WINE ROUND 15

PROFICIENCY TESTING PROGRAM

JANUARY 2007

REPORT NO. 532

ACKNOWLEDGMENTS

PTA wishes to acknowledge gratefully the technical assistance provided for this program by Mr Malcolm Gennoe of the Pernod Ricard Pacific Pty Ltd, SA, and Mr Marco Valessi of Vintessential Laboratories, VIC. This assistance included providing input into the design of the program, technical advice and discussion of the final report. PTA would also like to thank De Bortoli Wines for supplying the samples for the program and Vintessential Laboratories for conducting the homogeneity testing.

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CONTENTS

1. FOREWORD

This report summarises the results of a proficiency testing program on the chemical testing of wine.

The aim of the program was to assess laboratories' ability to competently perform the tests examined.

2. FEATURES OF THE PROGRAM

2.1 Results were received from 18 laboratories, with all laboratories submitting one set of results

2.2 The results, as reported by participants, are presented in Appendix A. Summary statistics are calculated from the results reported for each test. These are presented in Table A (page 2). Robust z-scores and z-score charts are presented in Appendix A. A listing of laboratories (by code number) identified as having reported outliers are presented in Table B (page 3).

2.3 Appendix B contains the results of the homogeneity testing on the samples used in the program.

2.4 Laboratories were requested to perform the tests according to the *Instructions to Participants* and to record their results on the accompanying *Results Sheet*, both of which were distributed to participants with their samples.

Copies of the *Instructions to Participants* and *Results Sheet* are given in Appendix C of this report.

2.5 Each laboratory was randomly allocated a code number for the program to allow for the confidential treatment of results. Reference to any laboratory in this report is made by its code number.

3. DESIGN OF PROGRAM

3.1 For this program each participant was provided with two 750 mL samples, one of white wine (sample 1) and one of red wine (sample 2).

The following tests were to be conducted on each sample:

- Total Acidity
- Volatile Acidity
- Actual Alcohol
- Total Sulfur Dioxide
- Residual Sugars
- pH
- Specific Gravity

3.2 Robust statistical procedures were used to generate the z-scores and summary statistics for each sample and for each test - number of results, median, normalised interquartile range, minimum, maximum and range.

3.3 Robust z-scores were calculated based on the average of results submitted for each test. Where only one result was submitted, this result was used to determine the z-score.

TABLE A: SUMMARY STATISTICS

Analysis	Sample No.	No. of Results	Median (g/L)	Normalised IQR
Total Acidity	1	17	5.96	0.07
	2	17	6.50	0.09
Volatile Acidity	1	15	0.385	0.043
	2	15	0.350	0.039
Actual Alcohol	1	18	13.40	0.06
	2	18	13.50	0.07
Total Sulfur Dioxide	1	17	161.0	3.3
	2	17	72.0	3.3
Residual Sugars	1	15	5.14	0.78
	2	15	3.10	1.87
pH	1	18	3.290	0.021
	2	18	3.488	0.028
Specific Gravity	1	17	0.99230	0.00050
	2	17	0.99670	0.00067

4. OUTLIER RESULTS

In order to achieve the program's aim of assessing laboratories' testing performance, a robust statistical approach, which uses z-scores to assess participants' performance, has been utilised. The z-score is a measure of how far the results are from the consensus value - a normalised value which gives a "score" to each result relative to the other results in the group. Therefore a z-score close to zero means that the result agrees well with those from other laboratories. An outlier is any result which has an absolute z-score value greater than three and is marked by the symbol **§**.

Each determination was examined for outliers with all methods pooled. Table B summarises the outliers detected.

TABLE B: OUTLIER RESULTS
(by laboratory code number)

Test	Sample 1	Sample 2
Total Acidity	1	3, 11
Volatile Acidity	9	5, 14
Actual Alcohol	-	11
Total Sulfur Dioxide	-	1
Residual Sugars	-	-
pH	5, 17	-
Specific Gravity	2, 6	-

5. STATISTICAL FORMAT

For each test, the following information is given:

(a) a table of results and calculated z-scores;

(b) a list of summary statistics; and

(c) ordered z-score charts;

(a) Table of Results and Z-Scores

Each of these tables contains the results returned by each laboratory, including the code number for the method used, and the laboratory z-scores calculated based on each laboratory's averaged results.

Note that 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.

Outliers are identified in the table by a marker (**§**) next to the relevant z-score. Please see reference [1] for details on how these z-scores are calculated.

(b) Summary Statistics

The list of summary statistics appears at the bottom of the table of results and consists of:

(i) the number of results for that test/sample (*No. of Results*);

(ii) the median of laboratory's results - i.e. the middle value (*Median*);

(iii) the normalised interquartile range of the results (*Normalised IQR*) - the interquartile range x 0.741;

(iv) the robust coefficient of variation, expressed as a percentage (*Robust CV*) - i.e. 100 x *Normalised IQR* / *Median*;

(v) the minimum and maximum laboratory results; and

(vi) the range (*Maximum* - *Minimum*).

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

(c) Ordered Z-Score Charts

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

These charts contain solid lines at +3 and -3, so the outliers are clearly identifiable as the laboratories whose "bar" extends beyond these "cut-off" lines. In some cases the y-axis of these charts has been limited, so very large or small (negative) z-scores appear to extend beyond the chart.

6. PTA & TECHNICAL ADVISER'S COMMENTS

Total Acidity
This analysis was well performed as shown by the low Robust CV values, with the majority of participants within 0.1 g/L of the median for Sample 1. However, results for Sample 2 tended to display a positive bias, which may be due to insufficient degassing. Outliers were reported for Sample 1 by Lab Code 1 only. Sample 2 outliers were reported by Lab Codes 3 and 11.

Volatile Acidity
A wide spread of results for both samples was observed, with Robust CV values above 10%. A contributing factor here is the range of methodologies used by participants. Lab Code 9 reported an outlier for Sample 1. Lab Codes 5 and 14 reported an outlier for Sample 2.

Actual Alcohol
This analysis was well performed with very low Robust CV values achieved. There were no outliers reported for Sample 1, and only one outlier reported for Sample 2 by Lab Code 11. Outliers associated with pycnometry which may be associated with cleanliness /temperature of the pycnometer.

Total Sulfur Dioxide
The analysis for Sample 1 was well performed, with low Robust CV values. Greater variation was seen for Sample 2, and seems to confirm a trend seen in previous PTA proficiency programs for red wine samples. Lab Code 1 reported an outlier for Sample 2. Error may be associated with condenser temperature, end point titration.

Residual Sugars
Robust CV values for both samples were at 15%. This is in part due the type of method used by participants. The Rebelein and Lane & Eynon methods (which measure all "Reducing" sugars including Pentoses) will overstate the actual fermentable sugar (Glucose+Fructose) levels by at 1 to 2 g/L. Due to the wide variation of results, no outliers were reported for either Sample 1 or 2.

pH
The analysis was generally well performed, with low Robust CV values observed. Lab Codes 5 and 17 reported outliers for Sample 1, while no outliers were reported for Sample 2.

Specific Gravity
The analysis was well performed, particularly for Sample 2, with low Robust CV values observed. Two outliers were reported for Sample 1; Lab Codes 2 and 6.

Conclusion

Overall this round of testing was well performed and compared favourably with previous rounds. The majority of labs reported measurement uncertainty for the tests they conducted.

7. REFERENCES

[1] "Guide to Proficiency Testing" – February 2004 (this document is located on the PTA website at www.pta.asn.au under "Publications").

APPENDIX A

All Results

Summary Results

Z-Score Charts

Section 1 –

Sample 1 (White Wine)

