

REPORT NO. 1018

**Hardness Testing of Metals
Proficiency Testing Program
Round 11**

March 2017

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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PO Box 7507 Silverwater NSW 2128 AUSTRALIA

CONTENTS

1. FOREWORD	1
2. FEATURES OF THE PROGRAM	1
3. FORMAT OF THE APPENDICES	2
4. STATISTICAL DESIGN OF THE PROGRAM	2
5. OUTLIER RESULTS	3
Table A: Summary Statistics for All Tests	3
Table B: Summary of Statistical Outliers	4
6. PTA AND TECHNICAL ADVISER'S COMMENTS	4
7. REFERENCES	8

APPENDICES

APPENDIX A

Summary of Results

Brinell Hardness A1.1

Vickers Hardness A2.1

Rockwell Hardness A3.1

Methods Used and Surface Preparation A4.1

APPENDIX B

Homogeneity Testing B1.1

APPENDIX C

Instructions to Participants C1.1

Results Sheet C2.1

1. FOREWORD

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

Proficiency Testing Australia (PTA) conducted the testing program in December 2016. 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 K Cividin, PTA Quality Coordinator / Senior Scientific Officer.

2. FEATURES OF THE PROGRAM

- (a) A total of 16 laboratories participated in the program, two of which did not return results for inclusion in the final report. Laboratories from the following countries received samples:

11	AUSTRALIA
1	INDIA
1	NEW ZEALAND
1	SAUDI ARABIA
1	TANZANIA
1	UNITED KINGDOM

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 14.5 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 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	13
	Median	179.0
	Normalised IQR	3.5
	Uncertainty (Median)	1.2
Vickers Hardness (HV)	Number of Results	14
	Median	178.5
	Normalised IQR	7.5
	Uncertainty (Median)	2.5
Rockwell B Hardness (HRB)	Number of Results	12
	Median	86.55
	Normalised IQR	1.11
	Uncertainty (Median)	0.40

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

Test	Outliers (Laboratory Code No.)
Brinell Hardness	1
Vickers Hardness	-
Rockwell B Hardness	8, 10

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

Of the 16 laboratories that participated in the program, 14 (88%) submitted results for inclusion in the final report. Of these 14 laboratories, 11 (79%) submitted results for all three tests.

The return rate for all tests is as follows:

- Brinell Hardness 13 out of 14 93%
- Vickers Hardness 14 out of 14 100%
- Rockwell B Hardness 12 out of 14 86%

6.2 Performance summary

One or more statistical outliers were reported by three of the 14 laboratories (21%) that returned results in this round of the program. For comparison, there were no statistical outliers reported by the participants in Round 10 of this program (see Report No. 990 for more details).

A total of 39 results were analysed in this program. Of these results, three (8%) were outlier results.

6.3 Brinell Hardness

A total of 13 laboratories tested the sample for Brinell hardness. Of these laboratories, eight reported using the AS 1816 method. One laboratory also indicated that they used an Australian Standard method. It is assumed that this was the AS 1816 method. One laboratory tested using the ISO 6506 method. Three laboratories tested using ASTM methods (see Appendix A4 for more details).

The methods were pooled when analysing the results.

For all methods pooled, the median and standard error of the Brinell hardness results was 179.0 ± 1.2 HBW.

The CV for the Brinell hardness results for this round was 1.9%. This is slightly higher than the CV of 1.4%, obtained for the Brinell hardness results in Round 10 of this program (see Report No. 990).

One laboratory (code 1) reported an outlier for Brinell hardness. Two other laboratories (codes 5 and 15) obtained absolute z-scores between 2.0 and 3.0.

The low Brinell readings reported by laboratory 1 and laboratory 5 might be due to these readings being taken near the middle of the sample. As reported by laboratory 3, the middle section of the sample was found to be softer, compared to the remainder of the sample. One of the ways to ensure accurate hardness values are obtained is to use the conversion charts to compare the values and analyse the results to ensure all the hardness readings represent the sample. Low results may be due to non flat surfaces (*e.g.* extremely rough or textured surfaces). Both surfaces need to be flat, clean and free from any scale, debris, dirt and oil before testing. Low results may also be reported when the surfaces are not perpendicular to the indenter. Surfaces should be flat within two degrees.

