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Department of Industry, Innovation and Science
National Measurement Institute

Proficiency Test Report
AQA 19-06
Solids in Water

June 2019

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I would like to thank the management and staff of the participating laboratories for supporting the study. It is only through widespread participation that we can provide an effective service to laboratories.

The assistance of the following NMI staff members in the planning, conduct and reporting of the study is acknowledged.

Luminita Antin

Luke Viskovic

Wei Huang

Hamish Lenton

Raluca Iavetz

A/g Manager, Chemical Proficiency Testing

Phone: 61-2-9449 0111
proficiency@measurement.gov.au

Accredited for compliance with ISO/IEC 17043

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1 SUMMARY

This report presents the results of the proficiency test AQA 19-06, solids in water. The study focused on the measurement of total solid (TS) dried at 103-105ºC, total suspended solid (TSS) dried at 103-105ºC and total dissolved solid (TDS) dried at 180ºC.

The sample set consisted of one ultrapure water sample spiked with glass fiber and potassium chloride.

The assigned values were the spiked values. The associated uncertainties were estimated from uncertainties associated with gravimetric measurement and the purity of material used for spiking.

The outcomes of the study were assessed against the aims as follows, to:

i. compare the performance of participant laboratories and assess their accuracy;

Laboratory performance was assessed using both z-scores and E_n-scores.

Of 14 results, 11 returned a satisfactory score of $|z| \leq 2$.

Of 14 E_n-scores, 11 were satisfactory with $|E_n| \leq 1$.

ii. evaluate the laboratories' methods used in determination of solids in water;

TSS was the test with the smallest between laboratories coefficient of variation, 9.8%.

iii. compare the performance of participant laboratories with their past performance;

All results reported in previous PT studies of solids in water returned satisfactory z-scores and 93% of those returned satisfactory E_n-scores.

iv. develop the practical application of traceability and measurement uncertainty and provide participants with information that will be useful in assessing their uncertainty estimates;

All results were reported with an expanded measurement uncertainty.

v. produce materials that can be used in method validation and as control samples.

The study samples were checked for homogeneity and are well characterised, through in-house testing and from the results of the proficiency round.

2 INTRODUCTION

2.1 NMI Proficiency Testing Program

The National Measurement Institute (NMI) is responsible for Australia's national measurement infrastructure providing a wide range of services, including a chemical proficiency testing program.

Proficiency testing (PT) "is evaluation of participant performance against pre-established criteria by means of interlaboratory comparison."[1] NMI PT studies target chemical testing in areas of high public significance such as trade, environment and food safety. NMI offers studies in:

- inorganic analytes in soil, water, food and pharmaceuticals;
- pesticide residues in fruit and vegetables, soil and water;
- petroleum hydrocarbons in soil and water;
- PFAS in water, soil, biota and food;
- allergens in food;
- controlled drug assay; and
- folic acid in flour.

AQA 19-06 is the 3rd NMI proficiency study of solids in water.

2.2 Study Aims

The aims of the study were to:

- compare the performance of participant laboratories and assess their accuracy;
- evaluate the laboratories methods used in the determination of solids in water;
- compare the performance of participant laboratories with their past performance;
- develop the practical application of traceability and measurement uncertainty; and
- produce materials that can be used in method validation and as control samples.

2.3 Study Conduct

The conduct of NMI proficiency tests is described in the NMI Chemical Proficiency Testing Study Protocol.[2] The statistical methods used are described in the NMI Chemical Proficiency Statistical Manual.[3] These documents have been prepared with reference to ISO Standard 17043[1] and The International Harmonized Protocol for Proficiency Testing of (Chemical) Analytical Laboratories.[4]

NMI is accredited by National Association of Testing Authorities, Australia (NATA) to ISO 17043 as a provider of proficiency testing schemes. This proficiency test is within the scope of NMI's accreditation.

The choice of the test method was left to the participating laboratories.

3 STUDY INFORMATION

3.1 Selection of Matrices and Inorganic Analytes

The tests were selected from those commonly measured in water.

3.2 Participation

Five laboratories participated and submitted results.

The timetable of the study was:

Invitations issued:	4 March 2019
Samples dispatched:	25 March 2019
Results due:	5 April 2019
Interim report issued:	10 April 2019

3.3 Test Material Specification

One sample was provided for analysis:

Sample S1 was 600 mL of unfiltered water spiked with glass fibre and potassium chloride.

3.4 Laboratory Code

All participant laboratories were assigned a confidential code number.

3.5 Sample Preparation, Analysis and Homogeneity Testing

Test samples from previous studies have been demonstrated to be sufficiently homogeneous for evaluation of participants' performance.[6] Therefore only a partial homogeneity test was conducted for TDS, TSS and TS in Sample S1 as the same preparation procedure was followed as in previous studies.[1] The results from the partial homogeneity testing are reported in the present study as the homogeneity value.

The preparation, analysis and homogeneity testing of the study samples are described in Appendix 1.

3.6 Stability of Analytes

No stability study was carried out for the present study. Sample stability was assumed based on the existing literature and on the results from the previous proficiency test of solids in water.[5,6]

3.7 Existing Sample Storage, Dispatch and Receipt

The test samples were stored at ambient temperature prior to dispatch.

The samples were dispatched by courier on 25 March 2019.

A description of the test samples, instructions for participants and a faxback form for participants to confirm the receipt of the test samples were sent with the samples.

An Excel spreadsheet for the electronic reporting of results was emailed to participants.

3.8 Instructions to Participants

Participants were instructed as follows:

- Participants will be asked to analyse the sample using their normal test method and to report results in units of mg/L for the following solids content: Total Solids (dried at 103-105ºC), Total Suspended Solids (dried at 103-105ºC) and Total Dissolved Solids (dried at 180ºC).
- Report results in mg/L using the electronic results sheet emailed to you. The approximate concentration range of the measurands in the test material is:

Sample S1	
Analyte	Approximate Conc. Range (mg/L)
TS (dried at 103-105ºC)	50-1250
TSS (dried at 103-105ºC)	50-1250
TDS (dried at 180ºC)	50-1250

- Report results as you would report to a client.
- Please send us all the requested details regarding the test method.
- Return the completed results sheet by 5 April 2019.

3.9 Interim Report

An interim report was emailed to participants on 10 April 2019.

4 PARTICIPANT LABORATORY INFORMATION

4.1 Test Method Summaries

The test methods used are described in Table 1.

Table 1 Methodology for Solids in Water

Lab. Code	Method Reference
1	APHA 2540
2	Standard methods for the examination of water and wastewater 23rd ed, 2017 APHA. AWWA, WEF. Section 2540C, 2540D
3	APHA 2540D
5	TSS 2540D, APHA AWWA (2012)

4.2 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about the basis of their uncertainty estimates (Table 2).

