Australian Government
Department of Industry, Innovation and Science
National Measurement Institute

Proficiency Test Report AQA 18-08 Methamphetamine and MDMA in Wipes

September 2018

ACKNOWLEDGMENTS

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

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 18-08 was conducted in July/August 2018. Four test samples of methamphetamine hydrochloride and MDMA hydrochloride in wipes were sent to thirteen laboratories. Two laboratories requested multiple sets of the test samples for the analysis to be performed by different analysts. Sixteen sets of results were submitted by the due date.

Test samples were prepared at the NMI laboratory in Sydney using methamphetamine hydrochloride and MDMA hydrochloride synthesised by NMI.

The four test samples were divided into 2 pairs of duplicates (S1/S2 and S3/S4), and the assigned values for each analyte were calculated as the robust averages of the pooled participant results in both samples in each duplicate pair. The associated uncertainties were estimated from the robust standard deviations of the participants' results for each duplicate pair.

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

- *assess the proficiency of laboratories measuring methamphetamine and MDMA in wipes;*

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

Laboratories **1**, **2**, **4**, **6**, **9** and **10** returned satisfactory z and E_n-scores for all analytes for which they submitted results.

Of the 84 results for which z-scores were calculated, 81 (96%) returned $|z| \leq 2$ indicating a satisfactory performance.

Of the 84 results for which $|E_n|$-scores were calculated, 68 (81%) returned $|E_n| \leq 1$ indicating agreement of the participants' results with the assigned value within their respective expanded uncertainties.

- *evaluate the laboratories methods used in the determination of methamphetamine and MDMA in wipes.*

Participants used a variety of methods for measurement of methamphetamine and MDMA in wipes and most produced comparable results.

- *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 expanded uncertainty.

The magnitude of reported uncertainties was within the range 3% to 35% relative.

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 and biota; and
- metals in soil, water, food and pharmaceuticals;

AQA 18-08 is the first NMI proficiency test of the analysis of methamphetamine and MDMA in wipes.

1.2 Study Background

Illicit laboratory sites (clandestine laboratories, 'clan labs') are places where illegal drugs have been manufactured. During the drug manufacturing process, toxic gases and aerosols are produced. These may be absorbed by flooring, walls, ducting and furnishings. Chemical contamination may remain in the property for many years. Field test kits are used to check the extent of contamination in the premises. Samples may be taken from non-porous surfaces inside a building using wipes.

This scheme was a pilot program to enable laboratories to assess their ability to measure methamphetamine and MDMA in wipes at investigation levels specified in Clandestine Drug Laboratory Remediation Guidelines 2011.

1.3 Study Aims

The aims of the study were to:

- assess the proficiency of laboratories measuring methamphetamine and MDMA in wipes;
- evaluate the laboratories methods used in the determination of methamphetamine and MDMA in wipes; and
- develop the practical application of traceability and measurement uncertainty and provide participants with information that will be useful in assessing their uncertainty estimates.

1.4 Study Conduct

NMI is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO 17043[1] as a provider of proficiency testing schemes. This controlled drug proficiency test is within the scope of NMI's accreditation.

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 Testing Statistical Manual.[3] These documents have been prepared with reference to ISO 17043 and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[4]

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitation issued:	24 April 2018
Samples dispatched:	09 July 2018
Results due:	10 August 2018
Interim report issued:	17 August 2018

2.2 Participation

A total of eighty-two international, national, state government and private laboratories were invited to participate.

Thirteen laboratories agreed to participate and eleven submitted results for at least one sample. These laboratories are listed in Appendix 1. Two laboratories requested additional sets of test samples to be analysed independently by different analysts.

2.3 Test Material Specification

Four samples were provided for analysis: AQA 18-08 S1, S2, S3 and S4. Each sample consisted of one wipe spiked with methamphetamine and MDMA.

Sample S1 was prepared to contain 3.24 µg/wipe methamphetamine base and 18.5 µg/wipe MDMA base.
Sample S2 was identical with Sample S1.
Sample S3 was prepared to contain 1.80 µg/wipe methamphetamine base and 9.86 µg/wipe MDMA base.
Sample S4 was identical with Sample S3.

2.4 Laboratory Code

Each participant was randomly assigned a confidential laboratory code.

2.5 Sample Preparation, Analysis and Homogeneity Testing

The preparation and homogeneity testing are described in Appendix 2. The study samples were found sufficiently homogeneous for the assessment of participants' results.

2.6 Stability of Analytes

Results of this study gave no reason to question the stability of the test samples. No correlation between reported results, the received date, the analysis date or the sample condition at arrival was observed.

2.7 Sample Storage, Dispatch and Receipt

The study samples were stored at 4°C and dispatched by courier on 9 July 2018.

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

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

2.8 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse each wipe for the amount of each drug using your normal test method.
- Store the samples refrigerated.

- For each analyte report results in µg/wipe drug as base. Report this figure as if reporting to a client.
- For each result report an estimate of your expanded uncertainty as µg/wipe drug as base.
- 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.
- Give brief details of your:
    - basis of uncertainty estimate (eg uncertainty budget, repeatability precision)
    - analytical method (eg sample treatment, instrument type and calibration method)
    - reference standard (eg source, purity)
- E-mail your results on this spreadsheet to proficiency@measurement.gov.au.
- Results are to be returned by COB Friday 10 August 2018.

2.9 Interim Report

An interim report was emailed to all participants on 17 August 2018.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Method Summaries

Summaries of test methods used by participants are transcribed in Table 1.

Table 1 Summary of Participants' test Methods

Lab. Code	Desorption Solution	Sample Treatment	Filtration	Equipment	Internal Standard	Method Reference
1	0.1 M sulfuric acid	30 minutes on rotatory mixer		LC-MS/MS	Methamphetamine-D14 MDMA-D5	NIOSH 9111
2	0.1 M sulfuric acid	30 minutes tumbling at 30rpm		LCMSMS	D5-Meth, D5-MDMA	NIOSH 9111 with pH adjustment prior to LC injection
3	Methanol extract for 30 minutes	Methanol extract boiled down to dryness	sample reconstituted in hexane/triethylacetate/ MBTFA	GC-MS	Eicosane	EFS-DM-625 Exam for the Presence of Trace Quantities of Controlled Substances
4	0.1 M Sulfuric acid	1 hour on shaker, then filtered	0.22 um Nylon filter	LCMSMS	Methamphetamine-D_5	Based on NIOSH 9111
5	0.1 M sulfuric acid	acid desorption, pH adjustment, liquid-liquid extraction, derivatisation		GCMS	Methamphetamine-D_{14}	NIOSH 9106 (with in-house modifications)
6	0.1M sulphuric acid	1 hour on a mechanical shaker	Nil	LCMSMS	Methamphetamine-D_9	Modified NIOSH 9111
7	0.2 N sulfuric acid	1 hour on a rotary mixer, pH adjustment		GCMS	Methamphetamine-D_{14}, MDMA-D_5	NIOSH 9111
8	MilliQ Water	10 minute vortex and then invert and vortex for an additional 10 minutes	0.20 um syringe filter	UPLC, LC QQQ		In house
9	0.1M Sulfuric acid	Addition of ISTD, desorption solution, 1 hour on rotary mixer, pH adjustment if required	0.2um Phenex RC filter	LCMSMS	Methamphetamine-D_{14}	based on NIOSH 9111
10	0.1M sulphuric acid	1 hour on a rotary mixer	Agilent PES 0.45 um filter	LCMS	Methamphetamine-D_{14}	NIOSH 9111
11	0.1 M Formic acid	Tumble for 1 hour, sonicate for 30mins, pH adjustment to approx pH 3	Filter 0.45um RC	LCMSMS	Methamphetamine-D_5, MDMA-D_5	NIOSH 9111
12	0.1M sulfuric acid	1 hour on a rotary mixer	0.22um syringe filter	UPLC-MSMS	Methamphetamine-D_{14}	NIOSH 9111 (Modified)
14	0.1M sulfuric acid	2 hours on rotary mixer, no pH adjustment	0.45 um PES filter	LCMS	Amphetamine-D_{11}	NIOSH 9111
15	MilliQ Water	Vials are vortexed for 10 minutes, then inverted and vortexed for a further 10 minutes	0.20 um syringe filter	UPLC, LC QQQ		In house

Table 1 Participant Summary of Test Methods (continued)

Lab. Code	Desorption Solution	Sample Treatment	Filtration	Equipment	Internal Standard	Method Reference
13	Each sample was extracted into separate solutions of methanol and hexane/triethylamine. Further derivatisation was conducted using trifluoroacetamide (TFA) agent. This was qualitative only.	Each sample was allowed to extract into solvent for approximately 15 minutes.		GCMS	Certified drugs reference standards for Methylampheta mine and MDMA run for Rt check	In-House temperature ramped drugs screen method / Agilent GCMS for qualitative screen
16	Water	Sample placed in i-chem vial 10 mL of water added, shaken on rotary mixer 10 min, inverted and shaken for further10 min	0.2 um Phenex syringe filter	LC for initial screening then LCMS QQQ for analysis		In-house

3.2 Reported Basis of Participants' Measurement Uncertainty Estimates

Participant approaches to measurement uncertainty are listed as received in Table 2.

