

REPORT NO. 1068

**Hardness Testing of Metals
Proficiency Testing Program
Round 13**

March 2018

ACKNOWLEDGMENTS

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Mr S Sameem, ARL Laboratory Services Pty Ltd. Also our thanks go to ARL Laboratory Services Pty Ltd, for the supply and homogeneity testing of the samples.

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1. FOREWORD

This report summarises the results of a proficiency testing program on the hardness properties of metals. It constitutes the thirteenth round of an ongoing series of programs.

Proficiency Testing Australia (PTA) conducted the testing program in December 2107 / January 2018. 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 Adviser was Mr S Sameem, ARL Laboratory Services Pty Ltd. This report was authorised by Mrs F Watton, PTA Quality Manager.

2. FEATURES OF THE PROGRAM

- (a) A total of 13 laboratories participated in the program, all of which returned results for inclusion in the final report. Laboratories from the following countries received samples:

10	AUSTRALIA
1	SAUDI ARABIA
1	SINGAPORE
1	TANZANIA

To ensure confidential treatment of results, each laboratory was allocated a unique random code number. Reference to each laboratory in this report is by its code number.

- (b) The results reported by participants are presented in Appendix A.
- (c) Each laboratory was provided with a steel sample, approximately 60 mm in diameter and approximately 30 mm thick. The sample was to be tested for Brinell, Vickers and Rockwell B hardness testing.
- (d) 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.
- (e) Prior to distribution, the samples were tested for homogeneity by ARL Laboratory Services Pty Ltd. Based on the results of this testing, the homogeneity of the samples was established (see Appendix B).

3. FORMAT OF THE APPENDICES

- (a) Appendix A is divided into four sections (A1-A4).

Sections A1-A3 contain the analysis of results reported by laboratories for Brinell, Vickers and Rockwell B hardness. These sections contain:

- i) a table of results reported by laboratories for each test, with estimates of their MUs and calculated z-scores;
- ii) a listing of the summary statistics; and
- iii) ordered z-score charts.

Section A4 contains information on the methods used by the participants and the surface preparations they performed.

- (b) Appendix B contains details of the homogeneity 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.* $100 \times \text{Normalised IQR} / \text{Median}$;
- *Minimum*: the lowest laboratory result;
- *Maximum*: the highest laboratory result; and
- *Range*: the difference between the *Maximum* and *Minimum*.

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:

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

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

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.* $2.0 < |z\text{-score}| < 3.0$). These results are considered to be questionable results.

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.

For further details on the calculation and interpretation of robust z-scores and ordered z-score charts, please see the *Guide to Proficiency Testing Australia (2016)*.

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	Average Result
Brinell Hardness (HBW)	Number of Results	12
	Median	197.5
	Normalised IQR	3.9
	Uncertainty (Median)	1.4
Vickers Hardness (HV)	Number of Results	12
	Median	206.8
	Normalised IQR	1.5
	Uncertainty (Median)	0.5
Rockwell B Hardness (HRB)	Number of Results	12
	Median	92.45
	Normalised IQR	0.80
	Uncertainty (Median)	0.29

Table B: Summary of Statistical Outliers
(By laboratory code number)

Test	Outliers (Laboratory Code No.)
Brinell Hardness	-
Vickers Hardness	3, 7, 10
Rockwell B Hardness	11

Notes:

1. For each test, the results for all test methods were pooled for analysis.
2. Summary statistics and z-scores were calculated for the average hardness value reported for each test.

6. PTA AND TECHNICAL ADVISER'S 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 of the 13 laboratories that participated in the program submitted results for inclusion in the final report. Of these 13 laboratories, 11 (85%) submitted results for all three tests.

The return rate for all tests is as follows:

- Brinell Hardness 12 out of 13 92%
- Vickers Hardness 12 out of 13 92%
- Rockwell B Hardness 12 out of 13 92%

6.2 Performance summary

Statistical outliers were reported by four of the 13 laboratories (31%) that returned results in this round of the program. For comparison, there were no statistical outliers reported by the participants in Round 12 of this program (see Report No. 1045 for more details).