Table 2 Basis of Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation		Guide Document for Estimating MU
		Precision[a]	Method Bias[a]	
2	Calculated from Standard Deviation and concentration of long term in house QC	Control Samples Duplicate analysis Instrument Calibration		NATA Technical Note 33
3	Top Down – precision and estimates of the method and laboratory bias	Control samples – RM	CRM Laboratory Bias from PT Studies	NMI Uncertainty course
4	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples Duplicate analysis	CRM	NATA Technical Note 33

5	Top Down – precision and estimates if the method and laboratory bias		CRM	5

[a] RM = Reference Material, CRM = Certified Reference Material, SS =Spiked samples.

4.3 Participant Comments on this PT Study or Suggestions for Future Studies

The study coordinator welcomes comments or suggestions from participants about this study or possible future studies. Such feedback may be useful in improving future studies. There were no comments made by the participants in this study

5 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

5.1 Results Summary

Participant results are listed in Tables 3 to 5 with resultant summary statistics: robust average, median, maximum, minimum, robust standard deviation (SD_{rob}) and robust coefficient of variation (CV_{rob}). Bar charts of results and performance scores are presented in Figures 2 to 4.

An example chart with interpretation guide is shown in Figure 1.

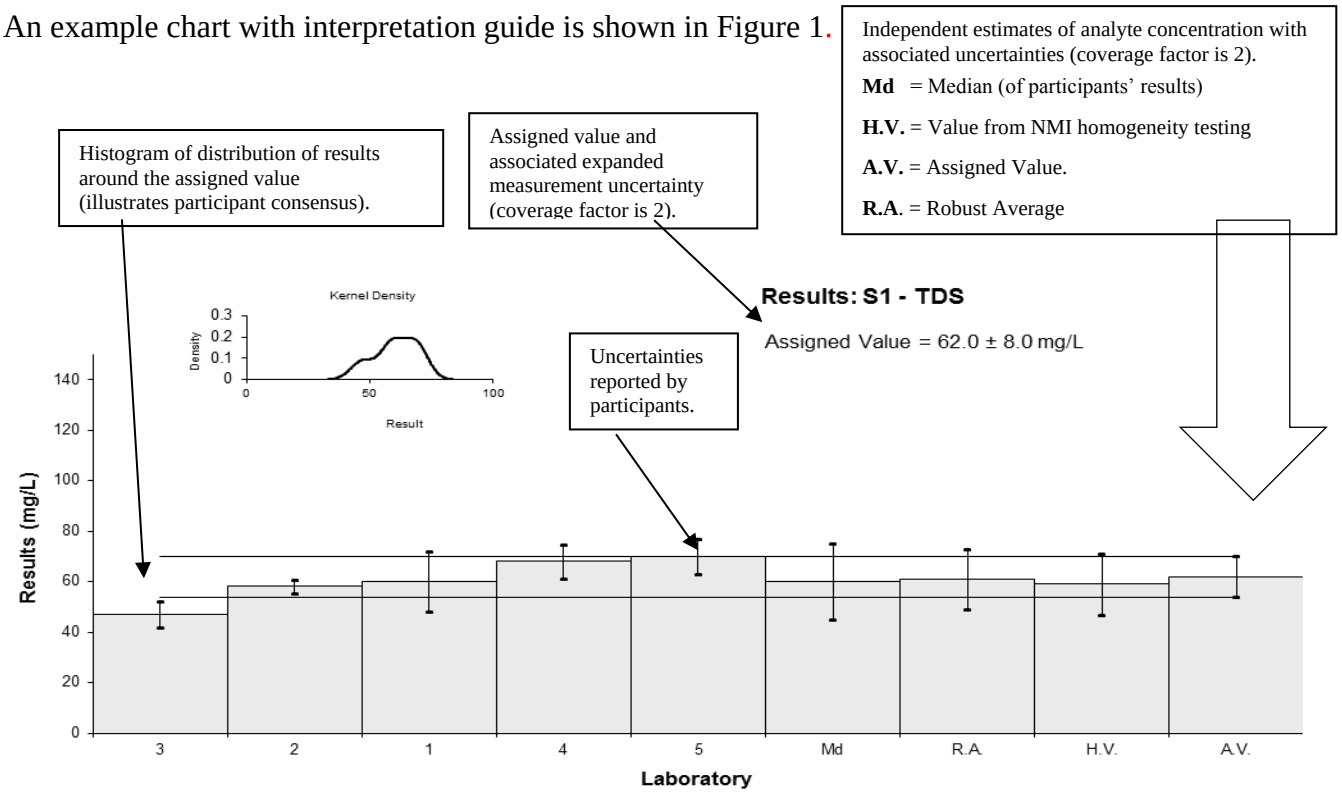


Figure 1 Guide to Presentation of Results

5.2 Assigned Value

An example of an assigned value calculation using data from the present study is given in Appendix 2. The assigned value is defined as: 'the value attributed to a particular property of a proficiency test item.'[1] In this study assigned values were the formulation values; the expanded uncertainties were estimated from uncertainties associated with gravimetric measurement and the purity of material used for spiking.

5.3 Robust Between-Laboratory Coefficient of Variation

The robust between-laboratory coefficient of variation (CV) is a measure of the variability of participants' results and was calculated using the procedure described in ISO 13528:2015(E).[7]

5.4 Target Standard Deviation

The target standard deviation (σ) is the product of the assigned value *(X)* and the performance coefficient of variation (PCV) as presented in Equation 1.

$$\sigma = (X) * \text{PCV} \qquad \text{Equation 1}$$

Where PCV is the performance coefficient of variation. The target standard deviation (σ) is used in the calculation of z-scores and provides scaling for laboratory deviation from the assigned value. It is important to note that the target standard deviation for this study is a

fixed value established by the study coordinator and is not the standard deviation of participants' results. The fixed value set for the target standard deviation is based on the existing regulation, the acceptance criteria indicated by the methods, the matrix, the concentration level of analyte and on experience from previous studies. By setting a fixed and realistic value for the performance standard deviation, the participant's performance (z-score) does not depend on other participants' performance and can be compared from study to study and against achievable performance. This provides a benchmark for progressive improvement.

5.5 z-Score

An example of z-score calculation using data from the present study is given in Appendix 2. For each participant's result a z-score is calculated according to Equation 2 below:

$$z = \frac{(\chi - X)}{\sigma} \qquad \text{Equation 2}$$

where:

z is z-score
χ is participants' result
X is the study assigned value
σ is the target standard deviation from Equation 1

A z-score with absolute value (|z|):

- $|z| \leq 2$ is satisfactory;
- $2 < |z| < 3$ is questionable;
- $|z| \geq 3$ is unsatisfactory.

