



**Report No. 1009**

**Metal Alloys Proficiency Testing  
Program**

**Round 31**

**Stainless Steel**

**February 2017**

**Acknowledgments**

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## CONTENTS

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

### ***APPENDIX A – Results and Data Analysis***

Carbon.....	A1
Manganese.....	A3
Phosphorus.....	A5
Sulfur.....	A7
Silicon.....	A9
Copper.....	A11
Nickel.....	A13
Chromium.....	A15
Molybdenum.....	A17
Cobalt.....	A19
Vanadium.....	A20

### ***APPENDIX B – Homogeneity and Stability Testing***

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Sample Preparation and Homogeneity Testing.....	B1
Stability Testing.....	B1

### ***APPENDIX C – Documentation***

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Instructions to Participants.....	C1
Results Sheet.....	C2

## 1. FOREWORD

This report summarises the results of a proficiency testing program on the chemical analysis of metal alloys. It constitutes the thirty first round of an ongoing series of programs.

The program was conducted in October/November 2016 by Proficiency Testing Australia (PTA). The aim of the program was to assess laboratories' abilities to competently perform the prescribed analyses.

The Program Coordinator was Mrs K Cividin and the Technical Adviser was Mr W Ting from Universal Scientific Laboratory. This report was authorised by Mr P Briggs, PTA General Manager.

## 2. FEATURES OF THE PROGRAM

(a) Participants were provided with one low alloy steel disc sample.

(b) A total of 13 laboratories received samples, comprising:

- 9 Australian participants; and

- 4 overseas participants, including:

- Colombia, Korea, Malaysia, and Singapore

All laboratories submitted their results by the due date.

(c) Laboratories were provided with the *Instructions to Participants* and *Results Sheet* (see Appendix C). Laboratories were requested to perform the tests according to their routine methods and to record their results on the *Results Sheet*.

(d) Prior to sample distribution, eight randomly selected samples were analysed for homogeneity. Based on the results of this testing (see Appendix B), the homogeneity of the samples was established.

(e) 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 code number only. Please note that two laboratories reported more than one set of results and, therefore, their code numbers (with letter) may appear several times in the same data set.

(f) Results (as reported by participants) with corresponding summary statistics (i.e. number of results, median, uncertainty of the median, normalised interquartile range, robust coefficient of variation, minimum, maximum and range) are presented in Appendix A (for each sample and for each of the analyses performed). Measurement Uncertainty (MU) is also presented where

supplied by participants. Please note that this information is presented for information purposes only and has not been used for the formal evaluation of results.

- (g) A robust statistical approach, using z-scores, was utilised to assess laboratories' testing performance (see Section 4). Robust z-scores, z-score charts relevant to each test are presented in Appendix A.
- (h) The document entitled *Guide to Proficiency Testing Australia, 2016* (reference [1]) defines the statistical terms and details the statistical procedures referred to in this report.
- (i) A tabulated listing of laboratories (by code number) identified as having outlier results can be found on page 8.

### **3. FORMAT OF THE APPENDICES**

- (a) Appendix A contains the analysis of results reported by laboratories for the samples by both all methods pooled and Technique Code 1 (AES Arc/Spark), except for Cobalt which is analysed only by all methods pooled. This section contains the following for each determinant, where appropriate:
  - a table of results and calculated z-scores;
  - a list of summary statistics;
  - ordered z-score charts; and
- (b) Appendix B contains details of the homogeneity and stability testing.
- (c) Appendix C contains copies of the *Instructions to Participants and Results Sheet*.

### **4. STATISTICAL DESIGN OF THE PROGRAM**

Please note that for Chromium, a target CV (coefficient of variation) was used to calculate the z-scores for both all methods pooled and technique code 1.

- (a) Outlier Results and Z-scores

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 close to zero indicates that the result agrees well with those from other laboratories, 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 “§”.

The table on page 8 summarises the outlier results detected.

(b) Results Tables and Summary Statistics

Each of these tables contains the results returned by each laboratory, including the code number for the method used, and the robust z-score calculated for each result.

Results have been entered exactly as reported by participants. That is, laboratories which did not report results to the precision (i.e. number of decimal places) requested on the Results Sheet have **not** been rounded to the requested precision before being included in the statistical analysis.

A list of summary statistics appears at the bottom of each of the tables of results and consists of:

- the number of results for that test/sample (*No. of Results*);
- the median of these results, i.e. the middle value (*Median*);
- the uncertainty of the median; a robust estimate of the standard deviation of the *Median*;
- the normalised interquartile range of the results (*Normalised IQR*);
- the robust coefficient of variation, expressed as a percentage (*Robust CV*) - i.e.  $100 \times \text{Normalised IQR} / \text{Median}$ ;
- the minimum and maximum laboratory results; and
- the range (*Maximum - 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:

$$\sqrt{\frac{\pi}{2}} \times \frac{\text{normIQR}}{\sqrt{n}} \quad n = \text{number of results}$$

Please see reference [1] for further details on these robust summary statistics.

## (c) Ordered Z-Score Charts

On these charts each laboratory's robust z-score is shown, in order of magnitude, and is marked with its code number. From these charts, each laboratory can readily compare its performance relative to the other laboratories.

These charts contain solid lines at +3.0 and -3.0, so that outliers are clearly identifiable as those laboratories whose "bar" extends beyond these "cut-off" lines. The y-axis of these charts has been limited, so very large z-scores appear to extend beyond the chart boundary.

The following table summarises the results submitted by participants for the program by all methods.

**TABLE A: SUMMARY STATISTICS**

<b>Test</b>	<b>No. of Results</b>	<b>Median</b>	<b>Normalised IQR</b>
Carbon	14	0.0230	0.0029
Manganese	14	1.3745	0.0204
Phosphorus	14	0.0295	0.0017
Sulfur	14	0.0290	0.0024
Silicon	14	0.3855	0.0095
Copper	14	0.5155	0.0287
Nickel	13	10.1195	0.1016
Chromium	13	16.7975	0.0667
Molybdenum	14	2.0363	0.0314
Cobalt	11	0.1505	0.0050
Vanadium	14	0.0795	0.0102

## 5. PTA AND TECHNICAL ADVISER'S COMMENTS

The alloy tested was Grade 316L stainless steel, the 'L' denoting a lower maximum carbon than Grade 316. This is a common grade with widespread usage. The salient alloying elements (chromium, nickel and molybdenum) are very expensive and manufacturers often aim for the low end of the specification as a cost control. It follows that testing of this grade requires a high level of competence, particularly for the three elements mentioned above and carbon. Sulfur, phosphorus, manganese and silicon should also be reported with confidence.

Most results have been generated by AES/Arc spark techniques. LECO OES is similar to AES Arc/spark. Before commencing any analysis by AES Arc/spark, the calibration status of the instrument should be established by analysing one or more Certified Reference Materials (CRMs). Laboratories that have previously participated in PTA programs should have discs from these programs which are relevant to Round 31. If one or more elements in the analysis of CRMs show unsatisfactory results, standard practice is to carry out a 'drift correction' to restore the instrument to its original calibration status. Then, one or more CRMs should be run to confirm the effectiveness of the corrective action. If the results from analysing the CRMs are still unsatisfactory, then the reason(s) must be established and addressed before any analysis resulting in the issue of a test report is carried out.

Where an instrument's calibration does not cover high levels of alloying elements (in this case Ni and Cr), care should be taken in reporting results for other elements as the matrix effect of lower Fe in the test samples may not be known. Laboratory code 12's instrument appears to be inoperative for high levels of Ni and Cr. When high concentrations of alloying elements are present, small changes near the zero point can drastically affect the results for (in this case), Ni and Cr, and this must be taken into account.

### Carbon

The specified maximum for carbon is 0.030%, therefore, accuracy is critical. With the exception of laboratory code 6, all results were acceptable. Laboratory code 6 should check their baseline or low point calibration as the reported result places the sample out of specification for 316L.

### Manganese

With the exception of the outliers, the results were satisfactory. It is likely that the calibrated ranges for Mn on the outliers' instruments do not cover Mn content >1.0%, but confidence in the reported results should have been established by analysis of a CRM.

### Nickel

With the exception of the outliers, these results were acceptable. Laboratory code 1 may have had a baseline drift resulting in the slightly high result and it is likely that laboratory code 12's instrument was not calibrated for this level of Ni.

### Chromium

With the exception of the outliers, these results were very good. Laboratories reporting outliers may have had instruments whose calibrations did not cover this level of Cr.

### Molybdenum

With the exception of the outliers, these results were very good. Laboratory codes 1 and 5 may have calibration curves that do not cover up to 3% Mo, resulting in flattening of the curves near the 2% mark. In practice, manufacturers would aim for a maximum of 2.2%. Laboratory code 12's instrument may not be calibrated for this level of Mo in a stainless steel.

### Sulfur

Most of the results were satisfactory. The results for laboratory code 12 suggest a matrix effect influence i.e. the instrument is not calibrated for high Ni and high Cr (stainless) steels.

### Phosphorus

With the exception of the outliers, these results were satisfactory. Laboratory code 12's calibration is questionable. It is rare to see this (0.05%) level of P in a stainless steel these days. Laboratory codes 5 and 13 may have a problem near the baseline resulting in a negative bias of 0.01%.

### Silicon

The number of outliers was disappointing. Again, the calibration status of the instrument should be established before running the test samples.

### Copper

Although Cu levels are not specified in this grade, it is important to generate accurate results as some stainless steels do have Cu specification limits. Accuracy is also important when trying to confirm or identify a particular heat (batch) of steel. It is likely that laboratory codes 6 and 9 Cu calibrations do not cover the range 0.5% to 1.0% and that laboratory code 12's instrument is not calibrated for this level of Cu in stainless steels.

### Cobalt

With the exception of the outliers, these results were very good. Laboratory code 10's instrument may not be calibrated to this level and Laboratory code 12's instrument may not be calibrated for Co in stainless steel.

### Vanadium

The results were satisfactory.

### Variations within and between laboratories

Generally, the variations within and between laboratories were minimal. Variations between laboratories were primarily due to calibration or calibration status issues.



#### Variation between methods

Most results were generated by AES Arc/spark, the exception being laboratory code 4, who employed AAS for most elements, LECO/Infrared for C and S and Photometry for Si. With the exception of the outliers, there was no appreciable variation between methods.

#### Possible sources of error

As mentioned above, laboratories which consistently yield outliers may be operating outside their normal parameters, or there may be errors of calibration. For any laboratory engaged in this sort of analysis, the parallel running of one or more appropriate CRMs is mandatory. All participants now have a CRM for Grade 316L which may be used for a range of austenitic stainless steels.

