Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

Proficiency Test Final Report AQA 21-21 Methamphetamine in Wipes

July 2021

ACKNOWLEDGMENTS

This study was conducted by the National Measurement Institute (NMI). Support funding was provided by the Australian Government Department of Industry, Science, Energy and Resources.

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.

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Accredited for compliance with ISO/IEC 17043

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SUMMARY

AQA 21-21 Methamphetamine in Wipes commenced in February 2021. Thirteen laboratories enrolled to participate, and eleven participants submitted results.

Three test samples were prepared by spiking wipes with varying amounts of methamphetamine.

The assigned values for all samples were the robust averages of participants' results. The associated uncertainties were estimated from the robust standard deviations of the participants' results.

Traceability: The consensus of participants' results is not traceable to any external reference, so although expressed in SI units, metrological traceability has not been established.

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

- *Assess the proficiency of laboratories measuring methamphetamine in wipes;*

Laboratory performance was assessed by both z-score and E_n-score.

Of 33 reported results, all returned a z-score of $|z| \leq 2.0$, indicating satisfactory performance.

Of 33 reported results, 27 (82%) returned a satisfactory E_n-score of $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratories **2**, **3**, **4**, **6**, **7**, **8**, **9** and **13** returned satisfactory z-scores and E_n-scores for all samples.

- *Evaluate the participants' methods for the measurement of methamphetamine in wipes;*

Participants used various methods for the measurement of methamphetamine in wipes and all produced compatible results.

- *Compare the performance of participants with their past performance;*

NMI has run 3 proficiency test studies for controlled substances in wipes. Over these studies, performance has remained very high, with the average proportion of satisfactory z-scores and E_n-scores being 98% and 80% respectively.

- *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 associated measurement uncertainty. Participants used a wide variety of procedures to estimate their uncertainty.

The magnitude of reported uncertainties was within the range of 1.0% to 3617% relative.

- *Produce materials that can be used in method validation and as control samples.*

The test samples produced for this study are homogeneous and are well characterised. Surplus of these samples is available for purchase and can be used for quality control and for method validation purposes.

1 INTRODUCTION

1.1 NMI Proficiency Testing Program

The National Measurement Institute (NMI) is responsible for Australia's national measurement infrastructure, providing a 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, law enforcement and food safety. NMI offers studies in:

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

1.2 Study Background

Clandestine laboratories ('clan labs') are places where illegal drugs have been manufactured. During the drug manufacturing process, toxic gases and aerosols are produced, which may be absorbed by the surroundings and may remain for many years. Field test kits are used to check the extent of contamination in the premises, and samples may be taken from non-porous surfaces using wipes. This PT scheme was developed to enable laboratories to assess their ability to measure methamphetamine in wipes at levels specified in the Clandestine Drug Laboratory Remediation Guidelines 2011.[2]

1.3 Study Aims

The aims of the study were to:

- assess the proficiency of laboratories measuring methamphetamine in wipes;
- evaluate the participants' methods for the measurement of methamphetamine in wipes;
- compare the performance of participants with their past performance;
- develop the practical application of traceability and measurement uncertainty, and provide participants with information that will be useful in assessing their uncertainty estimates; and
- produce materials that can be used in method validation and as control samples.

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

1.4 Study Conduct

The conduct of NMI PTs is described in the NMI Study Protocol for Proficiency Testing.[3] The statistical methods used are described in the NMI Chemical Proficiency Testing Statistical Manual.[4] These documents have been prepared with reference to ISO/IEC 17043 and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[1,5] NMI is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of PT schemes,[1] and this study is within the scope of NMI's accreditation.

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitation issued	22 February 2021
Samples dispatched	17 May 2021
Results due	15 June 2021
Interim report issued	16 June 2021

2.2 Participation and Laboratory Code

Thirteen laboratories enrolled to participate in this study. Each participant was randomly assigned a confidential laboratory code. Eleven participants submitted results.

2.3 Sample Preparation and Test Material Specification

Three test samples were prepared, each containing one wipe. Methamphetamine spiking solution was prepared by dissolving a known mass of methamphetamine hydrochloride (approximately 78.5% base (m/m) supplied by NMI Chemical Reference Materials) in methanol. Large Liv-Wipe alcohol wipes bought from a local supplier were removed from their packaging using tweezers and unfolded. The methamphetamine solution was spiked onto the wipes using calibrated positive displacement pipettes. After spiking, the methanol solvent was allowed to evaporate and the wipes were placed in amber glass jars, labelled, shrink-wrapped, and stored in a refrigerator before sample dispatch.

Sample S1 was prepared to contain 1.61 µg methamphetamine base/wipe.

Sample S2 was prepared to contain 0.402 µg methamphetamine base/wipe.

Sample S3 was prepared to contain 3.16 µg methamphetamine base/wipe.

2.4 Homogeneity of Samples

No homogeneity testing was conducted for this study. The same sample preparation procedure was followed as for previously conducted studies. The samples from previous studies were demonstrated to be sufficiently homogeneous for the evaluation of participants' performance. The results reported in this study gave no reason to question the samples' homogeneity.

2.5 Stability of Analytes

No assessment of the stability of the test samples was made before the samples were sent. To assess possible instability, the results returned by participants were considered. The results of this study gave no reason to question the stability of the test samples, with robust averages of participants' results being 99% to 113% of the spiked values. There was also no correlation observed between the reported results and days the samples spent in transit (see Appendix 1).

2.6 Sample Storage, Dispatch and Receipt

The test samples were stored at 4°C prior to dispatch. Samples were packed with cooler brick(s) and sent by courier on 17 May 2021. The following items were packaged with the samples:

- a letter which included a description of the test samples and instructions for participants; and
- a form for participants to return to confirm the receipt and condition of the samples.

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

2.7 Instructions to Participants

Participants were instructed as follows:

- If analyses cannot be commenced on the day of receipt, please store the samples refrigerated.
- Quantitatively analyse each wipe for the amount of methamphetamine using your routine test method.
- For each of Samples S1, S2 and S3, report a single result in units of µg methamphetamine as base/wipe expressed as if reporting to a client (i.e. corrected for recovery or not, according to your standard procedure). This figure will be used in all statistical analysis in the study report.
- For each result also report an estimate of your expanded uncertainty as µg methamphetamine as base/wipe, if determined.
- No limit of reporting has been set for this study. Report results as you would report to a client, applying the limit of reporting of the method used for analysis.
- Report any sample not tested as NT.
- Give brief details of your methodology and basis of uncertainty estimate as requested by the results sheet emailed to you.
- Please return your completed results sheet by 14 June 2021 by email to proficiency@measurement.gov.au.

The results due date was extended for all participants to 15 June 2021 due to a public holiday on the original due date.

2.8 Interim Report

An interim report was emailed to all participants on 16 June 2021.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Participants' Test Methods

Participants were requested to provide information about their test methods. Responses received are presented in Table 1. Some responses may be modified so that the participant cannot be identified.

Table 1 Summary of Participants' Test Methods

Lab. Code	Desorption Solution	Sample Treatment	Filtration	Technique	Detector	Method Reference
1	0.1M sulfuric acid	1 hr on rotary mixer	0.20 um syringe filter	LC	QQQ	In house, adapted from NIOSH 9111
2	0.1M sulfuric acid	30 mins on tumbler, pH adjustment	0.2 µm filter	HPLC	MS/MS	In house
3	0.1M sulfuric acid	30min on orbital shaker	Nil	UPLC	MS/MS	NIOSH 9111
4	0.1M Sulphuric acid	1 hour shaking	N/A	LCMS	MS/MS	NIOSH9111
5	0.1 M sulfuric acid	1 hr	Spun Down	HPLC	MS/MS	NIOSH 9111
6	0.1 M sulfuric acid	1 hr on rotary mixer	0.45 um PTFE filter	UPLC	MS/MS	in-house
7	0.1M sulfuric acid	30 mins on tumbler, pH adjustment	0.2 µm filter	HPLC	MS/MS	In-House method referencing NIOSH 9111
8	0.1 M sulfuric acid in UHP water	Samples shaken,1hr tumbled end over end and 20 min sonication	0.2 µm filter	HPLC	MS/MS	NIOSH 9111
9	0.1M Sulfuric Acid	1 hour on rotary mixer	No filtration	HPLC	MS	NIOSH 9111
11	0.1 M sulfuric acid	1 hr on rotary mixer	Centrifugation	HPLC	MS/MS	In-House
13	0.1M sulfuric acid	1 hr on rotary mixer	Agilent PES 0.45 um, 25 mm	HPLC	MS	Based on Niosh 9111

3.2 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about their basis of measurement uncertainty (MU). Responses received are presented in Table 2. Some responses may be modified so that the participant cannot be identified.

Table 2 Reported Basis of Uncertainty Estimates

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
1	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS Duplicate analysis	Instrument calibration Standard purity	NATA Technical Note 33
2	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM	Instrument calibration CRM	ISO/GUM

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
3	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Instrument calibration CRM Recoveries of SS Standard purity	Eurachem/CITAC Guide
4	Standard deviation of replicate analyses multiplied by 2 or 3	Standard deviation from PT studies only		
5	Plus Minus 10%	Duplicate analysis	CRM	NPAAC Requirements for the Estimation of Measurements of Uncertainty
6	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	CRM Recoveries of SS	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
7	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis	Instrument calibration CRM Recoveries of SS	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
8	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	Recoveries of SS	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
9	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Control samples - CRM Duplicate analysis	Instrument calibration CRM Standard purity	NMI Uncertainty Course
11	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Instrument calibration CRM Recoveries of SS Standard purity	NMI Uncertainty Course
13	NIOSH Method Accuracy Range (A)	Control samples - SS Duplicate analysis	Instrument calibration CRM Recoveries of SS Standard purity	NIOSH Manual of Analytical Methods 3/15/03 Page 208 Part P. Measurement Uncertainty and NIOSH Method Accuracy Range

* CRM = Certified Reference Material, RM = Reference Material, SS = Spiked Samples

3.3 Details of Participants' Standards

Participants were requested to provide information about their standards used. Responses received are presented in Table 3. Some responses may be modified so that that participant cannot be identified.