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

6.4 Vickers Hardness

Of the 14 laboratories that tested the sample for Vickers hardness, nine reported using the AS 1817 method. One laboratory also indicated that they used an Australian Standard method. It is assumed that this was the AS 1817 method. Two laboratories tested using the ISO 6507 method. Two laboratories tested using the ASTM E384 method (see Appendix A4 for more details).

The methods were pooled when analysing the results.

For all methods pooled, the median and standard error of the Vickers hardness results was 178.5 ± 2.5 HV.

The CV for the Vickers hardness results for this round was 4.2%. This is higher than the CV of 1.5%, obtained for the Vickers hardness results in Round 10 of this program (see Report No. 990).

There were no outliers reported for Vickers hardness.

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

6.5 Rockwell Hardness

A total of 12 laboratories tested the sample for Rockwell hardness. Of these laboratories, seven tested using the AS 1815 method. One laboratory also indicated that they used an Australian Standard method. It is assumed that this was the AS 1815 method. Two laboratories tested using the ISO 6508 method. Two laboratories tested using the ASTM E18 method (see Appendix A4 for more details).

The methods were pooled when analysing the results.

For all methods pooled, the median and standard error of the Rockwell B hardness results was 86.55 ± 0.40 HRB.

The CV for the Rockwell B hardness results for this round was 1.3%. This compares well with the CV of 1.2%, obtained for the Rockwell B hardness results in Round 10 of this program (see Report No. 990).

Two laboratories (codes 8 and 10) reported outliers for Rockwell B hardness. Both of these laboratories reported an average Rockwell hardness of 90.0 HRB, which was higher than the median of 86.55 HRB. Laboratories that reported high results may have taken their readings too close together. To obtain accurate results, indentations should be at least three diameters apart.

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

6.6 Measurement Uncertainty

The majority of participants in this round (67% – 71%) reported estimates of the measurement uncertainty associated with their results. The number and percentage of laboratories that reported estimates of their measurement uncertainty for each test is as follows:

- Brinell Hardness 9 out of 13 69%
- Vickers Hardness 10 out of 14 71%
- Rockwell B Hardness 8 out of 12 67%

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

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

All the 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

The overall performance of the participating laboratories in this round of the program is satisfactory. Although, compared to the previous round, where no outliers were reported, a total of three participating laboratories reported outliers in this round. By carefully analysing their testing methods and reporting, the results of these laboratories will surely improve.

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.

All hardness testing machines should be calibrated and maintained accordingly. As part of an effective maintenance plan, the indenters should be regularly removed and checked for signs of deterioration.

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: 2005 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 A370 – Standard Test Methods and Definitions for Mechanical Testing of Steel Products*.
9. *ASTM E10 – Standard Test Method for Brinell Hardness of Metallic Materials*.
10. *ASTM E18 – Standard Test Methods for Rockwell Hardness of Metallic Materials*.
11. *ASTM E110 – Standard Test Method for Rockwell and Brinell Hardness of Metallic Materials by Portable Hardness Testers*.
12. *ASTM E384 – Standard Test Method for Microindentation Hardness of 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	10/3000/12	24.3	163	160	161	161	5	-5.21 §
2	10/3000/15	21	180	180	179	180	-	0.29
3	10/3000/10	21	178	169	183	177	-	-0.58
4	10/3000/60	23	179	180	178	179	5%	0.00
5	10/3000	23	170	170, 170	170, 170	170	7	-2.61
6	10/3000/10	15	174	183	179	179	10	0.00
7	10/3000	20	180	180	178	179	1.6%	0.00
8	10/3000/22	22	183	183	187	184.3	1.8%	1.54
9	10/3000/10	20	179	179	179	179	4.8	0.00
10	10/3000/12	21	180	183	185	182.66	-	1.06
11	-	21	184	183	184	183.66	-	1.35
14	10/3000/W	20	177	177	179	178	3.9	-0.29
15	10/3000/12	19.0	187	186	186	186	7.0	2.03