#### Measurement Uncertainties

The measurement uncertainties were satisfactory with the exception of Ni, Cr and Mo for laboratory code 13, which were a little high.

This was an appropriate alloy for use in a proficiency test. With the exception of obvious and discounted outliers, the results were good. With laboratories increasingly reliant upon AES Arc/spark, it is critical that appropriate CRMs are used to confirm test results, and that laboratory operatives observe the appropriate protocols for this technique.

#### Metrological Traceability and Measurement Uncertainty of Assigned Values

Consensus values (median) derived from participants' results are used in this program. These values are not metrologically traceable to an external reference.

As the assigned value for this program is the median of the results submitted by the participants, the uncertainty of the median has been calculated and is presented as part of the summary statistics for each element.

#### Analysis of Results by Method Groups

In addition to the pooled method analysis, all tests, except Cobalt, have been analysed by Technique code 1 (AES - Arc/Spark).

## 6. OUTLIER RESULTS

Laboratories reporting outlier results by all methods are listed in the following table:

**TABLE B: SUMMARY OF STATISTICAL OUTLIERS**

<b>Test</b>	<b>Laboratory Code No.</b>
Carbon	6
Manganese	6, 12, 13
Phosphorus	5, 12, 13
Sulfur	5, 12
Silicon	1, 5, 6, 12, 13
Copper	6, 9, 12
Nickel	1
Chromium	1, 13
Molybdenum	1, 5, 12
Cobalt	10, 13
Vanadium	-

## 7. REFERENCE

- [1] *Guide to Proficiency Testing Australia*, 2016 (This document can be found on the PTA website, [www.pta.asn.au](http://www.pta.asn.au))

# APPENDIX A

## Results and Data Analysis

Carbon.....	A1
Manganese.....	A3
Phosphorus.....	A5
Sulfur.....	A7
Silicon.....	A9
Copper.....	A11
Nickel.....	A13
Chromium.....	A15
Molybdenum.....	A17
Cobalt.....	A19
Vanadium.....	A20

**Carbon (0.000%)**

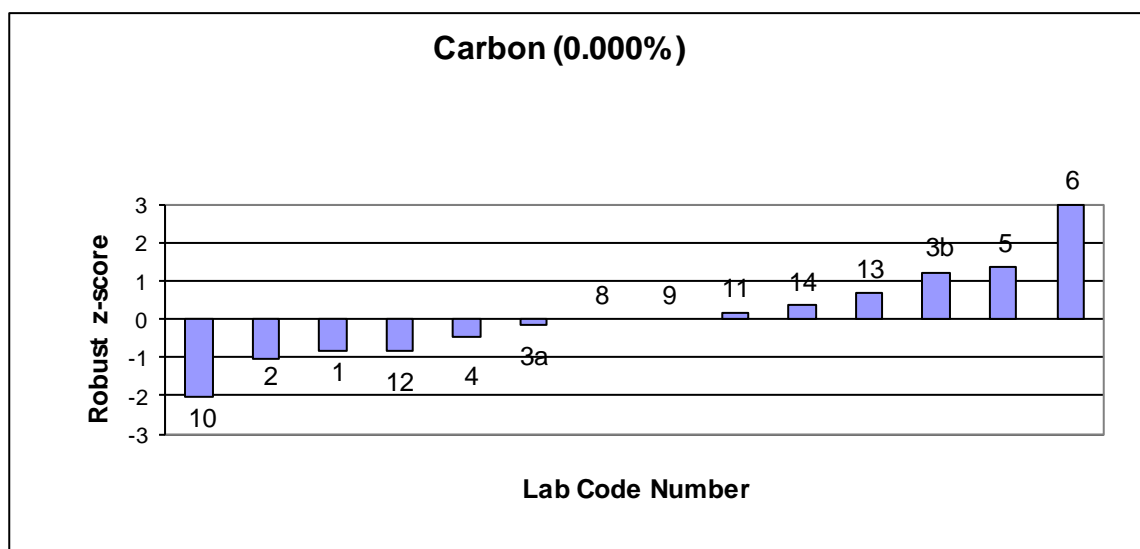
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.021	0.020	0.021	nr	-0.85	1
2	0.020	0.020	0.020	0.000	-1.02	1
3a	0.022	0.023	0.023	0.002	-0.17	1
3b	0.027	0.026	0.027	0.004	1.20	1
4	0.022	0.022	0.022	0.010	-0.44	6
5	0.027	0.027	0.027	0.002	1.37	1
6	0.047	0.047	0.047	0.003	8.20 §	1
8	0.023	0.023	0.023	0.001	0.00	1
9	0.023	0.023	0.023	0.002	0.00	1
10	0.017	0.017	0.017	0.000	-2.05	1
11	0.024	0.023	0.024	0.006	0.17	1
12	0.021	0.020	0.021	0.016	-0.85	1
13	0.024	0.026	0.025	0.005	0.68	6
14	0.024	0.024	0.024	0.005	0.34	1

nr = no result

§ = an outlier result i.e. |z-score| ≥ 3.0

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
6 LECO/OES

No. of Results	14
Median	0.0230
Norm IQR	0.0029
Uncertainty of the Median	0.0010
Robust CV	12.7%
Min	0.017
Max	0.047
Range	0.030



### Carbon (0.000%) - Analysed by Technique Code 1

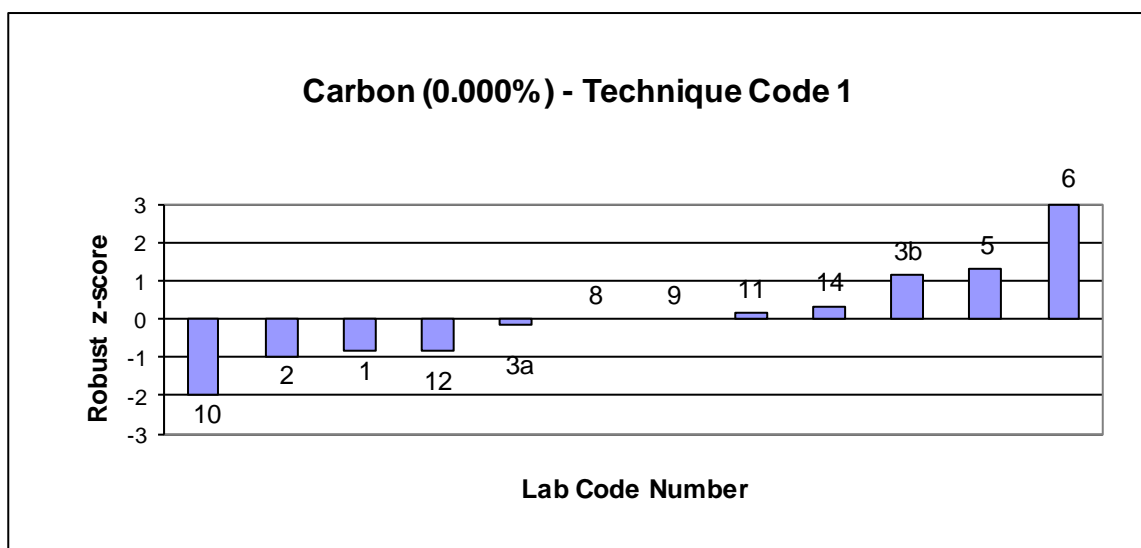
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.021	0.020	0.021	nr	-0.82	1
2	0.020	0.020	0.020	0.000	-0.98	1
3a	0.022	0.023	0.023	0.002	-0.16	1
3b	0.027	0.026	0.027	0.004	1.14	1
5	0.027	0.027	0.027	0.002	1.31	1
6	0.047	0.047	0.047	0.003	7.85 §	1
8	0.023	0.023	0.023	0.001	0.00	1
9	0.023	0.023	0.023	0.002	0.00	1
10	0.017	0.017	0.017	0.000	-1.96	1
11	0.024	0.023	0.024	0.006	0.16	1
12	0.021	0.020	0.021	0.016	-0.82	1
14	0.024	0.024	0.024	0.005	0.33	1

nr = no result

§ = an outlier result i.e.  $|z\text{-score}| \geq 3.0$

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)

No. of Results	12
Median	0.0230
Norm IQR	0.0031
Uncertainty of the Median	0.0011
Robust CV	13.3%
Min	0.017
Max	0.047
Range	0.030



**Manganese (0.000%)**

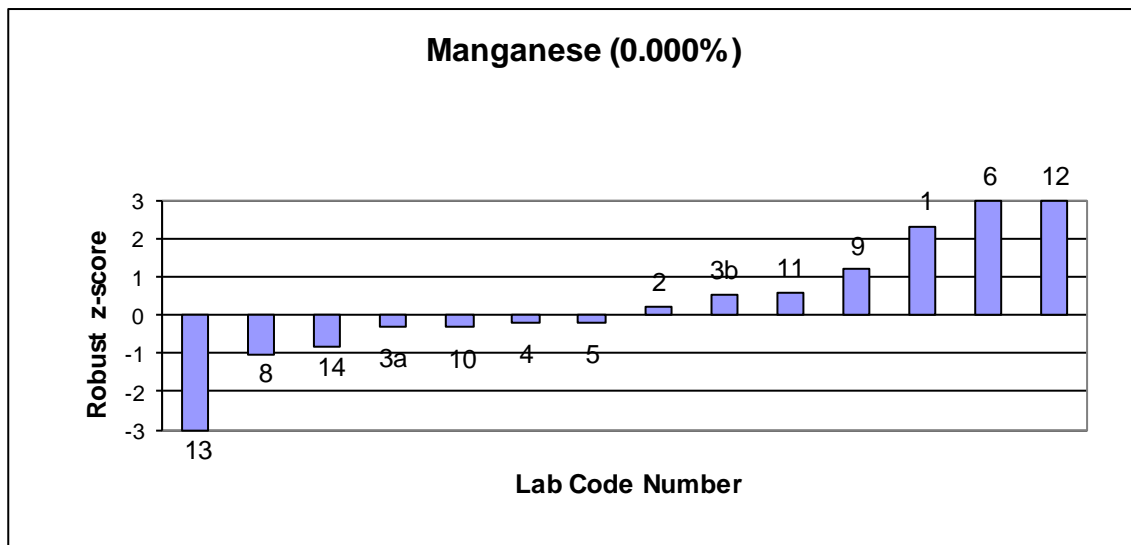
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	1.430	1.412	1.421	nr	2.28	1
2	1.375	1.383	1.379	0.01159	0.22	1
3a	1.360	1.376	1.368	0.017	-0.32	1
3b	1.380	1.391	1.386	0.018	0.54	1
4	1.36	1.38	1.370	0.02	-0.22	3
5	1.37	1.37	1.370	0.036	-0.22	1
6	1.552	1.551	1.552	0.0024	8.68 §	1
8	1.352	1.354	1.353	0.008	-1.05	1
9	1.401	1.396	1.399	0.045	1.18	1
10	1.369	1.367	1.368	0.001	-0.32	1
11	1.389	1.384	1.387	0.018	0.59	1
12	1.692	1.690	1.691	0.041	15.53 §	1
13	1.14	1.16	1.150	0.155	-11.01 §	6
14	1.364	1.351	1.358	0.05	-0.83	1

nr = no result

§ = an outlier result i.e. |z-score| ≥ 3.0

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 3 AAS (Atomic Absorption Spectrometry)  
 6 OES