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	CRM Recoveries of SS Instrument Calibration Standard Purity	Nata Technical Note 33
2	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS Duplicate Analysis	Recoveries of SS	ISO/GUM
3	Professional judgment	Instrument Calibration	CRM Instrument Calibration Standard Purity	N/A
4	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS Instrument Calibration	Standard Purity Recoveries of SS	Nata Technical Note 33
5	Top Down - precision and estimates of the method and laboratory bias	Instrument Calibration	Instrument Calibration	Eurachem/CITAC Guide
6	Standard uncertainty based on historical data	Control Samples – Laboratory Control Spikes Duplicate Analysis Instrument Calibration	Instrument Calibration Standard Purity	Eurachem/CITAC Guide
7	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS	Recoveries of SS	Nata Technical Note 33
8	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples – SS Duplicate Analysis Instrument Calibration	Recoveries of SS Instrument Calibration Standard Purity	Nata Technical Note 33

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
9	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM Duplicate Analysis Instrument Calibration	Standard Purity	Eurachem/CITAC Guide
10	NIOSH Method Accuracy Range (A)	Control Samples – SS Duplicate Analysis Instrument Calibration	CRM Recoveries of SS Instrument Calibration Standard Purity	NIOSH Manual of Analytical Methods 3/15/03 Page 208 Part P. Measurement Uncertainty and NIOSH Method Accuracy Range
11	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS	Recoveries of SS	Nata Technical Note 33
12	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM Duplicate Analysis Instrument Calibration	Recoveries of SS	Nata Technical Note 33
13	Examined as routine drug trace items. As this is a qualitative method only, a numerical value for uncertainty cannot be measured.			As detailed above, the items were tested on a qualitative basis only
14	Standard deviation of replicate analyses multiplied by 2 or 3	CRM Duplicate Analysis	CRM Recoveries of SS Instrument Calibration	Eurachem/CITAC Guide
15	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples (SS) Duplicate Analysis Instrument Calibration	Recoveries of SS Instrument Calibration Standard Purity	Nata Technical Note 33
16	Top Down - precision and estimates of the method and laboratory bias	Duplicate Analysis	CRM Instrument Calibration Recoveries of SS Standard Purity	Nata Technical Note 33

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

3.3 Details of Participant Calibration Standard

Reference standards used by laboratories are listed as received in Table 3.

Table 3 Participant Calibration Standard

Lab. Code	Calibration Standard	Purity (%)
1	(±)-Methamphetamine 1.0 mg/mL (±)-MDMA 1.0 mg/mL	
3	Methylamphetamine/MDMA	
4	CRM 1.0 mg/ml +- 0.005 mg/ml from Cerilliant	
5	Cerilliant, purity 99.9%	99.9
6	NMI standard, purity 99.8%	99.8
8	MA - NMI std 99.8%, MDMA - NMI std 95.6%	99.8, 95.6
9	Cerilliant 1.000 +/- 0.006 mg/mL	
10	CRM Ceriliant product M-009, 1mg/mL	
11	Chiron >99% as base	>99
12	NMI Standards	
14	Lipomed standard, purity 99.5%	99.5
15	MA - NMI std 99.8%, MDMA - NMI std 95.6%	99.8, 95.6
16	MA: NMI standard, purity 99.80 MDMA: NMI standard, purity 95.60	99.8, 95.6

3.4 Participants' Comments

The study manager welcomes comments or suggestions from participants as it provides information which will improve future studies. All returns are listed as received in Table 4 along with the study manager's response, where appropriate.

Table 4 Participant Comments

Lab. Code	Participant Comments	Study Manager's Comments
8	Provide a blank swab for analysis in addition to the unknown swabs.	These comments have been noted and we will consider including blank swabs in future PT studies of this nature. Thank you for your feedback.
15	As we test recovery as part of our analysis, it would have been helpful to have received a blank swab as part of the PT sample set.	
16	Blank swabs (2) to be included so we can perform more relevent recovery calculations. Format so this work sheet can be printed out on 2 pages as opposed to 5.	

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 5 to 12 with resultant summary statistics: mean, median, maximum, minimum, robust average, 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 9.

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

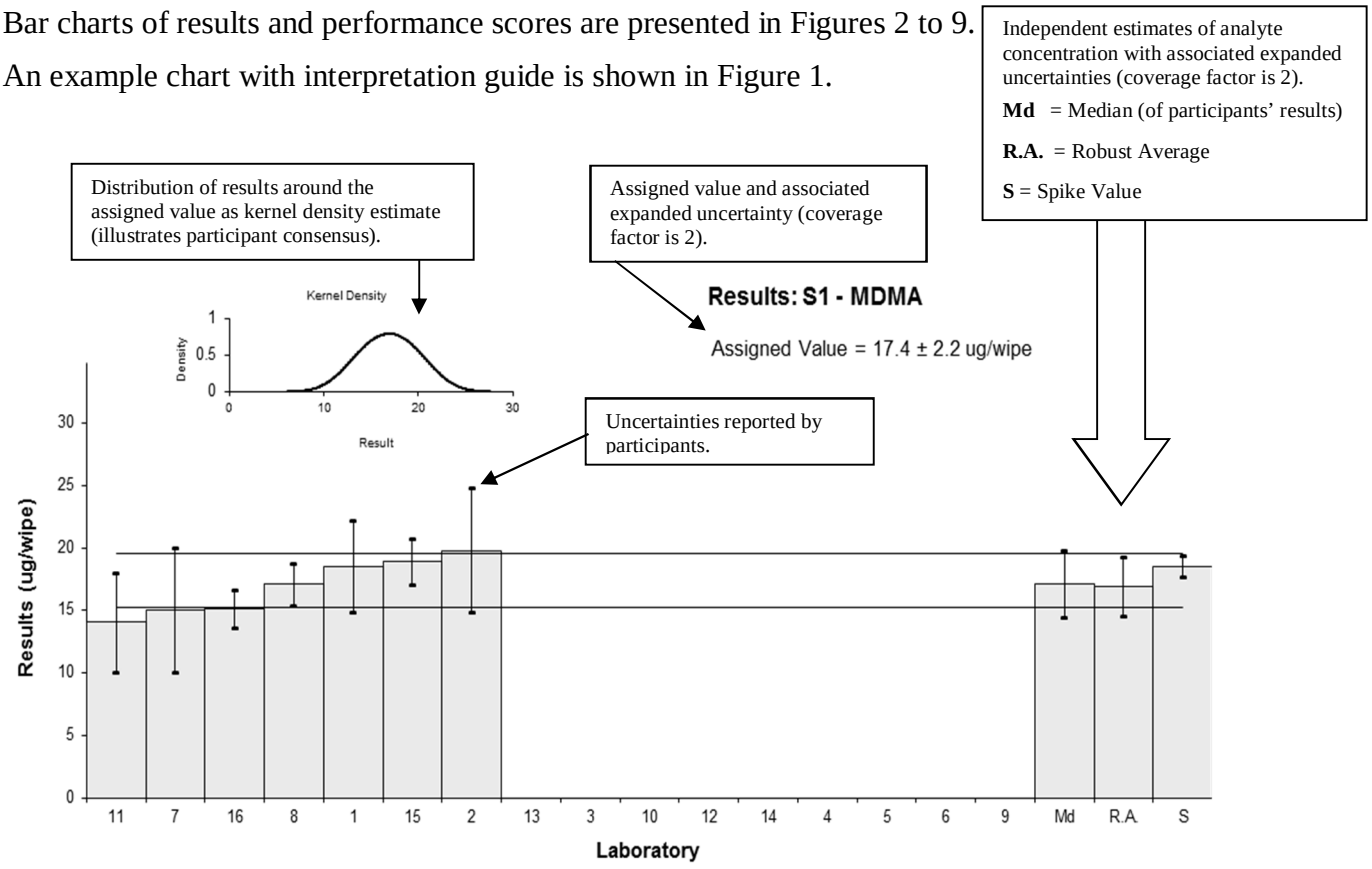


Figure 1 Guide to Presentation of Results

4.2 Assigned Value

Assigned value is defined as: 'the value attributed to a particular quantity and accepted, sometimes by convention, as having an uncertainty appropriate for a given purpose'.[1]

For a proficiency test, the assigned value is the best available measurement of the true concentration of an analyte in the test sample.