Summary Statistics

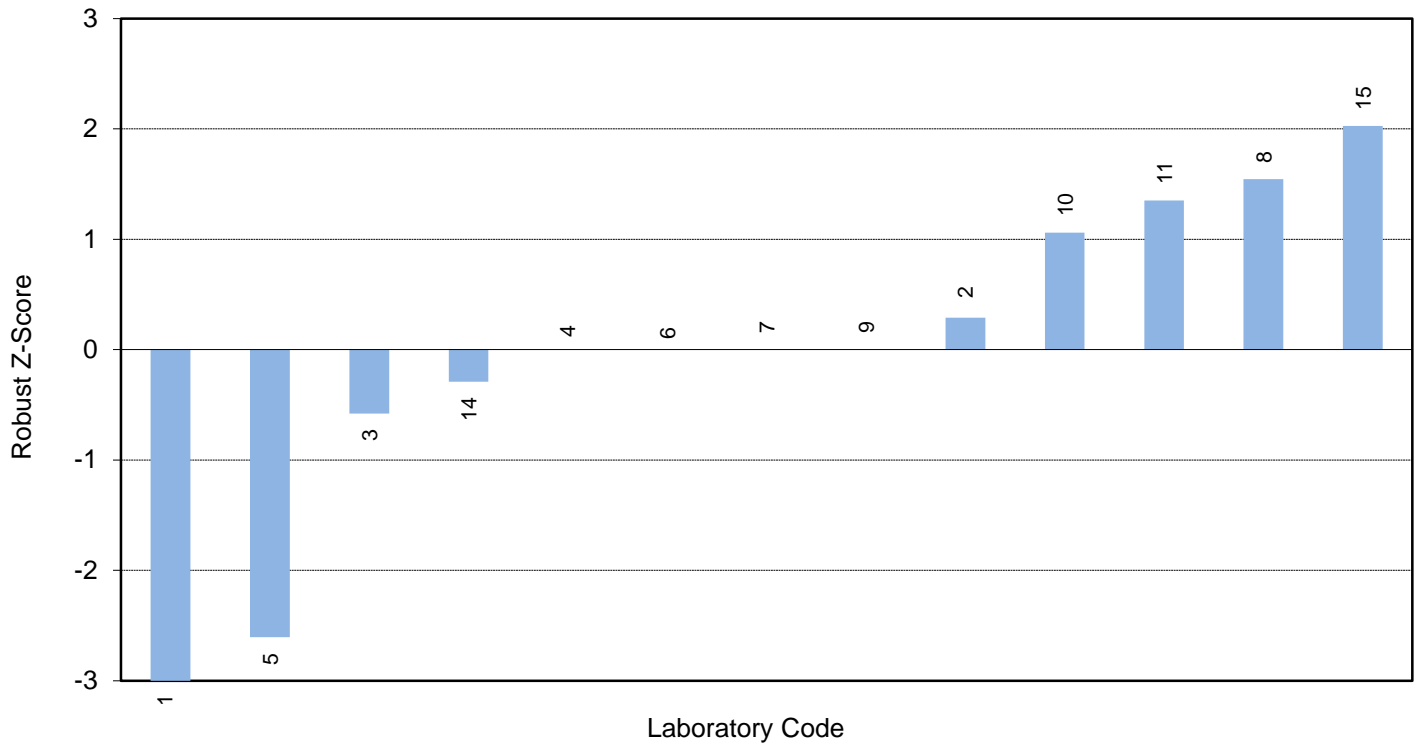
Statistic	Average Result
Number of Results	13
Median	179.0
Normalised IQR	3.5
Uncertainty (Median)	1.2
Robust CV	1.9%
Minimum	161
Maximum	186
Range	25

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.