Table 3 Participants' Standards

Lab. Code	Calibration Standard		Internal Standard	
	Origin	Purity (%)	Origin	Standard
1	NMI	99.8	CERILLIANT	Methamphetamine-D14
2	PM Separations	98.5	PM Separations	d l-Methamphetamine-D5

Lab. Code	Calibration Standard		Internal Standard	
	Origin	Purity (%)	Origin	Standard
3	Lipomed d,l-Methamphetamine.HCI	1mg/mL calibrated in methanol 1mL	Lipomed d,l-Methamphetamine-D14.HCl	Methamphetamine-D14
4	NMI	99.8	LGC Standards	rac-Methamphetamine-D9
5	Lipomed	99		Methamphetamine-D5
6	Supelco	1000mg/l	Cerilliant	Methamphetamine-D14
7	PM Separations	98.5	PM Separations	d l-Methamphetamine-D5
8	Lipomed	>98.5	PM Separations	(+/-)-Methamphetamine-D5
9	Lipomed	99.6	Lipomed	d,l-methylamphetamine-d_5
11	Lipomed	>99%	Lipomed	Methamphetamine-D5
13	Cerilliant M-009	1mg/mL	Cerilliant M-093, 1mg/mL	Methamphetamine-D14

3.4 Participants' Comments

The study coordinator welcomes comments or suggestions from participants as it provides information which can help improve future studies. Responses received are presented in Table 4, along with the study coordinator's response where appropriate.

Table 4 Participants' Comments

Lab. Code	Participant Comments	Study Coordinator's Response
1	(All samples) Methylamphetamine was detected above the limits specified in the guidelines and does require remediation. Methodology: Method validation has found recovery to be close to 100%. Hence, no recovery correction was applied. Comparison to the internal standard that is spiked onto each swab is used to monitor consistent recoveries. The limit of reporting for methylamphetamine was 0.37µg/swab.	
4	Methodology: Desorption with 0.1M Sulphuric by shaking, LCMSMS analysis. (modified NIOSH9111). Note: the default detection limit is 0.02 - 0.04 ug/sample depending on the sampling technique used.	
6	Real-world samples are always provided in 50ml polypropylene vials - perhaps this is an option in future instead of jars to avoid an extra handling step.	Thank you, we will take your suggestion into consideration for future studies. Samples in this study were packaged in glass containers with PTFE lined screw caps, as noted in the Australian Government Clandestine Drug Laboratory Remediation Guidelines.[2]
8	Methodology: Corrected on instrument but not for extraction.	
13	Ideally use wipes that are commonly used for sampling - these were too small. Methodology: Blank provided was analysed with samples, no methamphetamine was detected	Thank you, we will take your suggestion into consideration for future studies.

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 5 to 7 with resultant summary statistics: robust average, median, mean, numeric results (N), maximum (Max.), minimum (Min.), robust standard deviation (Robust SD) and robust coefficient of variation (Robust CV). 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.

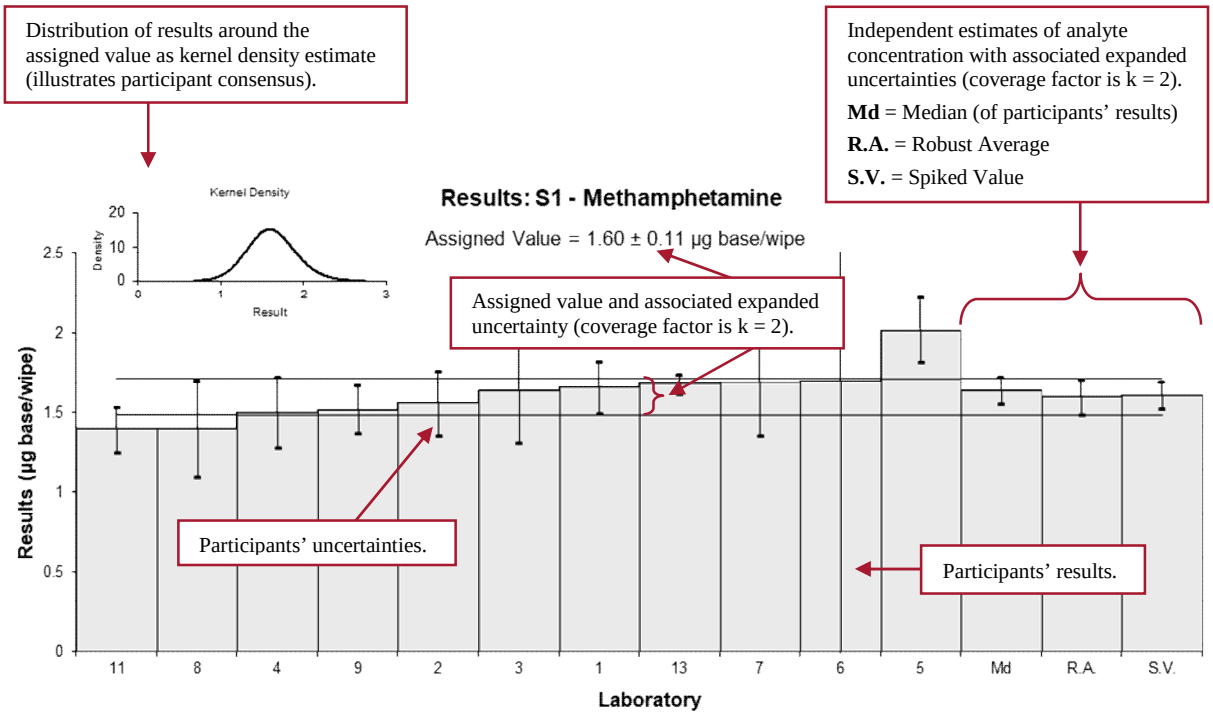


Figure 1 Guide to Presentation of Results

4.2 Assigned Value

Assigned value is defined as the: 'value attributed to a particular property of a proficiency test item'.[1] In this study, the property is the amount of methamphetamine per wipe in each sample. Assigned values were the robust averages of participants' results and the expanded uncertainties were estimated from the associated robust SDs (Appendix 2).

4.3 Robust Average and Robust Between Laboratories Coefficient of Variation

The robust averages and associated expanded MUs, and robust CVs (a measure of the variability of participants' results) were calculated using the procedure described in ISO 13528:2015.[6]

4.4 Performance Coefficient of Variation

The performance coefficient of variation (PCV) is a measure of the between laboratories variation that in the judgement of the study coordinator would be expected from participants, given the levels of analytes present. It is important to note that the PCV is a value set by the study coordinator; it is not calculated from the participants' results. It is based on the levels of analytes in the study and experience from previous studies. By setting a fixed and realistic value for the PCV, a participant's performance does not depend on other participants' performance and can be compared from study to study.

4.5 Target Standard Deviation

The target SD (σ) is the product of the assigned value (X) and the PCV, as presented in Equation 1. This value is used for calculation of z-scores.

$$\sigma = X \times PCV \qquad \textit{Equation 1}$$

4.6 z-Score

For each participant's result, a z-score is calculated according to Equation 2.

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

where:

z is z-score

χ is a participant's result

X is the assigned value

σ is the target standard deviation from Equation 1

For the absolute value of a z-score:

- $|z| \leq 2.0$ is satisfactory;
- $2.0 < |z| < 3.0$ is questionable;
- $|z| \geq 3.0$ is unsatisfactory.

4.7 E_n-Score

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.

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

where:

E_n is E_n-score

χ is a participant's 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

For the absolute value of an E_n-score:

- $|E_n| \leq 1.0$ is satisfactory;
- $|E_n| > 1.0$ is unsatisfactory.

4.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC 17025:2017 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.[7]

Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.[8]

5 TABLES AND FIGURES

Table 5

Sample Details

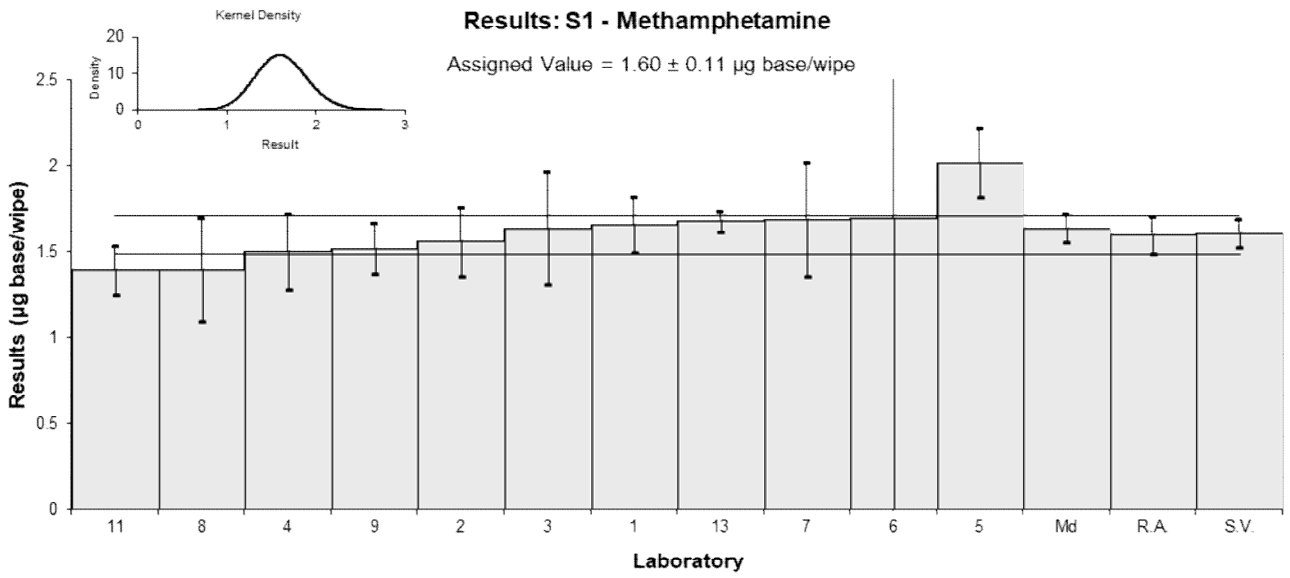
Sample No.	S1
Matrix	Wipe
Analyte	Methamphetamine
Units	µg base/wipe