A total of 36 results were analysed in this program. Of these results, four (11%) were outlier results.

6.3 Brinell Hardness

A total of 12 laboratories tested the sample for Brinell hardness. Of these laboratories, eight reported using the AS 1816 method. Two laboratories tested using the ISO 6506 method. Two laboratories tested using the ASTM E10 method (see Appendix A4 for more details).

There were not enough results submitted for any specific method to draw reliable conclusions from analysing grouped methods on this occasion. The methods were pooled when analysing the results.

For all methods pooled, the median and its uncertainty for the Brinell hardness results was 197.5 ± 1.4 HBW.

The CV for the Brinell hardness results for this round was 2.0%. This is slightly lower than the CV of 2.4%, obtained for the Brinell hardness results in Round 12 of this program (see Report No. 1045).

There were no outliers reported for Brinell hardness. Two laboratories (codes 3 and 5) obtained absolute z-scores between 2.0 and 3.0. The low hardness results reported by laboratories 3 and 5 suggest that these laboratories might need to review their test method procedures for Brinell testing. Their low hardness results may indicate inadequate surface preparation, including uneven test surfaces.

Seven laboratories reported measurement uncertainties associated with their Brinell hardness test results in this round.

6.4 Vickers Hardness

Of the 12 laboratories that tested the sample for Vickers hardness, nine reported using the AS 1817 method. One laboratory tested using the ISO 6507 method. Two laboratories tested using the ASTM E92 method (see Appendix A4 for more details).

There were not enough results submitted for any specific method to draw reliable conclusions from analysing grouped methods on this occasion. The methods were pooled when analysing the results.

For all methods pooled, the median and its uncertainty for the Vickers hardness results was 206.8 ± 0.5 HV.

The CV for the Vickers hardness results for this round was 0.7%. This is lower than the CV of 4.7%, obtained for the Vickers hardness results in Round 12 of this program (see Report No. 1045).

Three laboratories (codes 3, 7 and 10) reported outlier results for Vickers hardness.

Given the limited test surface, the sequence in which the hardness tests were carried out may have produced deceptive test results. This is especially true if the Vickers indentations were performed near the bigger Brinell indentations. Usually, in such circumstances, the Vickers results will be comparatively higher due to the localised deformation made by the Brinell tests. This could be the case for laboratory 7, whose average Vickers result of 212 HV is higher than the overall median of 206.8 HV.

The low Vickers results reported by laboratories 3 and 10 may suggest uneven test surfaces. The high degree of preparation required for Vickers hardness tests provides a better penetration for the indenter and, hence, the indentation. Although both laboratories reported adequate surface preparations, their test surfaces might still have been uneven. Additionally, other external factors, such as light, dirt, vibrations, temperature and humidity, play a contributing factor to erroneous results. These factors should always be controlled.

Seven laboratories reported measurement uncertainties associated with their Vickers hardness test results in this round.

6.5 Rockwell B Hardness

A total of 12 laboratories tested the sample for Rockwell B hardness. Of these laboratories, nine tested using the AS 1815 method. One laboratory tested using the ISO 6508 method. Two laboratories tested using the ASTM E18 method (see Appendix A4 for more details).

There were not enough results submitted for any specific method to draw reliable conclusions from analysing grouped methods on this occasion. The methods were pooled when analysing the results.

For all methods pooled, the median and its uncertainty for the Rockwell B hardness results was 92.45 ± 0.29 HRB.

The CV for the Rockwell B hardness results for this round was 0.9%. This is lower than the CV of 2.3%, obtained for the Rockwell B hardness results in Round 12 of this program (see Report No. 1045).

One laboratory (code 11) reported an outlier result for Rockwell B hardness. One laboratory (code 9) obtained an absolute z-score between 2.0 and 3.0.

The high results reported by laboratory 11 suggest that the sequence of hardness tests carried out by this laboratory might be a contributing factor for their outlier result. Alternatively their high results could be due to inadequate spacing for the hardness indentations, considering the limited test surface provided.