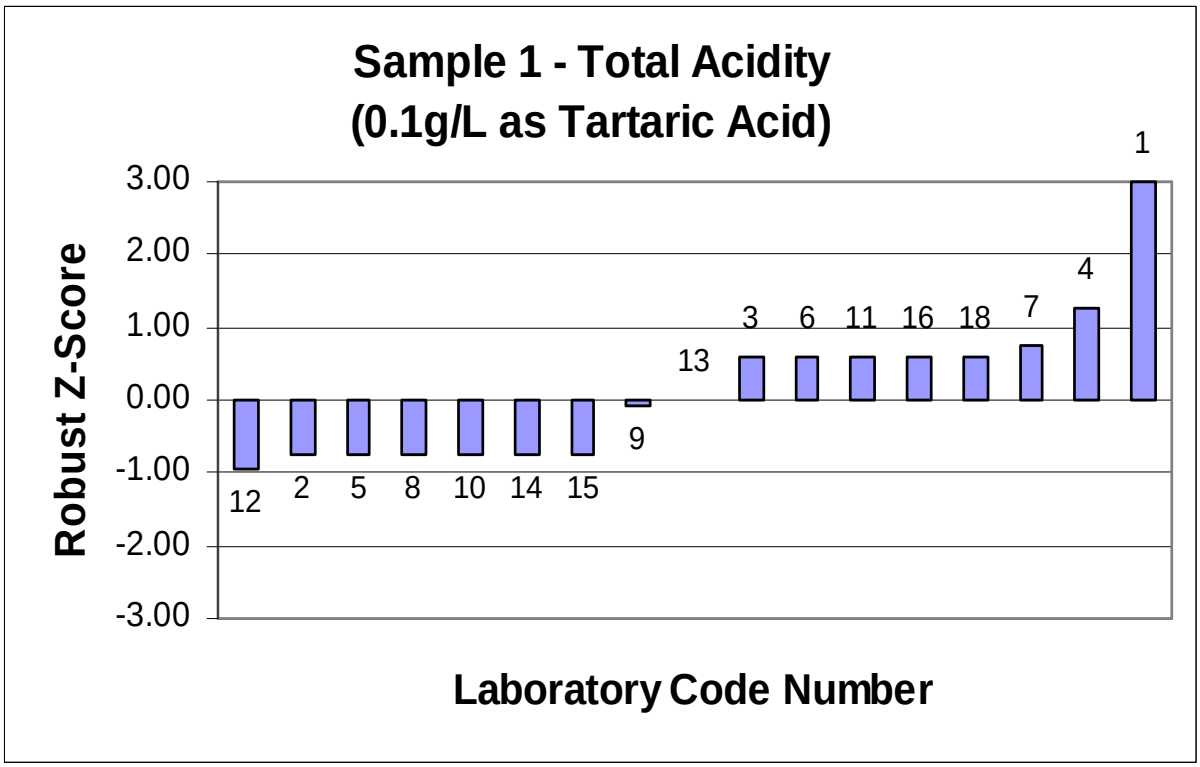
Total Acidity
0.1g/L as Tartaric Acid

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1	6.5	0.2	6.5	0.2	AOAC962.12	6.5	7.35 **§**
2	5.9	0.10	5.9	0.10	Titration to an end point	5.9	-0.74
3	5.9	0.08	6.1	0.08	Manual Titration	6.0	0.61
4	6.1	0.12	6.0	0.12	Autotitrator	6.1	1.28
5	5.9	0.1	5.9	0.1	Autotitration	5.9	-0.74
6	6.0	0.2	6.0	0.2	Autotitrator	6.0	0.61
7	5.99	0.15	6.03	0.15	Autotitration	6.0	0.74
8	5.9	0.11	5.9	0.11	Potentiometric/Autotitrator	5.9	-0.74
9	5.97	0.12	5.93	0.12	Manual Titration	6.0	-0.07
10	5.9	0.07	5.9	0.07	Automatic titrator	5.9	-0.74
11	6.0		6.0		Titration - end point 8.2	6.0	0.61
12	5.85	0.19	5.92	0.19	Autotitrator	5.9	-0.94
13	5.95	0.12	5.96	0.12	Autotitrator	6.0	0.00
14	5.9	0.18			Winescan	5.9	-0.74
15	5.9	0.1	5.9	0.1	Titration	5.9	-0.74
16	6.0	0.1	6.0	0.1	Autotitrator	6.0	0.61
17							
18	6.0	0.1	6.0			6.0	0.61

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	17
Median	5.96
NormIQR	0.07
Robust CV	1.24%
Min	5.9
Max	6.5
Range	0.6


Sample 1 - Total Acidity
(0.1g/L as Tartaric Acid)
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
12
2
5
8
10
14
15
9
13
3
6
11
16
18
7
4
1
Laboratory Code Number

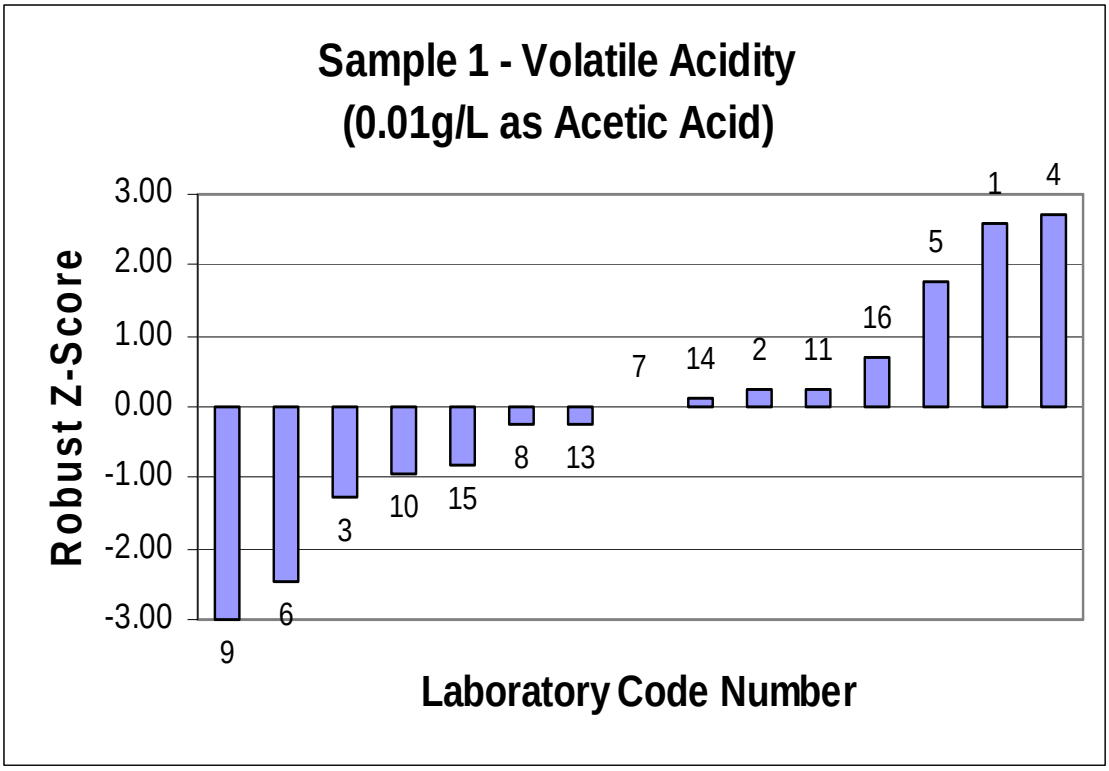
Volatile Acidity
0.05g/L as Acetic Acid

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1	0.53	0.05	0.48	0.05	AOAC940.19	0.51	2.58
2	0.41	0.10	0.40	0.10	Autoanalyser	0.41	0.23
3	0.34	0.10	0.34	0.10	Enzymatic	0.34	-1.29
4	0.52	0.065	0.50	0.065	Distillation	0.51	2.70
5	0.47	0.05	0.47	0.05	FTIR	0.47	1.76
6	0.29	0.05	0.29	0.05	Distillation	0.29	-2.46
7	0.41	0.03	0.38	0.03	Acetic Acid, enzymatic	0.40	0.00
8	0.39	0.04	0.38	0.04	Distillation/Titration	0.39	-0.23
9	0.24	0.01	0.25	0.01	Enzyme Kit	0.25	-3.52 **§**
10	0.36	0.12	0.35	0.12	HPLC	0.36	-0.94
11	0.41		0.40		Distillation/Titration	0.41	0.23
12							
13	0.38	0.04	0.39	0.04	Distillation	0.39	-0.23
14	0.40	0.09			Winescan	0.40	0.12
15	0.36	10%	0.36	10%	Enzymatic Acetic Acid	0.36	-0.82
16	0.42	0.05	0.43	0.05	Enzymatic	0.43	0.70
17							
18							

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	15
Median	0.395
NormIQR	0.043
Robust CV	10.79%
Min	0.25
Max	0.51
Range	0.265


Sample 1 - Volatile Acidity
(0.01g/L as Acetic Acid)
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
9
6
3
10
15
8
13
7
14
2
11
16
5
1
4
Laboratory Code Number

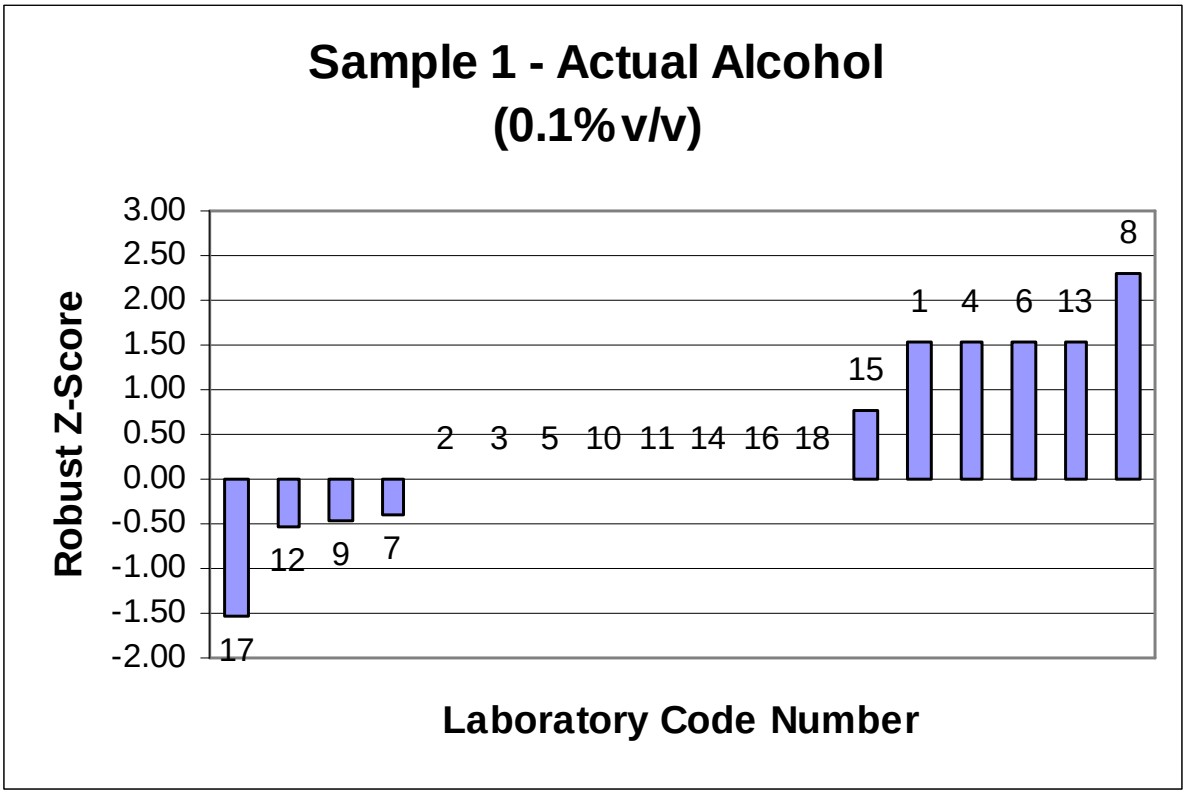
Actual Alcohol
0.1%v/v

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1	13.5	0.2	13.5	0.2	QIS12669	13.5	1.54
2	13.4	0.2	13.4	0.2	Distillation Hydrometry	13.4	0.00
3	13.4	0.1	13.4	0.1	Near Infra Red	13.4	0.00
4	13.5	0.046	13.5	0.046	Alcoholyser	13.5	1.54
5	13.4	0.2	13.4	0.2	FTIR	13.4	0.00
6	13.5	0.2	13.5	0.2	NIR	13.5	1.54
7	13.38	0.10	13.37	0.10	NIR	13.4	-0.39
8	13.5	0.3	13.6	0.3	Distillation Hydrometry	13.6	2.31
9	13.41	0.52	13.33	0.52	GC	13.4	-0.46
10	13.4	0.12	13.4	0.12	NIR	13.4	0.00
11	13.3		13.5		Distillation/Pycometry	13.4	0.00
12	13.37	0.1	13.36	0.1	NIR	13.4	-0.54
13	13.5	0.1	13.5	0.1	Alcoholyser NIR	13.5	1.54
14	13.4	0.1			Winescan	13.4	0.00
15	13.4	0.1	13.5	0.1	NIR	13.5	0.77
16	13.4	0.1			Distillation/Hydrometry	13.4	0.00
17	13.3	3%	13.3	3%	Based on AOAC984.14	13.3	-1.54
18	13.4	0.1	13.4			13.4	0.00