5.6 En-Score

An example of E_n-score calculation using data from the present study is given in Appendix 2. The E_n-score is complementary to the z-score in assessment of laboratory performance. E_n-score includes measurement uncertainty and is calculated according to Equation 3 below:

$$E_n = \frac{(\chi - X)}{\sqrt{U_\chi^2 + U_X^2}} \qquad \text{Equation 3}$$

where:

E_n is E_n-score
χ is a participants' result
X is the assigned value
U_χ is the expanded uncertainty of the participant's result
U_X is the expanded uncertainty of the assigned value

An E_n-score with absolute value ($|E_n|$):

- $|E_n| \leq 1$ is satisfactory;
- $|E_n| > 1$ is unsatisfactory.

5.7 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2017[8] must establish and demonstrate the traceability and measurement uncertainty associated with their test results. Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.[9]

6 TABLES AND FIGURES

Table 3

Sample Details

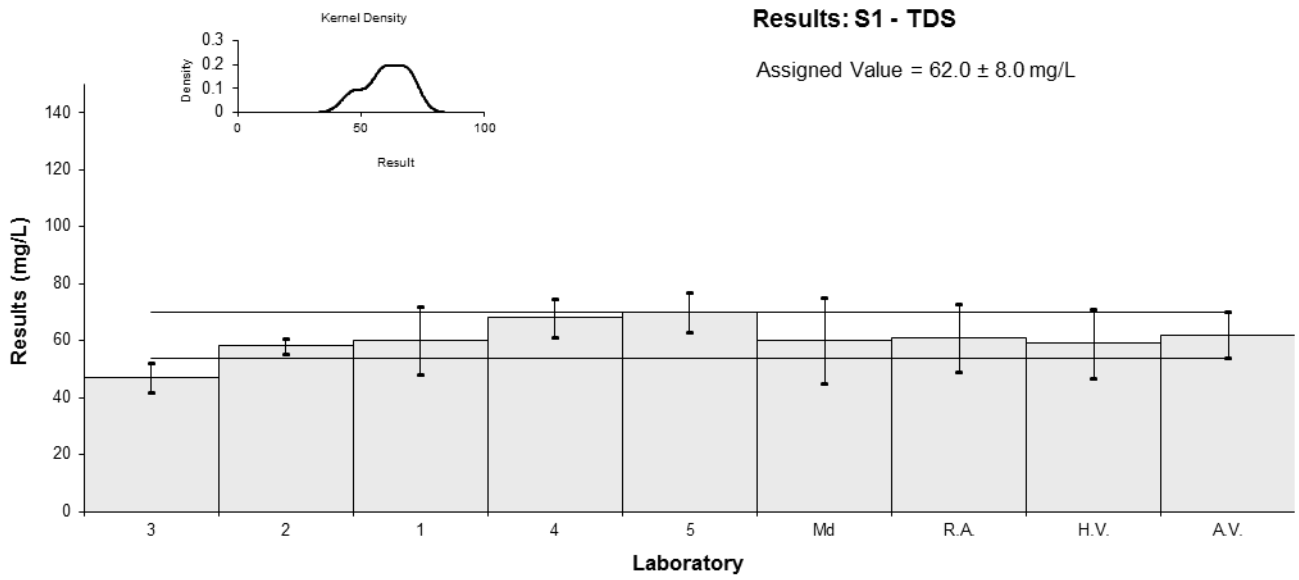
Sample No.	S1
Matrix.	Water
Analyte.	TDS
Units	mg/L

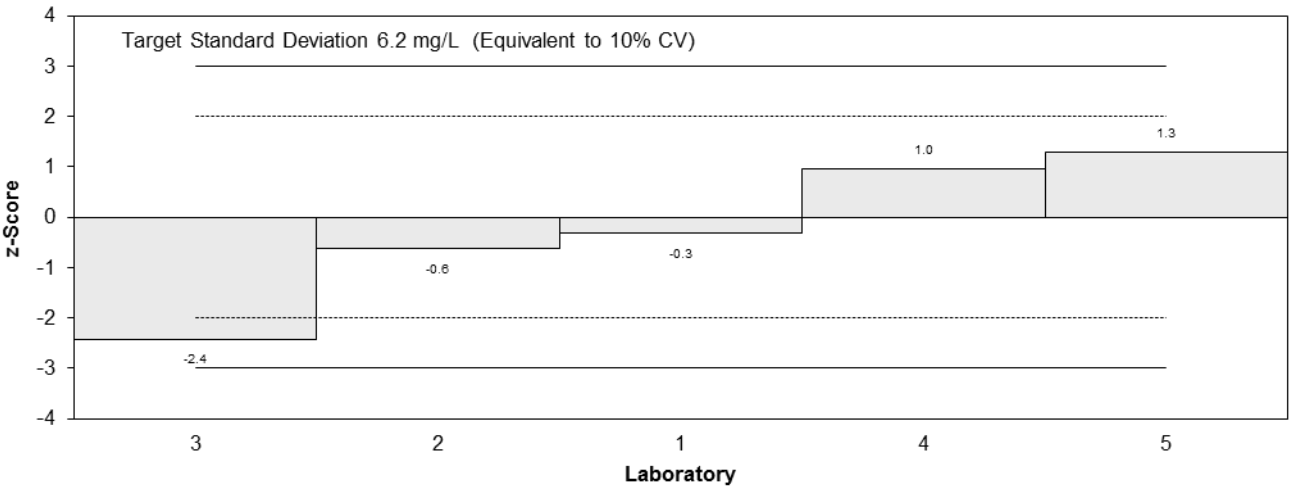
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	60	12	-0.32	-0.14
2	58.2	2.7	-0.61	-0.45
3	47	5	-2.42	-1.59
4	68	6.8	0.97	0.57
5	70	7.0	1.29	0.75

Statistics

Assigned Value	62.0	8.0
Spike	62.0	8.0
Homogeneity Value	59	12
Robust Average	61	12
Median	60	15
Mean	61	
N	5	
Max.	70	
Min.	47	
Robust SD	10	
Robust CV	17%	


Kernel Density
Density
Result
Results: S1 - TDS
Assigned Value = 62.0 ± 8.0 mg/L
Results (mg/L)
Laboratory
3
2
1
4
5
Md
R.A.
H.V.
A.V.


z-Scores: S1 - TDS
Target Standard Deviation 6.2 mg/L (Equivalent to 10% CV)
z-Score
Laboratory
1.3
1.0
-0.3
-0.6
-2.4