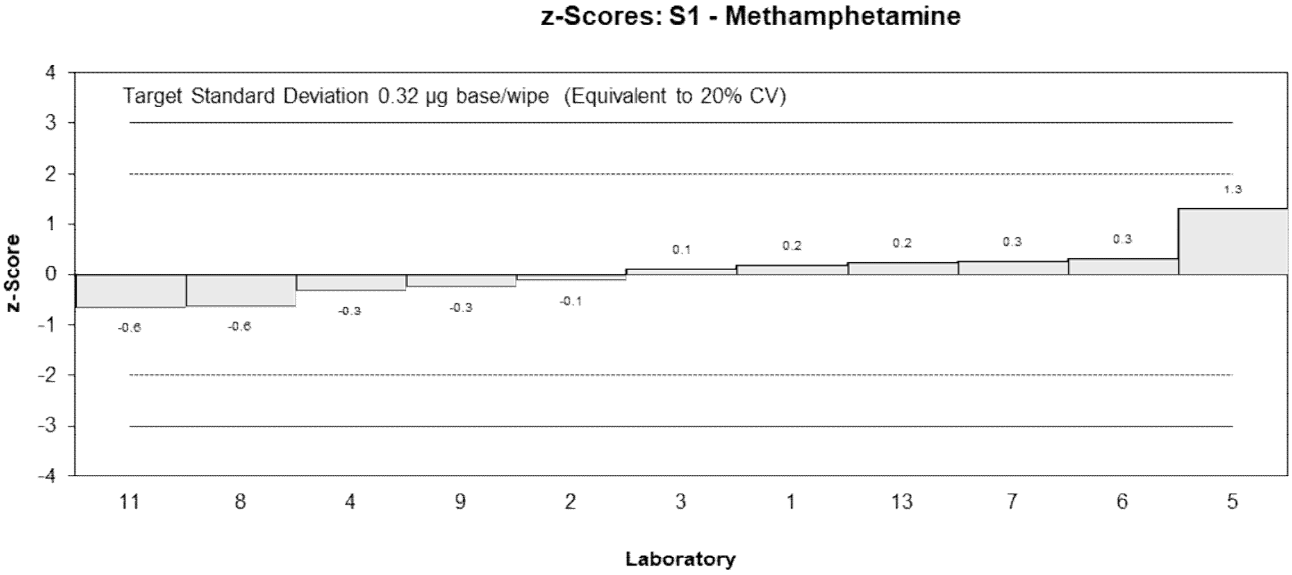
Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.66	0.16	NR	0.19	0.31
2	1.56	0.20	98	-0.13	-0.18
3	1.64	0.33	106	0.13	0.11
4	1.5	0.22	NR	-0.31	-0.41
5	2.02	0.20	NR	1.31	1.84
6	1.7	17	96	0.31	0.01
7	1.69	0.33	110	0.28	0.26
8	1.4	0.3	97	-0.63	-0.63
9	1.52	0.15	NR	-0.25	-0.43
11	1.396	0.14	NR	-0.64	-1.15
13	1.68	0.06	100.3	0.25	0.64

Statistics

Assigned Value	1.60	0.11
Spike	1.61	0.08
Robust Average	1.60	0.11
Median	1.64	0.08
Mean	1.62	
N	11	
Max.	2.02	
Min.	1.396	
Robust SD	0.15	
Robust CV	9.5%	


Kernel Density
Density
Result
Results: S1 - Methamphetamine
Assigned Value = 1.60 ± 0.11 µg base/wipe
Results (µg base/wipe)
Laboratory
11
8
4
9
2
3
1
13
7
6
5
Md
R.A.
S.V.


z-Scores: S1 - Methamphetamine
Target Standard Deviation 0.32 µg base/wipe (Equivalent to 20% CV)
z-Score
Laboratory
-0.6
-0.6
-0.3
-0.3
-0.1
0.1
0.2
0.2
0.3
0.3
1.3

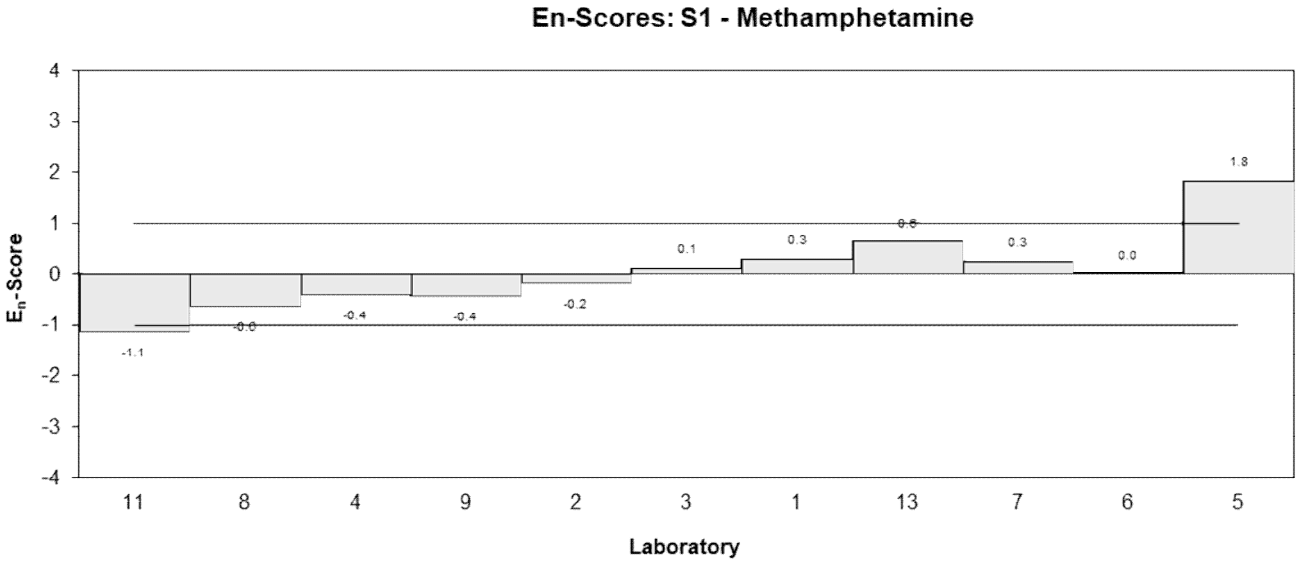

En-Scores: S1 - Methamphetamine
En-Score
Laboratory
-1.1
-0.6
-0.4
-0.4
-0.2
0.1
0.3
0.6
0.3
0.0
1.8

Figure 2

Table 6

Sample Details

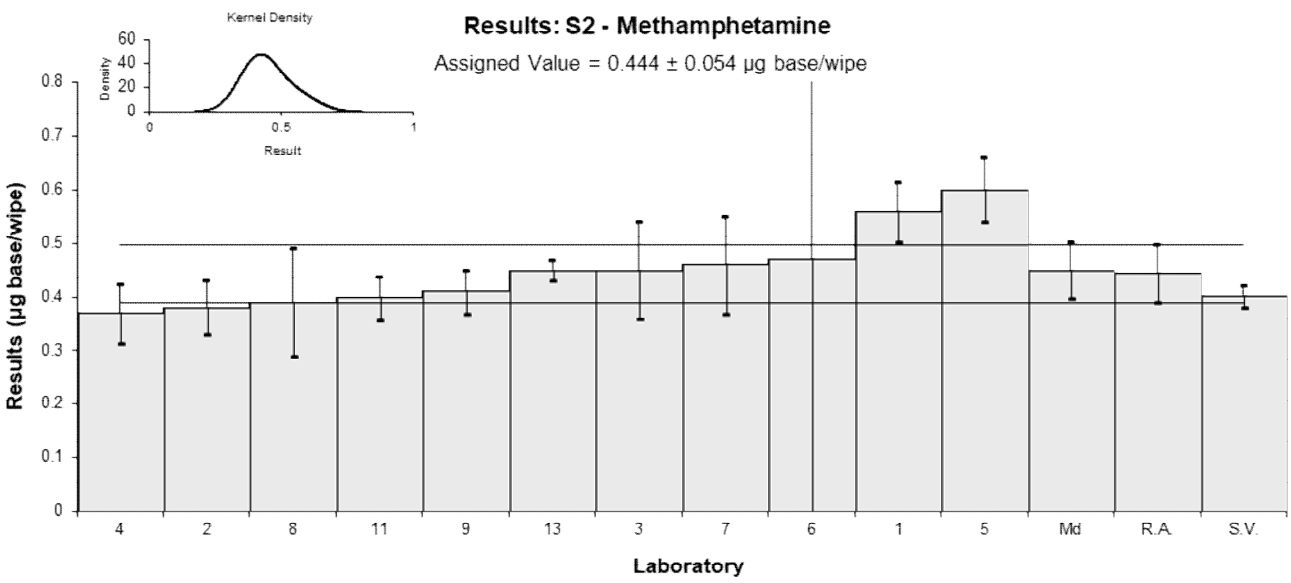
Sample No.	S2
Matrix	Wipe
Analyte	Methamphetamine
Units	µg base/wipe