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	30	24.3	171	172	174	172	6	-0.87
2	10	21	190	187	181	186	-	1.00
3	10	21	189	177	188	185	-	0.87
4	10	23	178	180	176	178	5%	-0.07
5	5	23	170	176, 172	174, 171	173	4	-0.74
6	30	16	185	181	188	185	7	0.87
7	20	20	191	187	187	188	1.1%	1.27
8	10	22	181	181	186	182	1.2%	0.47
9	10	20	177	176	178	177	4.4	-0.20
10	10	21	181.75	182.85	183.41	182.67	-	0.56
11	-	21	172	169	170	170.33	-	-1.09
12	30	24	174	174	178	175.3	5.3	-0.43
14	30	20	177	167	179	174	3.0	-0.60
15	10	18.9	178	181	178	179	3.7	0.07

Summary Statistics

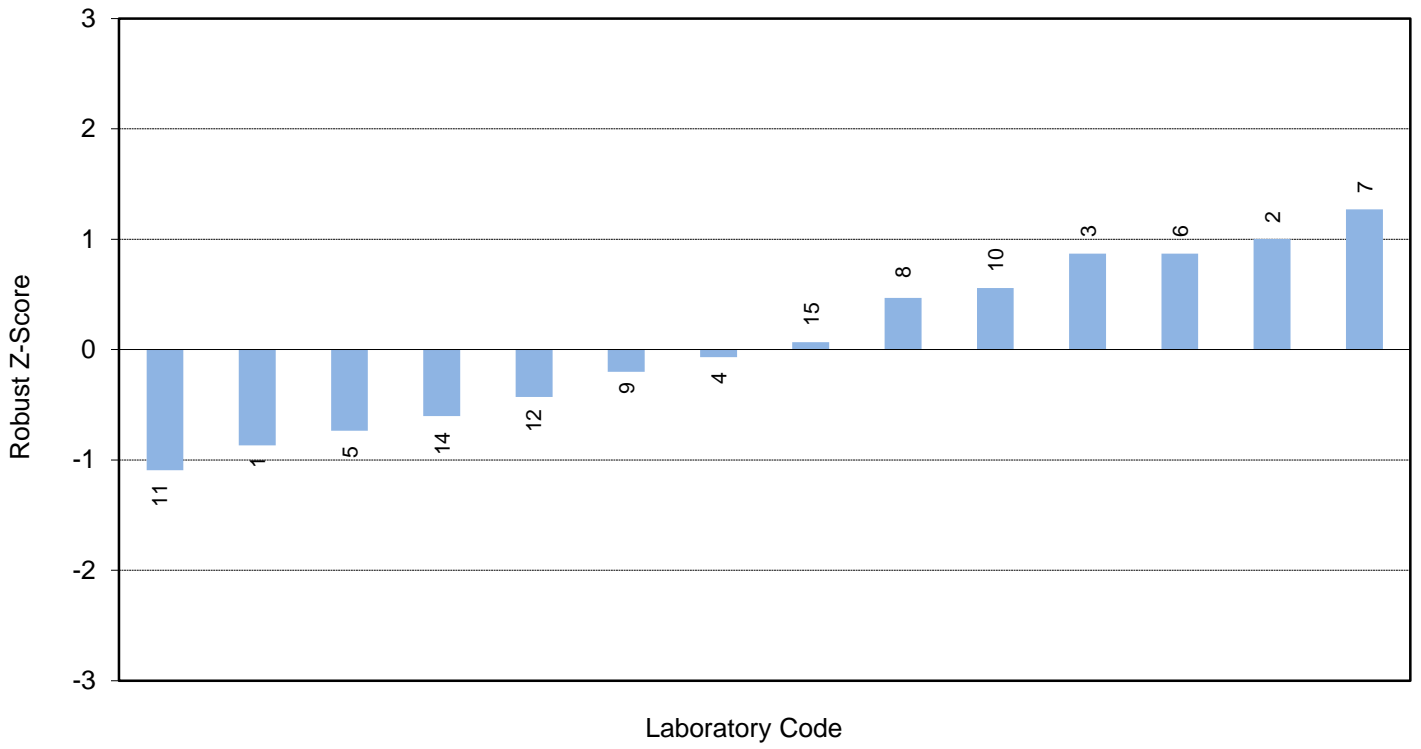
Statistic	Average Result
Number of Results	14
Median	178.5
Normalised IQR	7.5
Uncertainty (Median)	2.5
Robust CV	4.2%
Minimum	170
Maximum	188
Range	18

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.