No. of Results	14
Median	1.3745
Norm IQR	0.0204
Uncertainty of the Median	0.0068
Robust CV	1.5%
Min	1.150
Max	1.691
Range	0.541



### Manganese (0.000%) - Analysed by Technique Code 1

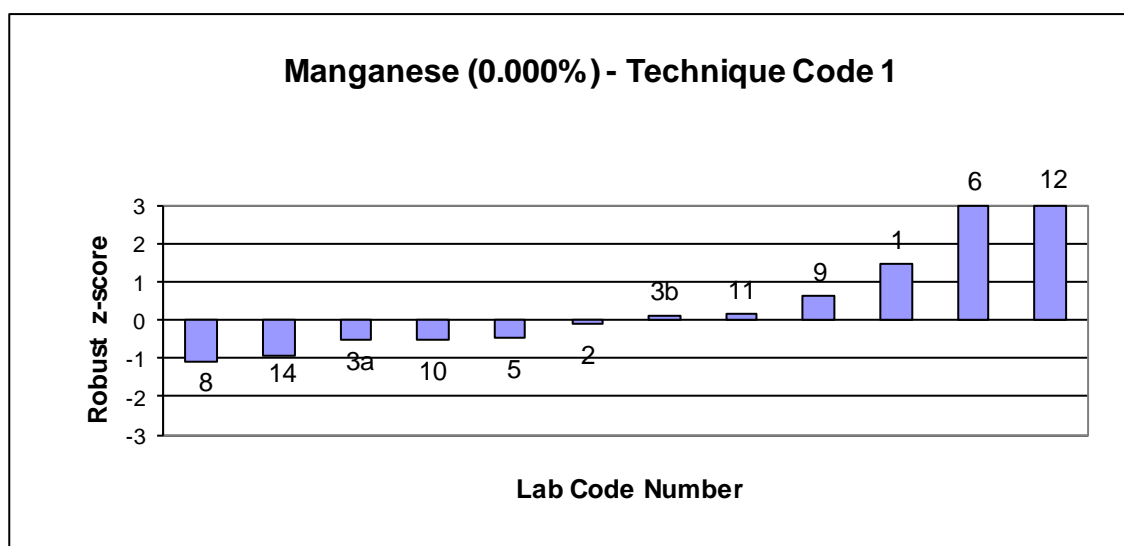
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	1.430	1.412	1.421	nr	1.45	1
2	1.375	1.383	1.379	0.01159	-0.12	1
3a	1.360	1.376	1.368	0.017	-0.53	1
3b	1.380	1.391	1.386	0.018	0.12	1
5	1.37	1.37	1.370	0.036	-0.46	1
6	1.552	1.551	1.552	0.0024	6.32	§
8	1.352	1.354	1.353	0.008	-1.09	1
9	1.401	1.396	1.399	0.045	0.61	1
10	1.369	1.367	1.368	0.001	-0.53	1
11	1.389	1.384	1.387	0.018	0.16	1
12	1.692	1.690	1.691	0.041	11.53	§
14	1.364	1.351	1.358	0.05	-0.92	1

nr = no result

§ = an outlier result i.e.  $|z\text{-score}| \geq 3.0$

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)

No. of Results	12
Median	1.3823
Norm IQR	0.0268
Uncertainty of the Median	0.0097
Robust CV	1.9%
Min	1.353
Max	1.691
Range	0.338



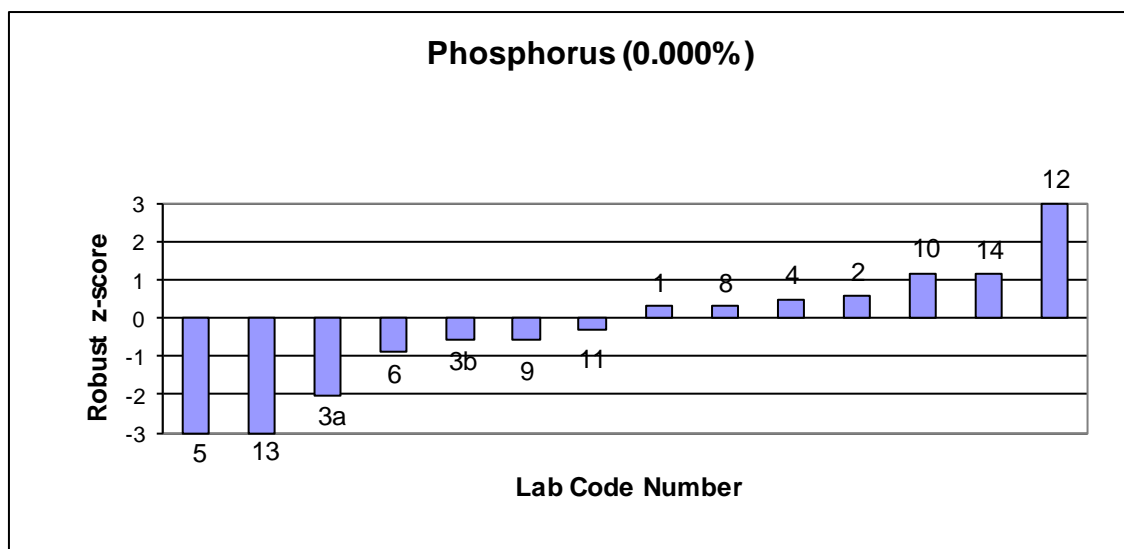
**Phosphorus (0.000%)**

Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.031	0.029	0.030	nr	0.29	1
2	0.03	0.031	0.031	0.00038	0.58	1
3a	0.026	0.026	0.026	0.002	-2.02	1
3b	0.026	0.031	0.029	0.003	-0.58	1
4	0.0302	0.0305	0.030	0.005	0.49	5
5	0.019	0.020	0.020	0.006	-5.77	§ 1
6	0.028	0.028	0.028	7.0 x 10 <sup>-4</sup>	-0.87	1
8	0.030	0.030	0.030	0.002	0.29	1
9	0.028	0.029	0.029	0.002	-0.58	1
10	0.033	0.03	0.032	0.002	1.15	1
11	0.029	0.029	0.029	0.006	-0.29	1
12	0.050	0.050	0.050	0.002	11.83	§ 1
13	0.02	0.023	0.022	0.004	-4.62	§ 6
14	0.032	0.031	0.032	0.003	1.15	1

nr = no result

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
5 Photometric  
6 OES

No. of Results	14
Median	0.0295
Norm IQR	0.0017
Uncertainty of the Median	0.0006
Robust CV	5.9%
Min	0.020
Max	0.050
Range	0.031





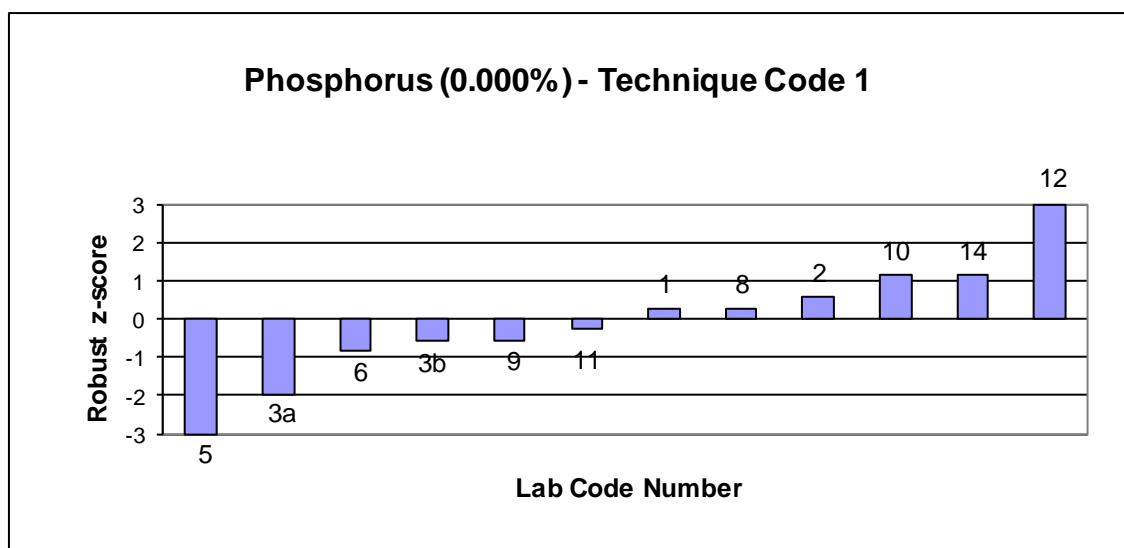
### Phosphorus (0.000%) - Analysed by Technique Code 1

Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.031	0.029	0.030	nr	0.28	1
2	0.03	0.031	0.031	0.00038	0.57	1
3a	0.026	0.026	0.026	0.002	-1.99	1
3b	0.026	0.031	0.029	0.003	-0.57	1
5	0.019	0.020	0.020	0.006	-5.68	1
6	0.028	0.028	0.028	7.0 x 10 <sup>-4</sup>	-0.85	1
8	0.030	0.030	0.030	0.002	0.28	1
9	0.028	0.029	0.029	0.002	-0.57	1
10	0.033	0.03	0.032	0.002	1.14	1
11	0.029	0.029	0.029	0.006	-0.28	1
12	0.050	0.050	0.050	0.002	11.64	1
14	0.032	0.031	0.032	0.003	1.14	1

nr = no result

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)

No. of Results	12
Median	0.0295
Norm IQR	0.0018
Uncertainty of the Median	0.0006
Robust CV	6.0%
Min	0.020
Max	0.050
Range	0.031