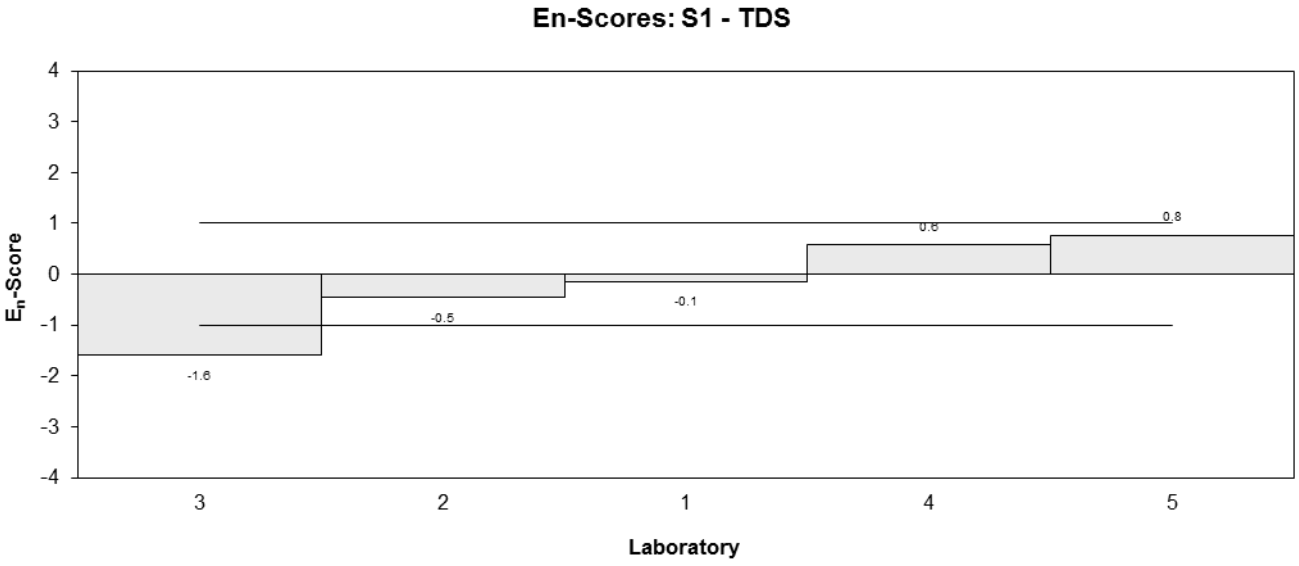

En-Scores: S1 - TDS
En-Score
Laboratory
0.8
0.6
-0.1
-0.5
-1.6

Figure 2

Table 4

Sample Details

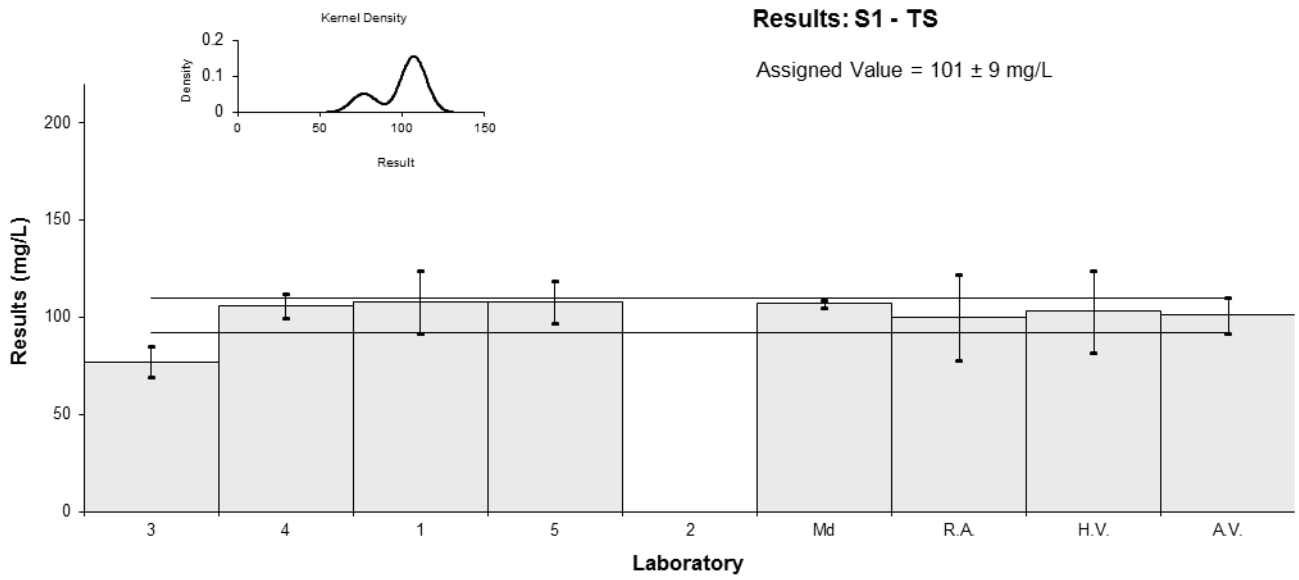
Sample No.	S1
Matrix.	Water
Analyte.	TS
Units	mg/L

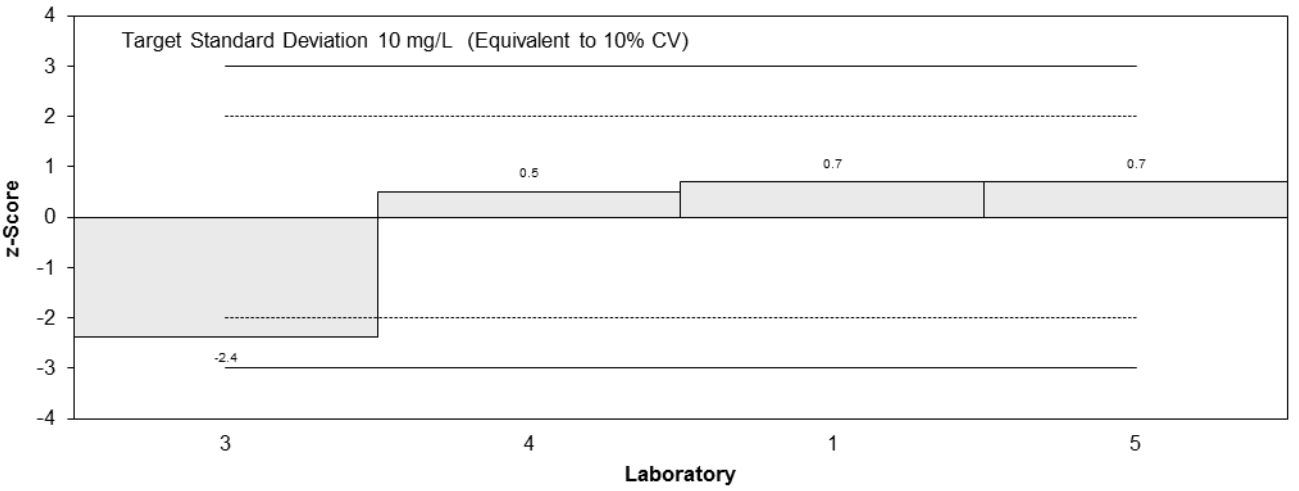
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	108	16	0.69	0.38
2	NT	NT		
3	77	8	-2.38	-1.99
4	106	6.4	0.50	0.45
5	108	10.8	0.69	0.50

