

**Report No. 803**

**Metal Alloys Proficiency Testing**

**Round 24**

**Stainless Steel**

**May 2013**

**Acknowledgments**

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## 1. FOREWORD

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

The program was conducted in February 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 Advisor was Mr W Ting from Universal Scientific Laboratory Pty Ltd. This report was authorised by Ms W Fajloun, PTA Quality Coordinator.

## 2. FEATURES OF THE PROGRAM

- (a) Participants were provided with one stainless steel disc sample.
- (b) A total of 15 laboratories received samples, comprising:
  - 10 Australian participants; and
  - 5 overseas participants, including:
    - New Zealand, Iran, Poland, Malaysia and Korea.

Of these 15 laboratories, 1 was unable to submit 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, a number of 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 some laboratories reported more than one set of results so may have more than one code number in the 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 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 and z-score charts relevant to each test are presented in Appendix A.
- (h) The document entitled *Guide to Proficiency Testing Australia, 2012* (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 7.

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

- (a) Appendix A contains the analysis of results reported by laboratories for the samples. This section contains the following for each determinant:
  - a table of results and calculated z-scores;
  - a list of summary statistics;
  - ordered z-score charts.
- (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**

- (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 "§".

(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 significant figures) 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;
- 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 0.7413, a 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).

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.

**TABLE A: SUMMARY STATISTICS**

<b>Test</b>	<b>No. of Results</b>	<b>Median</b>	<b>Normalised IQR</b>
Carbon	21	0.0450	0.0130
Sulphur	21	0.0260	0.0027
Phosphorus	21	0.0290	0.0030
Silicon	21	0.2895	0.0263
Manganese	21	1.5980	0.0385
Chromium	21	16.9500	0.1901
Nickel	21	10.0450	0.2361
Copper	21	0.3665	0.0234
Molybdenum	21	2.0160	0.0708
Vanadium	21	0.0799	0.0085
Titanium	19	0.0050	0.0024
Cobalt	20	0.1590	0.0093

## **5. PTA AND TECHNICAL ADVISOR'S COMMENTS**

The sample used in this round was Grade 316 stainless steel. This is a very common grade with widespread usage. The salient alloying elements (chromium, nickel and molybdenum) are very expensive and manufacturers 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 above-mentioned three elements, plus carbon, sulphur, manganese and silicon. Laboratory code 16 appeared to be unfamiliar with this material, but of more concern, may not have attempted to verify their results before submission.

It is likely that a laboratory which submitted generally acceptable results (with perhaps one or two questionable figures), may not have been able to verify results by recourse to one or more Certified Reference Materials (CRM's). Where a laboratory

has not reported a method of analysis, reasons for high, low or outlying results can only be speculated.

In the following summary of each test, results identified as outliers have been discounted.

#### Chromium

The results were generally good. The difference of 0.5% between laboratory code 20's results is not acceptable and laboratory code 9's results are slightly high. With these qualifications the submitted results are acceptable. For this level of chromium calibration, the status of AES - Arc/spark and AES - ICP instruments is critical. It is doubtful that many laboratories would still be capable of using the volumetric technique of ASTM E53.

#### Nickel

The results were generally good. Laboratory code 9's calibration at this level (10%) appears to be questionable. Laboratory code 22 reported an unacceptably high figure. Again, calibration of instrumentation must be spot on. Very few laboratories have the time or expertise to carry out a gravimetric analysis as did laboratory code 8.

#### Molybdenum

Probably the best set of results. It is unusual to see more than 2.1% in this grade and with the exception of laboratory code 9, all the results round to either 2.0 or 2.1%.

#### Manganese

These results were similar to Molybdenum. If laboratory code 17's results (even though acceptable) are omitted, the remaining figures are very good.

#### Carbon

Most of these results were good. The best results appear to have been generated by LECO. There is a variation of Grade 316 called 316L where the maximum carbon is 0.03%, so it follows that accuracy for this element is important. It is likely that the low end calibration for carbon on some of the instruments used is out by +/- 0.01%.

#### Phosphorus

All the results were good.

#### Sulphur

The results were very good.

#### Silicon

The results were mostly good with the exception of laboratory codes 18 and 20 whose results were low and high respectively.

### Copper

The results were generally good. Laboratory code 2 may have had a calibration problem for this element.

### Vanadium

The results were good. Results for laboratory codes 9 and 12 were fractionally high.

### Titanium

This element was present in a trace amount and tests out the low end of the calibration curves for AES Arc/spark, hence the spread of results.

### Cobalt

These results were good and indicate how comfortable this level is for analysis by AES (both techniques).

### Variations within and between laboratories

With the exception of carbon, variations within laboratories were acceptable. It is significant that the results shown by the two laboratories that used techniques other than AES Arc/spark gave generally good correlation. Calibration of AES Arc/spark instruments is always important, but critical when analysing for concentrations of 10% or more, and also when the concentration of the element of interest is near the baseline.

### Variations between methods

Most results appear to have been generated by AES Arc/spark.

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

Measurement uncertainties were generally good. Laboratory code 10's high MU could explain the difference in phosphorus results. The MU's reported by laboratory code 5 for manganese, chromium, nickel and molybdenum are quite high but the results for these elements are good.

This was an appropriate alloy for proficiency testing. A future consideration could be to give participants approximate concentrations of major elements before testing begins.

With the exception of the obvious outliers, the results, in the main were acceptable to good. With laboratories increasingly reliant upon AES Arc/spark, it is critical that appropriate CRM's are used to confirm test results.



### 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 in the Appendix A.

### Analysis of Results by Method Groups

In order for methods to be grouped for analysis, PTA requires at least 11 sets of results from the same method group. As there were less than 11 results submitted for each method, reliable conclusions cannot be drawn from analysing grouped methods on this occasion. Therefore, results from all method groups have been pooled for analysis.

## 6. OUTLIER RESULTS

Laboratories reporting outlier results are listed in the following table:

**TABLE B: SUMMARY OF STATISTICAL OUTLIERS**

Test	Laboratory Code No.
Carbon	-
Sulphur	12, 15, 16, 17
Phosphorus	9, 16, 18
Silicon	4*, 9, 16, 17
Manganese	16
Chromium	16, 17, 22
Nickel	16, 17
Copper	16
Molybdenum	16, 17
Vanadium	15, 16, 17
Titanium	10, 16
Cobalt	16, 18

\* This laboratory re-submitted their result (which was within the acceptable range) after a typographic error was detected.