Seven laboratories reported measurement uncertainties associated with their Rockwell B hardness test results in this round.

6.6 Measurement Uncertainty

The number and percentage of laboratories that reported estimates of their measurement uncertainty for each test is as follows:

- Brinell Hardness 7 out of 12 58%
- Vickers Hardness 7 out of 12 58%
- Rockwell B Hardness 7 out of 12 58%

The variation between the estimates of measurement uncertainty ranged from not reporting at all, to some reporting numerical values, while others reported percentages.

Any laboratory that reported a measurement uncertainty less than two times the uncertainty of the median may have underestimated their measurement uncertainty.

Any laboratory that reported a measurement uncertainty greater than three times the normalised IQR may have overestimated their measurement uncertainty.

All participants are highly encouraged to report and use measurement uncertainty, so that the program analysis can provide a better outlook of the overall performance for this program.

6.7 General Comments

For this round of hardness testing the overall performance of the participating laboratories was very good, with only four outlier results reported. Participating in these accredited proficiency testing programs should be very beneficial for these laboratories, so they can investigate the causes of their outlier results and review their operating procedures.

Since hardness tests are comparative in nature, it is very important to ensure that the area of testing is highlighted on the samples for proficiency testing purposes. This area is very important, as there may exist some variation in the hardness value from the middle section to the edges of the specimen. The samples were grooved this round to indicate the area to be tested. For the next round of this program, the hardness tests should be taken at the outer edge of the grooves. This will help get a better understanding of the hardness results based on the sample size, shape and location.

7. REFERENCES

1. *Guide to Proficiency Testing Australia (2016)*. (This document is located on the PTA website at www.pta.asn.au under Programs / Documents).
2. *AS 1815.1: 2007 Metallic materials – Rockwell hardness test – Test method (scales A, B, C, D, E, F, G, H, K, N, T)*.
3. *AS 1816.1: 2007 Metallic materials – Brinell hardness test – Test method (ISO 6506-1: 2005, MOD)*.
4. *AS 1817.1: 2003 Metallic materials – Vickers hardness test – Test method (ISO 6507-1: 1997, MOD)*.
5. *ISO 6506-1: 2014 Metallic materials – Brinell hardness test – Part 1: Test method*.
6. *ISO 6507-1: 2018 Metallic materials – Vickers hardness test – Part 1: Test method*.
7. *ISO 6508-1: 2016 Metallic materials – Rockwell hardness test – Part 1: Test method*.
8. *ASTM E10 – Standard Test Method for Brinell Hardness of Metallic Materials*.
9. *ASTM E18 – Standard Test Methods for Rockwell Hardness of Metallic Materials*.
10. *ASTM E92 – Standard Test Method for Vickers Hardness and Knoop Hardness of Metallic Materials*.

APPENDIX A

Summary of Results

Section A1

Brinell Hardness

A1.1

Brinell Hardness (HBW) – Results and Z-Scores

Lab Code	Scale	Temp. (°C)	Test 1	Test 2	Test 3	Average	MU (±)	Z-Score
1	-	21	197	197	197	197	-	-0.13
2	10/3000	-	199	199	197	198	-	0.13
3	10/3000/15	27	187	188	188	188	1.8	-2.41
4	10/3000/10	29.4	201	197	197	198.3	2.61	0.20
5	10/3000/15	-	187	191	187	188	3	-2.41
7	-	25.4	197	197	198	197	3.4	-0.13
8	10/3000/15	23	192	192	192	192	-	-1.39
9	10/3000/12	23	202	197	205	201.3	-	0.96
10	10/3000/12	24.2	197	201	200	199	5	0.38
11	10/3000/22	22	201	207	209	205	2.5%	1.90
12	10/3000/15	23	201	201	207	203	-	1.39
13	1/30/10	21.0	200	193	193	195	3.2	-0.63