4.3 Robust Average

The robust averages and associated expanded measurement uncertainties were calculated using the procedure described in 'Statistical methods for use in proficiency testing by inter-laboratory comparisons, ISO 13528:2015(E)'.[5]

4.4 Robust Between-Laboratory Coefficient of Variation

The robust between-laboratory coefficient of variation (robust CV) is a measure of the variability of participants' results and was calculated using the procedure described in ISO 13528:2015(E).[5]

4.5 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. This value is used for calculation of participant z-score.

$\sigma = X * PCV$ *Equation 1*

It is important to note that the PCV is a fixed value established by the study coordinator and is not the standard deviation of participants' results. By setting a fixed value for the PCV, the participants' performance can be compared from study to study.

4.6 z-Score

For each participant result a z-score is calculated according to Equation 2 below:

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

where:

z is z-score

χ is participants' result X is the study assigned value

σ is the target standard deviation

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

- $|z| \leq 2$ is satisfactory;
- $2 < |z| < 3$ is questionable;
- $|z| \geq 3$ 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 below:

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

where:

E_n is E_n-score

χ is participants' result X is the study 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.

4.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2017[6] 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. [7]

5 TABLES AND FIGURES

Table 5

Sample Details

Sample No.	S1
Matrix.	Wipe
Analyte.	MDMA
Units	µg/wipe

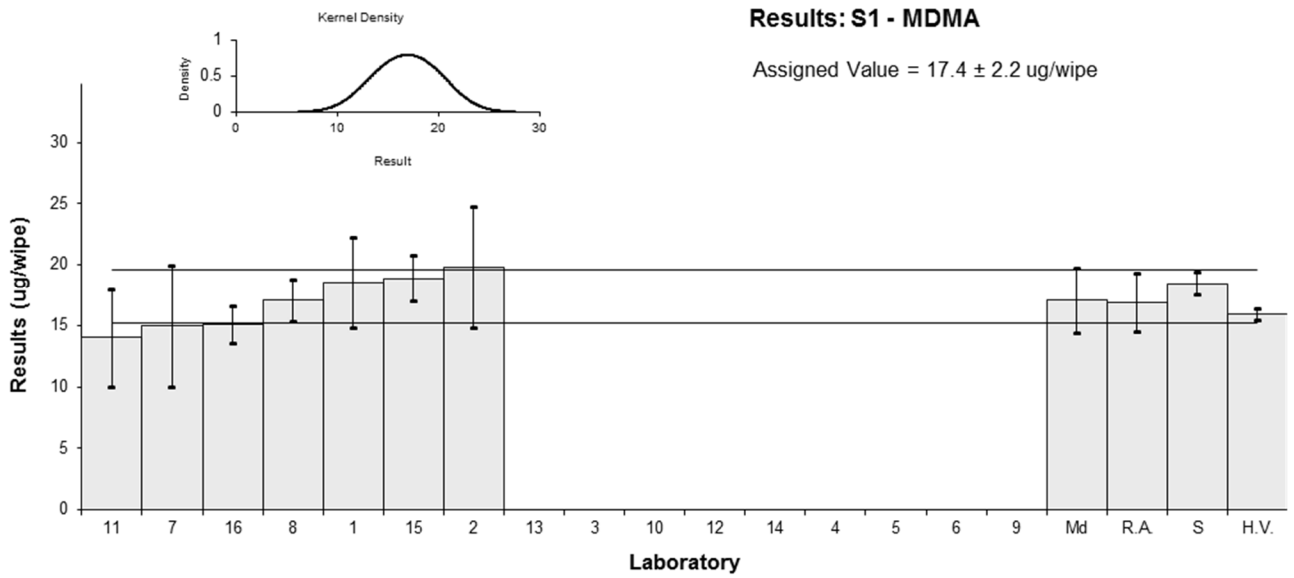
Participant Results

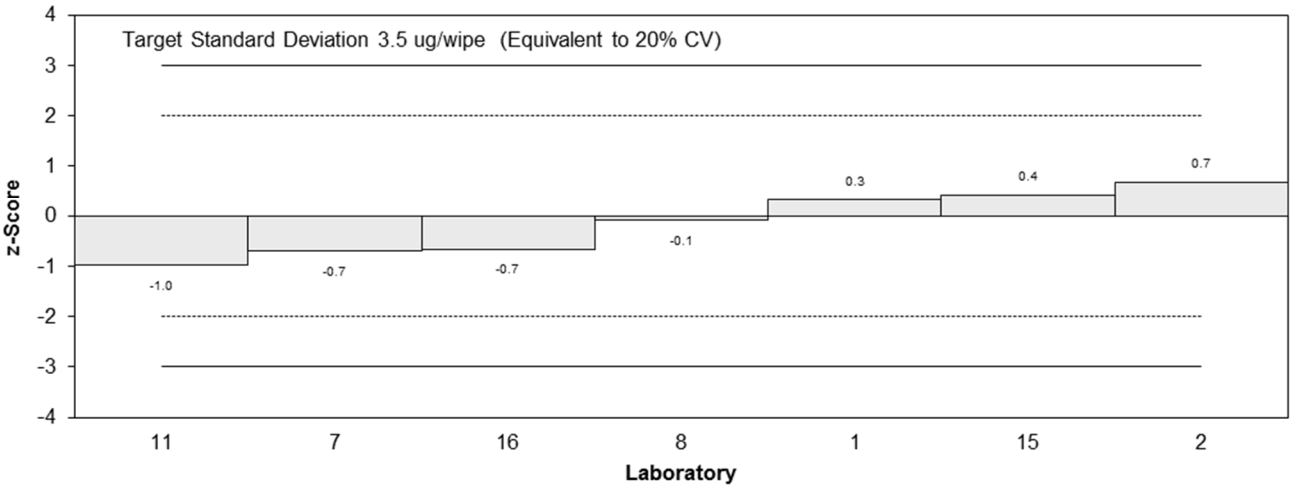
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	18.543	3.709	NR	0.33	0.27
2	19.8	5	95	0.69	0.44
3	NR	NR	NR		
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	15	5	NR	-0.69	-0.44
8	17.1	1.71	59	-0.09	-0.11
9	NT	NT	NT		
10	NT	NT	NT		
11	14	4	106	-0.98	-0.74
12	NT	NT	NT		
13	NR	NR	NR		
14	NT	NT	NT		
15	18.9	1.89	61	0.43	0.52
16	15.1	1.51	71	-0.66	-0.86

Statistics

Assigned Value[a]	17.4	2.2
Spike	18.5	0.9
Robust Average	16.9	2.4
NMI Homogeneity Value	15.9	0.4
Median	17.1	2.7
Mean	16.9	
N	7	
Max.	19.8	
Min.	14	
Robust SD	2.6	
Robust CV	15%	

[a]The assigned value was calculated as the robust average of the combined results of duplicate pair Samples S1 and S2.


Kernel Density
Density
Result
Results: S1 - MDMA
Assigned Value = 17.4 ± 2.2 ug/wipe
Results (ug/wipe)
Laboratory
11 7 16 8 1 15 2 13 3 10 12 14 4 5 6 9 Md R.A. S H.V.


z-Scores: S1 - MDMA
Target Standard Deviation 3.5 ug/wipe (Equivalent to 20% CV)
z-Score
Laboratory
11 7 16 8 1 15 2
-1.0 -0.7 -0.7 -0.1 0.3 0.4 0.7

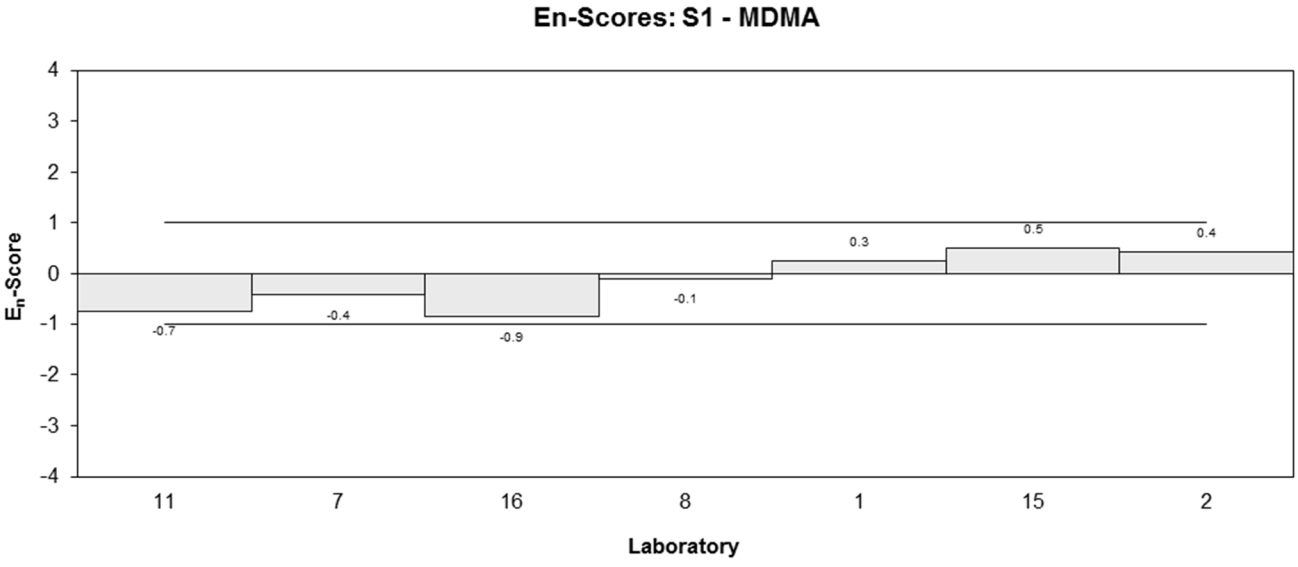

En-Scores: S1 - MDMA
En-Score
Laboratory
11 7 16 8 1 15 2
-0.7 -0.4 -0.9 -0.1 0.3 0.5 0.4