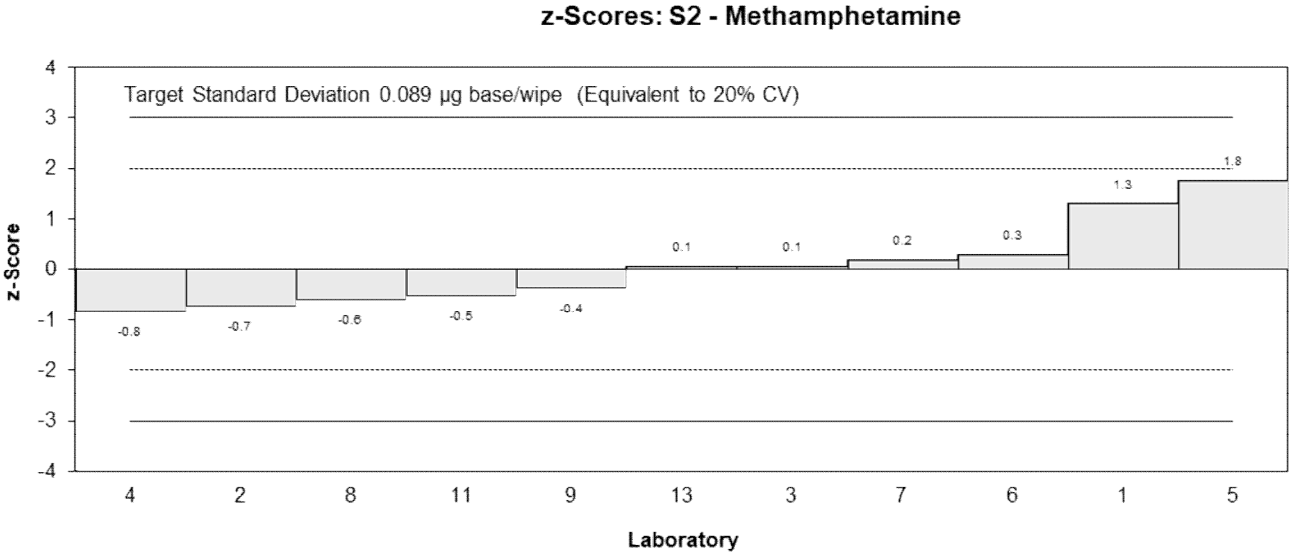
Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.56	0.056	NR	1.31	1.49
2	0.38	0.05	101	-0.72	-0.87
3	0.45	0.09	106	0.07	0.06
4	0.37	0.055	NR	-0.83	-0.96
5	0.60	0.06	NR	1.76	1.93
6	0.47	17	96	0.29	0.00
7	0.46	0.09	115	0.18	0.15
8	0.39	0.1	99	-0.61	-0.48
9	0.41	0.04	NR	-0.38	-0.51
11	0.398	0.040	NR	-0.52	-0.68
13	0.45	0.02	100.3	0.07	0.10

Statistics

Assigned Value	0.444	0.054
Spike	0.402	0.020
Robust Average	0.444	0.054
Median	0.450	0.052
Mean	0.449	
N	11	
Max.	0.6	
Min.	0.37	
Robust SD	0.071	
Robust CV	16%	


Kernel Density
Density
Result
Results: S2 - Methamphetamine
Assigned Value = 0.444 ± 0.054 µg base/wipe
Results (µg base/wipe)
Laboratory
4
2
8
11
9
13
3
7
6
1
5
Md
R.A.
S.V.


z-Scores: S2 - Methamphetamine
Target Standard Deviation 0.089 µg base/wipe (Equivalent to 20% CV)
z-Score
Laboratory
-0.8
-0.7
-0.6
-0.5
-0.4
0.1
0.1
0.2
0.3
1.3
1.8

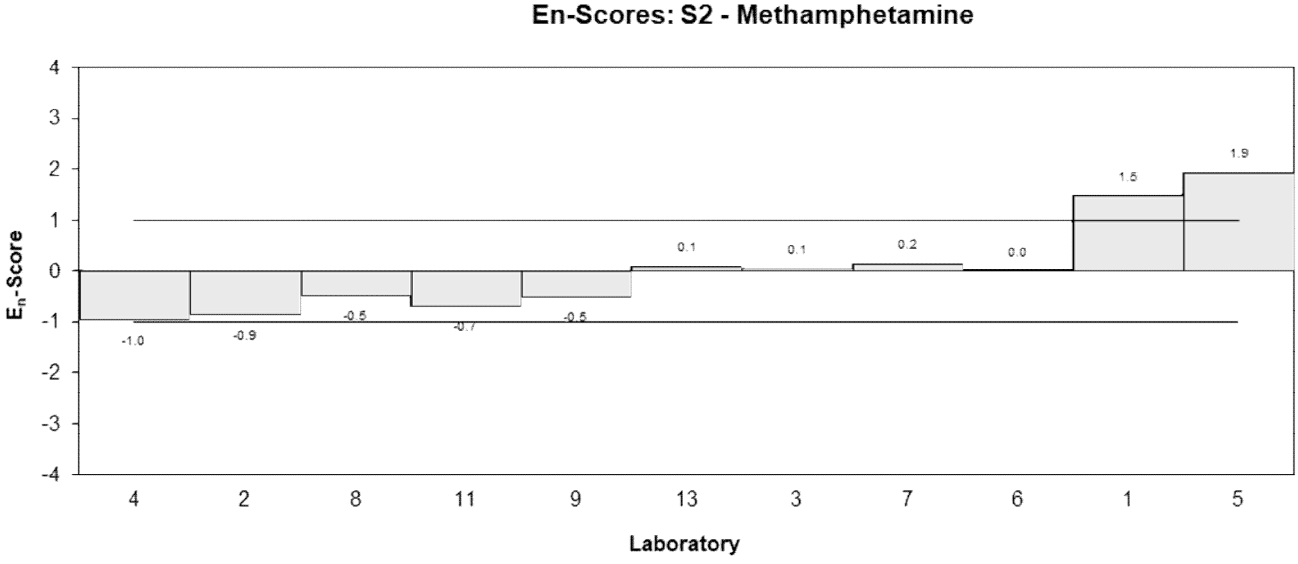

En-Scores: S2 - Methamphetamine
En-Score
Laboratory
-1.0
-0.9
-0.5
-0.7
-0.5
0.1
0.1
0.2
0.0
1.5
1.9

Figure 3

Table 7

Sample Details

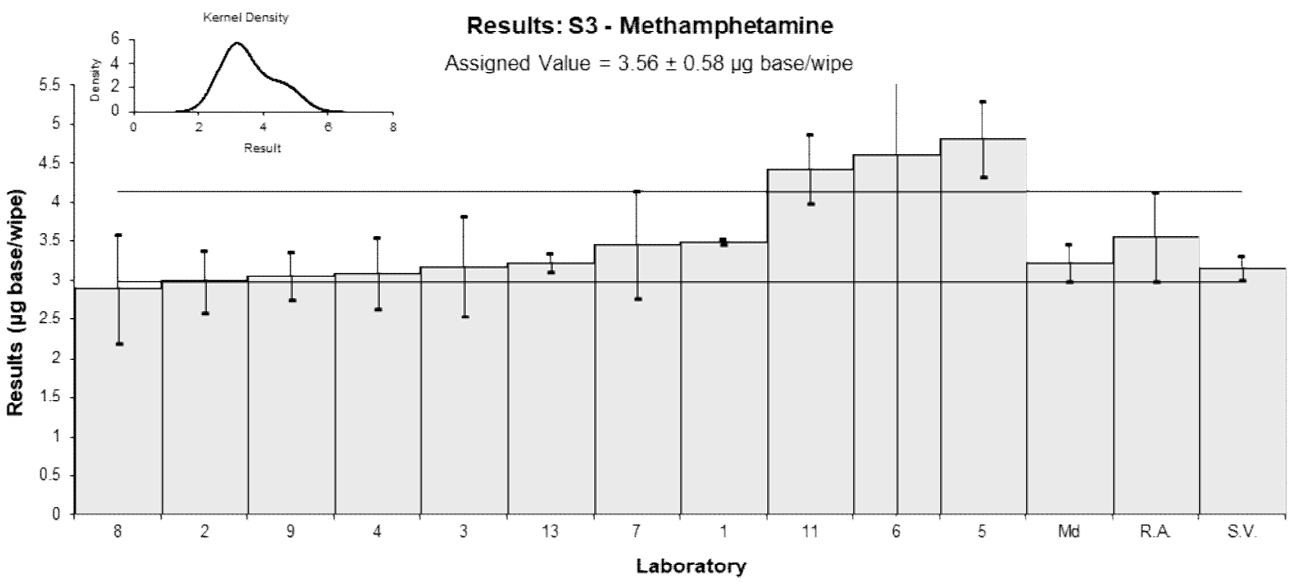
Sample No.	S3
Matrix	Wipe
Analyte	Methamphetamine
Units	µg base/wipe

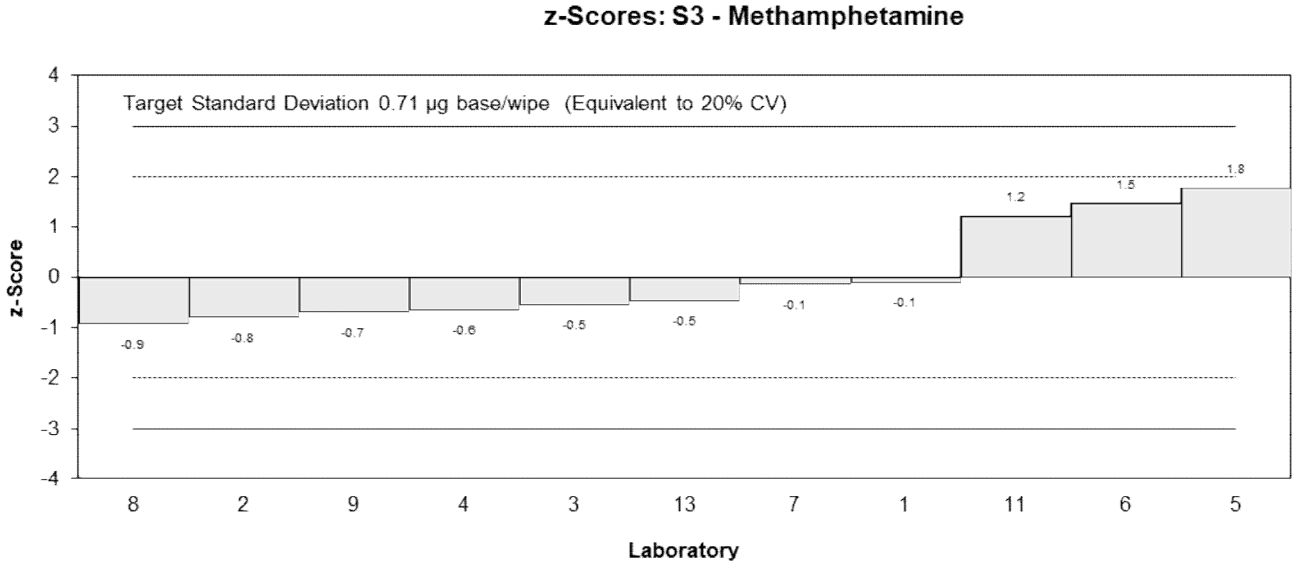
Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	3.49	0.034	NR	-0.10	-0.12
2	2.99	0.39	99	-0.80	-0.82
3	3.18	0.64	106	-0.53	-0.44
4	3.1	0.45	NR	-0.65	-0.63
5	4.82	0.48	NR	1.77	1.67
6	4.6	17	96	1.46	0.06
7	3.46	0.69	114	-0.14	-0.11
8	2.9	0.7	105	-0.93	-0.73
9	3.06	0.31	NR	-0.70	-0.76
11	4.429	0.44	NR	1.22	1.19
13	3.23	0.11	100.3	-0.46	-0.56