Summary Statistics

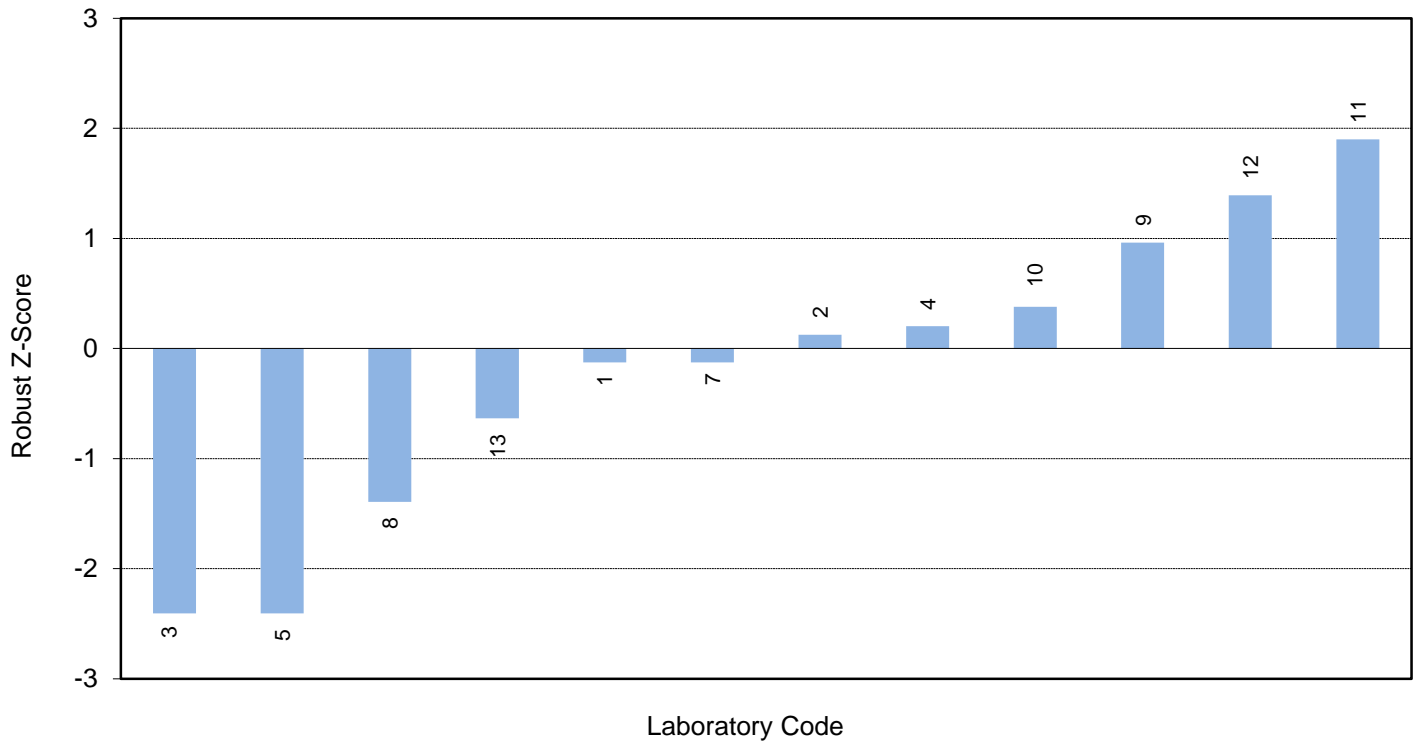
Statistic	Average Result
Number of Results	12
Median	197.5
Normalised IQR	3.9
Uncertainty (Median)	1.4
Robust CV	2.0%
Minimum	188
Maximum	205
Range	17

Note:

1. The results for all test methods were pooled for analysis.
2. Summary statistics and z-scores have been calculated for the average results reported.