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Results

No. results	18
Median	13.40
NormIQR	0.06
Robust CV	0.48%
Min	13.3
Max	13.6
Range	0.3


Sample 1 - Actual Alcohol
(0.1% v/v)
Robust Z-Score
3.00
2.50
2.00
1.50
1.00
0.50
0.00
-0.50
-1.00
-1.50
-2.00
17
12
9
7
2
3
5
10
11
14
16
18
15
1
4
6
13
8
Laboratory Code Number

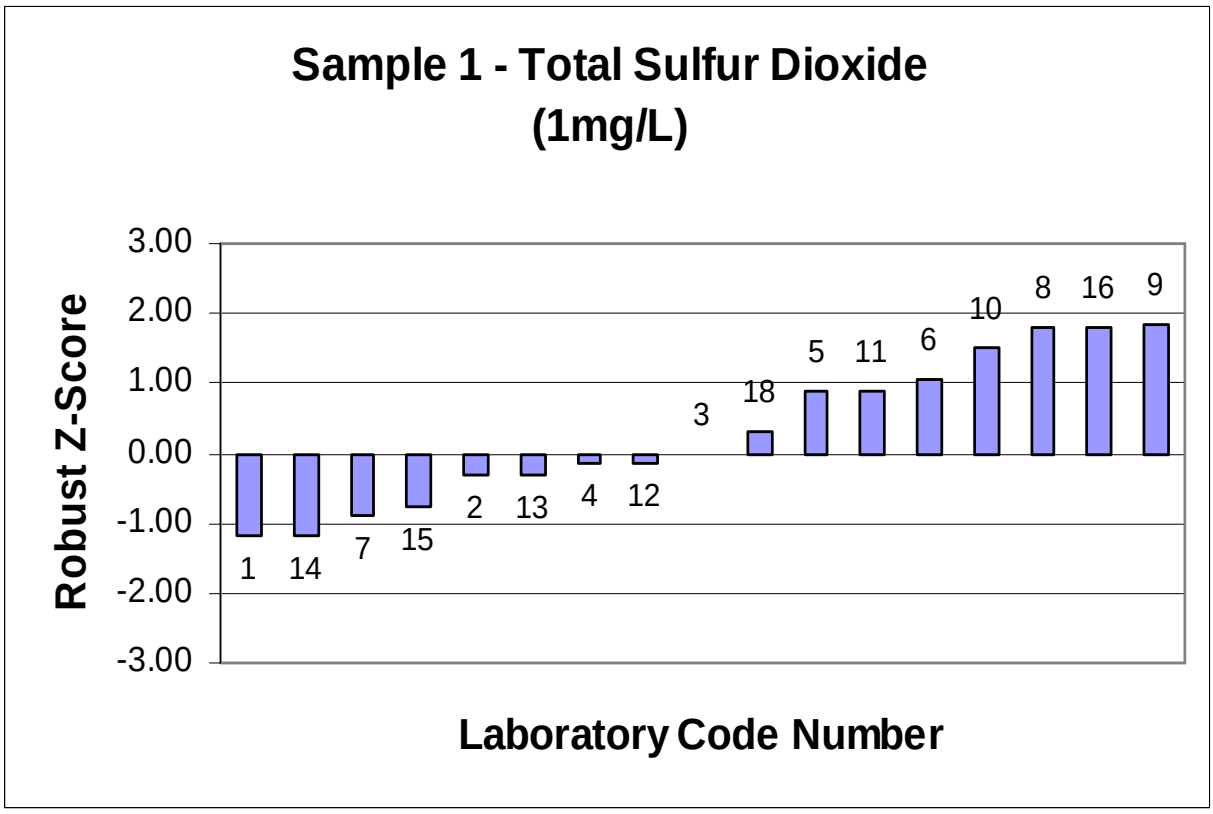
Total Sulfur Dioxide
1mg/L

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1	158	10	156	10	Monier Williams	157	-1.20
2	160	6	160	6	Aspiration	160	-0.30
3	160	4	162	4	Aspiration	161	0.00
4	161	6.8	160	6.8	Rankine	161	-0.15
5	166	8%	162	8%	Aspiration	164	0.90
6	165	6	164	6	Rankine Aspiration	165	1.05
7	157	3	159	3	Rankine Aspiration	158	-0.90
8	167	4	167	4	Aspiration/Titration	167	1.80
9	165.7	16.57	168.6	16.86	Aspiration	167	1.84
10	166	14	166	14	Aspiration/Oxidation	166	1.50
11	163		165		Monier Williams	164	0.90
12	159	6	162	6	Aspiration	161	-0.15
13	161	4	159	4	Aspiration	160	-0.30
14	157	5			FIA	157	-1.20
15	162	6	155	6	Aspiration	159	-0.75
16	165	5	169	5	Aspiration/Oxidation	167	1.80
17							
18	159	15	165			162	0.30

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	17
Median	161.0
NormIQR	3.3
Robust CV	2.1%
Min	157
Max	167
Range	10


Sample 1 - Total Sulfur Dioxide (1mg/L)
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
1
14
7
15
2
13
4
12
3
18
5
11
6
10
8
16
9
Laboratory Code Number

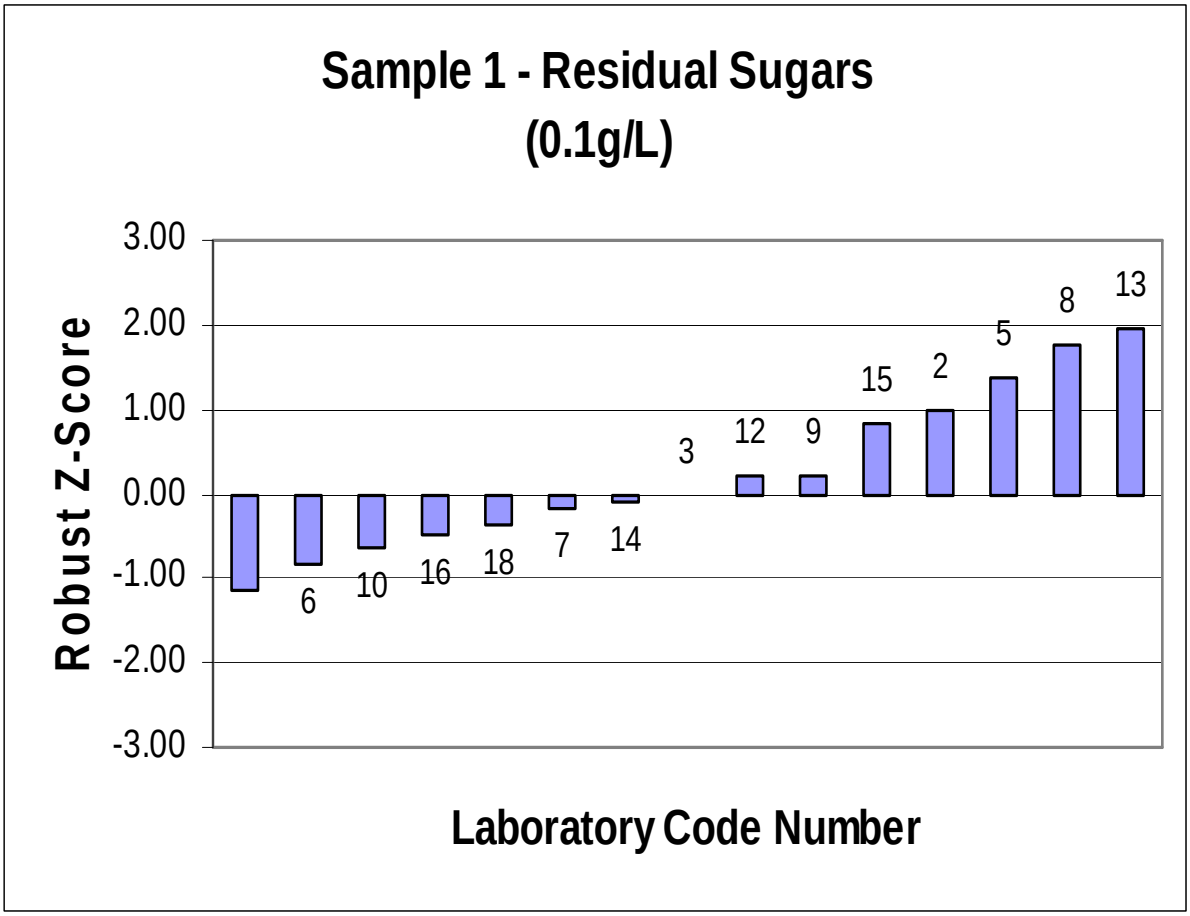
Residual Sugars
0.1g/L

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1							
2	5.8	0.2	5.8	0.2	HPLC	5.8	0.85
3	5.06	0.1	5.06	0.1	Enzymatic	5.1	-0.10
4	6.6	0.15	6.7	0.15	Rebelein	6.7	1.94
5	5.8	0.5	6.0	0.5	HPLC	5.9	0.98
6	4.3	0.5	4.2	0.5	Lane & Eynon	4.3	-1.15
7	4.86	0.12	4.85	0.12	Enzymatic, Glucose & Fructose	4.9	-0.37
8	6.2	0.3	6.2	0.3	Rebelein	6.2	1.37
9	5.385	0.48	5.205	0.47	Enzymatic Kit	5.3	0.20
10	4.5	0.13	4.5	0.13	Enzymatic	4.5	-0.82
11							
12	5.01	0.59	5.27	0.59	Enzymatic UV-Vis Spectrometer	5.1	0.00
13	6.6	0.4	6.4	0.4	Lane & Eynon	6.5	1.75
14	5.0	0.27			Winescan	5.0	-0.18
15	5.2	5%	5.4	5%	Enzymatic Glucose & Fructose	5.3	0.21
16	4.6	0.1	4.7	0.1	Enzymatic	4.7	-0.63
17							
18	4.7	0.5	4.8			4.8	-0.50