Figure 2

Table 6

Sample Details

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

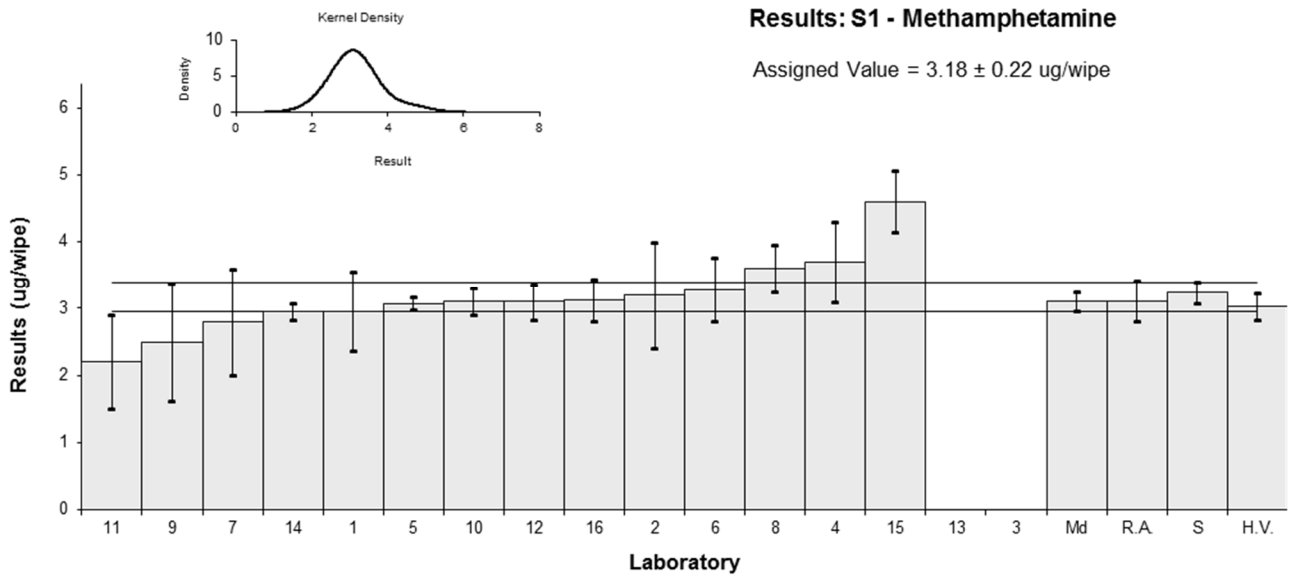
Participant Results

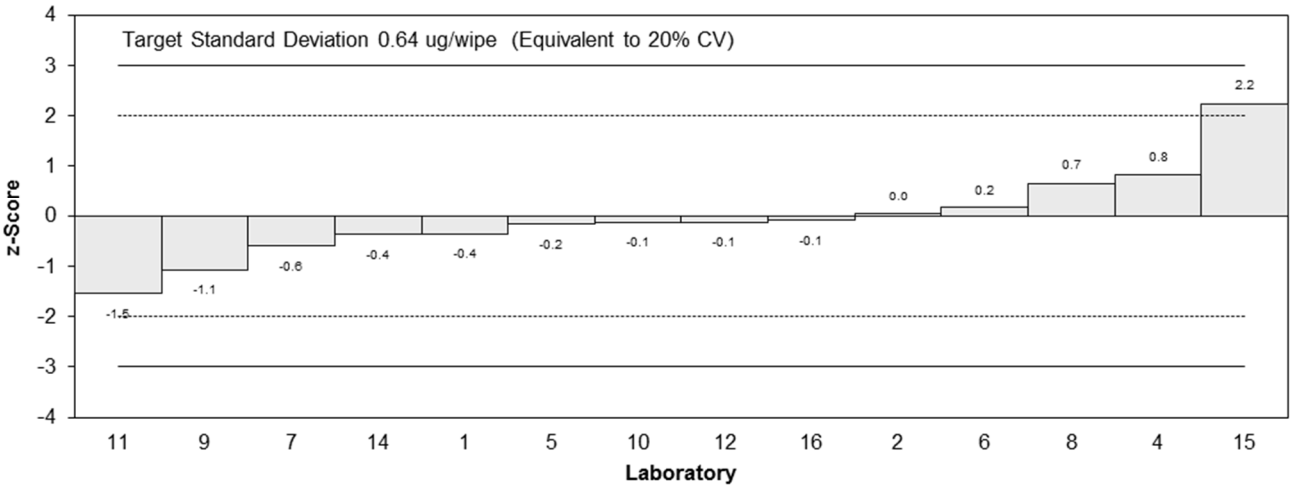
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2.954	0.591	NR	-0.36	-0.36
2	3.2	0.8	118	0.03	0.02
3	NR	NR	NR		
4	3.7	0.6	114	0.82	0.81
5	3.07	0.0976	NR	-0.17	-0.46
6	3.28	0.48	NR	0.16	0.19
7	2.8	0.8	NR	-0.60	-0.46
8	3.6	0.36	61	0.66	1.00
9	2.5	0.88	104	-1.07	-0.75
10	3.1	0.2	101	-0.13	-0.27
11	2.2	0.7	100	-1.54	-1.34
12	3.1	0.27	97	-0.13	-0.23
13	NR	NR	NR		
14	2.95	0.12	>98	-0.36	-0.92
15	4.6	0.46	62	2.23	2.78
16	3.12	0.31	71	-0.09	-0.16

Statistics

Assigned Value[a]	3.18	0.22
Spike	3.24	0.16
Robust Average	3.11	0.30
NMI Homogeneity Value	3.03	0.20
Median	3.10	0.14
Mean	3.16	
N	14	
Max.	4.6	
Min.	2.2	
Robust SD	0.45	
Robust CV	15%	

[a]The assigned value was calculated as the robust average of the combined results of duplicate pair Samples S1 and S2.


Kernel Density
Density
Result
Results: S1 - Methamphetamine
Assigned Value = 3.18 ± 0.22 ug/wipe
Results (ug/wipe)
Laboratory
11 9 7 14 1 5 10 12 16 2 6 8 4 15 13 3 Md R.A. S H.V.


z-Scores: S1 - Methamphetamine
Target Standard Deviation 0.64 ug/wipe (Equivalent to 20% CV)
z-Score
Laboratory
11 9 7 14 1 5 10 12 16 2 6 8 4 15
-1.5 -1.1 -0.6 -0.4 -0.4 -0.2 -0.1 -0.1 -0.1 0.0 0.2 0.7 0.8 2.2