**7. REFERENCE**

- [1] *Guide to Proficiency Testing Australia, 2012* (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
Sulphur.....	A2
Phosphorus.....	A3
Silicon.....	A4
Manganese.....	A5
Chromium.....	A6
Nickel.....	A7
Copper.....	A8
Molybdenum.....	A9
Vanadium.....	A10
Titanium.....	A11
Cobalt.....	A12

A1

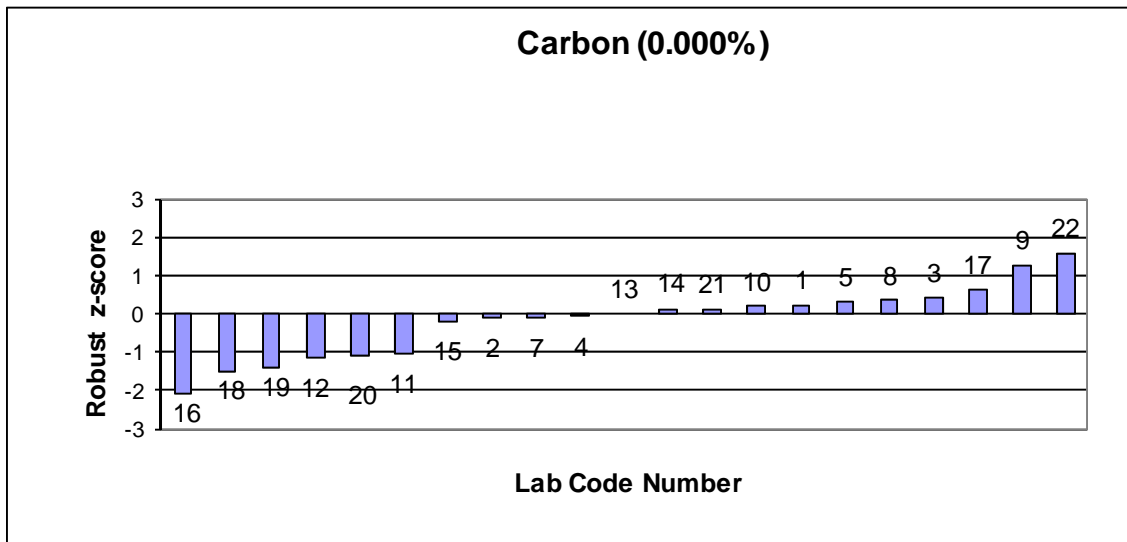
**Carbon (0.000%)**

Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.050	0.046	0.048	0.003	0.23	1
2	0.044	0.043	0.044	0.003	-0.12	1
3	0.050	0.051	0.051	0.0039	0.42	1
4	0.044	0.044	0.044	0.001	-0.08	1
5	0.0495	0.0485	0.049	0.0013	0.31	6
7	0.044	0.043	0.044	nr	-0.12	1
8	0.0493	0.0496	0.049	0.005	0.34	6
9	0.060	0.063	0.062	0.01	1.27	1
10	0.048	0.047	0.048	0.002	0.19	1
11	0.030	0.033	0.032	0.005	-1.04	1
12	0.030	0.030	0.030	0.002	-1.16	1
13	0.045	0.045	0.045	nr	0.00	1
14	0.047	0.046	0.047	0.020	0.12	1
15	0.043	0.042	0.043	nr	-0.19	nr
16	0.018	nr	0.018	nr	-2.08	nr
17	0.053	0.053	0.053	nr	0.62	nr
18	0.0251	0.0253	0.025	nr	-1.53	nr
19	0.0267	0.0273	0.027	nr	-1.39	nr
20	0.028	0.033	0.031	nr	-1.12	nr
21	0.046	0.047	0.047	nr	0.12	1
22	0.07011	0.06108	0.066	nr	1.59	nr

nr = no result

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

No. of Results	21
Median	0.0450
Norm IQR	0.0130
Uncertainty of the Median	0.0035
Robust CV	28.8%
Min	0.018
Max	0.066
Range	0.048



**Sulphur (0.000%)**

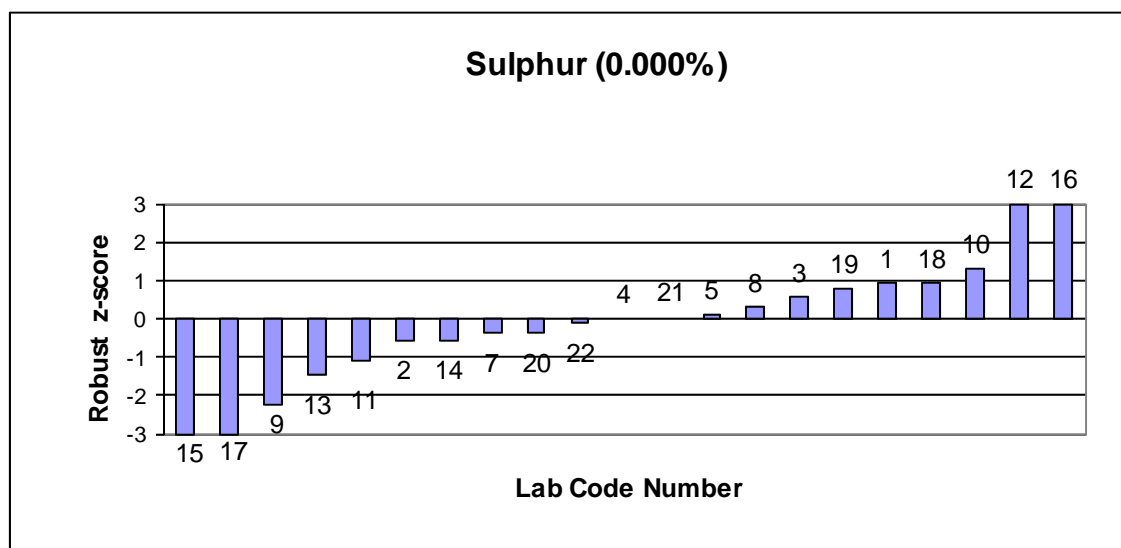
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.028	0.029	0.029	0.004	0.92	1
2	0.025	0.024	0.025	0.006	-0.55	1
3	0.027	0.028	0.028	0.0025	0.55	1
4	0.027	0.025	0.026	0.004	0.00	1
5	0.0261	0.0264	0.026	0.0017	0.09	6
7	0.025	0.025	0.025	nr	-0.37	1
8	0.0264	0.0272	0.027	0.005	0.30	6
9	0.020	0.020	0.020	0.01	-2.22	1
10	0.028	0.031	0.030	0.010	1.29	1
11	0.022	0.024	0.023	0.003	-1.11	1
12	0.040	0.039	0.040	0.005	4.99	§ 1
13	0.022	0.022	0.022	nr	-1.48	1
14	0.025	0.024	0.025	0.008	-0.55	1
15	0.012	0.012	0.012	nr	-5.17	§ nr
16	0.114	nr	0.114	nr	32.52	§ nr
17	0.013	0.012	0.013	nr	-4.99	§ nr
18	0.028	0.029	0.029	nr	0.92	nr
19	0.0288	0.0275	0.028	nr	0.79	nr
20	0.027	0.023	0.025	nr	-0.37	nr
21	0.025	0.027	0.026	nr	0.00	1
22	0.02556	0.02588	0.026	nr	-0.10	nr

nr = no result

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

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

No. of Results	21
Median	0.0260
Norm IQR	0.0027
Uncertainty of the Median	0.0007
Robust CV	10.4%
Min	0.012
Max	0.114
Range	0.102