**Sulfur (0.000%)**

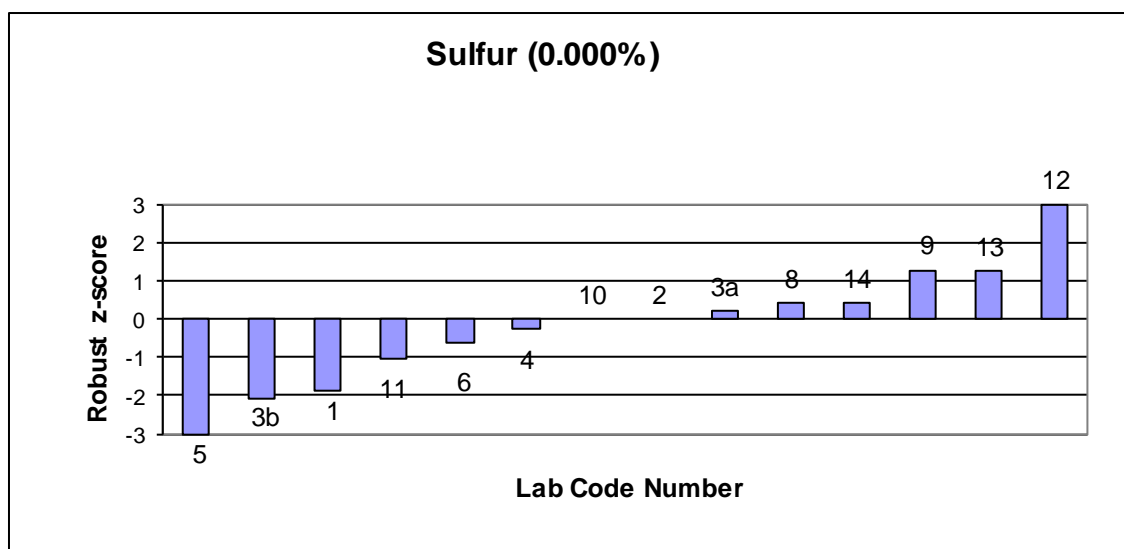
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.026	0.023	0.0245	nr	-1.87	1
2	0.029	0.029	0.029	0.00046	0.00	1
3a	0.030	0.029	0.0295	0.004	0.21	1
3b	0.022	0.026	0.024	0.002	-2.08	1
4	0.0282	0.0285	0.02835	0.005	-0.27	6
5	0.021	0.022	0.0215	0.006	-3.11	§
6	0.027	0.028	0.0275	5.8 x 10 <sup>-5</sup>	-0.62	1
8	0.030	0.030	0.03	0.003	0.42	1
9	0.032	0.032	0.032	0.005	1.25	1
10	0.030	0.028	0.029	0.001	0.00	1
11	0.027	0.026	0.0265	0.002	-1.04	1
12	0.051	0.055	0.053	0.004	9.96	§
13	0.030	0.034	0.032	0.006	1.25	6
14	0.029	0.031	0.03	0.003	0.42	1

nr = no result

§ = an outlier result i.e. |z-score| ≥ 3.0

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
6 LECO/OES

No. of Results	14
Median	0.0290
Norm IQR	0.0024
Uncertainty of the Median	0.0008
Robust CV	8.3%
Min	0.022
Max	0.053
Range	0.032



### Sulfur (0.000%) - Analysed by Technique Code 1

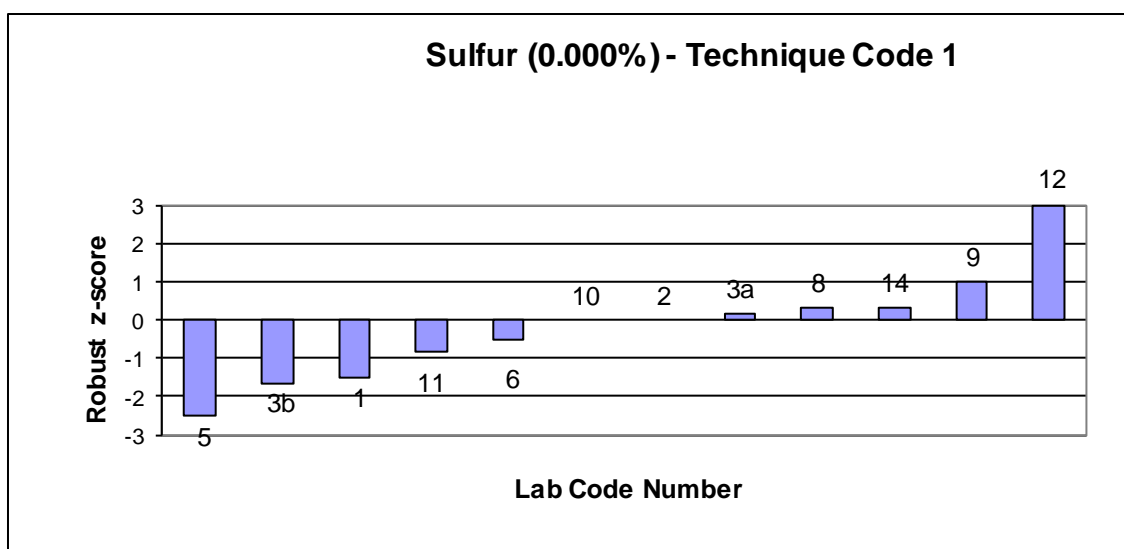
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.026	0.023	0.0245	nr	-1.52	1
2	0.029	0.029	0.029	0.00046	0.00	1
3a	0.030	0.029	0.0295	0.004	0.17	1
3b	0.022	0.026	0.024	0.002	-1.69	1
5	0.021	0.022	0.0215	0.006	-2.53	1
6	0.027	0.028	0.0275	$5.8 \times 10^{-5}$	-0.51	1
8	0.030	0.030	0.03	0.003	0.34	1
9	0.032	0.032	0.032	0.005	1.01	1
10	0.030	0.028	0.029	0.001	0.00	1
11	0.027	0.026	0.0265	0.002	-0.84	1
12	0.051	0.055	0.053	0.004	8.09 §	1
14	0.029	0.031	0.03	0.003	0.34	1

nr = no result

§ = an outlier result i.e.  $|z\text{-score}| \geq 3.0$

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)

No. of Results	12
Median	0.0290
Norm IQR	0.0030
Uncertainty of the Median	0.0011
Robust CV	10.2%
Min	0.022
Max	0.053
Range	0.032



**Silicon (0.000%)**

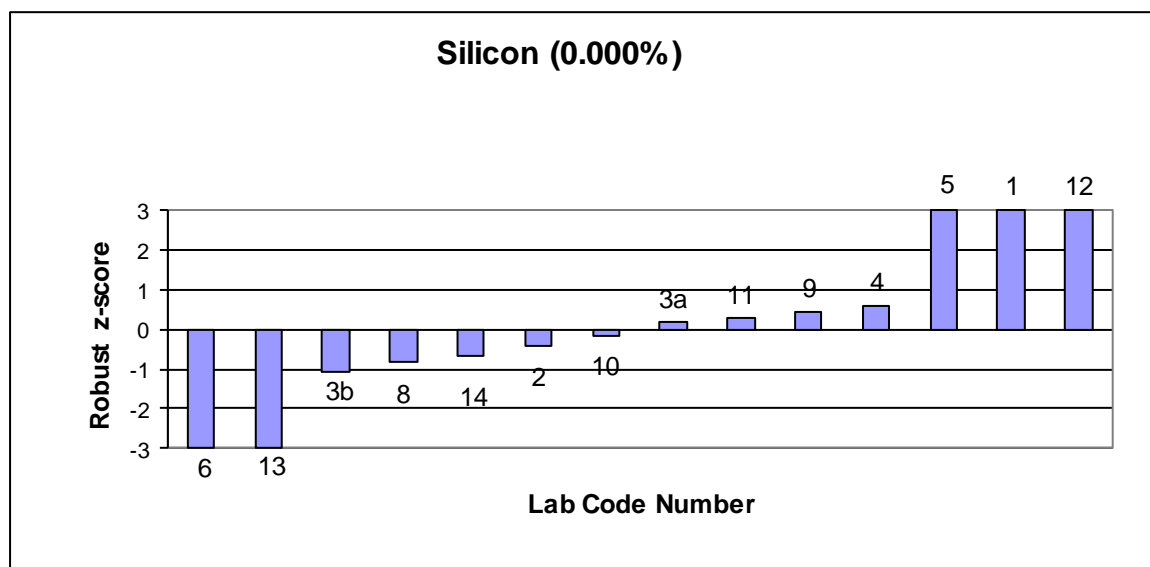
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.461	0.458	0.4595	nr	7.83 §	1
2	0.382	0.381	0.3815	0.00293	-0.42	1
3a	0.385	0.389	0.387	0.003	0.16	1
3b	0.375	0.376	0.3755	0.013	-1.06	1
4	0.390	0.392	0.391	0.01	0.58	6
5	0.425	0.423	0.424	0.044	4.07 §	1
6	0.271	0.275	0.273	0.017	-11.90 §	1
8	0.377	0.378	0.3775	0.006	-0.85	1
9	0.389	0.39	0.3895	0.009	0.42	1
10	0.384	0.384	0.384	0.0001	-0.16	1
11	0.389	0.387	0.388	0.036	0.26	1
12	0.488	0.491	0.4895	0.011	11.00 §	1
13	0.35	0.35	0.35	0.047	-3.76 §	6
14	0.386	0.372	0.379	0.03	-0.69	1

nr = no result

§ = an outlier result i.e.  $|z\text{-score}| \geq 3.0$ 

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
6 LECO/OES

No. of Results	14
Median	0.3855
Norm IQR	0.0095
Uncertainty of the Median	0.0032
Robust CV	2.5%
Min	0.273
Max	0.490
Range	0.217