Statistics

Assigned Value	101	9
Spike	101	9
Homogeneity Value	103	21
Robust Average	100	22
Median	107	2
Mean	99.8	
N	4	
Max.	108	
Min.	77	
Robust SD	17	
Robust CV	17%	


Kernel Density
Density
Result
Results: S1 - TS
Assigned Value = 101 ± 9 mg/L
Results (mg/L)
Laboratory
3
4
1
5
2
Md
R.A.
H.V.
A.V.


z-Scores: S1 - TS
Target Standard Deviation 10 mg/L (Equivalent to 10% CV)
z-Score
0.5
0.7
0.7
-2.4
Laboratory
3
4
1
5

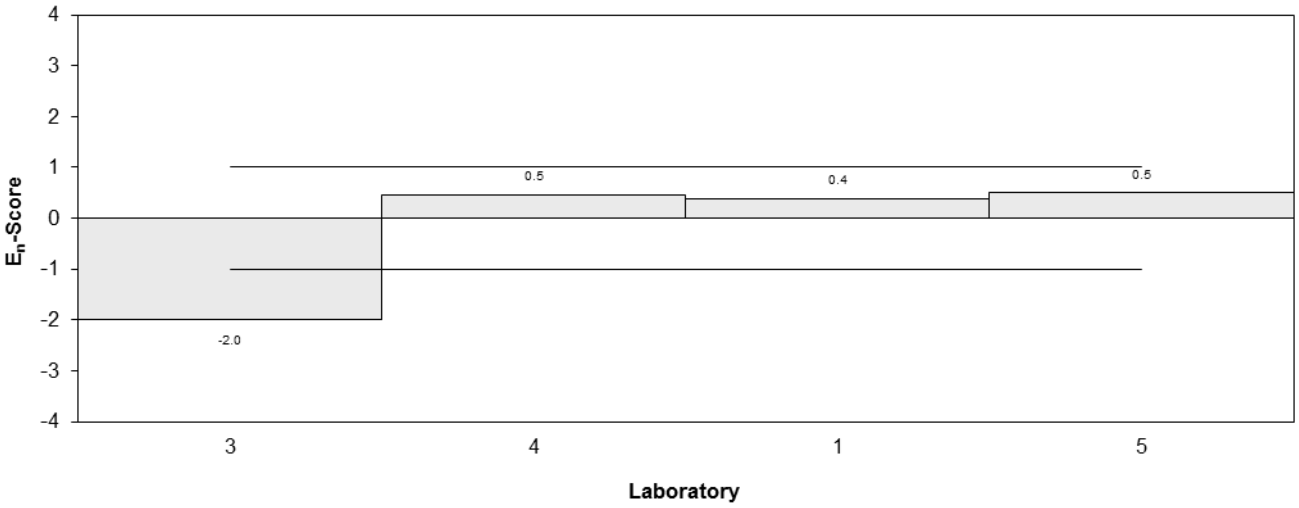

En-Scores: S1 - TS
En-Score
0.5
0.4
0.5
-2.0
Laboratory
3
4
1
5

Figure 3

Table 5

Sample Details

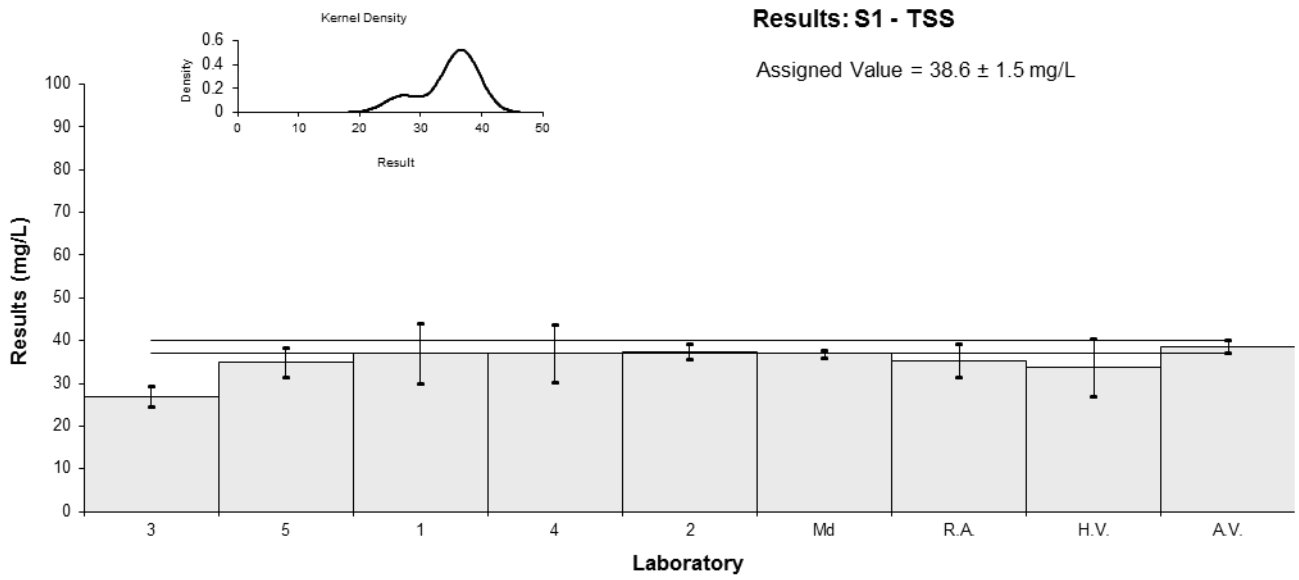
Sample No.	S1
Matrix.	Water
Analyte.	TSS
Units	mg/L