## Phosphorus (0.000%)

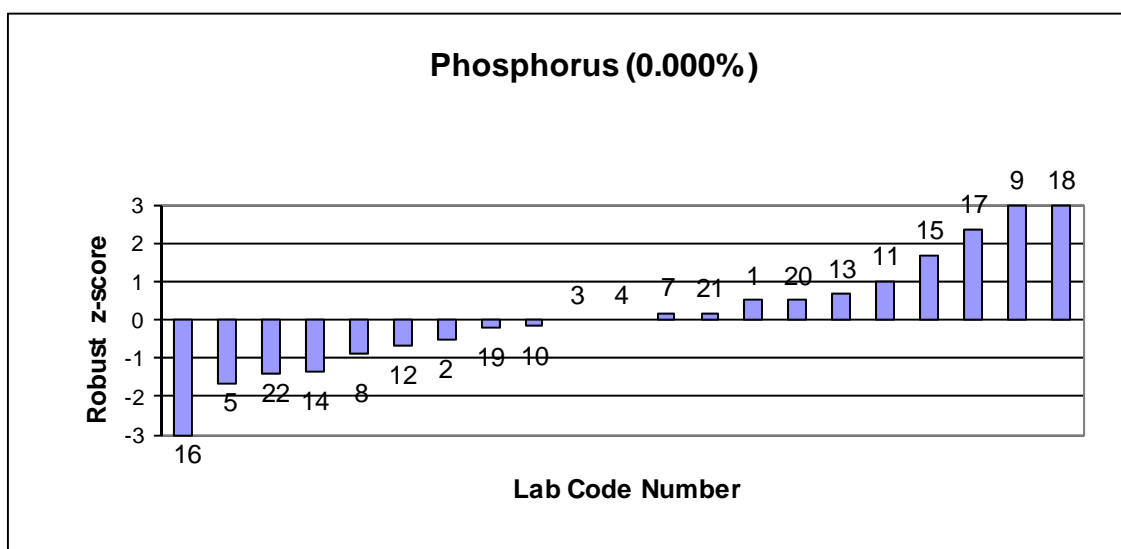
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.029	0.032	0.031	0.002	0.51	1
2	0.027	0.028	0.028	0.003	-0.51	1
3	0.029	0.029	0.029	0.0011	0.00	1
4	0.029	0.029	0.029	0.001	0.00	1
5	0.023	0.025	0.024	0.009	-1.69	6
7	0.029	0.03	0.030	nr	0.17	1
8	0.0263	0.0264	0.026	0.005	-0.89	6
9	0.04	0.04	0.040	0.01	3.71	§ 1
10	0.026	0.031	0.029	0.014	-0.17	1
11	0.032	0.032	0.032	0.001	1.01	1
12	0.027	0.027	0.027	0.003	-0.67	1
13	0.031	0.031	0.031	nr	0.67	1
14	0.026	0.024	0.025	0.006	-1.35	1
15	0.035	0.033	0.034	nr	1.69	nr
16	0.015	nr	0.015	nr	-4.72	§ nr
17	0.037	0.035	0.036	nr	2.36	nr
18	0.047	0.048	0.048	nr	6.24	§ nr
19	0.0281	0.0287	0.028	nr	-0.20	nr
20	0.032	0.029	0.031	nr	0.51	nr
21	0.029	0.03	0.030	nr	0.17	1
22	0.02541	0.02437	0.025	nr	-1.39	nr

nr = no result

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

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

No. of Results	21
Median	0.0290
Norm IQR	0.0030
Uncertainty of the Median	0.0008
Robust CV	10.2%
Min	0.015
Max	0.048
Range	0.033



**Silicon (0.000%)**

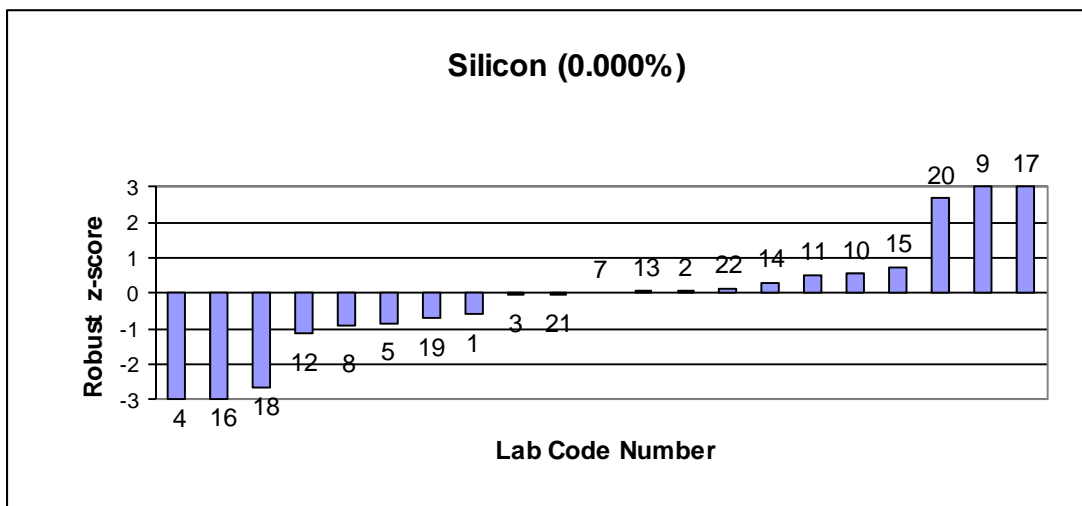
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.275	0.273	0.274	0.011	-0.59	1
2	0.288	0.294	0.291	0.02	0.06	1
3	0.288	0.290	0.289	0.0065	-0.02	1
4	0.028	0.028	0.028	0.002	-9.94	§ 1
5	0.271	0.264	0.2675	0.032	-0.84	2
7	0.289	0.290	0.2895	nr	0.00	1
8	0.263	0.267	0.265	0.01	-0.93	5
9	0.410	0.404	0.407	0.05	4.46	§ 1
10	0.307	0.300	0.3035	0.012	0.53	1
11	0.311	0.295	0.303	0.028	0.51	1
12	0.258	0.262	0.26	0.014	-1.12	1
13	0.289	0.291	0.29	nr	0.02	1
14	0.297	0.298	0.2975	0.084	0.30	1
15	0.309	0.309	0.309	nr	0.74	nr
16	0.085	nr	0.085	nr	-7.77	§ nr
17	0.502	0.481	0.4915	nr	7.68	§ nr
18	0.217	0.223	0.22	nr	-2.64	nr
19	0.2699	0.2717	0.2708	nr	-0.71	nr
20	0.353	0.366	0.3595	nr	2.66	nr
21	0.289	0.289	0.289	nr	-0.02	1
22	0.29458	0.29152	0.29305	nr	0.13	nr