**Silicon (0.000%) - Analysed by Technique Code 1**

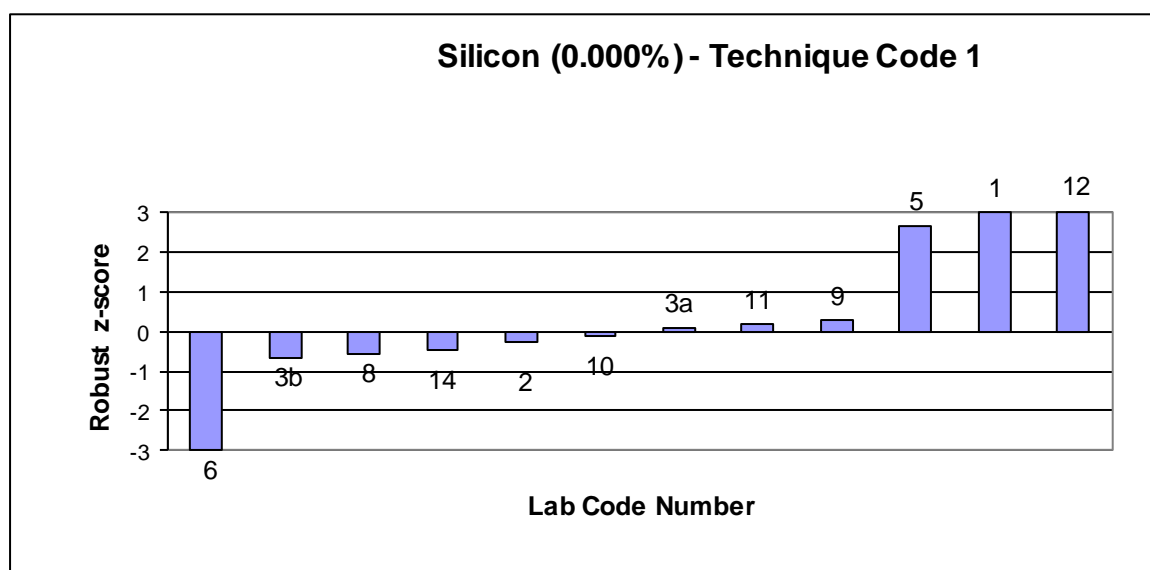
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.461	0.458	0.4595	nr	5.12 §	1
2	0.382	0.381	0.3815	0.00293	-0.28	1
3a	0.385	0.389	0.387	0.003	0.10	1
3b	0.375	0.376	0.3755	0.013	-0.69	1
5	0.425	0.423	0.424	0.044	2.66	1
6	0.271	0.275	0.273	0.017	-7.78 §	1
8	0.377	0.378	0.3775	0.006	-0.55	1
9	0.389	0.39	0.3895	0.009	0.28	1
10	0.384	0.384	0.384	0.0001	-0.10	1
11	0.389	0.387	0.388	0.036	0.17	1
12	0.488	0.491	0.4895	0.011	7.19 §	1
14	0.386	0.372	0.379	0.03	-0.45	1

nr = no result

§ = an outlier result i.e. |z-score| ≥ 3.0

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)

No. of Results	12
Median	0.3855
Norm IQR	0.0145
Uncertainty of the Median	0.0052
Robust CV	3.7%
Min	0.273
Max	0.490
Range	0.217



**Copper (0.000%)**

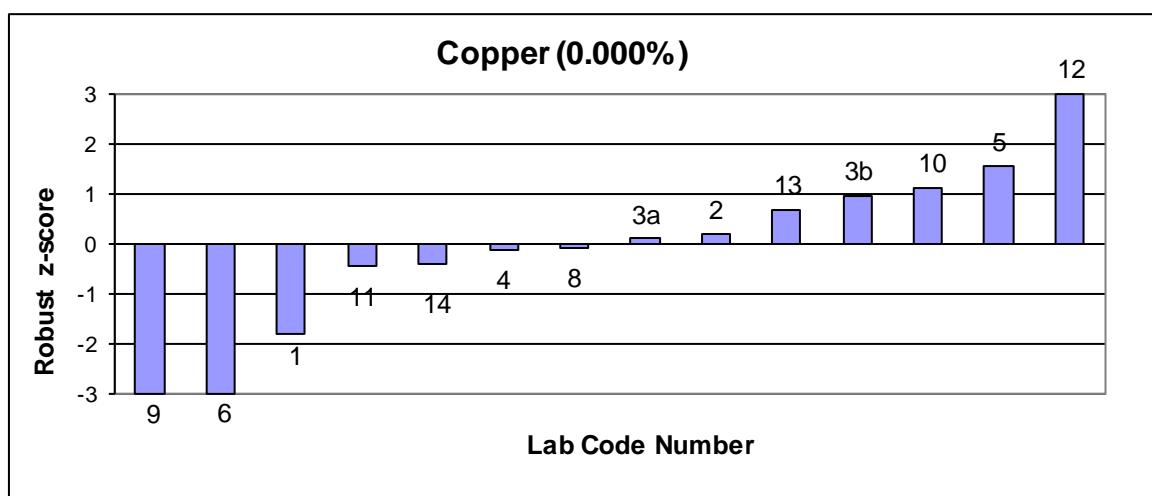
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.469	0.457	0.463	nr	-1.83	1
2	0.518	0.524	0.521	0.00139	0.19	1
3a	0.528	0.508	0.518	0.015	0.09	1
3b	0.544	0.542	0.543	0.028	0.96	1
4	0.511	0.513	0.512	0.01	-0.12	3
5	0.56	0.56	0.560	0.018	1.55	1
6	0.37	0.365	0.368	0.039	-5.15 §	1
8	0.515	0.511	0.513	0.005	-0.09	1
9	0.152	0.153	0.153	0.023	-12.64 §	1
10	0.548	0.546	0.547	0.001	1.10	1
11	0.502	0.502	0.502	0.008	-0.47	1
12	0.648	0.651	0.650	0.005	4.66 §	1
13	0.52	0.55	0.535	0.072	0.68	6
14	0.506	0.500	0.503	0.03	-0.44	1

nr = no result

§ = an outlier result i.e.  $|z\text{-score}| \geq 3.0$ 

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 3 AAS (Atomic Absorption Spectrometry)  
 6 OES

No. of Results	14
Median	0.5155
Norm IQR	0.0287
Uncertainty of the Median	0.0096
Robust CV	5.6%
Min	0.153
Max	0.650
Range	0.497



**Copper (0.000%) - Analysed by Technique Code 1**

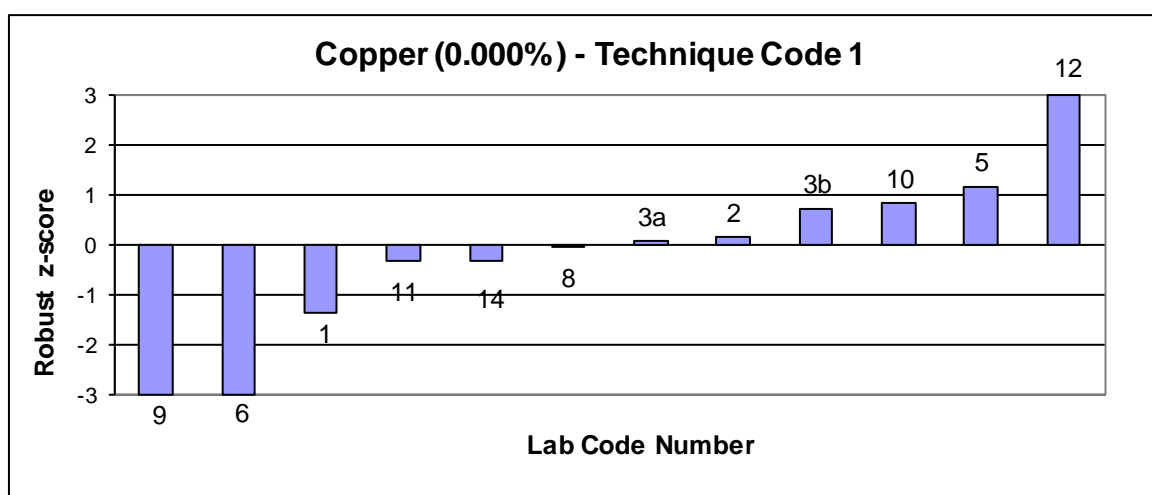
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.469	0.457	0.463	nr	-1.37	1
2	0.518	0.524	0.521	0.00139	0.14	1
3a	0.528	0.508	0.518	0.015	0.07	1
3b	0.544	0.542	0.543	0.028	0.72	1
5	0.56	0.56	0.560	0.018	1.16	1
6	0.37	0.365	0.368	0.039	-3.86 §	1
8	0.515	0.511	0.513	0.005	-0.07	1
9	0.152	0.153	0.153	0.023	-9.46 §	1
10	0.548	0.546	0.547	0.001	0.82	1
11	0.502	0.502	0.502	0.008	-0.35	1
12	0.648	0.651	0.650	0.005	3.49 §	1
14	0.506	0.500	0.503	0.03	-0.33	1

nr = no result

§ = an outlier result i.e. |z-score| ≥ 3.0

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)

No. of Results	12
Median	0.5155
Norm IQR	0.0384
Uncertainty of the Median	0.0139
Robust CV	7.4%
Min	0.153
Max	0.650
Range	0.497



**Nickel (0.000%)**

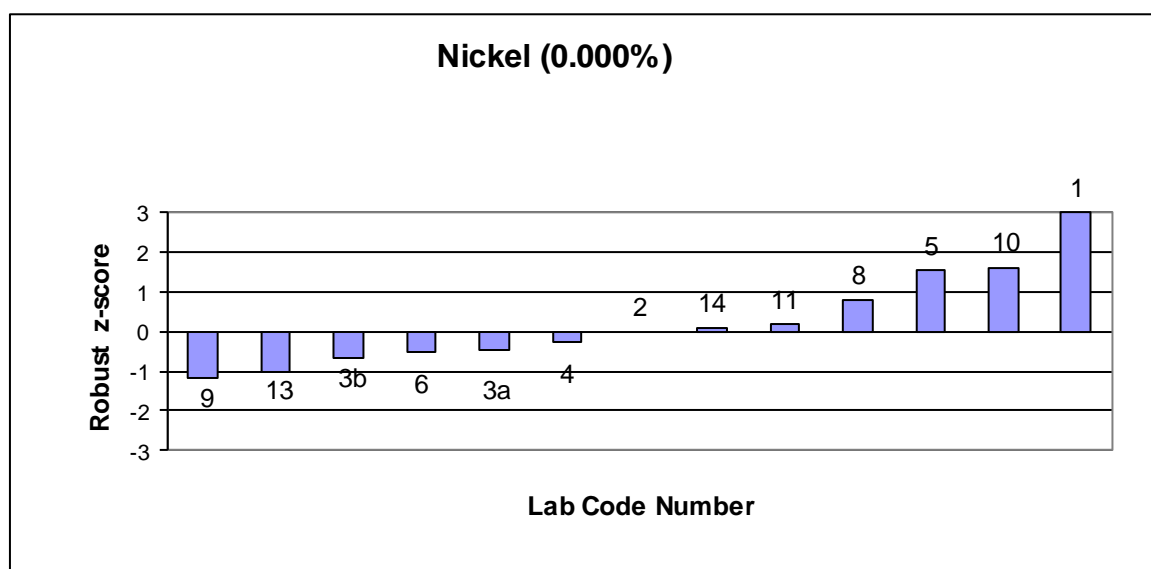
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	10.531	10.446	10.489	nr	3.63 §	1
2	10.117	10.122	10.120	0.01863	0.00	1
3a	10.059	10.082	10.071	0.144	-0.48	1
3b	10.040	10.062	10.051	0.150	-0.67	1
4	10.08	10.10	10.090	0.1	-0.29	3
5	10.27	10.28	10.275	0.214	1.53	1
6	10.055	10.074	10.065	0.190	-0.54	1
8	10.230	10.173	10.202	0.091	0.81	1
9	9.982	10.019	10.001	0.197	-1.17	1
10	10.325	10.239	10.282	0.06	1.60	1
11	10.147	10.130	10.139	0.153	0.19	1
12	>6.600	>6.600		0.020		1
13	9.99	10.04	10.015	1.232	-1.03	6
14	10.180	10.080	10.130	0.20	0.10	1

nr = no result

§ = an outlier result i.e. |z-score| ≥ 3.0

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 3 AAS (Atomic Absorption Spectrometry)  
 6 OES