Statistics

Assigned Value	3.56	0.58
Spike	3.16	0.16
Robust Average	3.56	0.58
Median	3.23	0.24
Mean	3.57	
N	11	
Max.	4.82	
Min.	2.9	
Robust SD	0.78	
Robust CV	22%	


Kernel Density
Density
Result
Results: S3 - Methamphetamine
Assigned Value = 3.56 ± 0.58 µg base/wipe
Results (µg base/wipe)
Laboratory
8
2
9
4
3
13
7
1
11
6
5
Md
R.A.
S.V.


z-Scores: S3 - Methamphetamine
Target Standard Deviation 0.71 µg base/wipe (Equivalent to 20% CV)
z-Score
Laboratory
-0.9
-0.8
-0.7
-0.6
-0.5
-0.5
-0.1
-0.1
1.2
1.5
1.8

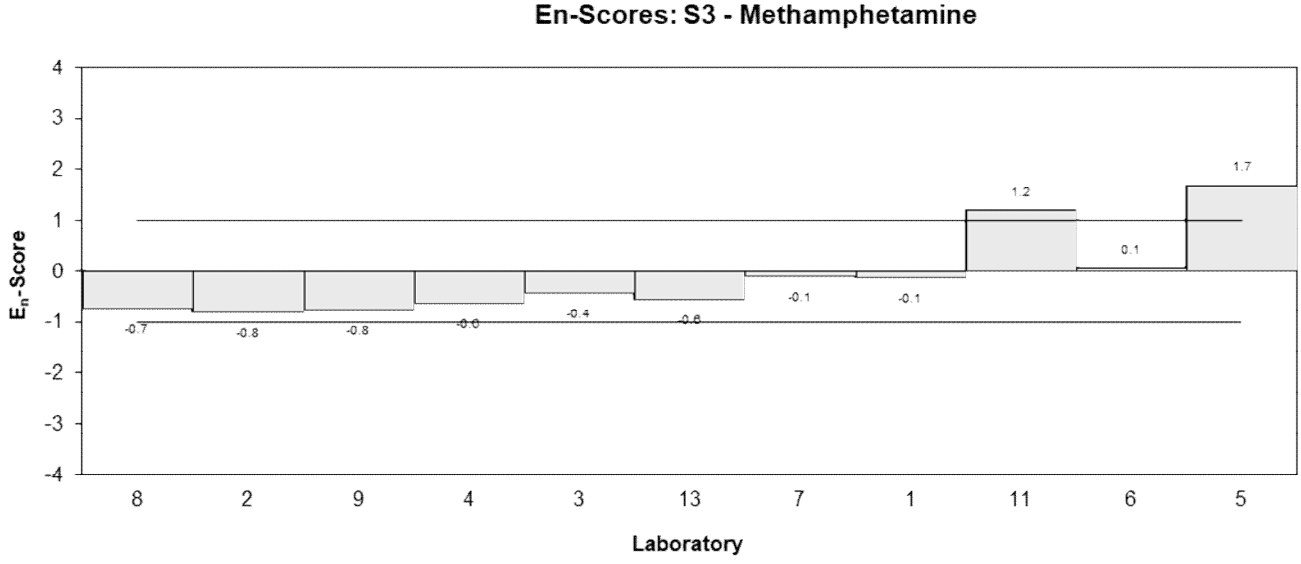

En-Scores: S3 - Methamphetamine
En-Score
Laboratory
-0.7
-0.8
-0.8
-0.6
-0.4
-0.6
-0.1
-0.1
1.2
0.1
1.7

Figure 4

6 DISCUSSION OF RESULTS

6.1 Assigned Value

The assigned values were the robust averages of the results reported by participants. The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528:2015.[6] Results less than 50% and greater than 150% of the robust average were removed before calculation of the assigned value, if applicable.[4,5] The calculation of the expanded uncertainty for robust averages is presented in Appendix 2, using Sample S2 as an example.

Traceability: The consensus of participants' results is not traceable to any external reference, so although expressed in SI units, metrological traceability has not been established.

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded MU associated with their results, and the basis of this uncertainty estimate. It is a requirement of ISO/IEC 17025:2017 that laboratories have procedures to estimate the uncertainty of chemical measurements and to report this uncertainty in specific circumstances, including when the client's instruction so requires.[7]

All results were reported with an associated MU. Participants used a wide variety of procedures to estimate their uncertainty (Table 2).

Laboratories **5**, **9** and **11** reported their uncertainties as a percentage rather than in µg base/wipe. These uncertainty values were modified accordingly by the study coordinator for this report.

The magnitude of reported uncertainties was within the range of 1.0% to 3617% relative. Laboratory **6** reported the same uncertainty for all samples, corresponding to 370% to 3617% relative; this participant may have reported uncertainties as relative instead of absolute values.

Laboratories with results having a satisfactory z-score and an unsatisfactory E_n-score may have underestimated the expanded MU associated with that result.

In some cases, results were reported with an inappropriate number of significant figures. Including too many significant figures may inaccurately reflect the precision of measurements. The recommended format is to write the 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 4.429 ± 0.44 µg base/wipe, it is recommended to report this as 4.43 ± 0.44 µg/wipe).[8]

6.3 z-Score

Target SDs equivalent to 20% PCV was used to calculate z-scores. CVs predicted by the Thompson-Horwitz equation,[9] target SDs (as PCV), and between laboratories CV obtained in this study are presented for comparison in Table 8.

Table 8 Thompson-Horwitz CV, Target SD (as PCV) and Between Laboratories CV

Sample	Analyte	Assigned Value (µg base/wipe)	Thompson-Horwitz CV (%)	Target SD (as PCV) (%)	Between Laboratories CV (%)
S1	Methamphetamine	1.60	22	20	9.5
S2	Methamphetamine	0.444	22	20	16
S3	Methamphetamine	3.56	22	20	22

Of 33 reported results, all returned a z-score of $|z| \leq 2.0$, indicating satisfactory performance.

The dispersal of participants' z-scores is presented graphically by laboratory in Figure 5.

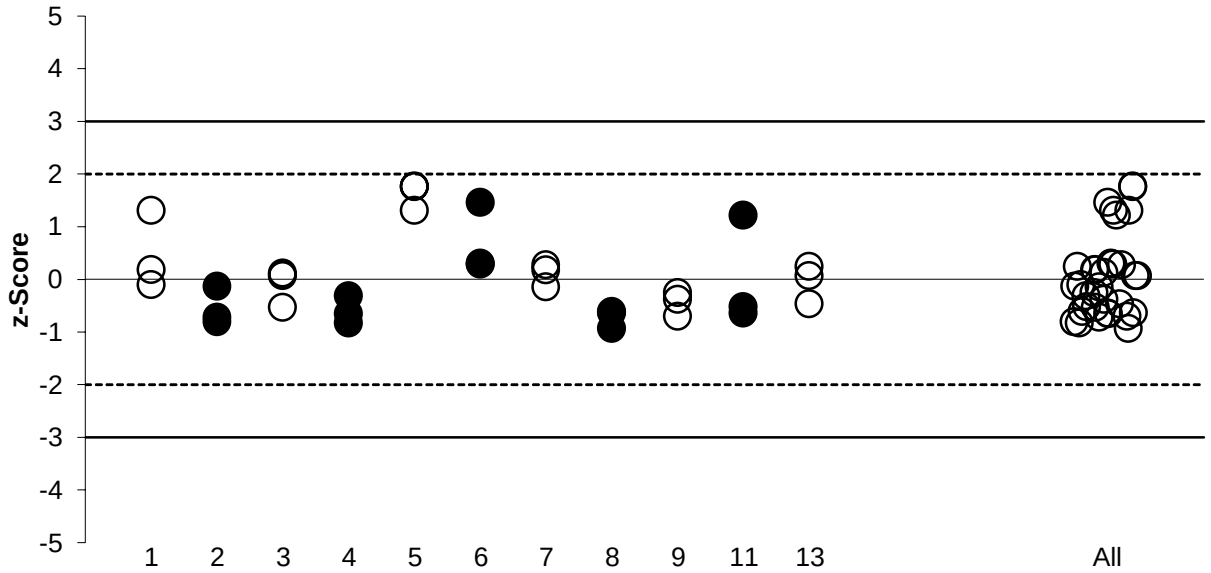


Figure 5 z-Score Dispersal by Laboratory

6.4 E_n-Score

If a participant did not report an uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score.

Of 33 reported results, 27 (82%) returned a satisfactory E_n-score of $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Eight participants: **2**, **3**, **4**, **6**, **7**, **8**, **9** and **13** returned satisfactory E_n-scores for all three samples.

Three participants returned at least one unsatisfactory E_n-score.

Laboratory **5** returned unsatisfactory E_n-scores for all three samples.

The dispersal of participants' E_n-scores is presented graphically by laboratory in Figure 6.