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	15
Median	5.14
NormIQR	0.78
Robust CV	15.11%
Min	4.3
Max	6.7
Range	2.4


Sample 1 - Residual Sugars
(0.1g/L)
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
6
10
16
18
7
14
3
12
9
15
2
5
8
13
Laboratory Code Number

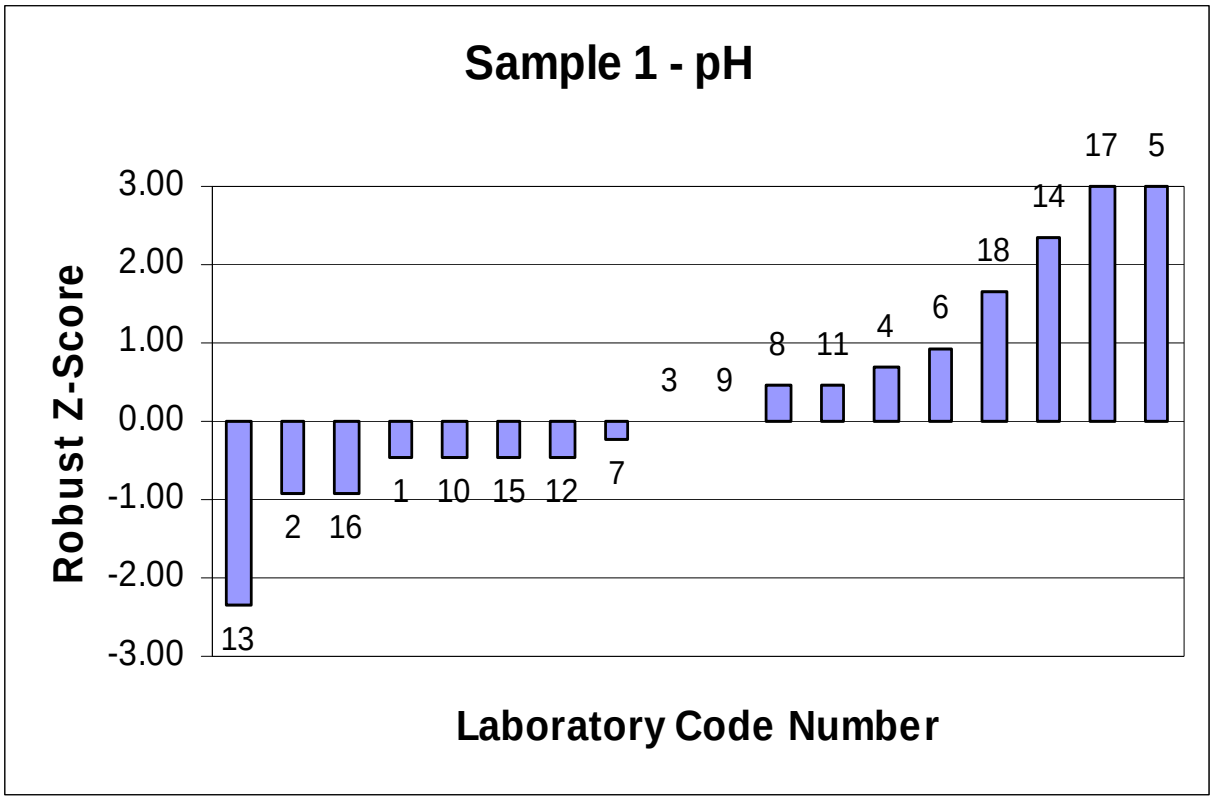
pH

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1	3.28	0.05	3.28	0.05		3.28	-0.47
2	3.27	0.05	3.27	0.05	pH Probe	3.27	-0.94
3	3.29	0.05	3.29	0.05	Meter	3.29	0.00
4	3.30		3.31		pH meter	3.31	0.70
5	3.38	0.07	3.38	0.07	pH electrode	3.38	4.22 §
6	3.31	0.2	3.31	0.2	pH meter	3.31	0.94
7	3.27	0.05	3.30	0.05	Potentiometric	3.29	-0.23
8	3.30	0.04	3.30	0.04	pH meter	3.30	0.47
9	3.29	0.03	3.29	0.03	pH meter	3.29	0.00
10	3.28	0.02	3.28	0.02	Autotitrator	3.28	-0.47
11	3.30		3.30		pH electrode	3.30	0.47
12	3.27	0.06	3.29	0.06	Autotitrator	3.28	-0.47
13	3.24	0.06	3.24	0.06	Autotitrator	3.24	-2.35
14	3.34	0.048			Winescan	3.34	2.35
15	3.28	0.1	3.28	0.1	pH meter	3.28	-0.47
16	3.27	0.1	3.27	0.1	Potentiometric	3.27	-0.94
17	3.37	0.3	3.38	0.3	Based on AOAC 16th ed. Vol II Method no 31.1.07	3.38	3.99 §
18	3.32		3.33			3.33	1.64

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	18
Median	3.290
NormIQR	0.021
Robust CV	0.65%
Min	3.24
Max	3.38
Range	0.14


Sample 1 - pH
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
13
2
16
1
10
15
12
7
3
9
8
11
4
6
18
14
17
5
Laboratory Code Number

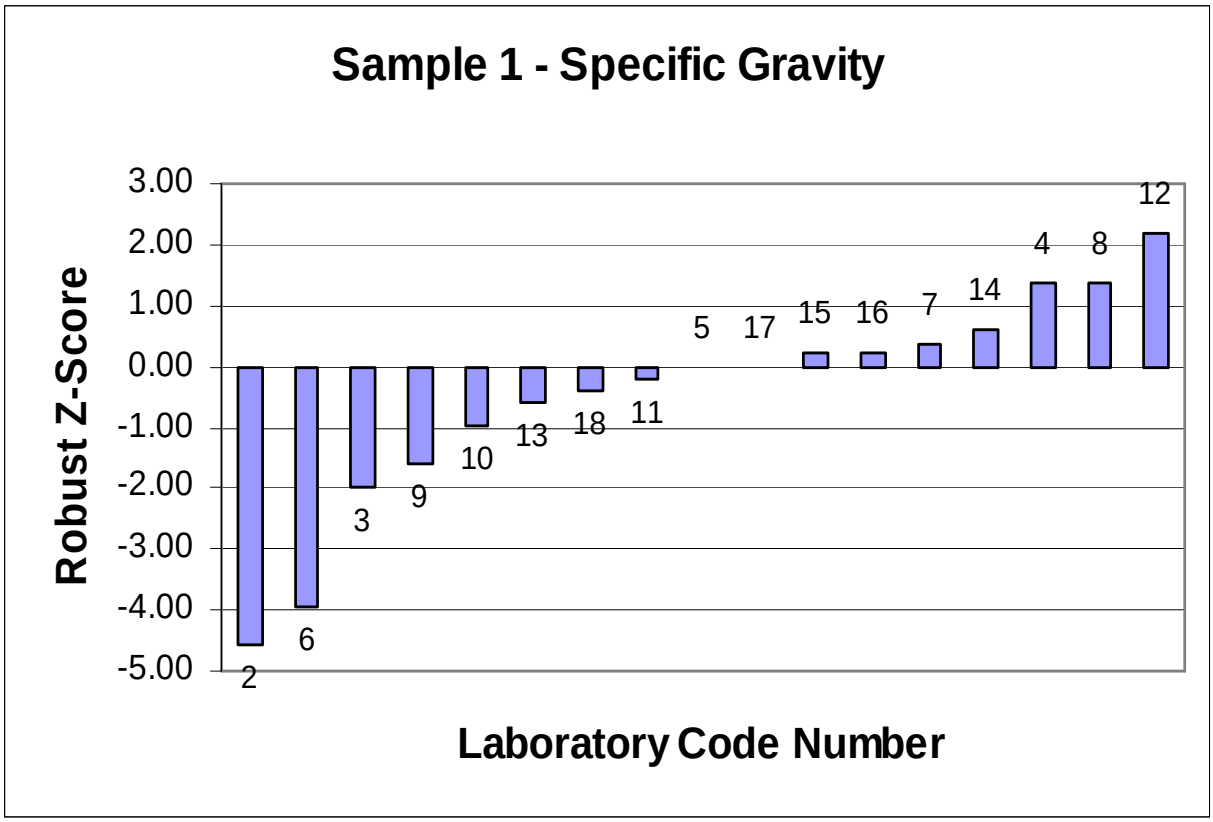
Specific Gravity

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1							
2	0.990	0.0005	0.990	0.0005	Hydrometry	0.9900	-4.56 **§**
3	0.9913	0.0008	0.9913	0.0008	Hydrometer	0.9913	-1.98
4	0.993	0.081	0.993	0.081	Density Meter	0.9930	1.39
5	0.9923	0.0006	0.9923	0.0006	Density Meter	0.9923	0.00
6	0.9897	0.002	0.9909	0.002	Density Meter	0.9903	-3.97 **§**
7	0.99248	0.0001	0.99248	0.0001	Density Meter	0.9925	0.36
8	0.993	0.002	0.993	0.002	Density Meter	0.9930	1.39
9	0.9915	0.001	0.9915	0.001	Hydrometer	0.9915	-1.59
10	0.9918	0.0012	0.9918	0.0012	Hydrometer	0.9918	-0.99
11	0.9923		0.9921		Pycnometry	0.9922	-0.20
12	0.9935	0.001	0.9933	0.001	DMS Meter	0.9934	2.18
13	0.992	0.001	0.992	0.001	Alcolyzer NIR	0.9920	-0.60
14	0.9926	0.00055			Winescan	0.9926	0.60
15	0.9924	0.0003	0.9924	0.0003	Density Meter	0.9924	0.20
16	0.9924	0.0003	0.9924	0.0003	Density Hydrometer	0.9924	0.20
17	0.9922	0.001	0.9924	0.001	Manufacturer's Manual	0.9923	0.00
18	0.9920		0.9922			0.9921	-0.40