No. of Results	13
Median	10.1195
Norm IQR	0.1016
Uncertainty of the Median	0.0353
Robust CV	1.0%
Min	10.001
Max	10.489
Range	0.488





### Nickel (0.000%) - Analysed by Technique Code 1

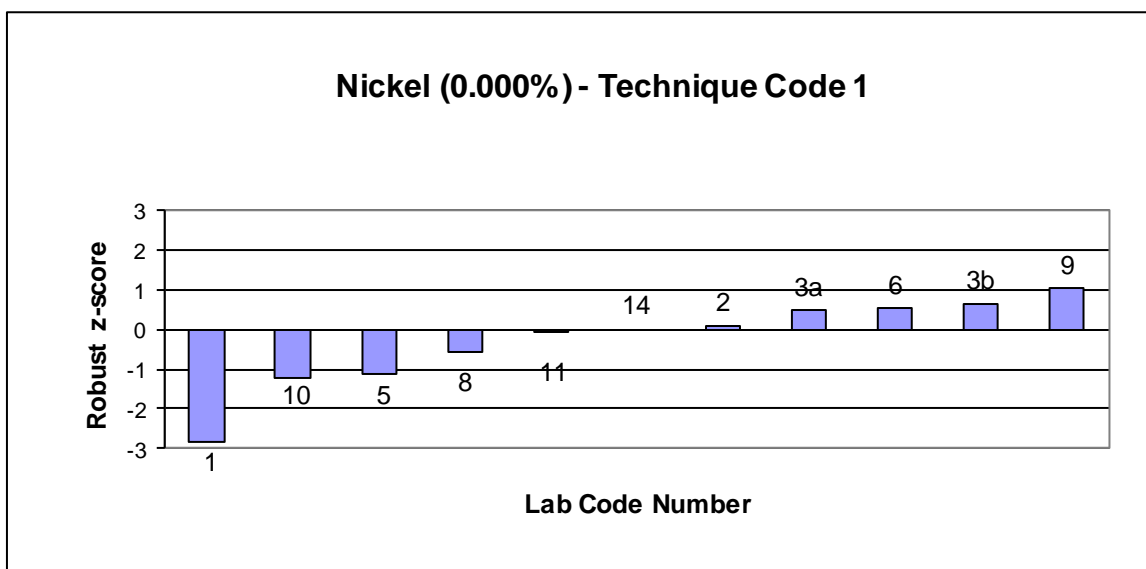
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	10.531	10.446	10.489	nr	2.83	1
2	10.117	10.122	10.120	0.01863	-0.08	1
3a	10.059	10.082	10.071	0.144	-0.47	1
3b	10.040	10.062	10.051	0.150	-0.62	1
5	10.27	10.28	10.275	0.214	1.15	1
6	10.055	10.074	10.065	0.190	-0.52	1
8	10.230	10.173	10.202	0.091	0.56	1
9	9.982	10.019	10.001	0.197	-1.02	1
10	10.325	10.239	10.282	0.06	1.20	1
11	10.147	10.130	10.139	0.153	0.07	1
12	>6.600	>6.600		0.020		1
14	10.180	10.080	10.130	0.20	0.00	1

nr = no result

§ = an outlier result i.e. |z-score| ≥ 3.0

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)

No. of Results	11
Median	10.1300
Norm IQR	0.1266
Uncertainty of the Median	0.0478
Robust CV	1.2%
Min	10.001
Max	10.489
Range	0.488



**Chromium (0.000%)**

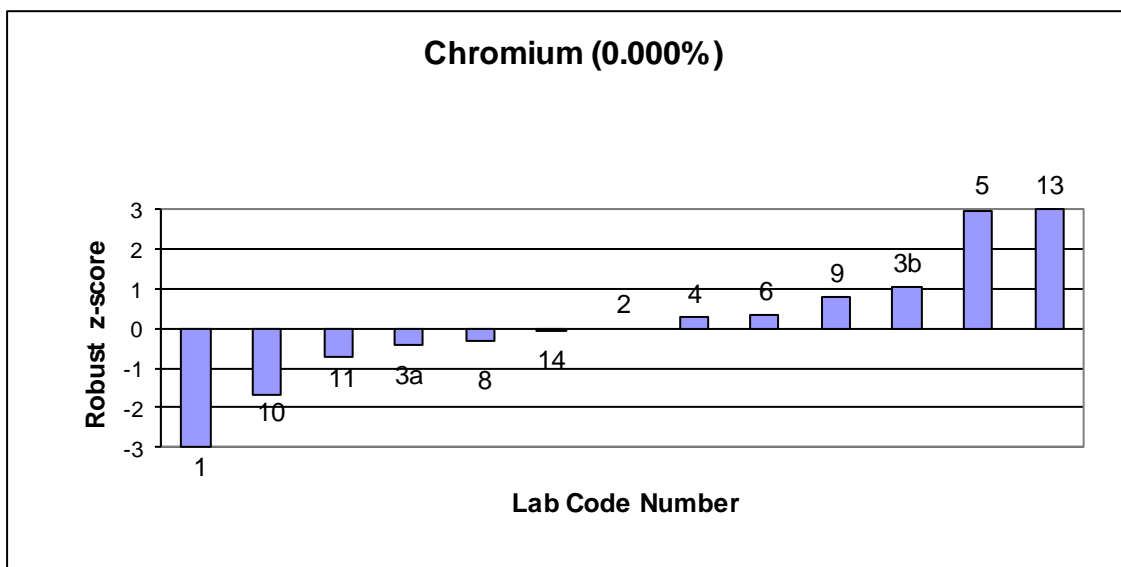
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	14.003	14.247	14.125	nr	-40.09 §	1
2	16.795	16.800	16.798	0.01159	0.00	1
3a	16.788	16.753	16.771	0.054	-0.41	1
3b	16.832	16.903	16.868	0.177	1.05	1
4	16.80	16.83	16.815	0.1	0.26	3
5	16.93	17.06	16.995	0.088	2.96	1
6	16.781	16.858	16.820	0.160	0.33	1
8	16.763	16.791	16.777	0.068	-0.31	1
9	16.817	16.883	16.850	0.092	0.79	1
10	16.664	16.708	16.686	0.03	-1.67	1
11	16.733	16.764	16.749	0.244	-0.73	1
12	>6.000	>6.000		0.003		1
13	17.33	17.33	17.330	2.132	7.99 §	6
14	16.78	16.81	16.795	0.20	-0.04	1

nr = no result

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
3 AAS (Atomic Absorption Spectrometry)  
6 OES

No. of Results	13
Median	16.7975
Norm IQR	0.0667
Uncertainty of the Median	0.0232
Robust CV	0.397%
Min	14.125
Max	17.330
Range	3.205

Please note that a target CV has been used to calculate the z-scores



**Chromium (0.000%) - Analysed by Technique Code 1**

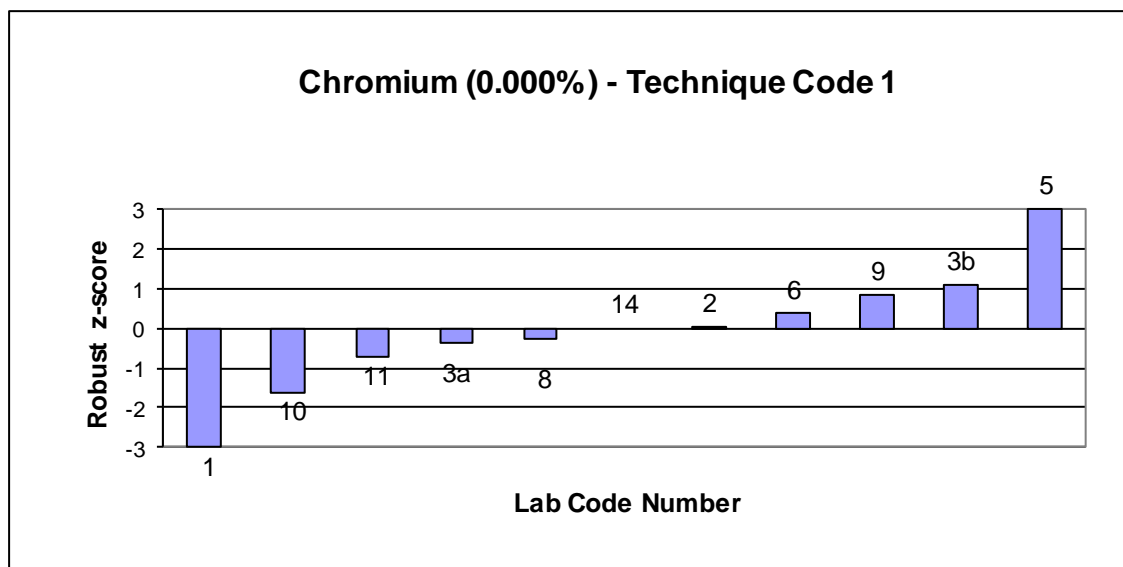
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	14.003	14.247	14.125	nr	-40.06	1
2	16.795	16.800	16.798	0.01159	0.04	1
3a	16.788	16.753	16.771	0.054	-0.37	1
3b	16.832	16.903	16.868	0.177	1.09	1
5	16.93	17.06	16.995	0.088	3.00	1
6	16.781	16.858	16.820	0.160	0.37	1
8	16.763	16.791	16.777	0.068	-0.27	1
9	16.817	16.883	16.850	0.092	0.83	1
10	16.664	16.708	16.686	0.03	-1.64	1
11	16.733	16.764	16.749	0.244	-0.70	1
12	>6.000	>6.000		0.003		1
14	16.78	16.81	16.795	0.20	0.00	1

nr = no result

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)