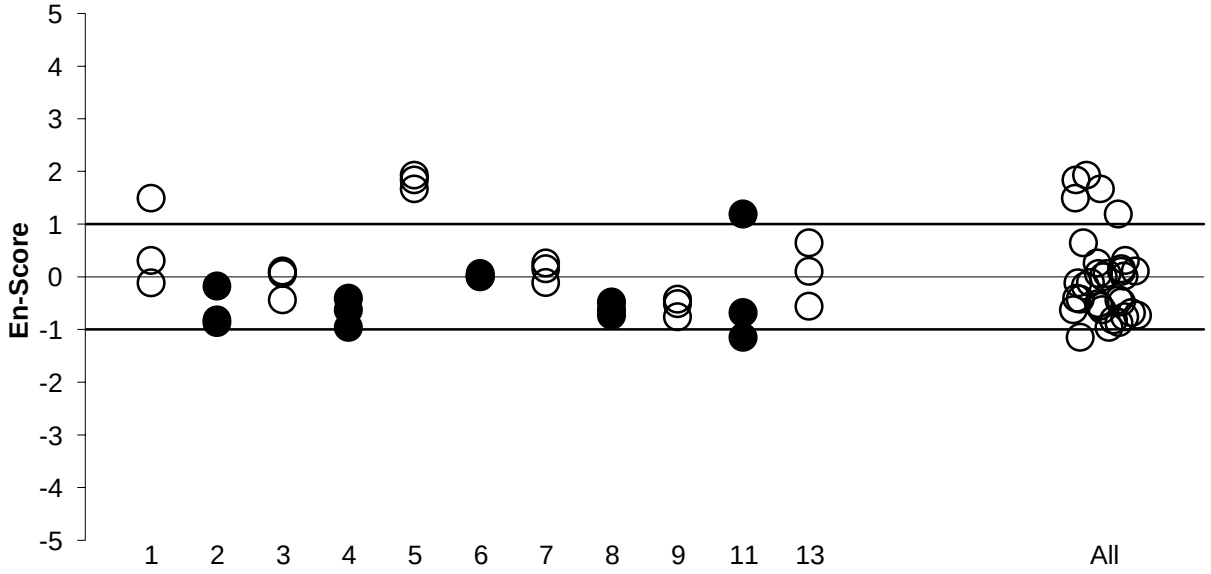


Figure 6 E_n-Score Dispersal by Laboratory

6.5 Participants' Analytical Methods

Participants were requested to analyse the samples using their normal test methods and to report a single result for each sample as they would normally report to a client. Results reported in this way reflect the true variability of results reported to laboratory clients. The method descriptions provided by participants are presented in Table 1.

A summary of participants' accreditation status, methods and reference standards is presented in Table 9. Eight participants reported that their method was based on the NIOSH 9111 method.

Table 9 Summary of Participants' Analyses

		Lab. Code
Accreditation	Yes to ISO/IEC 17025	1, 2, 3, 4, 5, 6, 7, 8, 9, 11, 13
Sample Treatment	Rotary Mixer / Shaking / Tumbling	1, 2, 3, 4, 6, 7, 8, 9, 11, 13
	Centrifuge	5, 11
	Sonication	8
	pH Adjustment	2, 7
Filter Used	Yes	1, 2, 6, 7, 8, 13
	No	3, 4, 5, 9, 11
Desorption Solution	0.1 M Sulfuric Acid	1, 2, 3, 4, 5, 6, 7, 8, 9, 11, 13
Instrument Used for Quantification	LC-MS/MS or LC-QQQ	1, 4
	HPLC-MS/MS	2, 5, 7, 8, 11
	UPLC-MS/MS	3, 6
	HPLC-MS	9, 13
Sources of Calibration Standard	NMI	1, 4
	PM Separations	2, 7
	Lipomed	3, 5, 8, 9, 11
	Supelco	6
	Cerilliant	13
Internal Standard	Methamphetamine-D5	2, 5, 7, 8, 9, 11
	Methamphetamine-D9	4
	Methamphetamine-D14	1, 3, 6, 13

A comparison of z-scores with a number of reported methodology parameters is given in Figures 7 to 9. Due to the wide variety of methods used, no significant trend was evident.