nr = no result

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

- Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 2 AES - ICP (Atomic Emission Spectroscopy - Inductively Coupled Plasma)  
 5 Photometric

No. of Results	21
Median	0.2895
Norm IQR	0.0263
Uncertainty of the Median	0.0072
Robust CV	9.1%
Min	0.028
Max	0.492
Range	0.464



**Manganese (0.000%)**

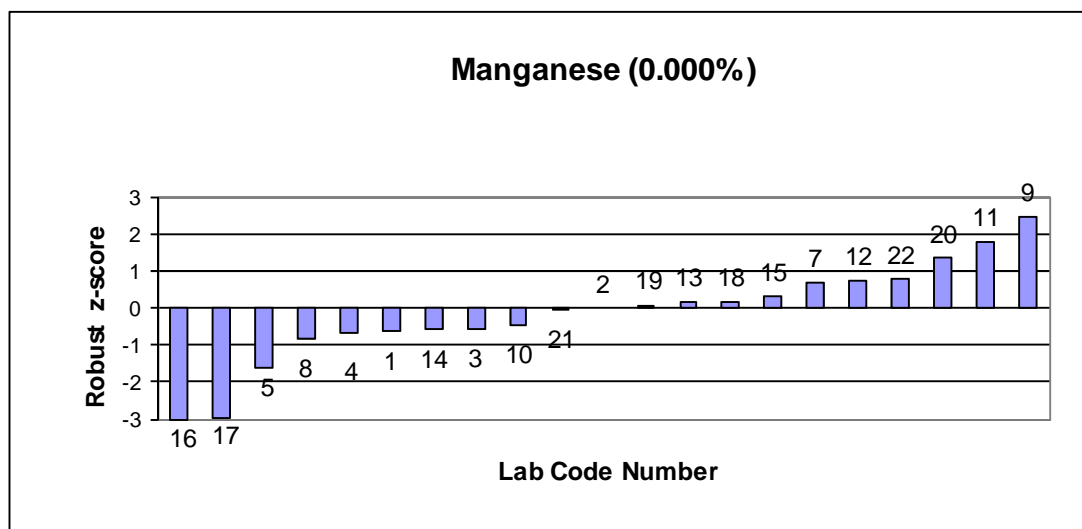
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	1.576	1.570	1.573	0.020	-0.65	1
2	1.592	1.604	1.598	0.04	0.00	1
3	1.575	1.577	1.576	0.0152	-0.57	1
4	1.574	1.568	1.571	0.015	-0.70	1
5	1.53	1.54	1.535	0.1	-1.63	2
7	1.61	1.64	1.625	nr	0.70	1
8	1.56	1.57	1.565	0.01	-0.86	2
9	1.682	1.705	1.694	0.05	2.48	1
10	1.580	1.580	1.580	0.020	-0.47	1
11	1.657	1.677	1.667	0.023	1.79	1
12	1.636	1.618	1.627	0.007	0.75	1
13	1.618	1.592	1.605	nr	0.18	1
14	1.570	1.580	1.575	0.050	-0.60	1
15	1.63	1.59	1.610	nr	0.31	nr
16	0.515	nr	0.515	nr	-28.10	§
17	1.478	1.489	1.484	nr	-2.97	nr
18	1.600	1.610	1.605	nr	0.18	nr
19	1.5965	1.6030	1.600	nr	0.05	nr
20	1.61	1.69	1.650	nr	1.35	nr
21	1.58	1.61	1.595	nr	-0.08	1
22	1.63827	1.61668	1.627	nr	0.76	nr

nr = no result

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

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 2 AES - ICP (Atomic Emission Spectroscopy - Inductively Coupled Plasma)

No. of Results	21
Median	1.5980
Norm IQR	0.0385
Uncertainty of the Median	0.0105
Robust CV	2.4%
Min	0.515
Max	1.694
Range	1.179