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	17
Median	0.99230
NormIQR	0.00050
Robust CV	0.05%
Min	0.9900
Max	0.9934
Range	0.0034


Sample 1 - Specific Gravity
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
-4.00
-5.00
2
6
3
9
10
13
18
11
5
17
15
16
7
14
4
8
12
Laboratory Code Number

Section 2 –

Sample 2 (Red Wine)

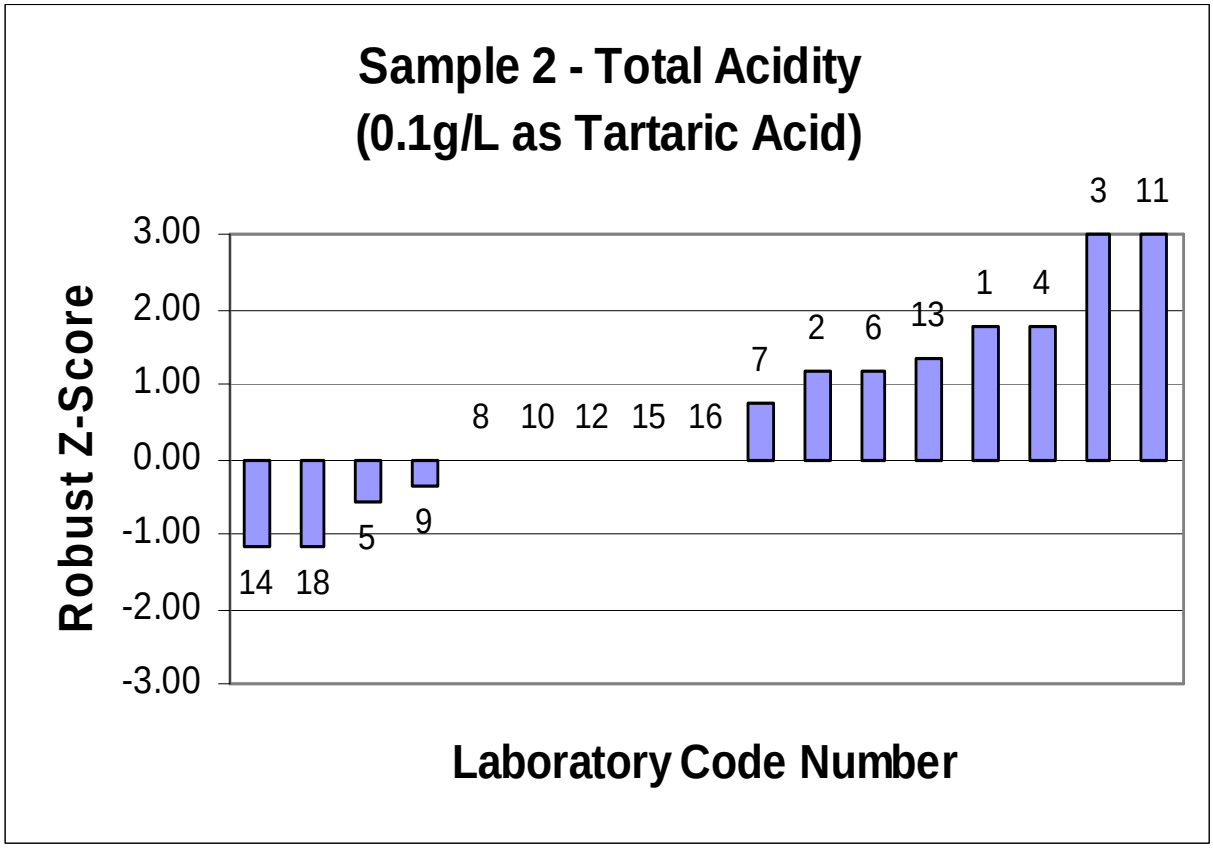
Total Acidity
0.1g/L as Tartaric Acid

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1	6.8	0.2	6.5	0.2	AOAC962.12	6.7	1.76
2	6.6	0.10	6.6	0.10	Titration to an end point	6.6	1.17
3	6.8	0.08	6.8	0.08	Manual Titration	6.8	3.52 **§**
4	6.6	0.12	6.7	0.12		6.7	1.76
5	6.5	0.1	6.4	0.1	Autotitration	6.5	-0.59
6	6.6	0.2	6.6	0.2	Autotitrator	6.6	1.17
7	6.55	0.15	6.58	0.15	Autotitration	6.6	0.76
8	6.5	0.11	6.5	0.11	Potentiometric/ Autotitrator	6.5	0.00
9	6.49	0.13	6.45	0.13	Manual Titration	6.5	-0.35
10	6.5	0.07	6.5	0.07	Autotitrator	6.5	0.00
11	7.0		7.0		Titration to end point 8.2	7.0	5.87 **§**
12	6.49	0.19	6.51	0.19	Autotitrator	6.5	0.00
13	6.63	0.12	6.60	0.12	Autotitrator	6.6	1.35
14	6.4	0.18				6.4	-1.17
15	6.5	0.1	6.5	0.1	Titration	6.5	0.00
16	6.5	0.1	6.5	0.1	Autotitrator	6.5	0.00
17							
18	6.4	0.1	6.4	0.1	AOAC947.05	6.4	-1.17

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	17
Median	6.50
NormIQR	0.09
Robust CV	1.31%
Min	6.4
Max	7.0
Range	0.6


Sample 2 - Total Acidity
(0.1g/L as Tartaric Acid)
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
14 18 5 9 8 10 12 15 16 7 2 6 13 1 4 3 11
Laboratory Code Number

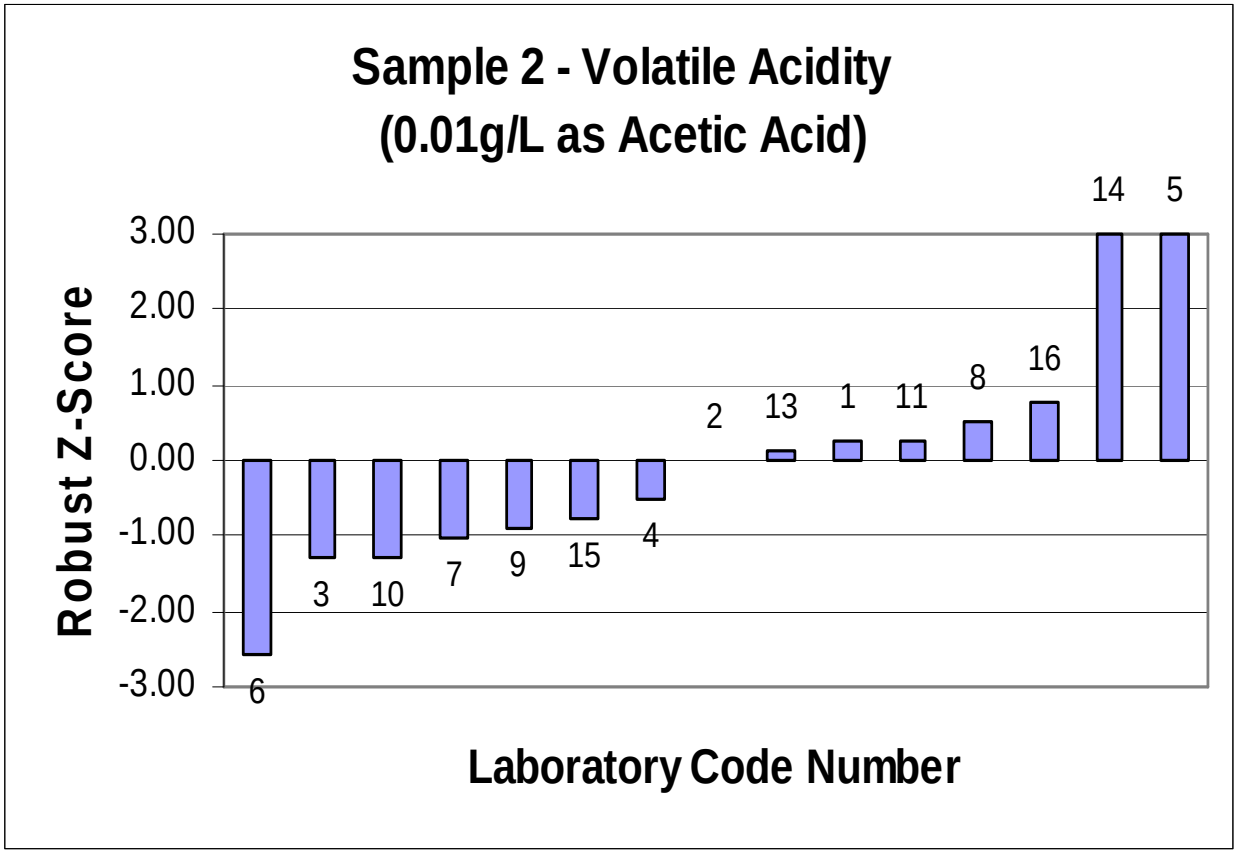
Volatile Acidity
0.05g/L as Acetic Acid

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1	0.36	0.05	0.36	0.05	AOAC940.19	0.36	0.26
2	0.35	0.10	0.35	0.10	Autoanalyser	0.35	0.00
3	0.30	0.10	0.30	0.10	Enzymatic	0.30	-1.28
4	0.32	0.065	0.34	0.065		0.33	-0.51
5	0.50	0.05	0.49	0.05	FTIR	0.50	3.73 §
6	0.25	0.05	0.25	0.05	Distillation	0.25	-2.57
7	0.32	0.03	0.30	0.03	Acetic Acid, enzymatic	0.31	-1.03
8	0.37	0.04	0.37	0.04	Distillation/ Titration	0.37	0.51
9	0.310	0.02	0.320	0.02	Enzymatic Kit	0.32	-0.90
10	0.30	0.12	0.30	0.12	HPLC	0.30	-1.28
11	0.36		0.36		Distillation Titration with indicator	0.36	0.26
12							
13	0.35	0.04	0.36	0.04	Distillation	0.36	0.13
14	0.48	0.09				0.48	3.34 §
15	0.32	10%	0.32	10%	Enzymatic Acetic Acid	0.32	-0.77
16	0.38	0.05	0.38	0.05	Enzymatic	0.38	0.77
17							
18							