A2.2

Vickers Hardness (HV)



Section A3

Rockwell 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	24.3	85.0	86.5	86	85.8	2	-0.67
2	21	85.7	85.6	87.1	86	-	-0.49
3	21	85.5	86.8	87.2	86.5	-	-0.04
4	23	89	88.5	88	88.5	5%	1.75
5	23	86.1	86.4, 85.8	86.5, 85.0	86.0	2.3	-0.49
8	22	89.7	90.2	90.2	90.0	1.01	3.10 §
9	25	85	85	85	85	1.9	-1.39
10	21	88.5	90.0	91.5	90.0	-	3.10 §
11	21	87	87	87.5	87.166	-	0.55
12	24	85.5	87.0	86.5	86.3	1.3	-0.22
14	20	87.48	86.28	86.98	86.9	0.8	0.31
15	19.0	86.5	86.0	87.2	86.6	1.4	0.04

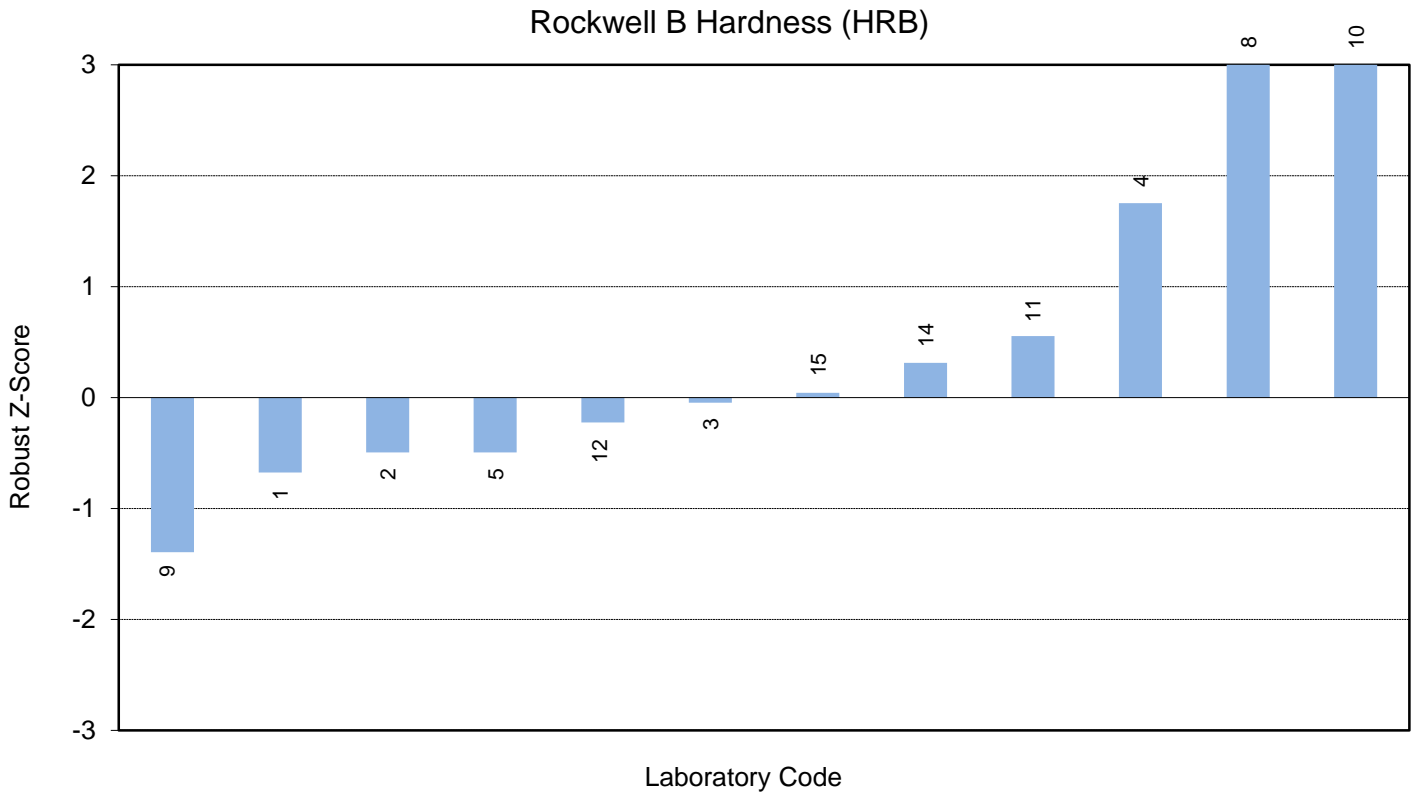
Summary Statistics

Statistic	Average Result
Number of Results	12
Median	86.55
Normalised IQR	1.11
Uncertainty (Median)	0.40
Robust CV	1.3%
Minimum	85.0
Maximum	90.0
Range	5.0

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



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	AS 1815.1
2	ASTM A370	ASTM E384	ASTM E18
3	AS 1816.1	AS 1817.1	AS 1815.1
4	AS 1816.1	AS 1817.1	AS 1815
5	AS 1816.1 - 2007	AS 1817.1 - 2007	AS 1815.1 - 2007
6	AS 1816.1 - 2007	AS 1817.1 - 2003	-
7	AS 1816	AS 1817	-
8	AS 1816	AS 1817	AS 1815.1
9	ASTM E110	AS 1817.1	AS 1815.1
10	ASTM E10	ASTM E384	ASTM E18
11	AS	AS	AS
12	-	ISO 6507-1: 2005	BS EN ISO 6508-1: 2015
14	AS 1816.1	AS 1817.1	AS 1815.1
15	6506-1	6507-1	6508-1

A4.2

Surface Preparation

Lab Code	Preparation Details
1	Grind and polish to 1 micron.
2	Test surface was ground by grinding machine.
3	Wet polish - P600. Note that areas in the middle of the sample tended to be softer.
4	Ground and polished to P1200.
5	Ground and polished to 1 micron finish.
6	The test face was surface ground then hand prepared using progressively finer 240, 400, 800 and 1000 grit Wet & Dry paper to produce a witness mark over the full surface. Preparation with each finer grade continued until the previous witness was removed.
7	Surface ground both surfaces to make parallel. Polished obverse side for better Brinell and Vickers readings.