A1.2

Brinell Hardness (HBW)



Section A2

Vickers Hardness

A2.1

Vickers Hardness (HV) – Results and Z-Scores

Lab Code	Load	Temp. (°C)	Test 1	Test 2	Test 3	Average	MU (±)	Z-Score
1	10	21.5	204.0	206.5	208.9	206.5	-	-0.17
2	10	-	200	208	212	207	-	0.17
3	-	27	198	200	203	200	1.6	-4.55 §
5	-	21	203	208	205	205	3	-1.18
6	10	22.6	205	213	200	206	4.92	-0.51
7	-	25.4	213	212	211	212	1.3	3.54 §
8	5	23	210	206	207	207.66	-	0.61
9	10	23	205.0	207.1	208.9	207.0	-	0.17
10	30	24.1	204	200	200	201	6	-3.88 §
11	-	22	202	216	205	207	1.2%	0.17
12	10	23	204	204	207	205	-	-1.18
13	30	21.0	208	207	206	207	11.2	0.17

Summary Statistics

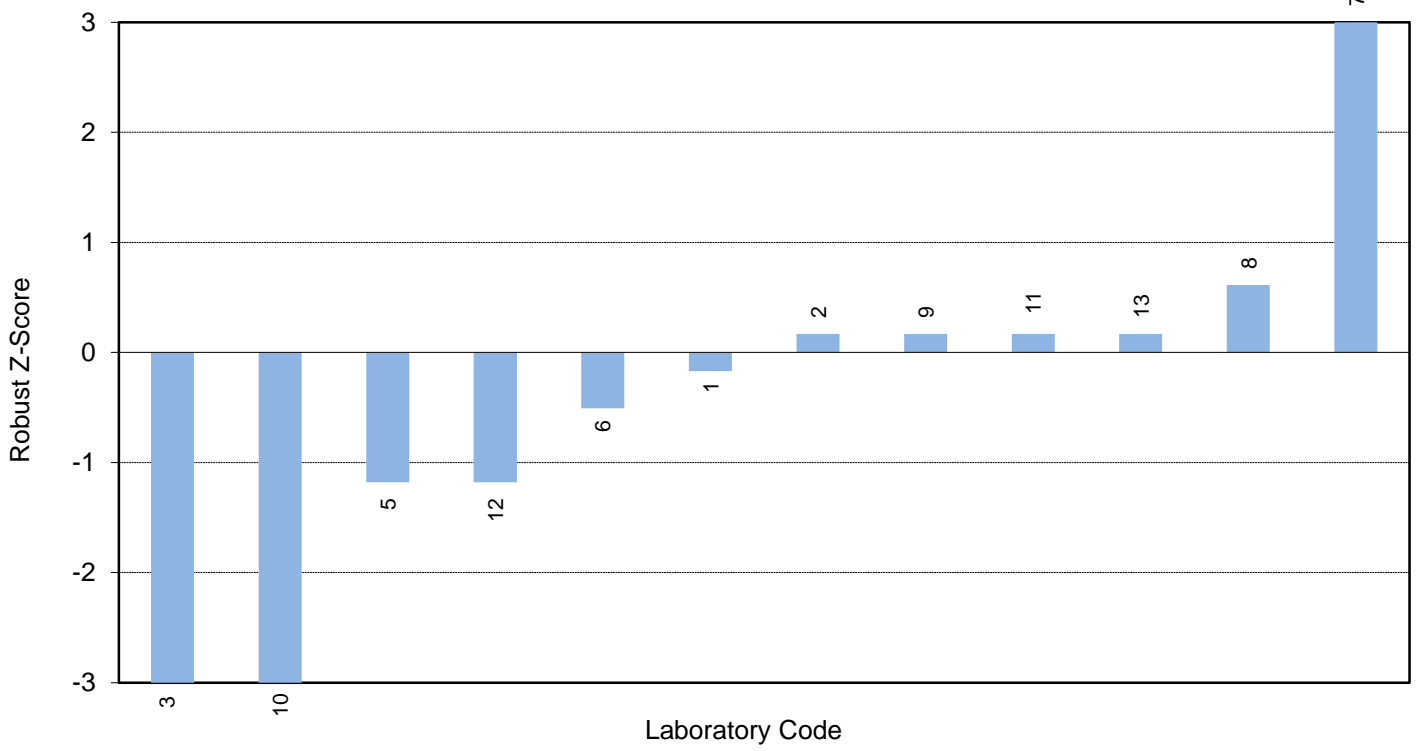
Statistic	Average Result
Number of Results	12
Median	206.8
Normalised IQR	1.5
Uncertainty (Median)	0.5
Robust CV	0.7%
Minimum	200
Maximum	212
Range	12