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	15
Median	0.350
NormIQR	0.039
Robust CV	11.120%
Min	0.25
Max	0.50
Range	0.25


Sample 2 - Volatile Acidity
(0.01g/L as Acetic Acid)
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
6
3
10
7
9
15
4
2
13
1
11
8
16
14
5
Laboratory Code Number

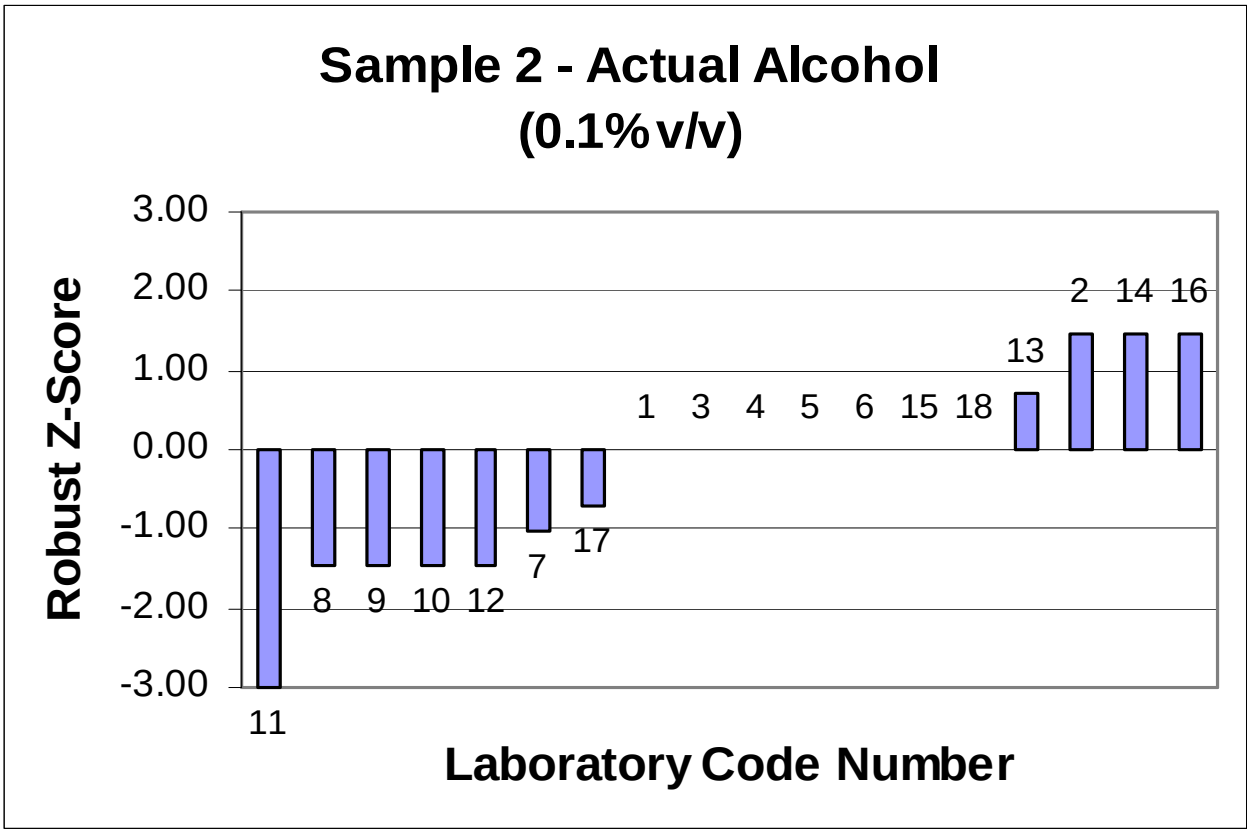
Actual Alcohol
0.1%v/v

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1	13.4	0.2	13.6	0.2	QIS12669	13.5	0.00
2	13.6	0.2	13.6	0.2	Distillation Hydrometry	13.6	1.46
3	13.5	0.1	13.5	0.1	Near Infra Red	13.5	0.00
4	13.5	0.046	13.5	0.046		13.5	0.00
5	13.5	0.2	13.5	0.2	FTIR	13.5	0.00
6	13.5	0.2	13.5	0.2	NIR	13.5	0.00
7	13.43	0.10	13.43	0.10	NIR	13.4	-1.02
8	13.4	0.3	13.4	0.3	Distillation/ Hydrometry	13.4	-1.46
9	13.40	0.52	13.40	0.52	GC	13.4	-1.46
10	13.4	0.12	13.4	0.12	NIR	13.4	-1.46
11	13.2		13.2		Distillation Pycnometry	13.2	-4.38 **§**
12	13.42	0.1	13.38	0.1	NIR	13.4	-1.46
13	13.5	0.1	13.6	0.1	Alcolyser NIR	13.6	0.73
14	13.6	0.1				13.6	1.46
15	13.5	0.1	13.5	0.1	NIR	13.5	0.00
16	13.6	0.1			Distillation/ Hydrometry	13.6	1.46
17	13.5	3%	13.4	3%	Based on AOAC984.14	13.5	-0.73
18	13.5	0.1	13.5		In House Method	13.5	0.00

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	18
Median	13.50
NormIQR	0.07
Robust CV	0.51%
Min	13.2
Max	13.6
Range	0.4


Sample 2 - Actual Alcohol
(0.1% v/v)
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
11
8
9
10
12
7
17
1
3
4
5
6
15
18
13
2
14
16
Laboratory Code Number

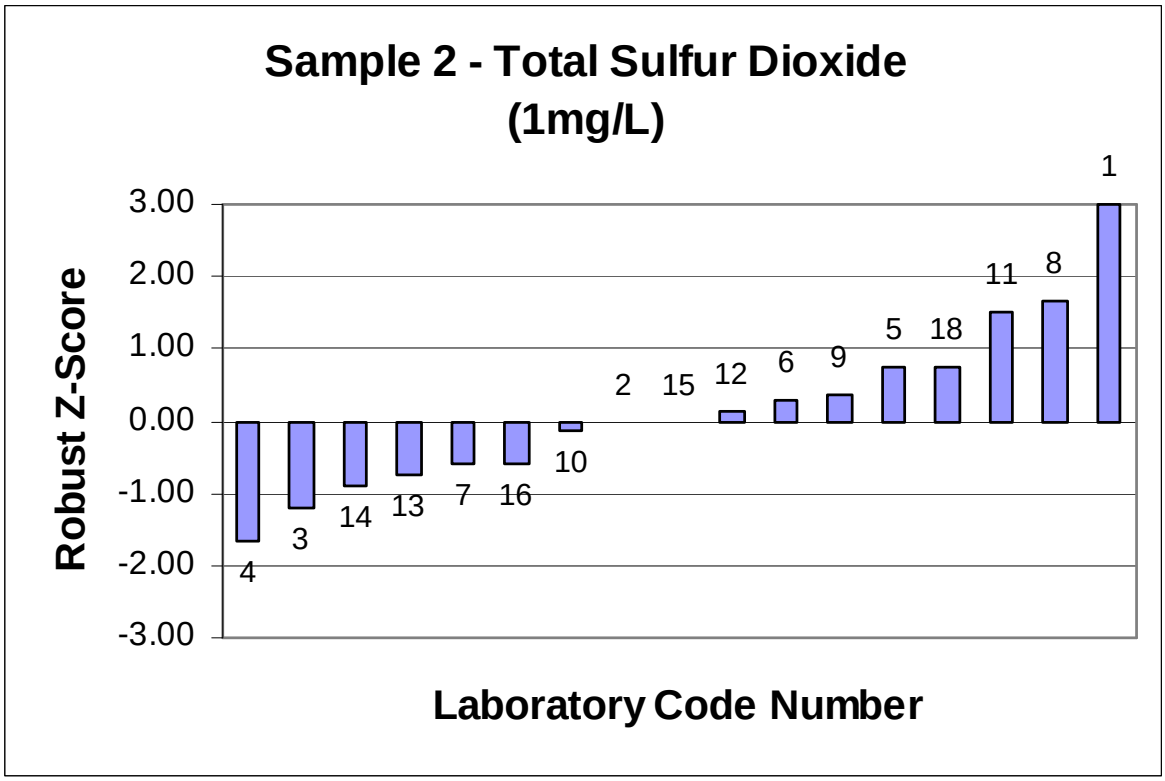
Total Sulfur Dioxide
1mg/L

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1	88	10	81	10	Monier Williams	85	3.75 **§**
2	73	6	71	6	Aspiration	72	0.00
3	68	4	68	4	Aspiration	68	-1.20
4	67	6.8	66	6.8		67	-1.65
5	74	4	75	4	Aspiration	75	0.75
6	74	6	72	6	Rankine Aspiration	73	0.30
7	70	3	70	3	Rankine Aspiration	70	-0.60
8	76	4	79	4	Aspiration/ Titration	78	1.65
9	72.76	7.28	73.60	7.36	Aspiration	73	0.35
10	73	6	70	6	Aspiration/Oxidation	72	-0.15
11	77		77		Monier Williams	77	1.50
12	72	6	73	6	Aspiration	73	0.15
13	70	4	69	4	Aspiration	70	-0.75
14	69	4				69	-0.90
15	72	6	72	6	Aspiration	72	0.00
16	70	5	70	5	Aspiration/Oxidation	70	-0.60
17							
18	75	7	74		AOAC990.28	75	0.75

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	17
Median	72.0
NormIQR	3.3
Robust CV	4.6%
Min	67
Max	85
Range	18