**Chromium (0.000%)**

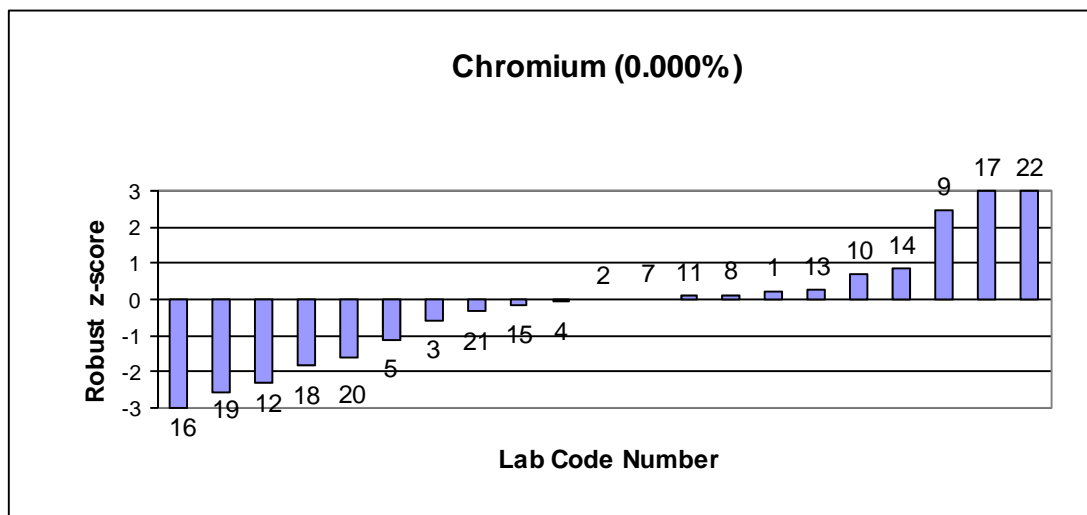
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	16.974	17.010	16.992	0.090	0.22	1
2	17.00	16.90	16.950	0.3	0.00	1
3	16.871	16.816	16.844	0.1854	-0.56	1
4	16.904	16.970	16.937	0.159	-0.07	1
5	16.69	16.79	16.740	0.67	-1.10	2
7	16.92	16.98	16.950	nr	0.00	1
8	16.95	17.00	16.975	0.10	0.13	6
9	17.420	17.420	17.420	0.02	2.47	1
10	17.080	17.080	17.080	0.118	0.68	1
11	16.977	16.957	16.967	0.018	0.09	1
12	16.515	16.502	16.509	0.061	-2.32	1
13	17.02	16.973	16.997	nr	0.24	1
14	17.050	17.170	17.110	0.442	0.84	1
15	16.92	16.92	16.920	nr	-0.16	nr
16	3.757	nr	3.757	nr	-69.38	§
17	17.81	17.698	17.754	nr	4.23	§
18	16.60	16.60	16.600	nr	-1.84	nr
19	16.449	16.476	16.463	nr	-2.56	nr
20	16.4	16.9	16.650	nr	-1.58	nr
21	16.89	16.88	16.885	nr	-0.34	1
22	17.9822	17.81452	17.898	nr	4.99	§

nr = no result

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

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 2 AES - ICP (Atomic Emission Spectroscopy - Inductively Coupled Plasma)  
 6 ASTM E353

No. of Results	21
Median	16.9500
Norm IQR	0.1901
Uncertainty of the Median	0.0520
Robust CV	1.1%
Min	3.757
Max	17.898
Range	14.141



**Nickel (0.000%)**

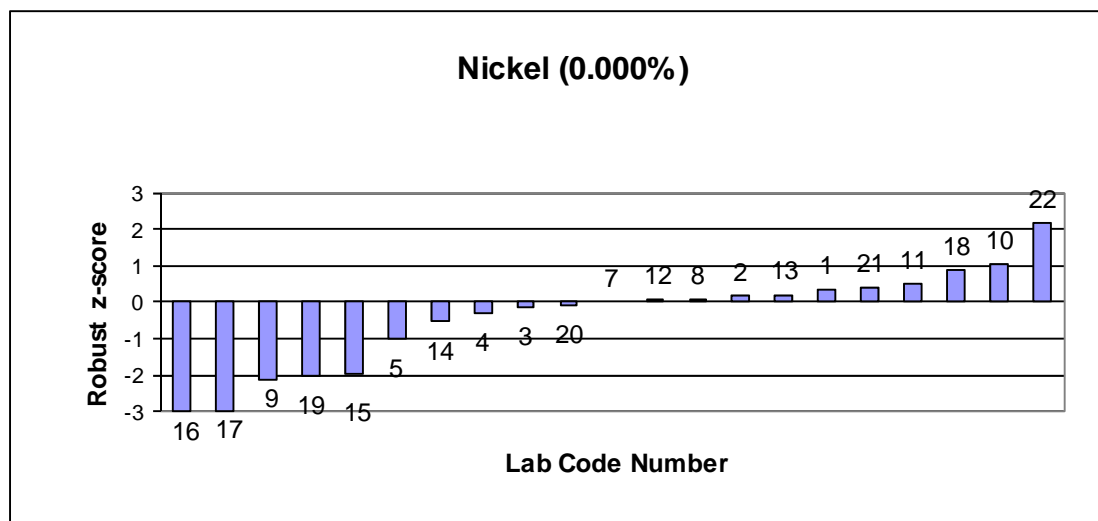
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	10.139	10.118	10.129	0.115	0.35	1
2	10.04	10.13	10.085	0.2	0.17	1
3	10	10.035	10.018	0.152	-0.12	1
4	9.986	9.968	9.977	0.061	-0.29	1
5	9.80	9.82	9.810	0.40	-1.00	2
7	10.08	10.01	10.045	nr	0.00	1
8	10.06	10.07	10.065	0.10	0.08	4
9	9.501	9.597	9.549	0.050	-2.10	1
10	10.300	10.280	10.290	0.114	1.04	1
11	10.190	10.130	10.160	0.056	0.49	1
12	10.064	10.044	10.054	0.028	0.04	1
13	10.07	10.116	10.093	nr	0.20	1
14	9.900	9.940	9.920	0.229	-0.53	1
15	9.59	9.57	9.580	nr	-1.97	nr
16	3.291	nr	3.291	nr	-28.61	§
17	8.824	8.947	8.886	nr	-4.91	§
18	10.20	10.30	10.250	nr	0.87	nr
19	9.5710	9.5732	9.572	nr	-2.00	nr
20	9.96	10.1	10.030	nr	-0.06	nr
21	10.1	10.18	10.140	nr	0.40	1
22	10.62225	10.49555	10.559	nr	2.18	nr

nr = no result

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

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 2 AES - ICP (Atomic Emission Spectroscopy - Inductively Coupled Plasma)  
 4 Gravimetric

No. of Results	21
Median	10.0450
Norm IQR	0.2361
Uncertainty of the Median	0.0646
Robust CV	2.4%
Min	3.291
Max	10.559
Range	7.268