Note:

1. § denotes an outlier (i.e. $|z\text{-score}| \geq 3.0$).
2. The results for all test methods were pooled for analysis.
3. Summary statistics and z-scores have been calculated for the average results reported.

A2.2

Vickers Hardness (HV)



Section A3

Rockwell B Hardness

A3.1

Rockwell B Hardness (HRB) – Results and Z-Scores

Lab Code	Temp. (°C)	Test 1	Test 2	Test 3	Average	MU (±)	Z-Score
1	22	93	92	93	92.6	-	0.19
2	-	90	92	91	91	-	-1.82
3	27	92.1	92.4	93.0	92.5	0.49	0.06
5	-	92.2	92.5	92.5	92.4	1.0	-0.06
6	22.6	92	94	93	93	1	0.69
7	25.5	91.7	90.3	92.3	91.4	2.16	-1.32
8	28	91	90	91	91	-	-1.82
9	23	92.8	95.0	96.0	94.6	-	2.70
10	24.2	92.5	92.7	91.9	92.3	2	-0.19
11	22	99.2	98.9	98.9	99.0	1.6	8.22 §
12	23	92.7	92.0	92.3	92.3	-	-0.19
13	21.0	93.7	93.3	93.9	93.6	1.0	1.44

Summary Statistics

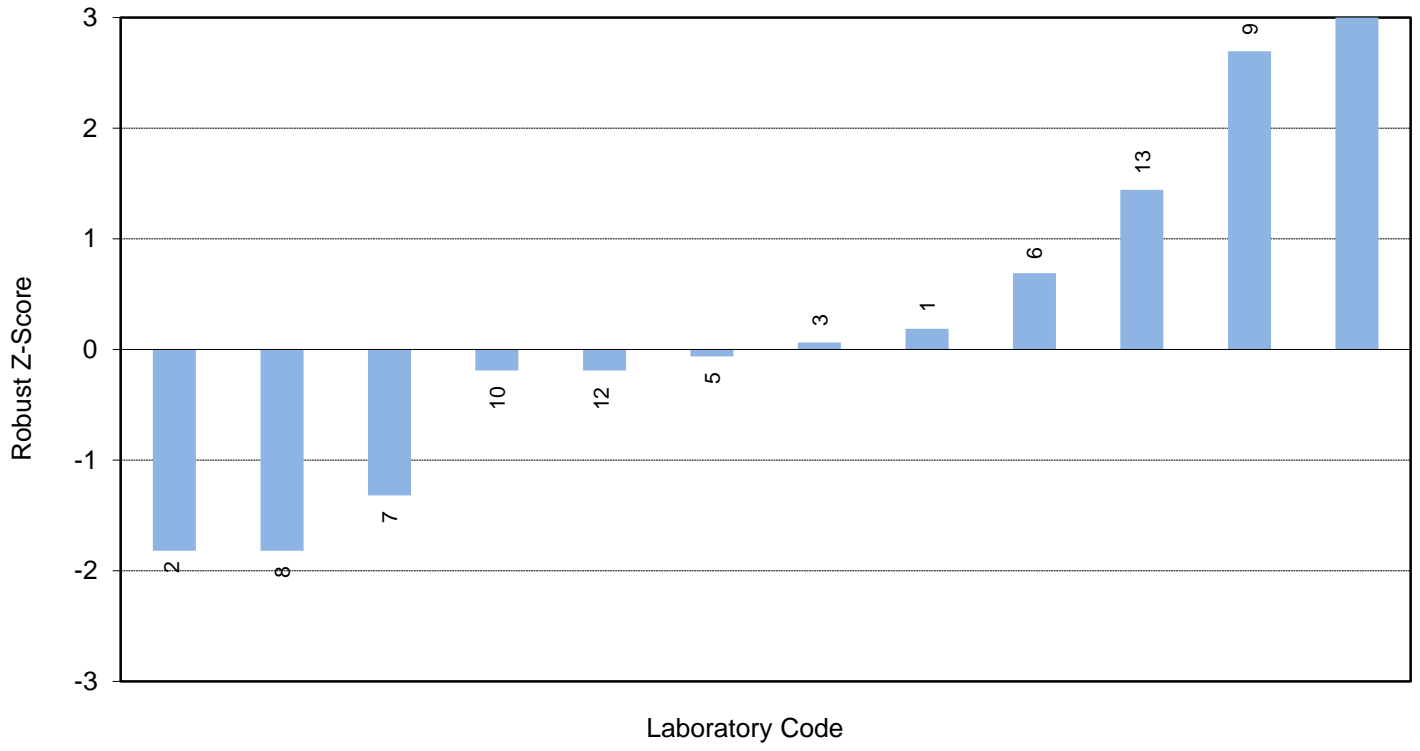
Statistic	Average Result
Number of Results	12
Median	92.45
Normalised IQR	0.80
Uncertainty (Median)	0.29
Robust CV	0.9%
Minimum	91.0
Maximum	99.0
Range	8.0