No. of Results	11
Median	16.7950
Norm IQR	0.0667
Uncertainty of the Median	0.0252
Robust CV	0.397%
Min	14.125
Max	16.995
Range	2.870

Please note that a target CV has been used to calculate the z-scores



**Molybdenum (0.000%)**

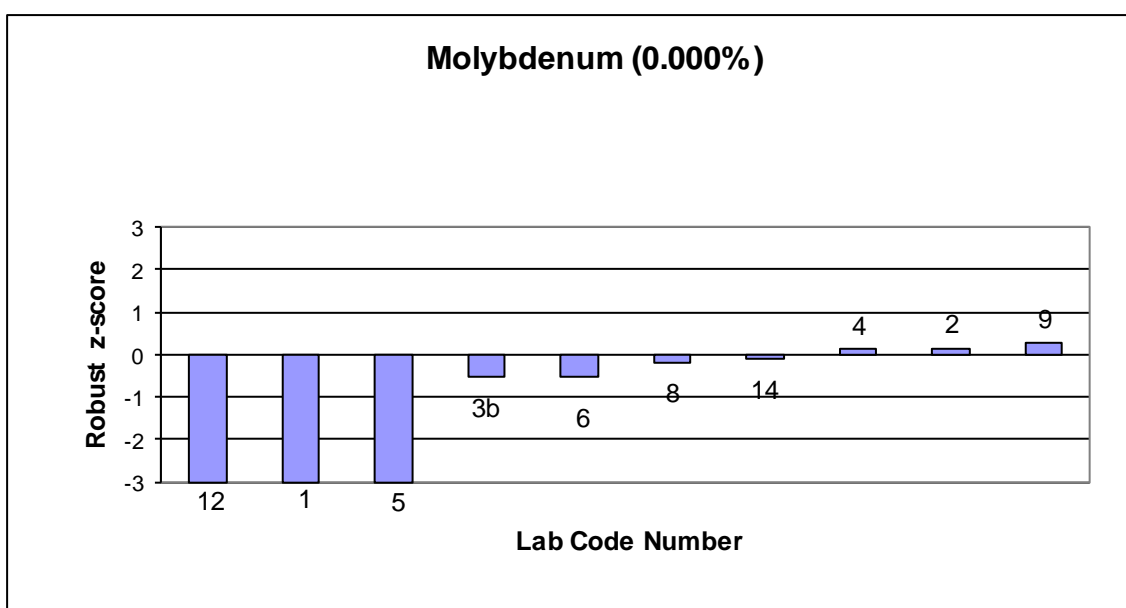
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	1.922	1.889	1.906	nr	-4.16 §	1
2	2.049	2.033	2.041	0.00591	0.15	1
3a	2.074	2.061	2.068	0.035	0.99	1
3b	2.019	2.020	2.020	0.049	-0.53	1
4	2.04	2.04	2.040	0.01	0.12	3
5	1.92	1.90	1.910	0.054	-4.02 §	1
6	2.021	2.018	2.020	0.023	-0.53	1
8	2.032	2.029	2.031	0.016	-0.18	1
9	2.052	2.038	2.045	0.044	0.28	1
10	2.077	2.069	2.073	0.005	1.17	1
11	2.072	2.066	2.069	0.074	1.04	1
12	1.642	1.608	1.625	0.003	-13.09 §	1
13	2.07	2.11	2.090	2.282	1.71	6
14	2.056	2.009	2.033	0.05	-0.12	1

nr = no result

§ = an outlier result i.e.  $|z\text{-score}| \geq 3.0$ 

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 3 AAS (Atomic Absorption Spectrometry)  
 6 OES

No. of Results	14
Median	2.0363
Norm IQR	0.0314
Uncertainty of the Median	0.0105
Robust CV	1.5%
Min	1.906
Max	2.090
Range	0.185



**Molybdenum (0.000%) - Analysed by Technique Code 1**

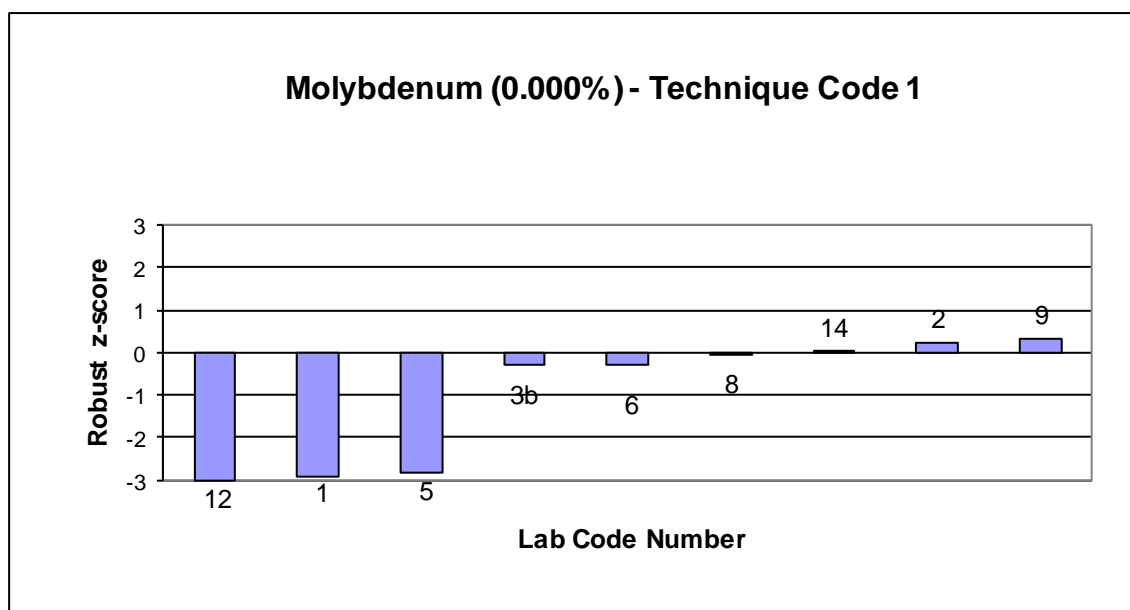
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	1.922	1.889	1.906	nr	-2.91	1
2	2.049	2.033	2.041	0.00591	0.22	1
3a	2.074	2.061	2.068	0.035	0.83	1
3b	2.019	2.020	2.020	0.049	-0.28	1
5	1.92	1.90	1.910	0.054	-2.80	1
6	2.021	2.018	2.020	0.023	-0.28	1
8	2.032	2.029	2.031	0.016	-0.02	1
9	2.052	2.038	2.045	0.044	0.31	1
10	2.077	2.069	2.073	0.005	0.96	1
11	2.072	2.066	2.069	0.074	0.86	1
12	1.642	1.608	1.625	0.003	-9.37	§
14	2.056	2.009	2.033	0.05	0.02	1

nr = no result

§ = an outlier result i.e.  $|z\text{-score}| \geq 3.0$ 

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)

No. of Results	12
Median	2.0315
Norm IQR	0.0434
Uncertainty of the Median	0.0157
Robust CV	2.1%
Min	1.906
Max	2.073
Range	0.168



**Cobalt (0.000%)**

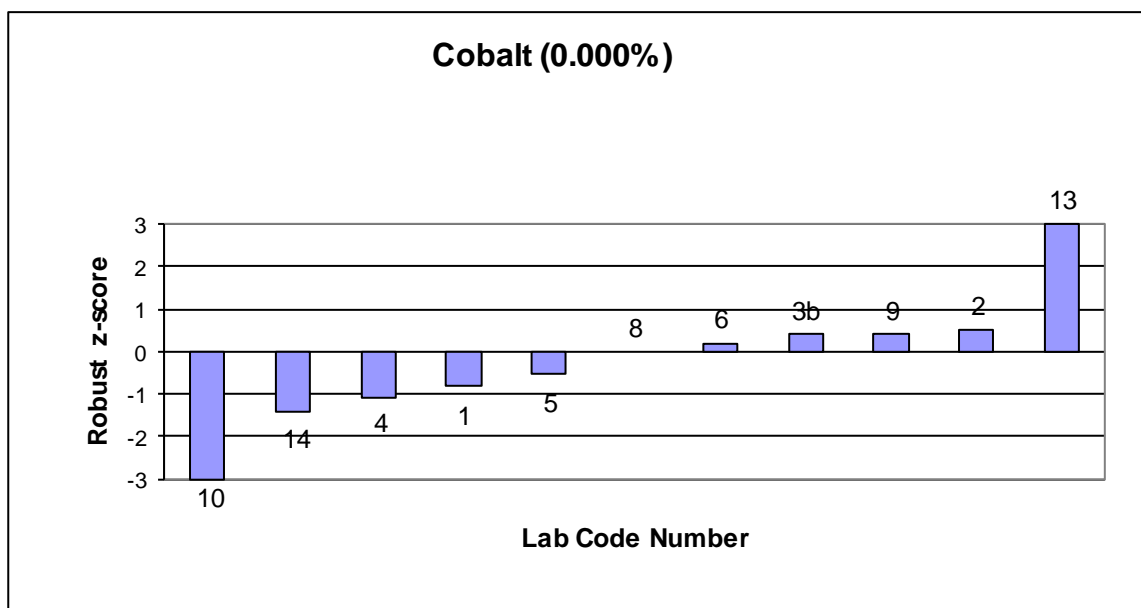
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.148	0.145	0.1465	nr	-0.80	1
2	0.153	0.153	0.153	0.00054	0.50	1
3b	0.153	0.152	0.1525	0.007	0.40	1
4	0.144	0.146	0.145	0.01	-1.10	3
5	0.147	0.149	0.148	0.006	-0.50	1
6	0.151	0.152	0.1515	0.00026	0.20	1
8	0.151	0.15	0.1505	0.002	0.00	1
9	0.152	0.153	0.1525	0.002	0.40	1
10	0.117	0.118	0.1175	0.0003	-6.60	§ 1
13	0.18	0.18	0.18	0.024	5.90	§ 6
14	0.142	0.145	0.1435	0.02	-1.40	1

nr = no result

§ = an outlier result i.e. |z-score| ≥ 3.0

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 3 AAS (Atomic Absorption Spectrometry)  
 6 OES

No. of Results	11
Median	0.1505
Norm IQR	0.0050
Uncertainty of the Median	0.0019
Robust CV	3.3%
Min	0.118
Max	0.180
Range	0.063