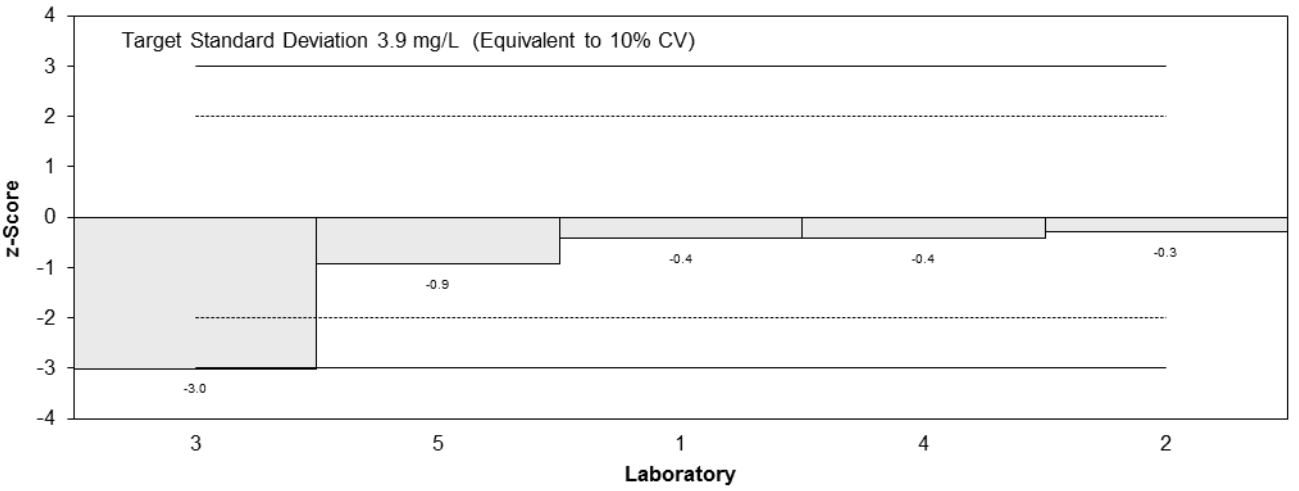
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	37	7	-0.41	-0.22
2	37.5	1.8	-0.28	-0.47
3	27	2.5	-3.01	-3.98
4	37	6.7	-0.41	-0.23
5	35	3.5	-0.93	-0.95

Statistics

Assigned Value	38.6	1.5
Spike	38.6	1.5
Homogeneity Value	33.7	6.7
Robust Average	35.3	3.9
Median	37.0	0.9
Mean	34.7	
N	5	
Max.	37.5	
Min.	27	
Robust SD	3.5	
Robust CV	9.8%	


Kernel Density
Density
Result
Results: S1 - TSS
Assigned Value = 38.6 ± 1.5 mg/L
Results (mg/L)
Laboratory
3
5
1
4
2
Md
R.A.
H.V.
A.V.


z-Scores: S1 - TSS
Target Standard Deviation 3.9 mg/L (Equivalent to 10% CV)
z-Score
-3.0
-0.9
-0.4
-0.4
-0.3
Laboratory
3
5
1
4
2

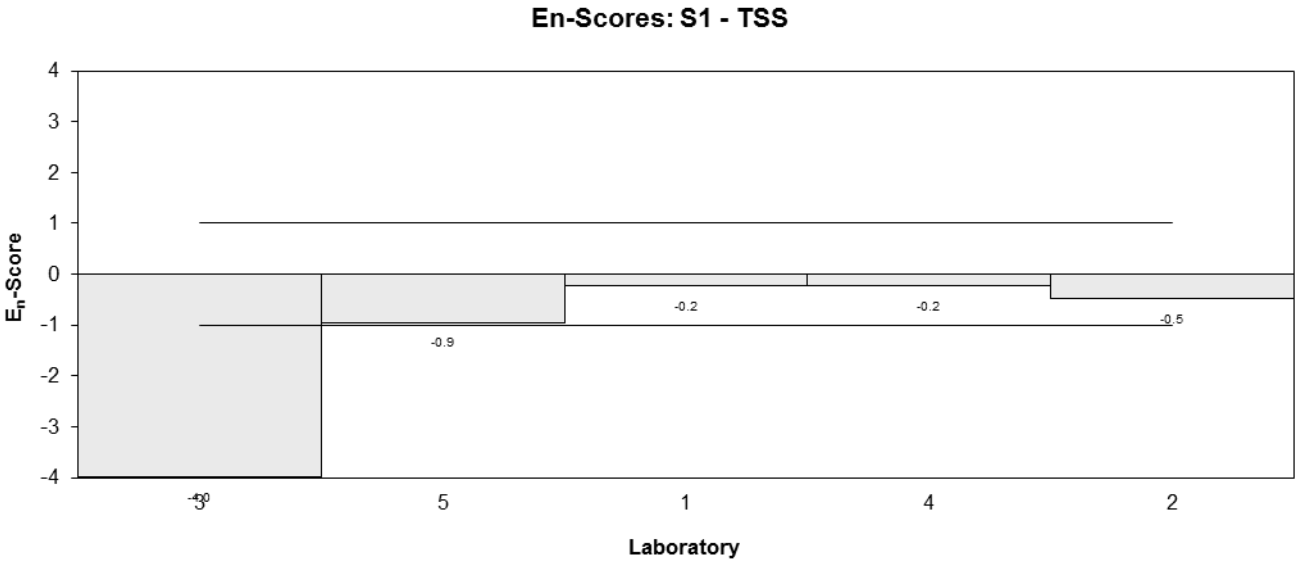

En-Scores: S1 - TSS
En-Score
-0.9
-0.2
-0.2
-0.5
Laboratory
5
1
4
2

Figure 4

7 DISCUSSION OF RESULTS

7.1 Assigned Value

Sample S1 was ultrapure water to which a known amount of glass fibre and potassium chloride was added.

Assigned Values were the formulation values. When samples are prepared by spiking an analyte-free matrix with a solution of a pure chemical, the formulated concentration may be used as the assigned value. The associated expanded uncertainties were calculated using the procedure described in ISO 13528:2015, Statistical methods for use in proficiency testing by interlaboratory comparisons.[7] Participants' results were also in good agreement, providing further support for the assigned values.

Traceability The assigned values for TDS, TSS and TS rely on gravimetric sample preparation. Gravimetric measurements were calibrated using Australian standards for mass and are traceable to the SI unit for mass (kg).

7.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded measurement uncertainty associated with their results. All results were reported with an expanded measurement uncertainty. The participants' bases of their uncertainty estimates are presented in Table 2. Most participants correctly covered all sources of errors in their estimates of measurement uncertainty.

Proficiency tests allow a check of participants' uncertainty estimates. Results and the expanded MU are presented in the bar charts for each analyte (Figures 2 to 4).

In some cases the results were reported with an inappropriate number of significant figures. The recommended format is to write uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places. For example, instead of 108 ± 10.8 mg/L, it is better to report 108 ± 11 mg/L.[10]

7.3 E_n-score

The E_n-score indicates how closely a result agrees with the assigned value taking into account the respective uncertainties. The dispersal of participants' E_n-scores is graphically presented in Figure 5. Of 14 results for which E_n-scores were calculated, 11 returned a satisfactory score of $|E_n| \leq 1$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.

7.4 z-Score

The target standard deviation defines satisfactory performance in a proficiency test.

Target standard deviations equivalent to 10% were used to calculate the z-scores. The fixed value set for the target standard deviation is based on the existing regulation, the acceptance criteria indicated by the methods and on experience from previous studies. Unlike the standard deviation based on between laboratories CV, by setting a fixed and realistic value for the performance standard deviation, the participant's performance (z-score) does not depend on other participants' performance.

The dispersal of participants' z-scores is presented in Figure 6 (by laboratory code) and in Figure 7 (by test).