**Copper (0.000%)**

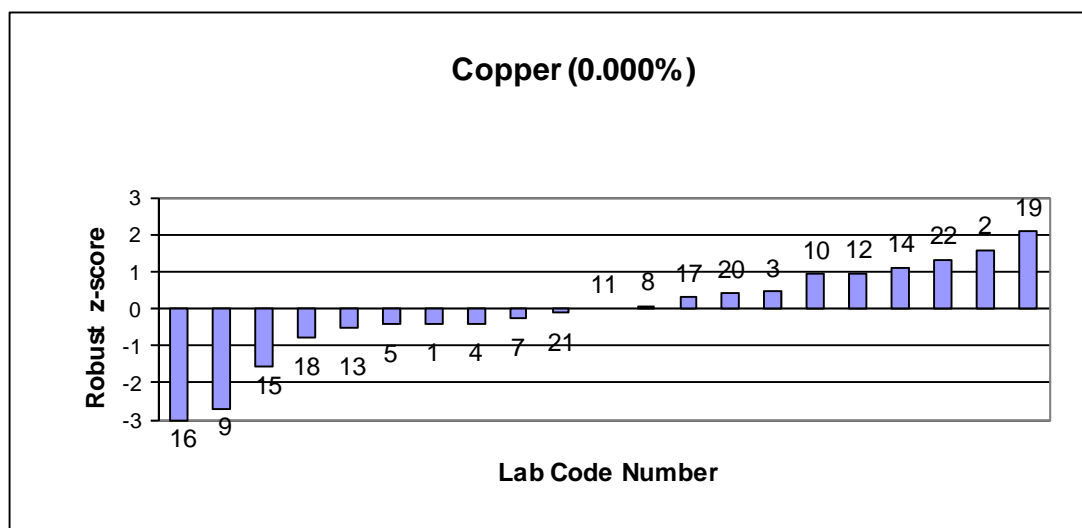
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.357	0.357	0.357	0.008	-0.41	1
2	0.400	0.407	0.404	0.02	1.58	1
3	0.374	0.380	0.377	0.0087	0.45	1
4	0.357	0.357	0.357	0.159	-0.41	1
5	0.353	0.360	0.357	0.042	-0.43	2
7	0.361	0.359	0.360	nr	-0.28	1
8	0.370	0.365	0.368	0.01	0.04	2
9	0.300	0.306	0.303	0.05	-2.72	1
10	0.389	0.387	0.388	0.006	0.92	1
11	0.364	0.369	0.367	0.003	0.00	1
12	0.389	0.388	0.389	0.002	0.94	1
13	0.355	0.353	0.354	nr	-0.54	1
14	0.391	0.394	0.393	0.050	1.11	1
15	0.39	0.27	0.330	nr	-1.56	nr
16	0.123	nr	0.123	nr	-10.43	§
17	0.373	0.374	0.374	nr	0.30	nr
18	0.347	0.349	0.348	nr	-0.79	nr
19	0.4134	0.4166	0.415	nr	2.08	nr
20	0.378	0.374	0.376	nr	0.41	nr
21	0.365	0.362	0.364	nr	-0.13	1
22	0.39653	0.39859	0.398	nr	1.33	nr

nr = no result

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

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 2 AES - ICP (Atomic Emission Spectroscopy - Inductively Coupled Plasma)

No. of Results	21
Median	0.3665
Norm IQR	0.0234
Uncertainty of the Median	0.0064
Robust CV	6.4%
Min	0.123
Max	0.415
Range	0.292



**Molybdenum (0.000%)**

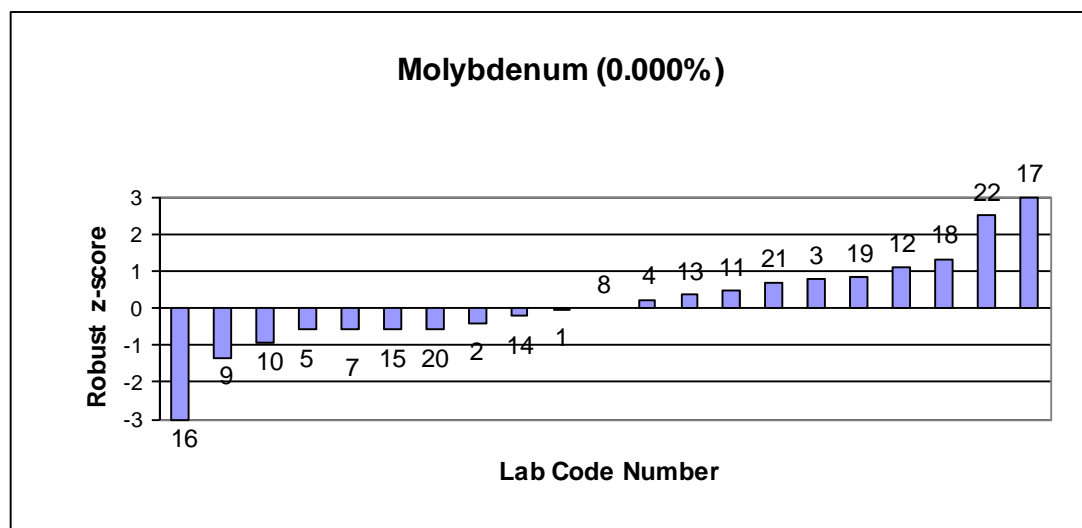
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	2.025	2.004	2.0145	0.047	-0.02	1
2	1.992	1.983	1.9875	0.1	-0.40	1
3	2.066	2.075	2.0705	0.0352	0.77	1
4	2.033	2.027	2.03	0.041	0.20	1
5	1.98	1.97	1.975	0.29	-0.58	2
7	1.99	1.96	1.975	nr	-0.58	1
8	2.014	2.018	2.016	0.01	0.00	2
9	1.914	1.926	1.92	0.05	-1.36	1
10	1.960	1.940	1.95	0.036	-0.93	1
11	2.043	2.053	2.048	0.027	0.45	1
12	2.101	2.086	2.0935	0.025	1.09	1
13	2.065	2.017	2.041	nr	0.35	1
14	2.010	1.990	2	0.106	-0.23	1
15	1.97	1.98	1.975	nr	-0.58	nr
16	0.655	nr	0.655	nr	-19.22	§
17	2.272	2.265	2.2685	nr	3.57	§
18	2.10	2.12	2.11	nr	1.33	nr
19	2.0839	2.0659	2.0749	nr	0.83	nr
20	1.95	2	1.975	nr	-0.58	nr
21	2.08	2.05	2.065	nr	0.69	1
22	2.19485	2.18909	2.19197	nr	2.49	nr

nr = no result

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

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 2 AES - ICP (Atomic Emission Spectroscopy - Inductively Coupled Plasma)

No. of Results	21
Median	2.0160
Norm IQR	0.0708
Uncertainty of the Median	0.0194
Robust CV	3.5%
Min	0.655
Max	2.269
Range	1.614