**Vanadium (0.000%)**

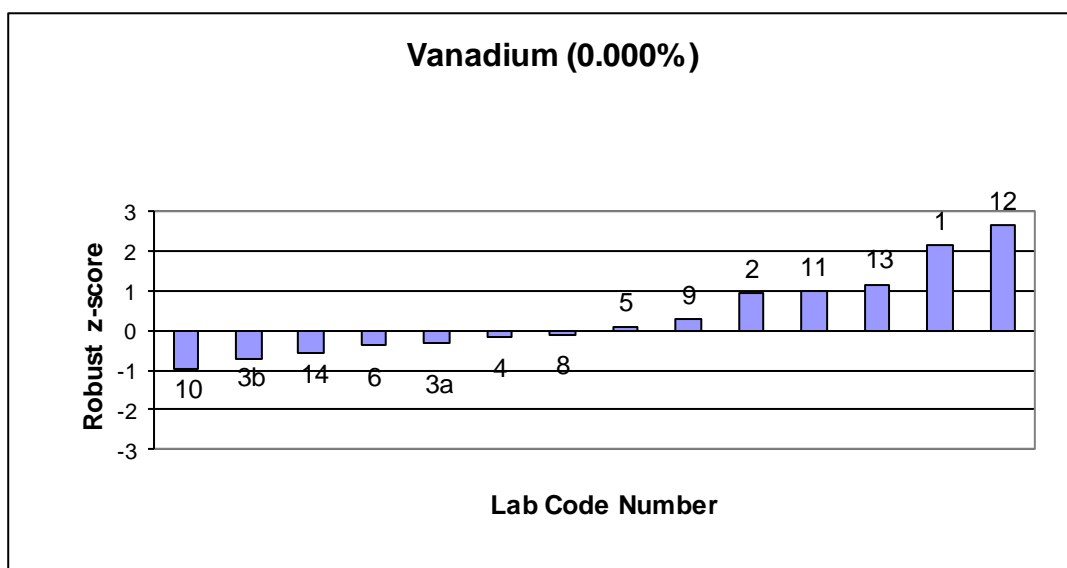
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.102	0.101	0.102	nr	2.16	1
2	0.089	0.089	0.089	0.00022	0.93	1
3a	0.076	0.076	0.076	0.003	-0.34	1
3b	0.072	0.072	0.072	0.005	-0.74	1
4	0.0773	0.0779	0.078	0.01	-0.19	3
5	0.080	0.081	0.081	0.002	0.10	1
6	0.075	0.076	0.076	0.001	-0.39	1
8	0.078	0.079	0.079	0.002	-0.10	1
9	0.083	0.082	0.083	0.001	0.29	1
10	0.069	0.070	0.070	0.0005	-0.98	1
11	0.090	0.089	0.090	0.001	0.98	1
12	0.106	0.107	0.107	0.001	2.65	1
13	0.090	0.092	0.091	0.012	1.13	6
14	0.073	0.074	0.074	0.003	-0.59	1

nr = no result

§ = an outlier result i.e. |z-score| ≥ 3.0

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
3 AAS (Atomic Absorption Spectrometry)  
6 OES

No. of Results	14
Median	0.0795
Norm IQR	0.0102
Uncertainty of the Median	0.0034
Robust CV	12.8%
Min	0.070
Max	0.107
Range	0.037



### Vanadium (0.000%) - Analysed by Technique Code 1

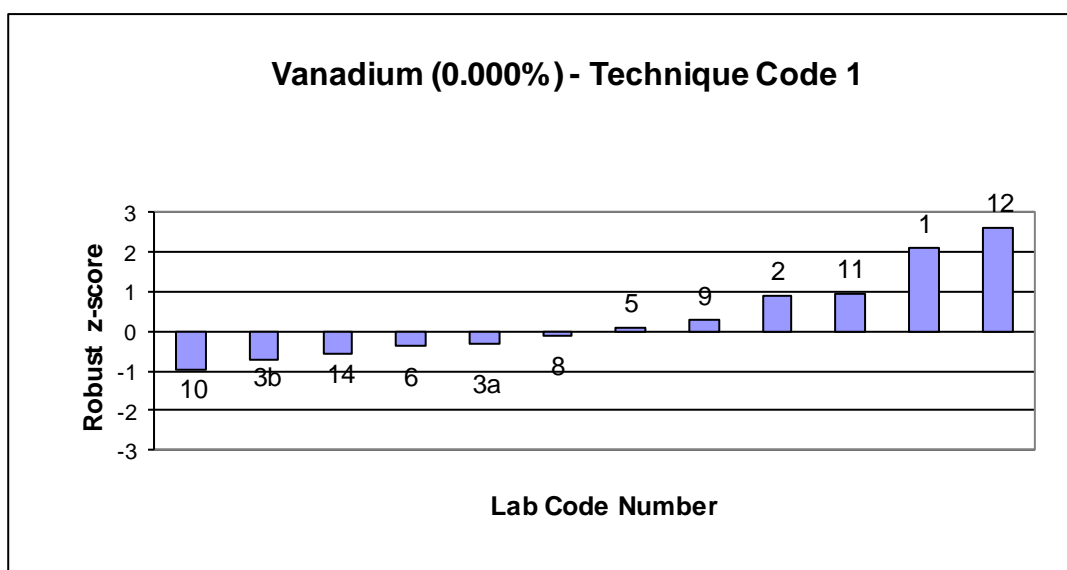
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.102	0.101	0.102	nr	2.10	1
2	0.089	0.089	0.089	0.00022	0.91	1
3a	0.076	0.076	0.076	0.003	-0.33	1
3b	0.072	0.072	0.072	0.005	-0.72	1
5	0.080	0.081	0.081	0.002	0.10	1
6	0.075	0.076	0.076	0.001	-0.38	1
8	0.078	0.079	0.079	0.002	-0.10	1
9	0.083	0.082	0.083	0.001	0.29	1
10	0.069	0.070	0.070	0.0005	-0.96	1
11	0.090	0.089	0.090	0.001	0.96	1
12	0.106	0.107	0.107	0.001	2.58	1
14	0.073	0.074	0.074	0.003	-0.57	1

nr = no result

§ = an outlier result i.e. |z-score| ≥ 3.0

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)

No. of Results	12
Median	0.0795
Norm IQR	0.0105
Uncertainty of the Median	0.0038
Robust CV	13.2%
Min	0.070
Max	0.107
Range	0.037





# APPENDIX B

## Homogeneity and Stability Testing

Sample Preparation and Homogeneity Testing.....	B1
Stability Testing.....	B1

### **Sample Preparation and Homogeneity**

The samples were supplied by Universal Scientific Laboratory Pty Ltd.

Eight discs were selected and tested for each element and the results are shown in the following table:

Carbon	Sulfur	Phosphorus	Silicon
0.0223	0.0289	0.0254	0.392
0.0212	0.0290	0.0247	0.392
0.0208	0.0294	0.0258	0.393
0.0209	0.0281	0.0260	0.388
0.0213	0.0286	0.0263	0.391
0.0201	0.0295	0.0265	0.383
0.0221	0.0299	0.0254	0.384
0.0201	0.0299	0.0263	0.386
Manganese	Chromium	Nickel	Copper
1.363	16.81	10.03	0.510
1.390	16.88	10.02	0.524
1.380	16.72	10.01	0.520
1.381	16.82	10.07	0.518
1.367	16.81	10.06	0.527
1.392	16.72	10.05	0.522
1.376	16.58	10.08	0.520
1.375	16.78	10.02	0.521
Molybdenum	Vanadium	Cobalt	
2.048	0.0768	0.155	
2.060	0.0780	0.156	
2.075	0.0779	0.156	
2.073	0.0783	0.156	
2.056	0.0782	0.154	
2.083	0.0794	0.155	
2.094	0.0782	0.156	
2.068	0.0777	0.154	

Analysis of this data indicated that the samples were sufficiently homogenous and, therefore, any results later identified as outliers could not be attributed to sample variability.

### **Stability Testing**

Due to the nature of the samples it was not considered necessary to perform stability testing.

# APPENDIX C

## Documentation

Instructions to Participants .....	C1
Results Sheet .....	C2



Proficiency Testing Australia

## Proficiency Testing Program

### Metal Alloys (Round 31) – October 2016

#### **INSTRUCTIONS TO PARTICIPANTS**

Please read instructions carefully **BEFORE** commencing testing.

1. For this round each participant will be supplied with one steel disc.
2. Participants are asked to test the percentage composition (in duplicate) for each sample for the following elements:  
Carbon, Sulfur, Phosphorus, Silicon, Manganese, Chromium, Nickel, Copper, Molybdenum, Vanadium and Cobalt.  
If the analysis of any element is not possible, please note this on the results sheet.  
Please be advised that the initial measurement recorded is to be noted as "Result 1" and the following measurement is to be recorded as "Result 2" on the results sheet.
3. These tests are to be conducted by the methods used routinely in your laboratory. The sample should be treated as a routine sample.
4. Results are to be reported as a % to three decimal places. **Do not report any values as "<".** The method used for each test should also be noted.
5. For each test note the appropriate technique code no. on the Results Sheet:
  1. AES – Arc/Spark (Atomic Emission Spectroscopy – Arc\Spark)
  2. AES – ICP (Atomic Emission spectroscopy – Inductively Coupled Plasma)
  3. AAS (Atomic Absorption Spectrometry)
  4. Gravimetric
  5. Photometric
  6. Other (please specify)
6. Laboratories are also requested to calculate and report an estimate of measurement uncertainty (MU) for each reported measurement result. All estimates of measurement uncertainty must be given as a 95% confidence interval (coverage factor  $k=2$ )
7. Testing may commence as soon as samples are received. All laboratories are to return their results by **Friday 11<sup>th</sup> November 2016** to:  
Karen Cividin  
Proficiency Testing Australia  
PO Box 7507  
Silverwater NSW 2128  
AUSTRALIA  
Phone: +61 2 9736 8295 Fax: +61 2 9743 6664
8. To allow for the confidential treatment of your results in the final report, you have been allocated a code number which appears on your results sheet.



Proficiency Testing Australia

## Proficiency Testing Program

Metal Alloys (Round 31) – October 2016

<b>RESULTS SHEET</b>
----------------------

Date sample was received: \_\_\_\_\_

Lab Code: 

TEST (report % to three decimal places)	SAMPLE		MU ( $\pm$ )	Technique Code No.
	Result 1	Result 2		
Carbon				
Sulfur				
Phosphorus				
Silicon				
Manganese				
Chromium				
Nickel				
Copper				
Molybdenum				
Vanadium				
Cobalt				

Signed: \_\_\_\_\_

Date: \_\_\_\_\_

Please return no later than **Friday 11<sup>th</sup> November 2016**, to:

Karen Cividin  
 Proficiency Testing Australia  
 PO Box 7507, Silverwater NSW 2128  
 phone: +61 2 9736 8295  
 fax: +61 9743 6664

- End of Report -