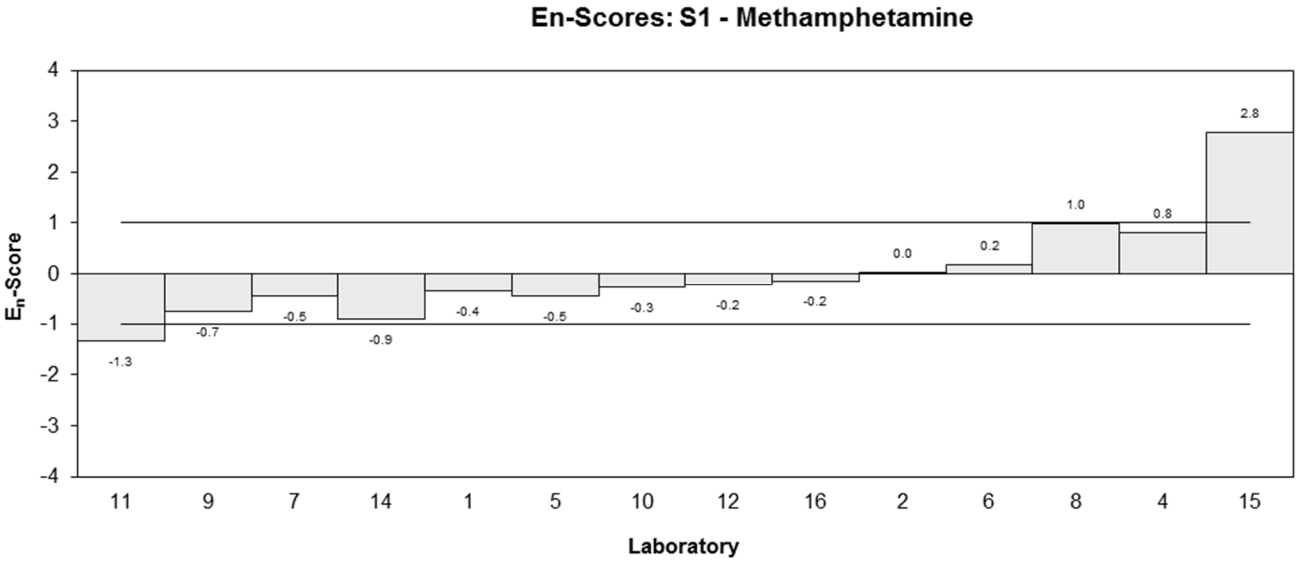

En-Scores: S1 - Methamphetamine
En-Score
Laboratory
11 9 7 14 1 5 10 12 16 2 6 8 4 15
-1.3 -0.7 -0.5 -0.9 -0.4 -0.5 -0.3 -0.2 -0.2 0.0 0.2 1.0 0.8 2.8

Figure 3

Table 7

Sample Details

Sample No.	S2
Matrix.	Wipe
Analyte.	MDMA
Units	ug/wipe as base

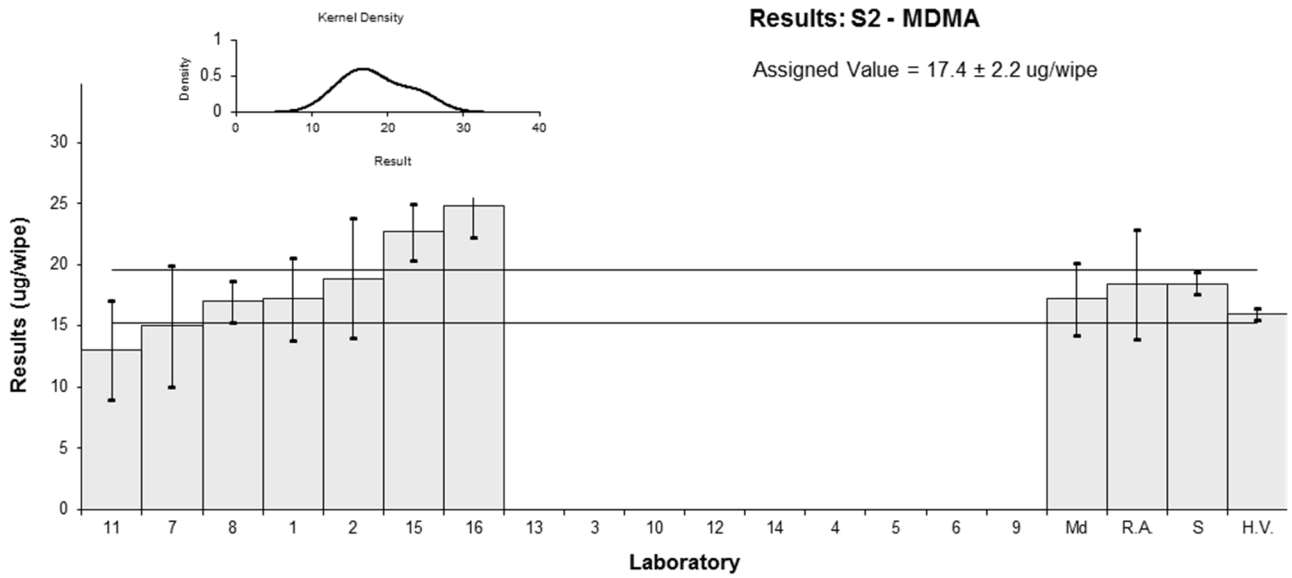
Participant Results

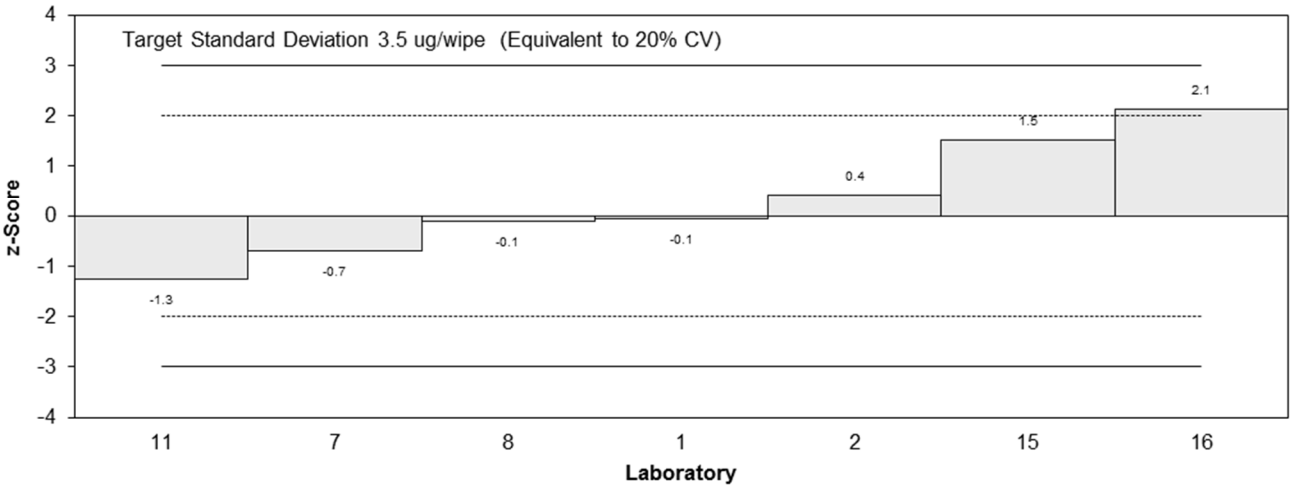
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	17.206	3.441	NR	-0.06	-0.05
2	18.9	4.9	97	0.43	0.28
3	NR	NR	NR		
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	15	5	NR	-0.69	-0.44
8	17.0	1.70	59	-0.11	-0.14
9	NT	NT	NT		
10	NT	NT	NT		
11	13	4	109	-1.26	-0.96
12	NT	NT	NT		
13	NR	NR	NR		
14	NT	NT	NT		
15	22.7	2.27	61	1.52	1.68
16	24.8	2.48	71	2.13	2.23

Statistics

Assigned Value[a]	17.4	2.2
Spike	18.5	0.9
Robust Average	18.4	4.5
NMI Homogeneity Value	15.9	0.4
Median	17.2	3.0
Mean	18.4	
N	7	
Max.	24.8	
Min.	13	
Robust SD	4.7	
Robust CV	26%	

[a]The assigned value was calculated as the robust average of the combined results of duplicate pair Samples S1 and S2.