**Vanadium (0.000%)**

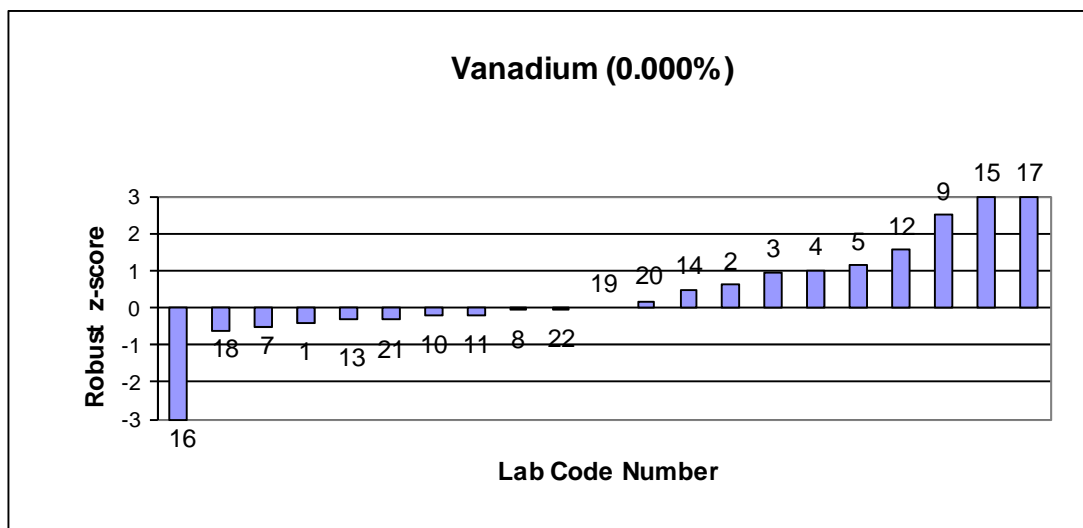
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.076	0.077	0.0765	0.016	-0.39	1
2	0.086	0.084	0.085	0.008	0.60	1
3	0.088	0.088	0.088	0.0010	0.96	1
4	0.088	0.089	0.0885	0.002	1.01	1
5	0.089	0.090	0.0895	0.005	1.13	2
7	0.075	0.076	0.0755	nr	-0.51	1
8	0.0794	0.0792	0.0793	0.005	-0.06	2
9	0.101	0.101	0.101	0.01	2.48	1
10	0.078	0.078	0.078	0.002	-0.22	1
11	0.078	0.078	0.078	0.001	-0.22	1
12	0.094	0.092	0.093	0.001	1.54	1
13	0.077	0.077	0.077	nr	-0.33	1
14	0.084	0.084	0.084	0.015	0.49	1
15	0.11	0.11	0.11	nr	3.54	§
16	0.031	nr	0.031	nr	-5.73	§
17	0.088	0.87	0.479	nr	46.82	§
18	0.0742	0.0746	0.0744	nr	-0.64	nr
19	0.0796	0.0801	0.07985	nr	0.00	nr
20	0.0792	0.0834	0.0813	nr	0.17	nr
21	0.078	0.076	0.077	nr	-0.33	1
22	0.07935	0.07944	0.079395	nr	-0.05	nr

nr = no result

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

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 2 AES - ICP (Atomic Emission Spectroscopy - Inductively Coupled Plasma)

No. of Results	21
Median	0.0799
Norm IQR	0.0085
Uncertainty of the Median	0.0023
Robust CV	10.7%
Min	0.031
Max	0.479
Range	0.448



**Titanium (0.000%)**

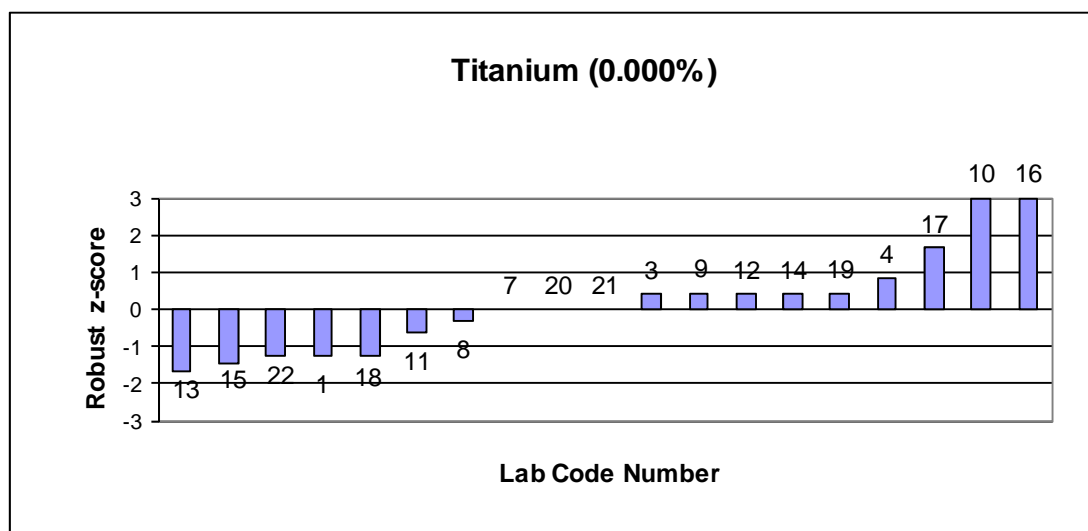
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.002	0.002	0.002	0.0003	-1.24	1
3	0.006	0.006	0.006	0.0007	0.41	1
4	0.007	0.007	0.007	0.001	0.82	1
5	nr	nr	nr	nr	nr	2
7	0.005	0.005	0.005	nr	0.00	1
8	0.0044	0.0042	0.004	0.001	-0.29	2
10	0.014	0.014	0.014	0.001	3.71	1
9	0.006	0.006	0.006	0.01	0.41	1
11	0.004	0.003	0.004	0.001	-0.62	1
12	0.006	0.006	0.006	0.001	0.41	1
13	0.001	0.001	0.001	nr	-1.65	1
14	0.006	0.006	0.006	0.042	0.41	1
15	0.002	0.001	0.002	nr	-1.44	nr
16	0.046	nr	0.046	nr	16.89	nr
17	0.009	0.009	0.009	nr	1.65	nr
18	0.002	0.002	0.002	nr	-1.24	nr
19	0.0061	0.0060	0.006	nr	0.43	nr
20	0.005	0.005	0.005	nr	0.00	nr
21	0.005	0.005	0.005	nr	0.00	1
22	0.00196	0.00193	0.002	nr	-1.26	nr

nr = no result

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

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 2 AES - ICP (Atomic Emission Spectroscopy - Inductively Coupled Plasma)

No. of Results	19
Median	0.0050
Norm IQR	0.0024
Uncertainty of the Median	0.0007
Robust CV	48.6%
Min	0.001
Max	0.046
Range	0.045