Notes:

1. § denotes an outlier (i.e. $|z\text{-score}| \geq 3.0$).
2. The results for all test methods were pooled for analysis.
3. Summary statistics and z-scores have been calculated for the average results reported.

A3.2

Rockwell B Hardness (HRB)



Section A4

Methods Used and Surface Preparation

A4.1

Methods Used

Lab Code	Brinell Hardness	Vickers Hardness	Rockwell B Hardness
1	AS 1816.1	AS 1817.1 - 2003	AS 1815.1 - 2007
2	ISO 6506.1 - 2014	AS 1817.1 - 2003	AS 1815.1 - 2007
3	AS 1816.1 - 2007	AS 1817.1 - 2007	AS 1815.1 - 2007
4	AS 1816.1	-	-
5	AS 1816.1	AS 1817.1	AS 1815.1
6	-	AS 1817.1	AS 1815.1
7	ASTM E10-15a	ASTM E92-16	ASTM E18-16
8	AS 1816.1	AS 1817.1	AS 1815.1
9	ASTM E10	ASTM E92	ASTM E18
10	AS 1816.1	AS 1817.1	AS 1815.1
11	AS 1816.1	AS 1817.1	AS 1815.1
12	AS 1816.1	AS 1817.1	AS 1815.1
13	ISO 6506-1: 2014	ISO 6507-1: 2005	ISO 6508-1: 2015

A4.2

Surface Preparation

Lab Code	Preparation Details
1	No surface preparation performed.
2	For Brinell and Vickers testing: The surface was ground and polished to P2500 grit. For Rockwell testing: The test piece was machined on top and bottom surfaces to ensure section was flat and parallel. The top surface was then hand polished to P2500 grit.
3	The surface has been polished using SiC abrasive paper (2000 grit).
4	No surface preparation performed.
5	No additional preparation performed.
6	Wet hand ground sample on 240, 400, 800 and 1200 grit papers then diamond polished on 6 μm and 1 μm polishing cloths.
7	No surface preparation performed.
8	No surface preparation performed.
9	Light surface grinding.
10	Grind and polish to 1 micron.
11	Finish was enhanced with metallographic polishing compound (3 μm diamond).
12	Wet surface ground. Wet polish to 600 g.
13	Surface polishing was done on test surface.