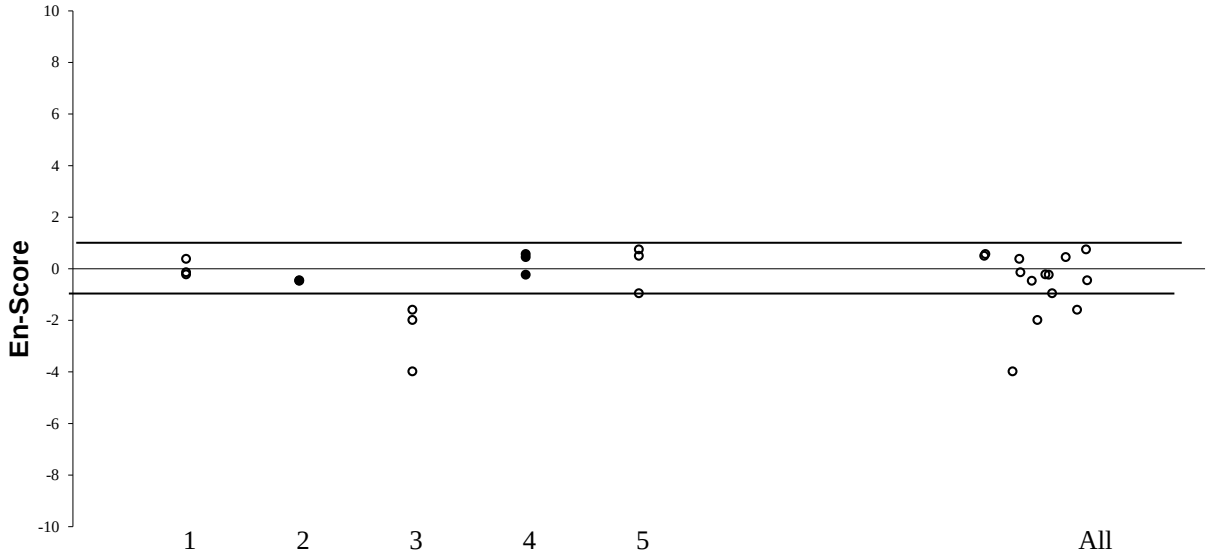


Figure 5 E_n-Score Dispersal by Laboratory

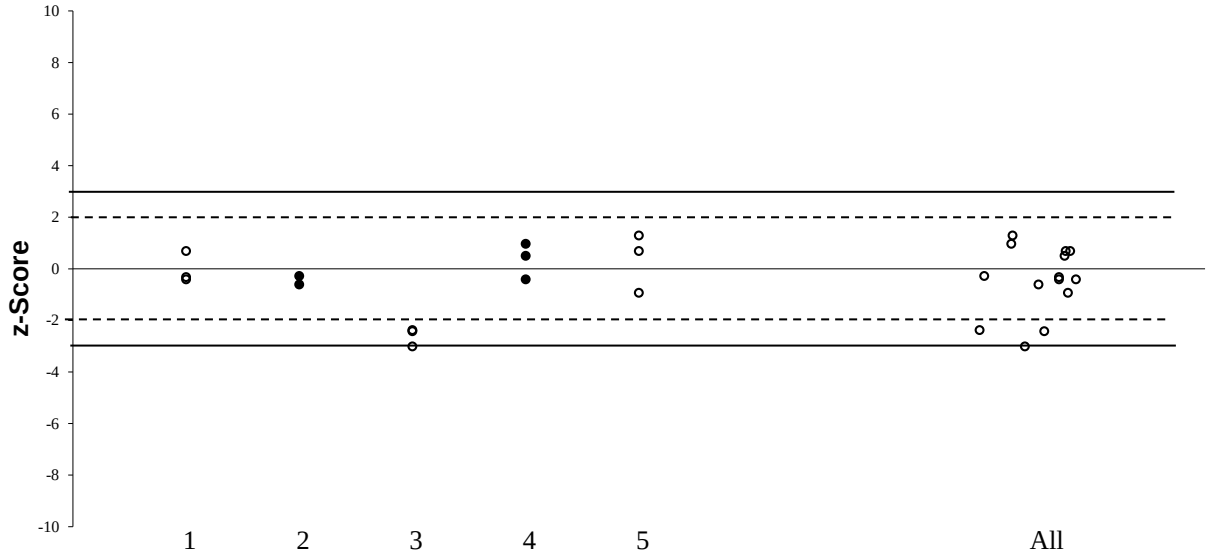


Figure 6 z-Score Dispersal by Laboratory

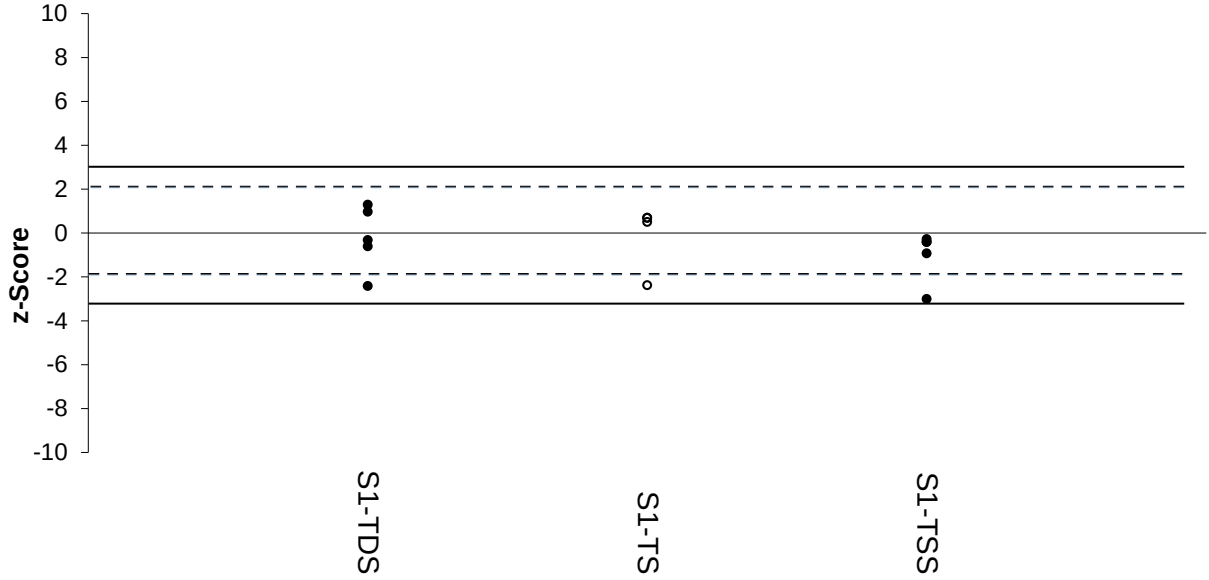


Figure 7 z-Score Dispersal by Test

The method description provided by participants is presented in Table 1. All participants used APHA Method 2540.