8	Both faces machined to give parallel surfaces providing good visual imprint reading surface.
9	Linish 1000#.
10	Milling and finish grinding.
11	Surface preparation to P1200.
12	Both faces faced off in lathe. Obverse surface ground on SiC paper to 600 grit.
14	Surface painted red was metallographically prepared using SiC paper and 3 micron diamond finish.
15	Sample was prepared on 80 grit and 240 grit pads prior to hardness testing.

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

Test 1	Test 2	Test 3	Average
176	179	179	178.0
179	179	179	179.0
182	182	182	182.0
180	180	181	180.3
198	177	180	185.0
180	180	179	179.7
182	181	179	180.7
179	180	180	179.7

Vickers HV 10

Test 1	Test 2	Test 3	Average
175	177	169	173.7
171	171	178	173.3
174	175	176	175.0
185	174	184	181.0
171	171	173	171.7
168	186	179	177.7
187	176	175	179.3
178	177	174	176.3

B1.2

Rockwell HRB

Test 1	Test 2	Test 3	Average
87	87.5	87	87.2
87	87	87	87.0
88.5	88.5	87	88.0
87	87	88	87.3
88	87.5	87.5	87.7
87.5	88.5	87.5	87.8
88.5	88	87.5	88.0
89	87	86.5	87.5

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 11, December 2016

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 approximately 14.5 mm thick. The sample has been painted red, and has a number labelled on the circumference.
- 2) The sample is to be tested for Brinell, Vickers and Rockwell 'B' hardness. Participants are advised to improve the surface to provide a better testing surface. The side painted red is the side that should be prepared and tested.
- 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 **Thursday 22 December 2016**. Results reported later than this date may not be analysed in the final report.

Hardness Testing Of Metals Proficiency Testing Program

Round 11, December 2016

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.

.....

.....

.....

.....

.....

.....

Print Name: _____

Signature: _____

Date: _____

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