APPENDIX B

Homogeneity Testing

B1.1

HOMOGENEITY TESTING

Before the samples were distributed to participants, eight randomly selected samples were tested for homogeneity by ARL Laboratory Services Pty Ltd. The results of the homogeneity testing are displayed below:

Homogeneity Testing Results

Brinell HBW 10/3000

Sample No.	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Average
1	187	186	185	185	183	187	185.5
5	185	185	186	186	184	185	185.2
10	183	187	185	183	184	186	184.7
15	185	185	186	185	184	185	185.0
21	184	183	185	184	185	186	184.5
27	185	185	184	186	187	186	185.5
29	185	185	185	185	186	188	185.7
36	186	185	187	186	184	184	185.3

Vickers HV 10

Sample No.	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Average
1	200	199	195	197	197	197	197.5
5	198	202	203	196	199	202	200.0
10	196	203	198	200	204	203	200.7
15	202	199	204	201	202	200	201.3
21	199	202	204	199	203	204	201.8
27	203	198	205	205	201	202	202.3
29	202	204	204	205	205	203	203.8
36	204	204	200	199	204	203	202.3

B1.2

Rockwell B HRB

Sample No.	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Average
1	93.3	92.5	90.5	91.2	92.2	90.8	91.75
5	92	88.7	92.4	90.5	87.8	90.4	90.30
10	93.4	92.4	88.7	91.3	92	92.3	91.68
15	92.8	91.6	90.7	90.5	90.4	92.5	91.42
21	90.5	91.9	92.7	91.7	93.5	92.5	92.13
27	90.2	92.6	92.2	92.1	92.5	93.6	92.20
29	92.9	93.2	91.3	92.0	93.6	92.3	92.55
36	92	91.4	91.3	91.2	91.7	93.5	91.85

Analysis of the homogeneity testing data indicated that the samples were sufficiently homogeneous for the program and, therefore, any participant results identified as outliers cannot be attributed to sample variability.

APPENDIX C

Instructions to Participants and Results Sheet

Hardness Testing Of Metals Proficiency Testing Program Round 13, November 2017

Instructions to Participants

To ensure that the results of this program can be analysed correctly, participants are asked to adhere carefully to these instructions.

- 1) The sample for this hardness testing program consists of a steel sample, approximately 60 mm in diameter and 30 mm thick. The sample has a number labelled on the circumference.
- 2) The sample is to be tested for Brinell, Vickers and Rockwell 'B' hardness. Although both surfaces of the test sample are polished, the testing area is enclosed with a 0.5 mm groove. Participants may wish to improve the surface to provide a better testing surface.
- 3) The sample should be treated as a routine laboratory sample. All testing, recording and reporting is to be performed in accordance with your routine test methods.
- 4) Please use the attached Result Sheet to record and report your results to Proficiency Testing Australia. Please also report the method used for testing (e.g. AS 1816.1, ISO 6506.1, etc. for Brinell hardness testing, AS 1817.1, ISO 6507.1, etc. for Vickers hardness testing, AS 1815.1, ISO 6508.1, etc. for Rockwell HRB hardness testing).
- 5) Do not discard the hardness test sample until you have received the final report. You may be asked to carry out a retest or to return the sample to Proficiency Testing Australia for retesting at the laboratory that performed the homogeneity tests.
- 6) For this program, your laboratory has been allocated the code number on the attached Results Sheet. All reference to your laboratory in reports associated with this program will be via this code number, thus ensuring the confidentiality of your results.
- 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 $k \approx 2$).
- 8) Return the Results Sheet, either by mail, email or facsimile, 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 **Wednesday 20 December 2017**. Results reported later than this date may not be analysed in the final report.

Hardness Testing Of Metals Proficiency Testing Program

Round 13, November 2017

RESULTS SHEET

Laboratory Code:

Sample I.D.	Scale	Report to nearest	Test Temp °C	Results					Standard (AS, ISO, etc.)
				Test 1	Test 2	Test 3	Average	MU (±)	
	Brinell (/ /) (mm/kg/s)	1 BHN							
	HV () Insert load used	1 HV							
	HRB	0.1 HRB							

Did you carry out surface preparation on the obverse surfaces of the samples for the hardness tests?
Yes / No

If Yes, please give details of preparation.

.....

.....

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.....

.....

.....

Print Name: _____

Signature: _____

Date: _____

-----End of Report-----