TSS was the test with the smallest between laboratories coefficient of variation, 9.8%.

All results reported by Laboratory 3 were lower than the assigned value by almost the same factor 0.75. This is an indication of method or laboratory bias.

7.5 Comparison with Previous NMI Proficiency Tests of Metals in Soil

AQA 19-06 is the third NMI proficiency test of solids in water. The same fixed target standard deviation was used in the present study as in the previous studies of solids in water. This allowed a comparison of participants' performance (z-score) over time and provided a benchmark for progressive improvement. Participants' performance in measurement of solids in water over time is presented in Figure 8.

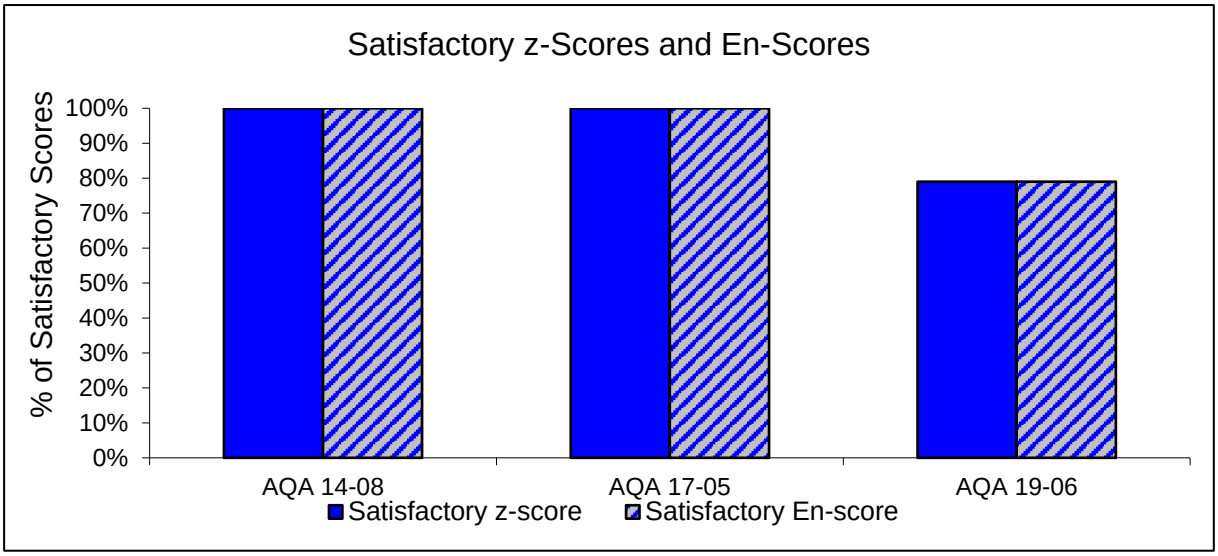


Figure 8 Participants' Performance in Solids in Water PT Studies

Individual performance history reports are emailed to each participant at the end of the study; the consideration of z-scores for an analyte over time provides much more useful information than a single z-score. Over time, laboratories should expect at least 95% of their scores to lie within the range $|z| \leq 2$. Scores in the range $2 < |z| < 3$ can occasionally occur, however these should be interpreted in conjunction with the other scores obtained by that laboratory. For example, a trend of z-scores on one side of the zero line is an indication of method or laboratory bias.

7.6 Reference Materials and Certified Reference Materials

Participants reported whether control samples (spiked samples, certified reference materials-CRMs or matrix specific reference materials-RMs) had been used (Table 6).

Table 6 Control Samples Used by Participants

Lab. Code	Description of Control Samples
2	Control Samples
3	Control Samples-Reference Material
4	Control Samples

When a CRM is not available, matrix matched control samples taken through all steps of the analytical process, are the most valuable quality control tools for assessing a method's performance.

8 REFERENCES

[1] ISO 17043:2010, Conformity assessment – *General requirements for proficiency testing*.

[2] NMI 2016, *NMI Chemical Proficiency Testing Study Protocol*, viewed 22 March 2017, <http://www.measurement.gov.au>.

[3] NMI 2016, *NMI Chemical Proficiency Testing Statistical Manual*, viewed 22 March 2017, <http://www.measurement.gov.au>.

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APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

A1.1 Sample Preparation

Sample S1 was ultrapure water. To 10015 g of ultra-high purity water has been added 0.386 g of glass fibre filter disintegrated and dispersed and 0.62 g of 99.95% pure potassium chloride.

A1.2 Sample Analysis and Homogeneity Testing

A partial homogeneity test was conducted for TDS, TS and TSS in Samples S1.[5] Three bottles were analysed in duplicate and the average of the results was reported as the homogeneity value.

Sample Analysis for Solids in Water

A well-mixed sample was filtered through a pre-weighed glass fibre filter; the residue retained on the filter was dried at 104°C weighed and reported as TSS. The filtrate was collected in a pre-weighed container then dried at 180ºC weighed and reported as TDS.

For TS measurements the unfiltered sample was dried at 104ºC into a pre-weighed container. After drying the residue was weighed and reported as TS.

APPENDIX 2 - Z-SCORE AND E_N SCORE CALCULATION

z-Score and E_n-score

For each participant's result the z-score and E_n-score are calculated according to Equation 2 and Equation 3 respectively (see page 9).

A worked example is set out below in Table 7.

Table 7 z-Score and E_n-score for TS result reported by Laboratory 1 in S1

TS Result mg/L	Assigned Value mg/L	Set Target Standard Deviation	z-Score	E_n-Score
108±16	101±9	10% as CV or 0.10x101= =10.1 mg/L	$z = \frac{(108 - 101)}{10.1}$ z = 0.69	$En = \frac{(108 - 101)}{\sqrt{16^2 + 9^2}}$ E_n=0.38

APPENDIX 3 - ACRONYMS AND ABBREVIATIONS

APHA	American Public Health Association
A.V.	Assigned Value
CRM	Certified Reference Material
CV	Coefficient of Variation
H.V.	Homogeneity Value
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
NMI	National Measurement Institute (of Australia)
NR	Not Reported
NT	Not Tested
PT	Proficiency Test
PCV	Performance Coefficient of Variation
RA	Robust Average
RM	Reference Material
Robust CV	Robust Coefficient of Variation
Robust SD	Robust Standard Deviation
S	Spiked or formulated concentration of a PT sample
SS	Spiked sample
SI	The International System of Units
s^2_{sam}	Sampling variance
s_a/σ	Analytical standard deviation divided by the target standard deviation
SFA	Segment Flow Analyser
SRM	Standard Reference Material (Trademark of NIST)
TDS	Total dissolved solid
TS	Total solids
TSS	Total suspended solid
Target SD	Target standard deviation
σ	Target standard deviation