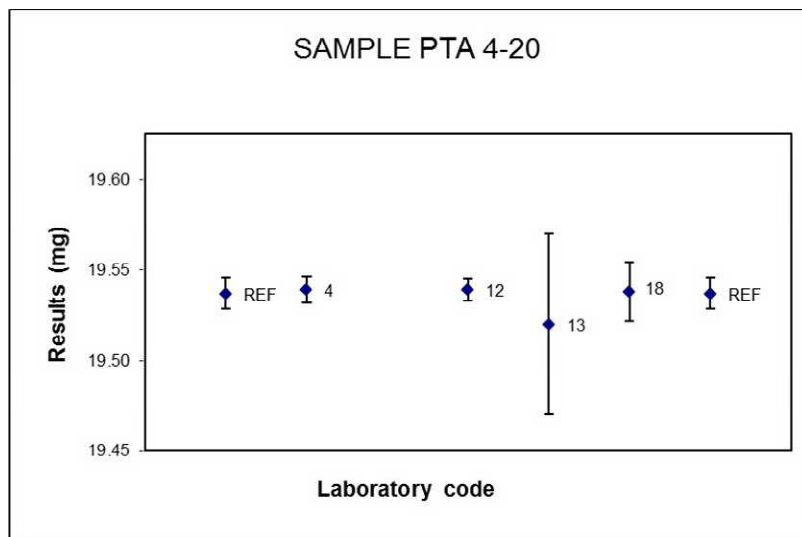
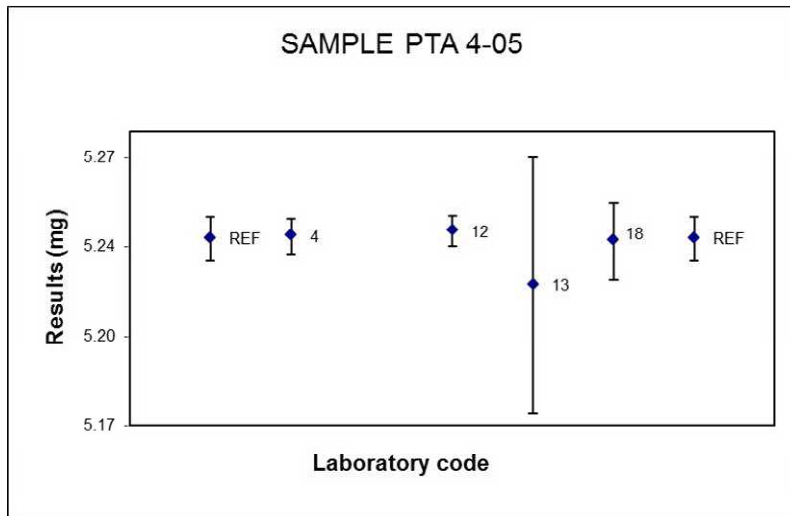
### A5.3

## Graphical Presentation of Participants' Results



## A5.4

### Graphical Presentation of Participants' Results



Note : The results and uncertainty bars for Laboratory Code 9 extend beyond the chart limits.



## Appendix A6

### ADDITIONAL INFORMATION (Microbalances used by Participants)

Lab Code	Microbalance		
	Brand	Model	Range
1	Mettler Toledo	MX-5	6 dec place
2A	Mettler Toledo	XS 105 DUAL RANGE	MAX 41g
2B	Mettler	XSE105	MAX 41g
3A	Mettler Toledo	XS 105 DU	Range 2:41g /0.00001g
3B	Mettler Toledo	X5205 DU	Range 2:81g
3C	Mettler Toledo	XS 205	Low range 0-80g
4	Mettler Toledo	XP6	0.001mg-6.1g
5	Mettler	Toledo XP6	0-6g
6	Mettler Toledo	MX5	0.000-5,000mg
7	AND	GH-252	5 point balance
8	Mettler Toledo	XP6	0-6g
9	AND	GR.202	0-100mg
10	Ohaus	DV215CD	0-210g
11	Mettler Toledo	XS105DU	2
12	Mettler Toledo	XP6	
13	Sartorius	MSA225S	0.01-220g
15	Mettler Toledo	AG135	0.01
16	Mettler Toledo	XP2U	0.0001mg-2.1g
17	Mettler Toledo	MT5	5.1g
18	Mettler Toledo	XP6	3000mg
19	Mettler Toledo	MX5	0-500mg

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 5)**

**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  $k \approx 2$ .) *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.  
7 Leeds Street, Rhodes, NSW 2138. Australia.  
Phone: +61 2 9736 8397 Fax: +61 2 9743 6664  
Email: michael.li@pta.asn.au

## PROFICIENCY TESTING AUSTRALIA

## Gravimetric Round 5

**Results Sheet**Lab Code: 

Sample Identification	Weight (mg)	Uncertainty ( $U_{95}$ )* (mg)
<i>Nominal 5 mg</i>		
<i>Nominal 20 mg</i>		

\*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.

7 Leeds Street, Rhodes, NSW 2138 Australia.

Phone: +61 2 9736 8397 Fax: +61 2 9743 6664 Email: michael.li@pta.asn.au

---- End of Report ----