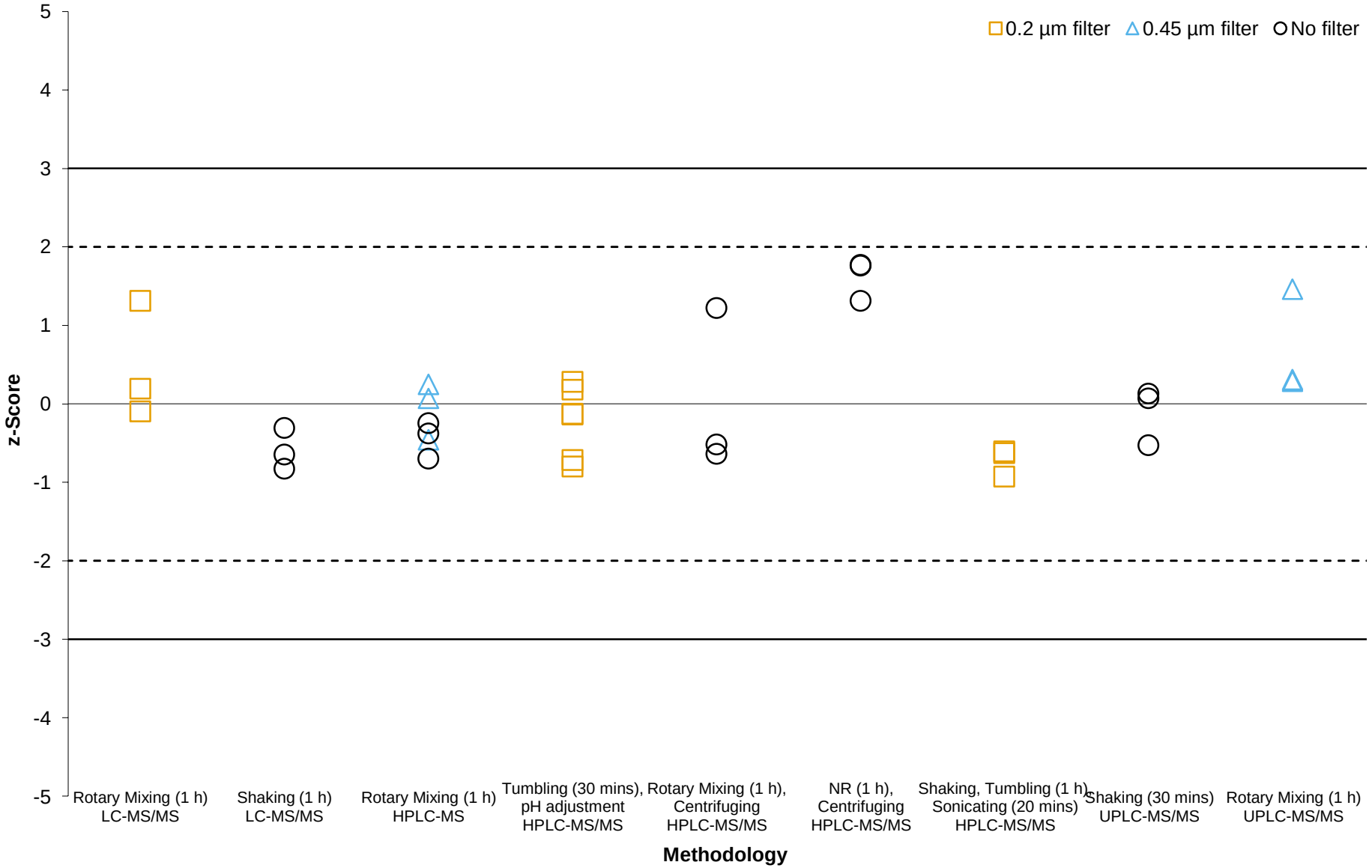


Figure 7 z-Scores vs Methodology

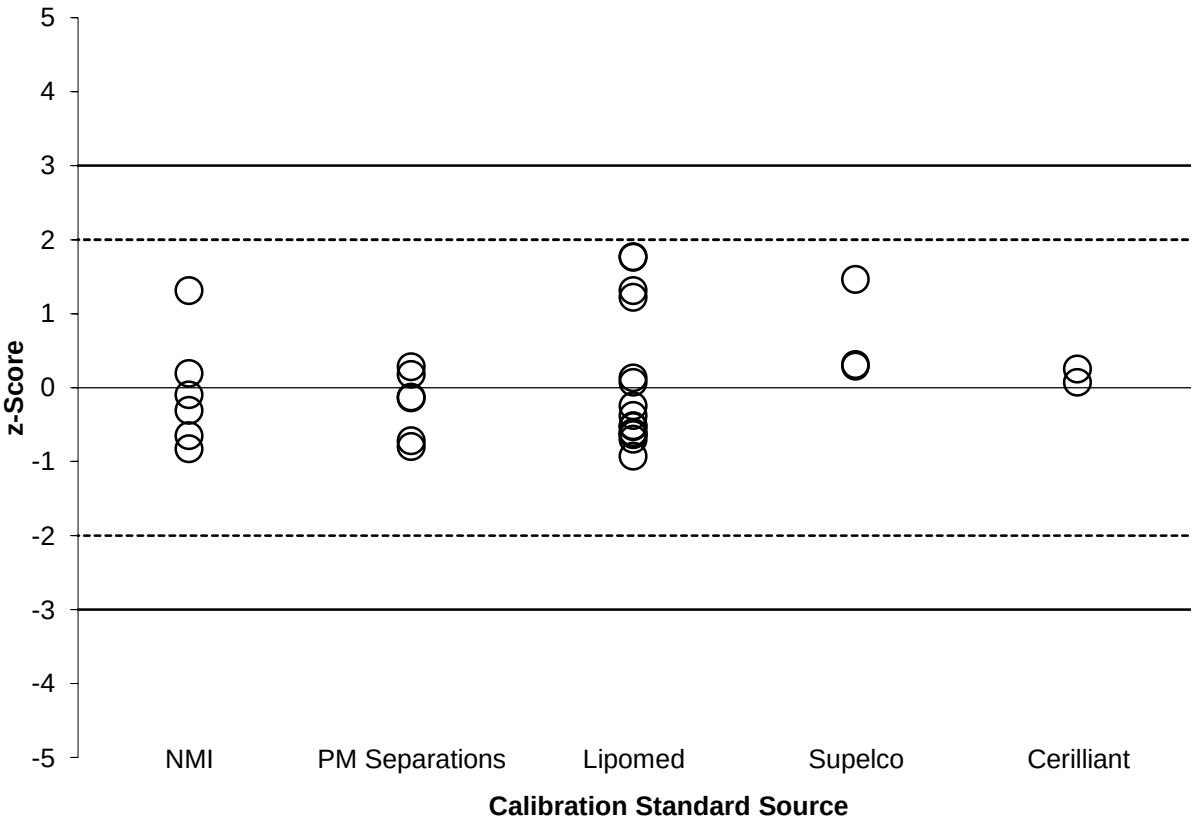


Figure 8 z-Scores vs Calibration Standard Source

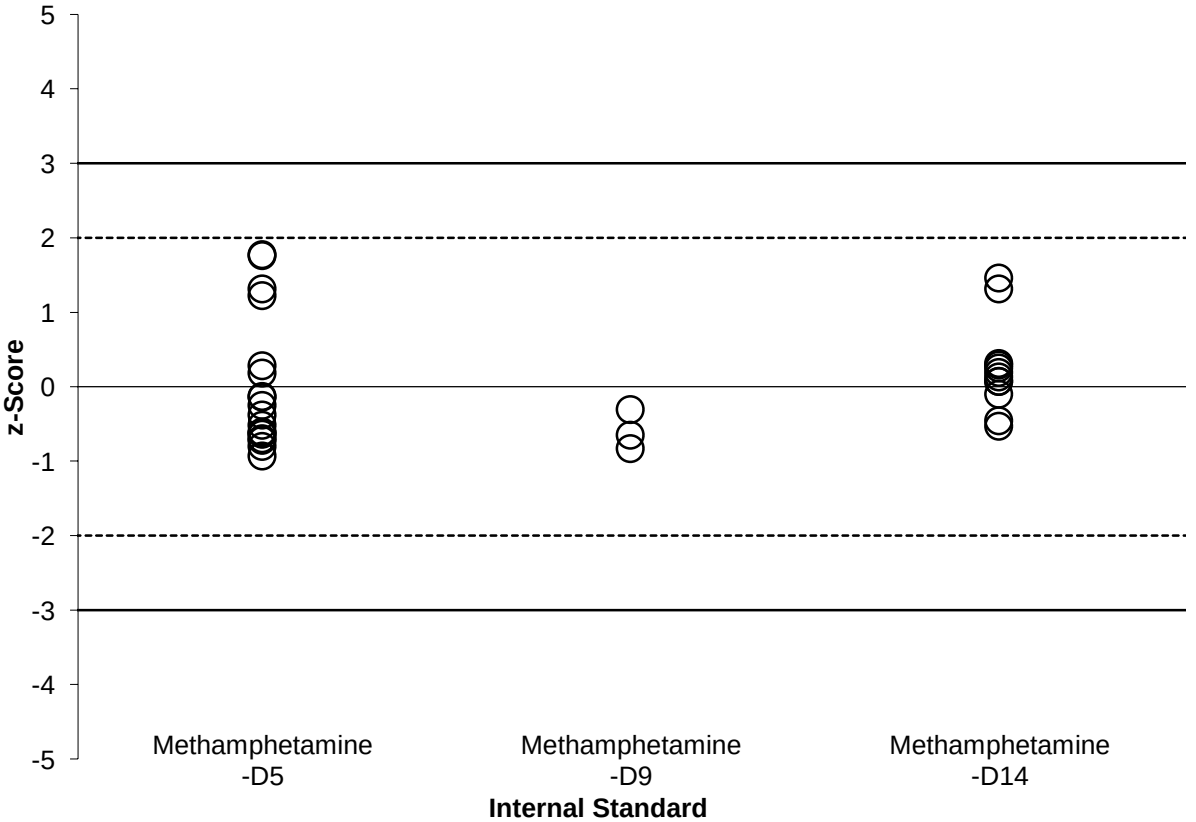


Figure 9 z-Scores vs Internal Standard

6.6 Clandestine Laboratory Remediation Investigation Levels

The Australian Government Clandestine Drug Laboratory Remediation Guidelines specifies the investigation levels (ILs) for various chemicals at clandestine laboratory sites.[2]

For methamphetamine in indoor residential surface areas, the IL criteria is 0.5 µg/100 cm^2 (corresponding in this study to 0.5 µg/wipe). Laboratories should be able to identify if the sample is above or below this IL.

For this study, Sample S2 was prepared to contain 0.402 µg methamphetamine/wipe. The final assigned value for this sample was 0.444 ± 0.054 µg methamphetamine/wipe, which including uncertainty, is below the IL, and therefore the theoretical location which was sampled would not require further remediation.

A summary of results and uncertainties for Sample S2 is presented graphically in Figure 10. The shaded area corresponds to methamphetamine values that are greater than the IL and therefore the site would require further remediation. As Sample S2 was fortified to be below the IL, reported results should all have been below the red shaded area.

In this study for Sample S2, Laboratories **2**, **4**, **8**, **9**, **11** and **13** reported results (including uncertainties) lower than the IL, consistent with the final assigned value for this sample and indicating that no further remediation of the site would be required. Laboratories **3**, **6** and **7** reported results lower than the investigation level, however their expanded uncertainties span the IL. Laboratories **1** and **5** reported results (including uncertainties) greater than the IL, which would indicate that further remediation would be required. It can be seen that there are inconsistencies for results obtained (and therefore further actions required) by different laboratories, particularly for this sample that had a methamphetamine level very close to the IL.

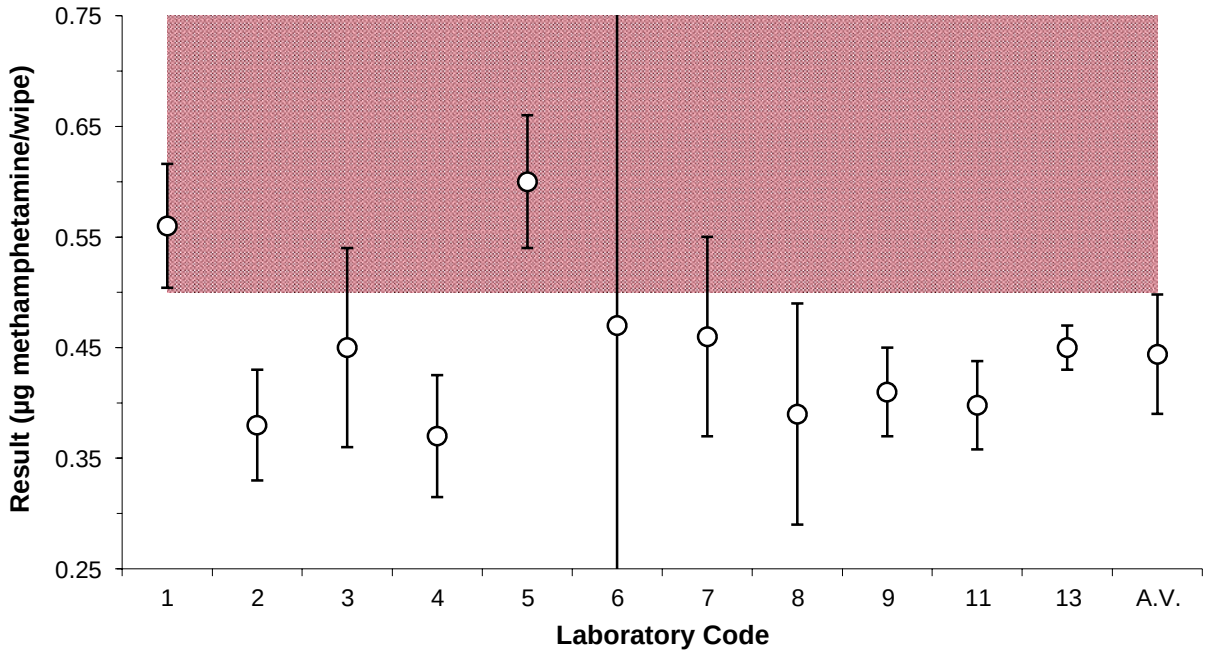


Figure 10 Sample S2 Participant Results and Assigned Value (A.V.). The shaded area corresponds to methamphetamine values greater than the IL.

6.7 Comparison with Previous Controlled Substances in Wipes PT Studies

NMI has run 3 controlled substances in wipes PT studies. A summary of the participation and satisfactory performance (presented as a percentage of the total number of scores for each study) obtained by participants in these studies is presented in Figure 11. To enable direct

comparison, the target SD used to calculate z-scores has been kept constant at 20% PCV. Over these studies, performance has remained very high, with the average proportion of satisfactory z-scores and E_n-scores being 98% and 80% respectively.