Kernel Density
Density
Result
Results: S2 - MDMA
Assigned Value = 17.4 ± 2.2 ug/wipe
Results (ug/wipe)
11 7 8 1 2 15 16 13 3 10 12 14 4 5 6 9 Md R.A. S H.V.
Laboratory


z-Scores: S2 - MDMA
Target Standard Deviation 3.5 ug/wipe (Equivalent to 20% CV)
z-Score
-1.3
-0.7
-0.1
-0.1
0.4
1.5
2.1
11 7 8 1 2 15 16
Laboratory

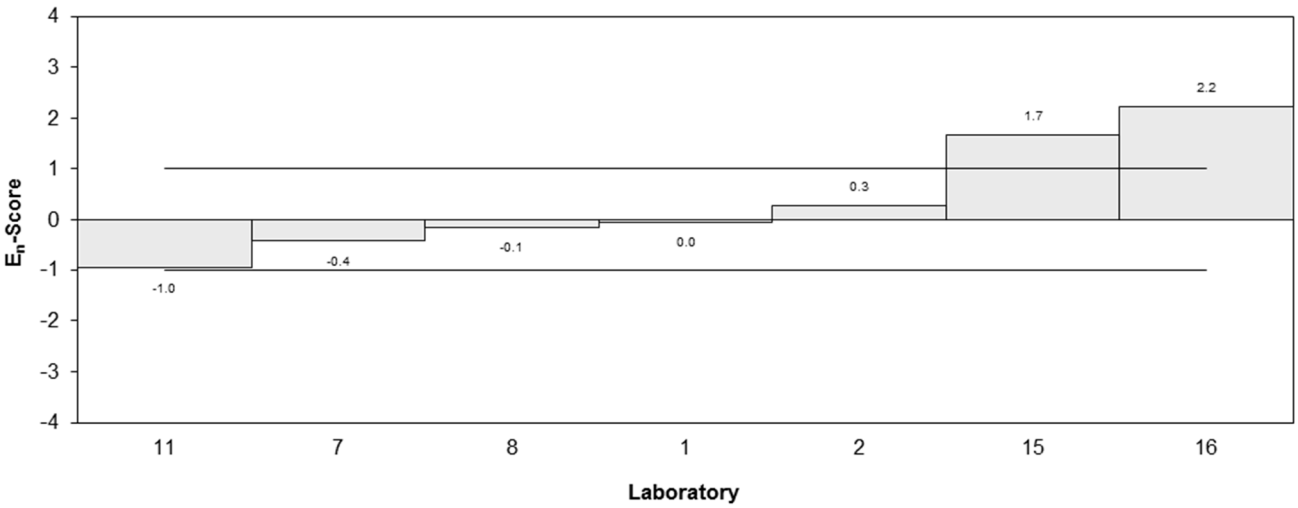

En-Scores: S2 - MDMA
En-Score
-1.0
-0.4
-0.1
0.0
0.3
1.7
2.2
11 7 8 1 2 15 16
Laboratory

Figure 4

Table 8

Sample Details

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

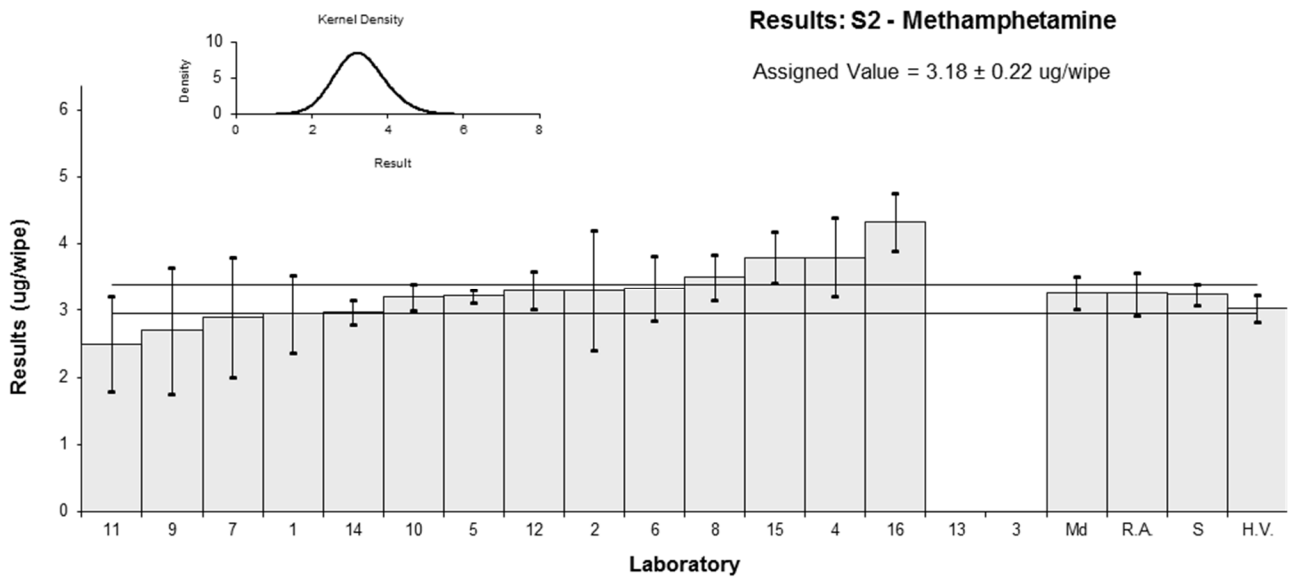
Participant Results

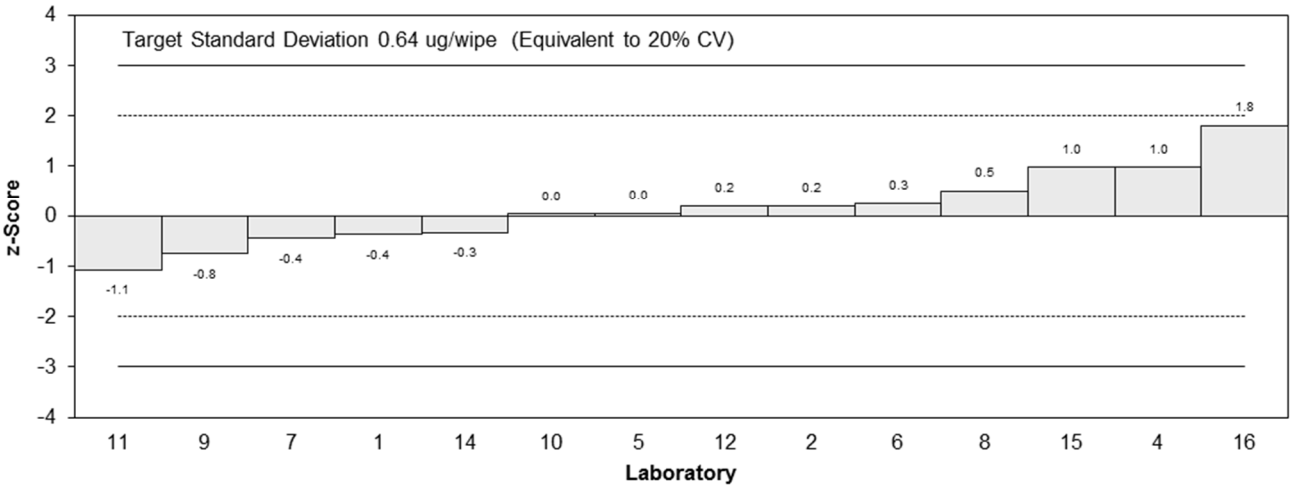
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2.948	0.590	NR	-0.36	-0.37
2	3.3	0.9	120	0.19	0.13
3	NR	NR	NR		
4	3.8	0.6	114	0.97	0.97
5	3.21	0.100	NR	0.05	0.12
6	3.34	0.49	NR	0.25	0.30
7	2.9	0.9	NR	-0.44	-0.30
8	3.5	0.35	61	0.50	0.77
9	2.7	0.94	104	-0.75	-0.50
10	3.2	0.2	101	0.03	0.07
11	2.5	0.7	101	-1.07	-0.93
12	3.3	0.29	97	0.19	0.33
13	NR	NR	NR		
14	2.97	0.19	>98	-0.33	-0.72
15	3.8	0.38	62	0.97	1.41
16	4.32	0.43	71	1.79	2.36

Statistics

Assigned Value[a]	3.18	0.22
Spike	3.24	0.16
Robust Average	3.25	0.32
NMI Homogeneity Value	3.03	0.20
Median	3.26	0.25
Mean	3.27	
N	14	
Max.	4.32	
Min.	2.5	
Robust SD	0.48	
Robust CV	15%	

[a]The assigned value was calculated as the robust average of the combined results of duplicate pair Samples S1 and S2.