Sample 2 - Total Sulfur Dioxide (1mg/L)
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
4
3
14
13
7
16
10
2
15
12
6
9
5
18
11
8
1
Laboratory Code Number

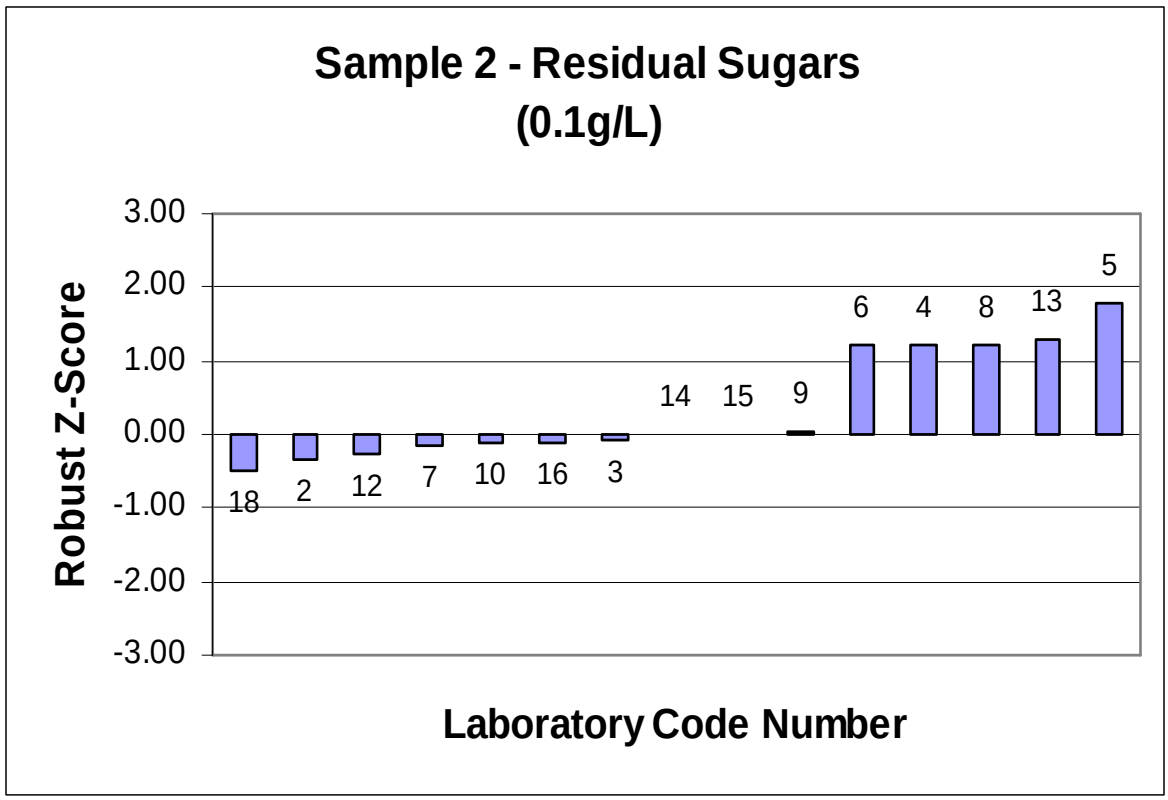
Residual Sugars
0.1g/L

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1							
2	2.5	0.2	2.4	0.2	HPLC	2.5	-0.35
3	2.96	0.1	2.96	0.1	Enzymatic	3.0	-0.07
4	5.3	0.15	5.5	0.15		5.4	1.23
5	6.4	0.5	6.5	0.5	HPLC	6.5	1.79
6	5.4	0.5	5.3	0.5	Lane & Eynon	5.4	1.20
7	2.80	0.12	2.79	0.12	Glucose & Fructose, Enzymatic	2.8	-0.16
8	5.4	0.3	5.4	0.3	Rebelein	5.4	1.23
9	3.14	0.28	3.145	0.28	Enzymatic Kit	3.1	0.02
10	2.8	0.13	3.0	0.13	Enzymatic	2.9	-0.11
11							
12	2.57	0.59	2.63	0.59	Enzymatic UV Vis Spectrophotometer	2.6	-0.27
13	5.6	0.4	5.5	0.4	Lane & Eynon	5.6	1.31
14	3.1	0.27				3.1	0.00
15	3.2	5%	3.0	5%	Enzymatic Glucose & Fructose	3.1	0.00
16	2.9	0.1	2.9	0.1	Enzymatic	2.9	-0.11
17							
18	2.2	0.3	2.2		In House Method	2.2	-0.48

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	15
Median	3.10
NormIQR	1.87
Robust CV	60.44%
Min	2.2
Max	6.5
Range	4.3


Sample 2 - Residual Sugars
(0.1g/L)
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
18
2
12
7
10
16
3
14
15
9
6
4
8
13
5
Laboratory Code Number

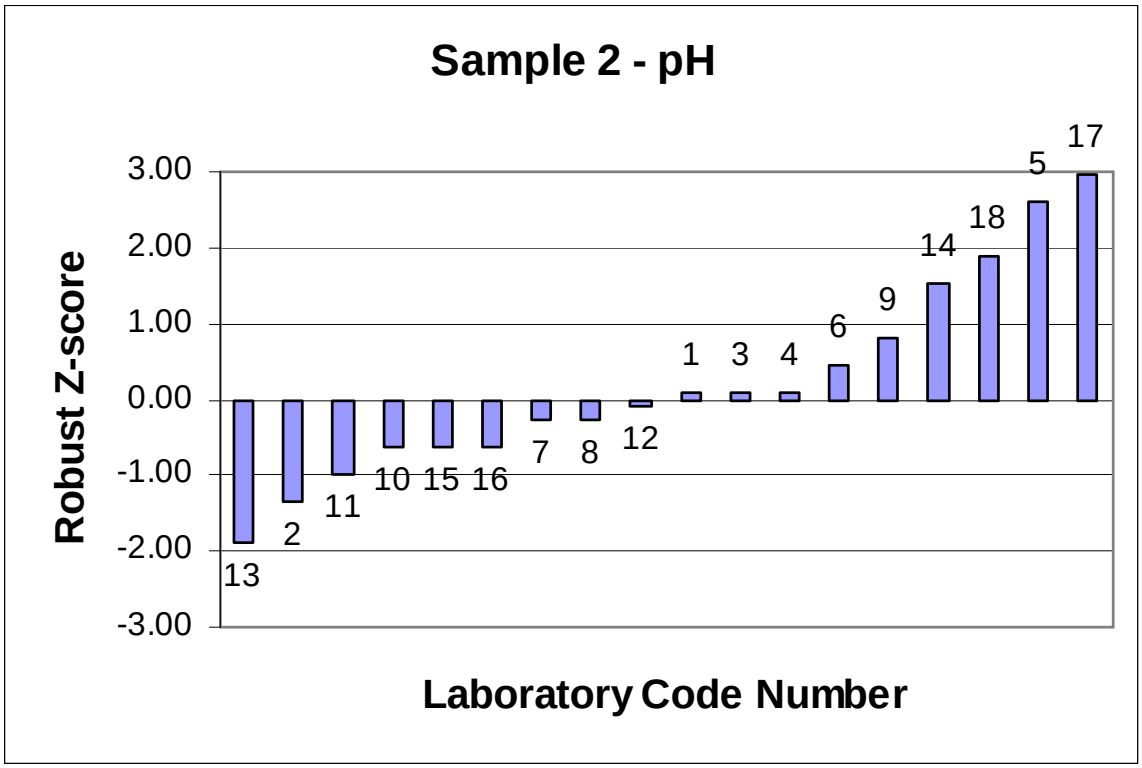
pH

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1	3.49	0.05	3.49	0.05		3.49	0.09
2	3.45	0.05	3.45	0.05	pH Probe	3.45	-1.35
3	3.49	0.05	3.49	0.05	Meter	3.49	0.09
4	3.49		3.49			3.49	0.09
5	3.56	0.07	3.56	0.07	pH electrode	3.56	2.61
6	3.50	0.2	3.50	0.2	pH meter	3.50	0.45
7	3.46	0.05	3.50	0.05	Potentiometric	3.48	-0.27
8	3.49	0.04	3.47	0.04	pH meter	3.48	-0.27
9	3.51	0.04	3.51	0.04	pH meter	3.51	0.81
10	3.47	0.02	3.47	0.02	Autotitrator	3.47	-0.63
11	3.46		3.46		pH electrode	3.46	-0.99
12	3.46	0.06	3.51	0.06	Autotitrator	3.49	-0.09
13	3.44	0.06	3.43	0.06	Autotitrator	3.44	-1.89
14	3.53	0.048				3.53	1.53
15	3.47	0.1	3.47	0.1	pH meter	3.47	-0.63
16	3.47	0.1	3.47	0.1	Potentiometric	3.47	-0.63
17	3.57	0.3	3.57	0.3	Based on AOAC 16th ed. Vol II Method no 31.1.07	3.57	2.97
18	3.54		3.54			3.54	1.89

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	18
Median	3.488
NormIQR	0.028
Robust CV	0.80%
Min	3.44
Max	3.57
Range	0.13


Sample 2 - pH
Robust Z-score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
13
2
11
10
15
16
7
8
12
1
3
4
6
9
14
18
5
17
Laboratory Code Number