**Cobalt (0.000%)**

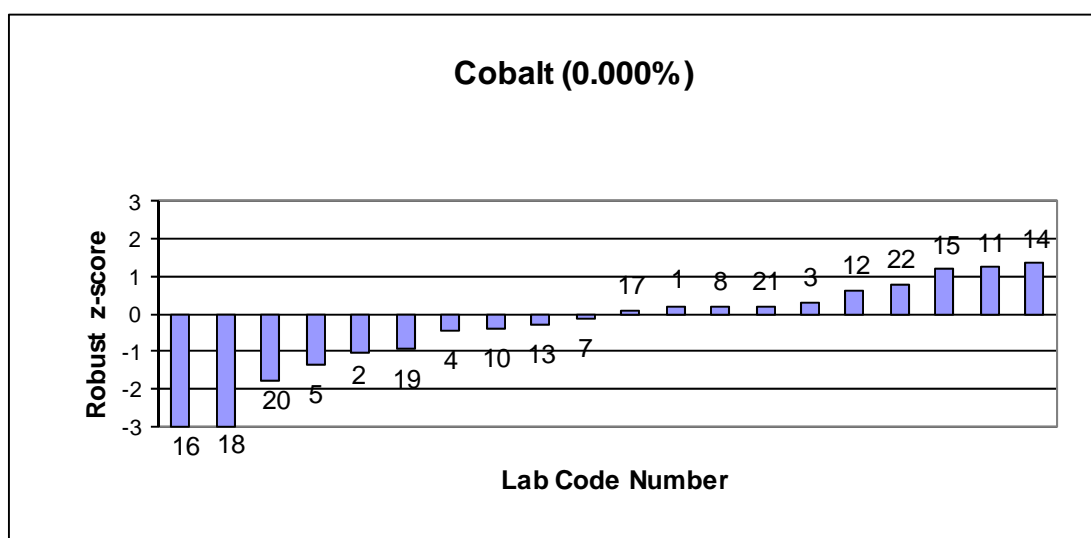
Lab Code	Result 1	Result 2	Average	MU	Robust Z-score	Technique
1	0.159	0.163	0.161	0.006	0.21	1
2	0.148	0.151	0.150	0.01	-1.02	1
3	0.161	0.163	0.162	0.0055	0.32	1
4	0.155	0.155	0.155	0.003	-0.43	1
5	0.147	0.146	0.147	0.012	-1.34	2
7	0.158	0.158	0.158	nr	-0.11	1
8	0.162	0.160	0.161	0.005	0.21	2
10	0.156	0.155	0.156	0.014	-0.38	1
11	0.174	0.167	0.171	0.006	1.23	1
12	0.165	0.165	0.165	0.002	0.64	1
13	0.159	0.154	0.157	nr	-0.27	1
14	0.170	0.173	0.172	0.026	1.34	1
15	0.17	0.17	0.170	nr	1.18	nr
16	0.077	nr	0.077	nr	-8.80	§
17	0.162	0.158	0.160	nr	0.11	nr
18	0.107	0.108	0.108	nr	-5.52	§
19	0.1500	0.1508	0.150	nr	-0.92	nr
20	0.142	0.143	0.143	nr	-1.77	nr
21	0.161	0.161	0.161	nr	0.21	1
22	0.16721	0.1657	0.166	nr	0.80	nr

nr = no result

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

Technique: 1 AES - Arc/spark (Atomic Emission Spectroscopy - Arc/Spark)  
 2 AES - ICP (Atomic Emission Spectroscopy - Inductively Coupled Plasma)

No. of Results	20
Median	0.1590
Norm IQR	0.0093
Uncertainty of the Median	0.0026
Robust CV	5.9%
Min	0.077
Max	0.172
Range	0.095



# **APPENDIX B**

## **Homogeneity and Stability Testing**

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



### **Sample Preparation and Homogeneity Testing**

The stainless steel 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 tables:

Sample	Carbon	Sulphur	Phosphorus	Silicon	Manganese	Chromium
1	0.04999	0.0283	0.0269	0.267	1.560	16.94
2	0.04817	0.0286	0.0270	0.279	1.601	16.96
3	0.04812	0.0287	0.0271	0.283	1.567	16.99
4	0.04926	0.0284	0.0270	0.281	1.566	17.00
5	0.04989	0.0272	0.0300	0.279	1.558	16.95
6	0.04955	0.0260	0.0270	0.284	1.539	17.00
7	0.04790	0.0264	0.0263	0.281	1.534	16.83
8	0.05000	0.0262	0.0264	0.263	1.531	16.83
Average	0.04911	0.027475	0.027213	0.277125	1.557	16.9375
SD	0.000904	0.001155	0.001166	0.007754	0.022854	0.070051
CV	1.84%	4.20%	4.28%	2.80%	1.47%	0.41%

Sample	Nickel	Copper	Molybdenum	Vanadium	Titanium	Cobalt
1	10.03	0.374	2.0100	0.080	0.0038	0.161
2	10.02	0.371	1.9970	0.079	0.0045	0.163
3	10.10	0.375	2.0040	0.0799	0.0044	0.159
4	10.07	0.364	2.0120	0.0782	0.0043	0.156
5	10.06	0.370	2.0650	0.0786	0.0044	0.161
6	10.09	0.365	2.0490	0.0797	0.0044	0.1625
7	10.08	0.361	2.0390	0.0790	0.0043	0.1617
8	10.10	0.358	2.0500	0.0771	0.0050	0.162
Average	10.06875	0.36725	2.028025	0.078938	0.004388	0.160775
SD	0.030443	0.006182	0.025615	0.000977	0.000327	0.002278
CV	0.30%	1.68%	1.26%	1.24%	7.45%	1.42%

Analysis of this data indicated that the samples were sufficiently homogeneous 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.....	C3



Proficiency Testing Australia

**Proficiency Testing Program**

**Metal Alloys (Round 24) – January 2013**

***INSTRUCTIONS TO PARTICIPANTS***

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

1. For this round each participant will be supplied with one stainless steel disc.
2. Participants are asked to test the percentage composition (in duplicate) for each sample for the following elements:

Carbon, Sulphur, Phosphorus, Silicon, Manganese, Chromium, Nickel, Copper, Molybdenum, Vanadium, Titanium 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  $k \approx 2$ )

7. Testing may commence as soon as samples are received. All laboratories are to return their results by **Friday 22<sup>nd</sup> February 2013** 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.

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*m:\ptalkaren\metalalloys\round24\instructionsandresultssheet*



Proficiency Testing Australia  
**Proficiency Testing Program**  
**Metal Alloys (Round 24) – January 2013**

**RESULTS SHEET**

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

Lab Code:

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

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

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

Please return no later than **Friday 22<sup>nd</sup> February 2013**, to: Karen Cividin, Proficiency Testing Australia  
 PO Box 7507, Silverwater NSW 2128  
ph: +61 2 9736 8295, fx: +61 9743 6664