Kernel Density
Density
Result
Results: S2 - Methamphetamine
Assigned Value = 3.18 ± 0.22 ug/wipe
Results (ug/wipe)
Laboratory
11 9 7 1 14 10 5 12 2 6 8 15 4 16 13 3 Md R.A. S H.V.


z-Scores: S2 - Methamphetamine
Target Standard Deviation 0.64 ug/wipe (Equivalent to 20% CV)
z-Score
Laboratory
11 9 7 1 14 10 5 12 2 6 8 15 4 16
-1.1 -0.8 -0.4 -0.4 -0.3 0.0 0.0 0.2 0.2 0.3 0.5 1.0 1.0 1.8

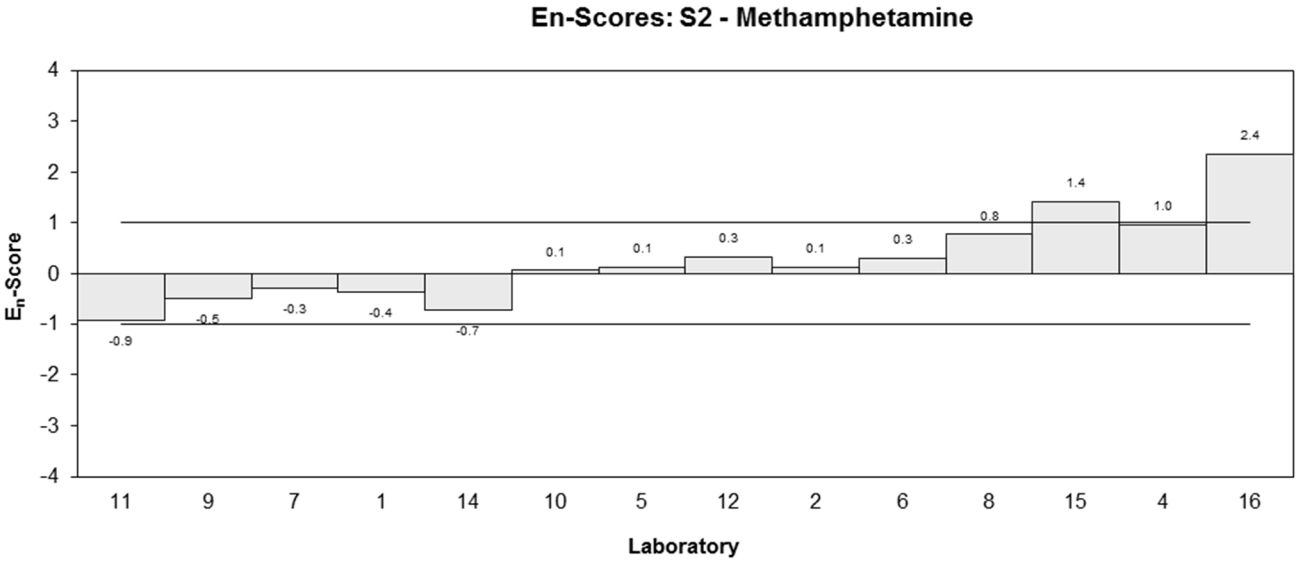

En-Scores: S2 - Methamphetamine
En-Score
Laboratory
11 9 7 1 14 10 5 12 2 6 8 15 4 16
-0.9 -0.5 -0.3 -0.4 -0.7 0.1 0.1 0.3 0.1 0.3 0.8 1.4 1.0 2.4

Figure 5

Table 9

Sample Details

Sample No.	S3
Matrix.	Wipe
Analyte.	MDMA
Units	ug/wipe as base

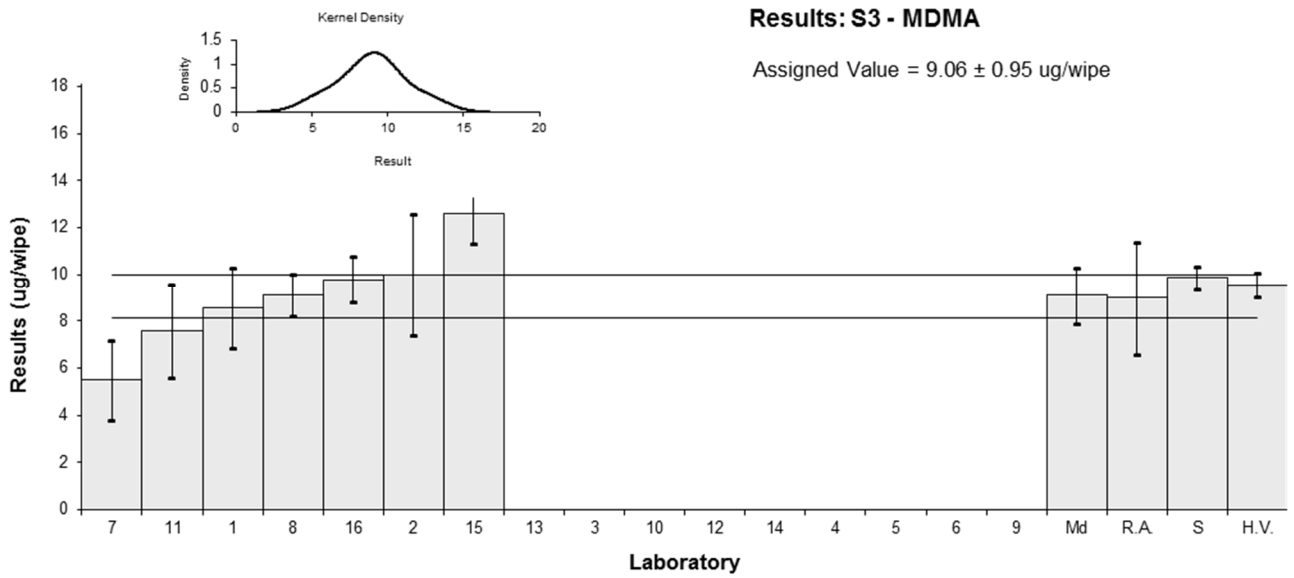
Participant Results

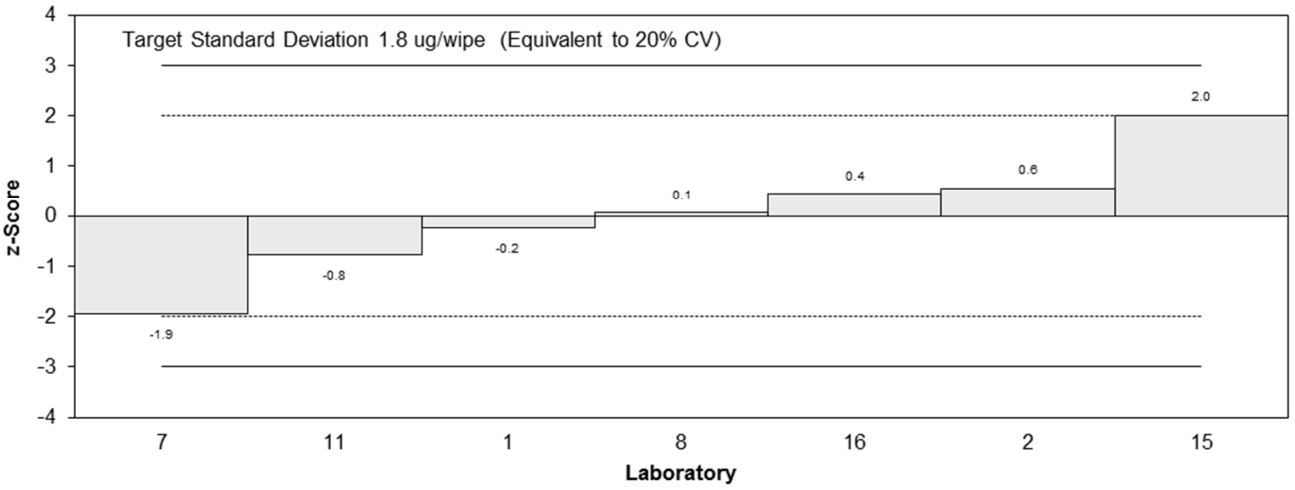
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	8.576	1.715	NR	-0.27	-0.25
2	10.0	2.6	108	0.52	0.34
3	NR	NR	NR		
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	5.5	1.7	NR	-1.96	-1.83
8	9.1	0.91	59	0.02	0.03
9	NT	NT	NT		
10	NT	NT	NT		
11	7.6	2	105	-0.81	-0.66
12	NT	NT	NT		
13	NR	NR	NR		
14	NT	NT	NT		
15	12.6	1.26	60	1.95	2.24
16	9.79	0.97	71	0.40	0.54

Statistics

Assigned Value[a]	9.06	0.95
Spike	9.86	0.49
NMI Homogeneity Value	9.56	0.52
Robust Average[a]	9.0	2.4
Median	9.1	1.2
Mean	9.0	
N	7	
Max.	12.6	
Min.	5.5	
Robust SD	2.5	
Robust CV	28%	

[a]The assigned value was calculated as the robust average of the combined results of duplicate pair Samples S3 and S4.