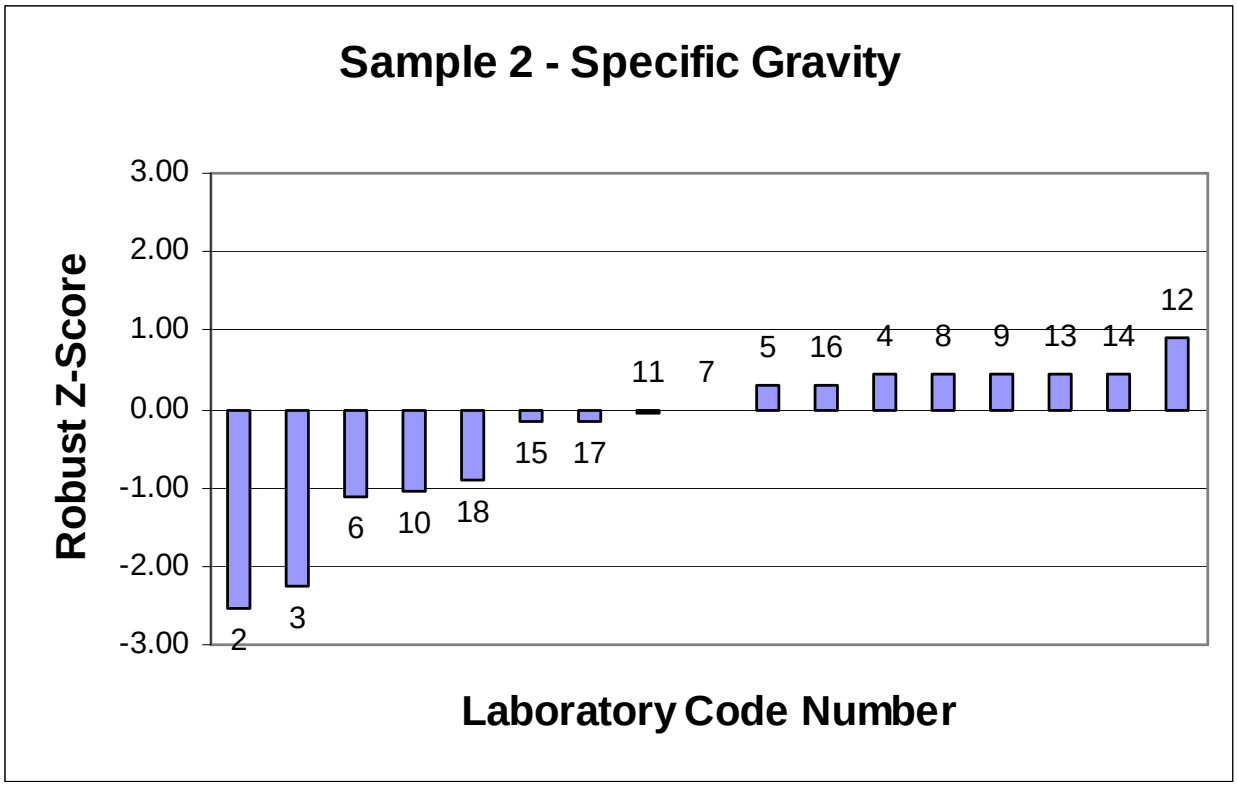
Specific Gravity

Lab Code	Result 1	MU±	Result 2	MU±	Method	Averaged Results	Robust Z-Score
1							
2	0.995	0.0005	0.995	0.0005	Hydrometer	0.9950	-2.54
3	0.9952	0.0008	0.9952	0.0008	Hydrometer	0.9952	-2.24
4	0.997	0.081	0.997	0.081		0.9970	0.46
5	0.9969	0.0006	0.9969	0.0006	Density Meter	0.9969	0.31
6	0.9956	0.002	0.9963	0.002	Density Meter	0.9960	-1.12
7	0.99669	0.0001	0.99670	0.0001	Density Meter	0.9967	0.00
8	0.997	0.002	0.997	0.002	Density Meter	0.9970	0.46
9	0.9970	0.001	0.9970	0.001	Hydrometer	0.9970	0.46
10	0.9960	0.0012	0.9960	0.0012	Hydrometer	0.9960	-1.04
11	0.9968		0.9965		Pycometry	0.9967	-0.07
12	0.9974	0.001	0.9972	0.001	DMS Meter	0.9973	0.91
13	0.997	0.001	0.997	0.001	Alcolyser NIR	0.9970	0.46
14	0.9970	0.00055				0.9970	0.46
15	0.9966	0.0003	0.9966	0.0003	Density Meter	0.9966	-0.14
16	0.9969	0.0003	0.9969	0.0003	Density Hydrometer	0.9969	0.31
17	0.9966	0.001	0.9966	0.001	Manufacturer's Manual	0.9966	-0.14
18	0.9962	0.0005	0.9960		AS2378 & AOAC920.56	0.9961	-0.89

Notes:
MU = Measurement Uncertainty
§ denotes an outlier

Summary Statistics

No. results	17
Median	0.99670
NormIQR	0.00067
Robust CV	0.07%
Min	0.9950
Max	0.9973
Range	0.0023


Sample 2 - Specific Gravity
Robust Z-Score
3.00
2.00
1.00
0.00
-1.00
-2.00
-3.00
2
3
6
10
18
15
17
11
7
5
16
4
8
9
13
14
12
Laboratory Code Number

APPENDIX B

Homogeneity Testing

HOMOGENEITY TESTING

As mentioned in the introduction of this report, a number of samples were selected for preliminary testing analyses to monitor the homogeneity of the samples.

For this, 10 of each sample type were randomly selected and tested under repeatability conditions (i.e. same operator, same method etc). The results are given below. Statistical Analysis using an ANOVA indicate that there was no significant difference between the samples.

White Wine Sample

Sample	Alc	RS	TA	pH	VA	SG	Total SO2
1	13.4	4.9	5.9	3.37	0.35	0.9919	168
2	13.4	4.9	5.9	3.38	0.35	0.9918	162
3	13.4	4.9	5.9	3.38	0.35	0.9918	165
4	13.4	4.9	5.8	3.38	0.35	0.9918	169
5	13.4	4.8	5.9	3.38	0.36	0.9918	166
6	13.4	4.9	5.9	3.38	0.35	0.9918	170
7	13.4	4.9	5.8	3.38	0.35	0.9917	170
8	13.4	4.9	5.8	3.38	0.35	0.9918	168
9	13.4	4.9	5.8	3.38	0.35	0.9918	168
10	13.4	5	5.9	3.37	0.35	0.9918	162

Red Wine Sample

Sample	Alc	RS	TA	pH	VA	SG	Total SO2
1	13.4	2.6	6.5	3.56	0.51	0.9961	75
2	13.4	2.5	6.5	3.56	0.51	0.9961	74
3	13.5	2.6	6.5	3.55	0.51	0.9962	76
4	13.5	2.6	6.6	3.56	0.50	0.996	76
5	13.5	2.6	6.6	3.56	0.49	0.996	74
6	13.5	2.4	6.6	3.54	0.50	0.9962	76
7	13.5	2.7	6.5	3.53	0.50	0.9961	76
8	13.5	2.5	6.6	3.53	0.49	0.9962	76
9	13.5	2.6	6.5	3.53	0.50	0.996	76
10	13.5	2.6	6.6	3.53	0.48	0.9961	74

APPENDIX C

Instructions to Participants

and

Results Sheet

PROFICIENCY TESTING AUSTRALIA
WINE PROFICIENCY PROGRAM – NOVEMBER 2006
ROUND 15
INSTRUCTIONS TO PARTICIPANTS

Please read instructions carefully BEFORE commencing testing.

To ensure that the results of this program can be analysed properly participants are asked to note carefully the following:

1. For this program each participant is provided with two samples, one of white wine and one of red wine labelled sample 1 and sample 2.

2. The following tests are to be conducted in duplicate on both samples:

   Total Acidity
   Volatile Acidity
   Actual Alcohol
   Total Sulfur Dioxide
   Reducing Sugars
   pH
   Specific Gravity

   Laboratories should perform these tests by their usual methods.

   All results are to be reported on the attached Results Sheet. Please ensure that the method used is entered on the Results Sheet for each set of analyses. If the method is not a standard method, please provide a precise description.

3. For Total Acidity, participants should titrate to end point pH 8.2. It is recognised that some methods for total acidity (e.g. EEC Regulation 000/90; Method 13, page 81) prescribe an end point at pH 7.0. However, for the purpose of this program it is necessary that all values be obtained at the same end point, namely pH 8.2.

4. 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 give as a 95% confidence interval (coverage factor $k \approx 2$)

5. All tests may be conducted as soon as the samples are received. Results should be forwarded to:

   Ms Kate Wiggins
   Senior Scientific Officer
   Proficiency Testing Australia
   PO Box 7507
   SILVERWATER NSW 2128
   ph: (02) 9736 8397 fax: (02) 9743 6664
   e-mail: kwiggins@pta.asn.au
   please no later than **1 December 2006.**

6. For this program your laboratory has been allocated a code number which will allow for confidential treatment of your results in any reports prepared for this round of testing.
   Your code number for this round of testing is ☐

C1

PROFICIENCY TESTING AUSTRALIA
WINE PROFICIENCY PROGRAM ROUND 15– NOVEMBER 2006
RESULTS SHEET

Laboratory Code

SAMPLE 1 – WHITE WINE						
Test	Report To Nearest	Result 1	Measurement Uncertainty	Result 2	Measurement Uncertainty	Method
Total Acidity	0.1 g/L as Tartaric Acid					
Volatile Acidity	0.01g/L as Acetic Acid					
Actual Alcohol	0.1%v/v					
Total Sulfur Dioxide	1mg/L					
Residual Sugars	0.1g/L					
pH	0.01					
Specific Gravity	0.0001					

C1

PROFICIENCY TESTING AUSTRALIA

WINE PROFICIENCY PROGRAM ROUND 15– NOVEMBER 2006

RESULTS SHEET

Laboratory Code

SAMPLE 2 – RED WINE						
Test	Report To Nearest	Result 1	Measurement Uncertainty	Result 2	Measurement Uncertainty	Method
Total Acidity	0.1 g/L as Tartaric Acid					
Volatile Acidity	0.01g/L as Acetic Acid					
Actual Alcohol	0.1%v/v					
Total Sulfur Dioxide	1mg/L					
Residual Sugars	0.1g/L					
pH	0.01					
Specific Gravity	0.0001					

Date(s) of Tests(s):_____

Signature:_____

C1

PTA WINE PROFICIENCY TESTING PROGRAM – ROUND 15

MEASUREMENT UNCERTAINTY COMMENTS

Laboratory Code

Please use the space below to briefly describe the methods used to determine the measurement uncertainty for each reported measurement results.

Please return results no later than **1 December 2006 to:**

Ms Kate Wiggins
Senior Scientific Officer
Proficiency Testing Australia
PO Box 7507
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