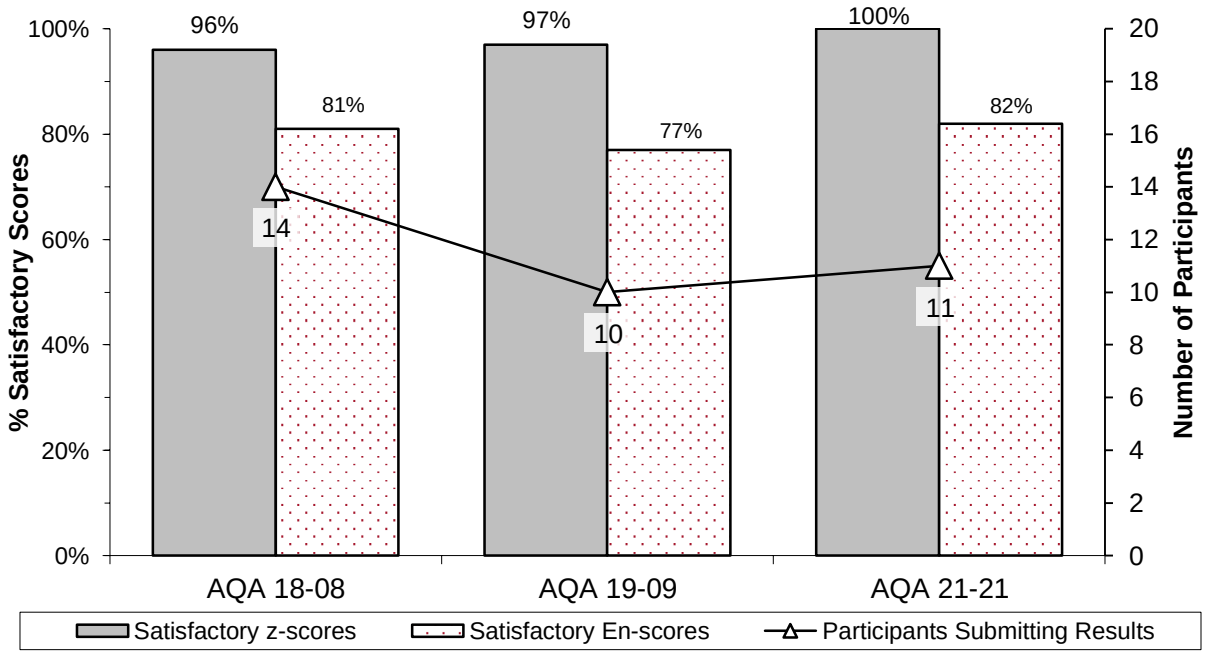


Figure 11 Summary of Participation and Satisfactory Performance in Controlled Substances in Wipes PT Studies

Individual performance history reports are emailed to participants at the end of each study; the consideration of z-scores over time provides much more useful information than a single score. Over time, laboratories should expect at least 95% of their scores to lie within the range $|z| \leq 2.0$. Scores in the range $2.0 < |z| < 3.0$ 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 REFERENCES

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

[2] Commonwealth of Australia, 2011, *Clandestine Drug Laboratory Remediation Guidelines*.

[3] NMI, 2020, *Study Protocol for Proficiency Testing*, viewed June 2021, <https://www.industry.gov.au/sites/default/files/2020-10/cpt_study_protocol.pdf>.

[4] NMI, 2021, *Chemical Proficiency Testing Statistical Manual*, viewed June 2021, <https://www.industry.gov.au/sites/default/files/2019-07/cpt_statistical_manual.pdf>.

[5] Thompson, M., Ellison, S.L.R. & Wood, R., 2006, 'The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories', *Pure Appl. Chem.*, vol. 78, pp. 145-196.

[6] ISO 13528:2015, *Statistical methods for use in proficiency testing by interlaboratory comparison.*

[7] ISO/IEC 17025:2017, *General requirements for the competence of testing and calibration laboratories.*

[8] Eurachem/CITAC Guide CG 4, QUAM:2012.P1, *Quantifying Uncertainty in Analytical Measurement*, 3rd edition, viewed June 2021, <http://www.eurachem.org/images/stories/Guides/pdf/QUAM2012_P1.pdf>.

[9] Thompson, M., 2000, 'Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing', *Analyst*, vol. 125, pp. 385-386.

APPENDIX 1 – STABILITY ASSESSMENT

Samples were stored at 4°C after preparation and before dispatch. Samples were dispatched with cooler brick(s) on 17 May 2021, and participants were advised to store the samples refrigerated if analyses could not be commenced on the day of receipt.

Sample receipt date and reported sample condition on receipt are presented in Table 10. All participants reported that their samples arrived in at least an acceptable condition.

Table 10 Summary of Sample Receipt Date and Reported Condition

Lab. Code	Received Date	Reported Arrival Condition
1	18/05/2021	Fit for analysis
2	18/05/2021	Good
3	20/05/2021	Acceptable
4	20/05/2021	Intact, in polystyrene container with ice pack
5	18/05/2021	Good
6	18/05/2021	Good
7	18/05/2021	Good
8	18/05/2021	Fit for analysis
9	18/05/2021	Excellent - Still cold
11	18/05/2021	Good
13	20/05/2021	Acceptable

A comparison of z-scores obtained to days spent in transit is presented in Figure 12. No statistically significant correlation between results and the days spent in transit was observed.

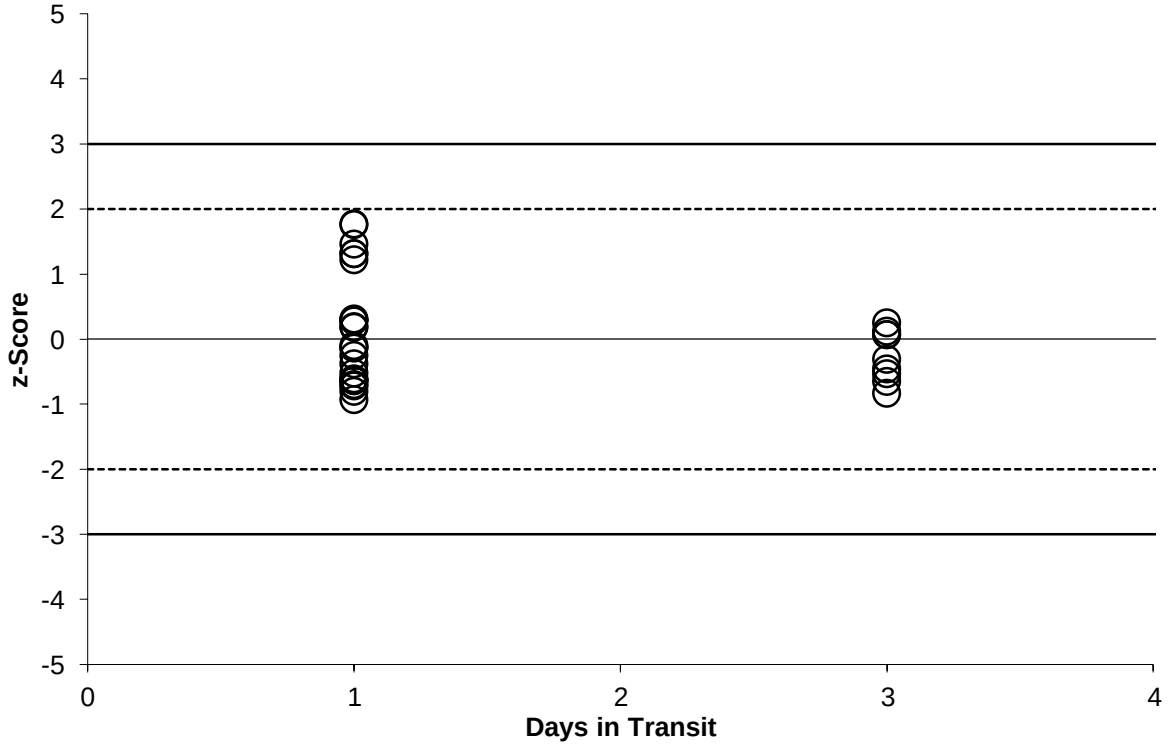


Figure 12 z-Scores vs Days in Transit

APPENDIX 2 – ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, Z-SCORE AND E_N-SCORE CALCULATIONS

A2.1 Robust Average and Associated Uncertainty

When the robust average is calculated using the procedure described in ISO 13528:2015,[6] the uncertainty is estimated as:

$$u_{rob\ av} = \frac{1.25 \times S_{rob\ av}}{\sqrt{p}} \qquad \textit{Equation 4}$$

where:

$u_{rob\ av}$ is the standard uncertainty of the robust average

$S_{rob\ av}$ is the standard deviation of the robust average

p is the number of results

The expanded uncertainty ($U_{rob\ av}$) is the standard uncertainty multiplied by a coverage factor of 2 at approximately 95% confidence level.

A worked example is set out below in Table 11.

Table 11 Uncertainty of Robust Average for Methamphetamine in Sample S2

No. results (p)	11
Robust Average	0.444 µg base/wipe
$S_{rob\ average}$	0.071 µg base/wipe
$u_{rob\ average}$	0.027 µg base/wipe
k	2
$U_{rob\ average}$	0.054 µg base/wipe

Hence, the robust average for methamphetamine in Sample S2 is 0.444 ± 0.054 µg base/wipe.

A2.2 z-Score and E_n-Score Calculations

For each participant's result, a z-score and E_n-score are calculated according to Equations 2 and 3 respectively (Sections 4.6 and 4.7).

A worked example is set out below in Table 12.

Table 12 z-Score and E_n-Score Calculation for Sample S1 Result Reported by Laboratory 1

Participant Result (µg base/wipe)	Assigned Value (µg base/wipe)	Target SD	z-Score	E_n-Score
1.66 ± 0.16	1.60 ± 0.11	20% as PCV, or: 0.2 × 1.60 = 0.32 µg base/wipe	z-Score = $\frac{1.66 - 1.60}{0.32}$ = 0.19	E_n-Score = $\frac{1.66-1.60}{\sqrt{0.16^2+0.11^2}}$ = 0.31

APPENDIX 3 – ACRONYMS AND ABBREVIATIONS

A.V.	Assigned Value
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
GAG	General Accreditation Guidance (NATA)
GUM	Guide to the expression of Uncertainty in Measurement
HPLC	High Performance Liquid Chromatography
IEC	International Electrotechnical Commission
IL	Investigation Level (Clandestine Laboratory Remediation Guidelines)
ISO	International Organization for Standardization
LC	Liquid Chromatography
Max.	Maximum value in a set of results
Md	Median
Min.	Minimum value in a set of results
MS	Mass Spectrometry
MS/MS	Tandem Mass Spectrometry
MU	Measurement Uncertainty
N	Number of reported results
NATA	National Association of Testing Authorities, Australia
NIOSH	National Institute for Occupational Safety & Health
NMI	National Measurement Institute (Australia)
NPAAC	National Pathology Accreditation Advisory Council
PT	Proficiency Test
PCV	Performance Coefficient of Variation
QQQ	Triple Quadruple (Mass Spectrometry)
R.A.	Robust Average
RM	Reference Material
SD	Standard Deviation
SI	International System of Units
SS	Spiked Samples
S.V.	Spiked Value
UPLC	Ultra Performance Liquid Chromatography

END OF REPORT