Kernel Density
Density
Result
Results: S3 - MDMA
Assigned Value = 9.06 ± 0.95 ug/wipe
Results (ug/wipe)
7 11 1 8 16 2 15 13 3 10 12 14 4 5 6 9 Md R.A. S H.V.
Laboratory


z-Scores: S3 - MDMA
Target Standard Deviation 1.8 ug/wipe (Equivalent to 20% CV)
z-Score
-1.9
-0.8
-0.2
0.1
0.4
0.6
2.0
7 11 1 8 16 2 15
Laboratory

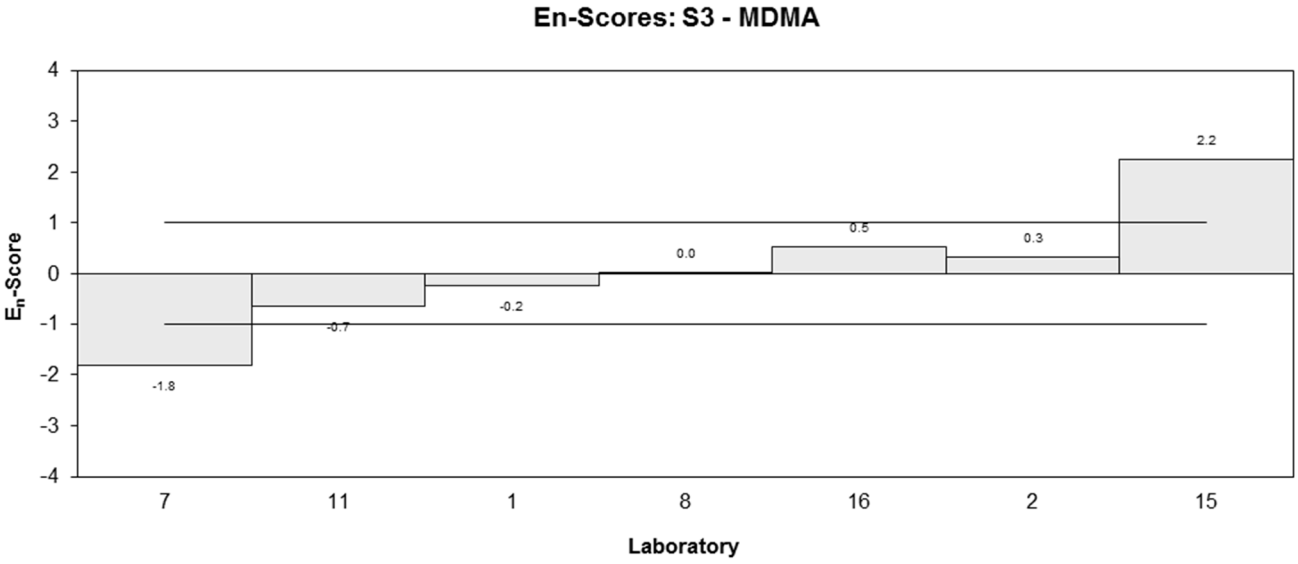

En-Scores: S3 - MDMA
En-Score
-1.8
-0.7
-0.2
0.0
0.5
0.3
2.2
7 11 1 8 16 2 15
Laboratory

Figure 6

Table 10

Sample Details

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

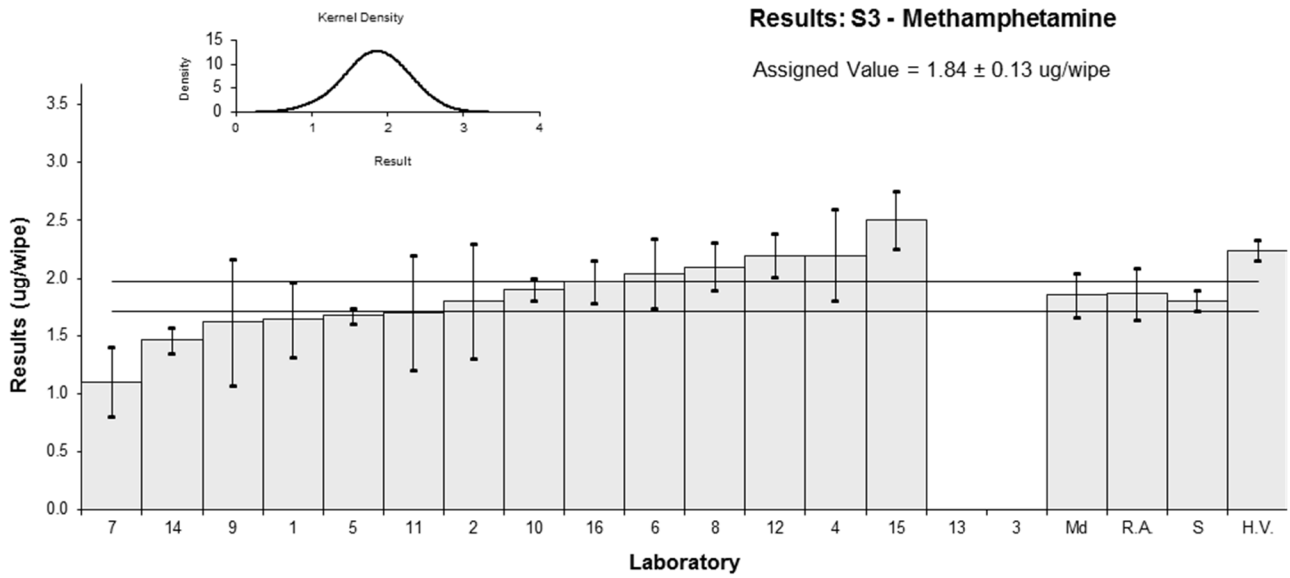
Participant Results

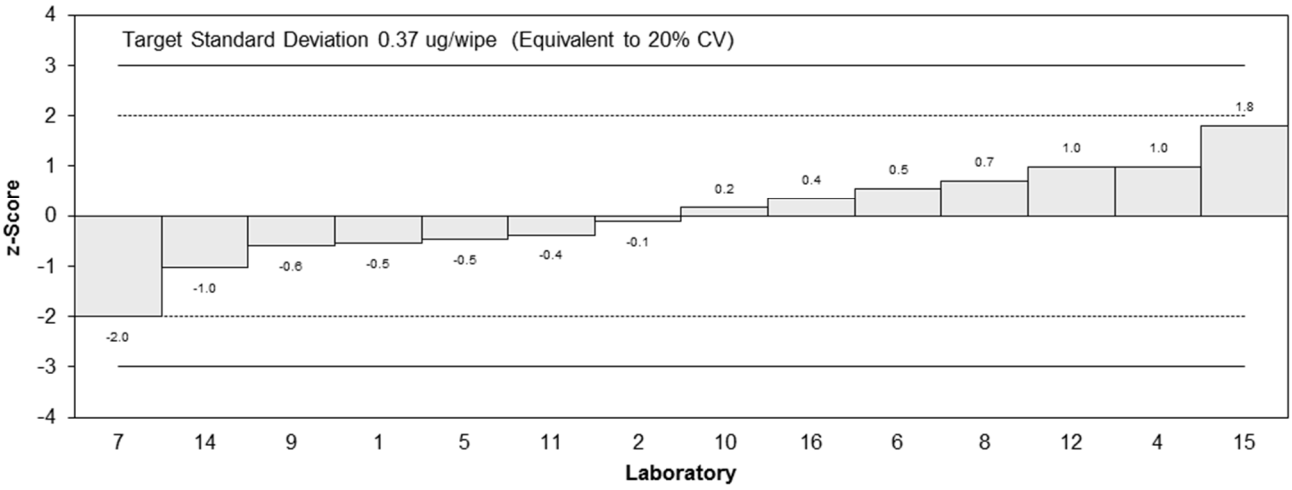
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.641	0.328	NR	-0.54	-0.56
2	1.8	0.5	123	-0.11	-0.08
3	NR	NR	NR		
4	2.2	0.4	114	0.98	0.86
5	1.67	0.0633	NR	-0.46	-1.18
6	2.04	0.30	NR	0.54	0.61
7	1.1	0.3	NR	-2.01	-2.26
8	2.1	0.21	61	0.71	1.05
9	1.62	0.55	104	-0.60	-0.39
10	1.9	0.1	101	0.16	0.37
11	1.7	0.5	98	-0.38	-0.27
12	2.2	0.19	97	0.98	1.56
13	NR	NR	NR		
14	1.46	0.11	>98	-1.03	-2.23
15	2.5	0.25	62	1.79	2.34
16	1.97	0.19	71	0.35	0.56

Statistics

Assigned Value[a]	1.84	0.13
Spike	1.80	0.09
NMI Homogeneity Value	2.24	0.09
Robust Average[a]	1.86	0.23
Median	1.85	0.19
Mean	1.85	
N	14	
Max.	2.5	
Min.	1.1	
Robust SD	0.34	
Robust CV	18%	

[a]The assigned value was calculated as the robust average of the combined results of duplicate pair Samples S3 and S4.


Kernel Density
Density
Result
Results: S3 - Methamphetamine
Assigned Value = 1.84 ± 0.13 ug/wipe
Results (ug/wipe)
Laboratory
7 14 9 1 5 11 2 10 16 6 8 12 4 15 13 3 Md R.A. S H.V.


z-Scores: S3 - Methamphetamine
Target Standard Deviation 0.37 ug/wipe (Equivalent to 20% CV)
z-Score
Laboratory
7 14 9 1 5 11 2 10 16 6 8 12 4 15
-2.0 -1.0 -0.6 -0.5 -0.5 -0.4 -0.1 0.2 0.4 0.5 0.7 1.0 1.0 1.8

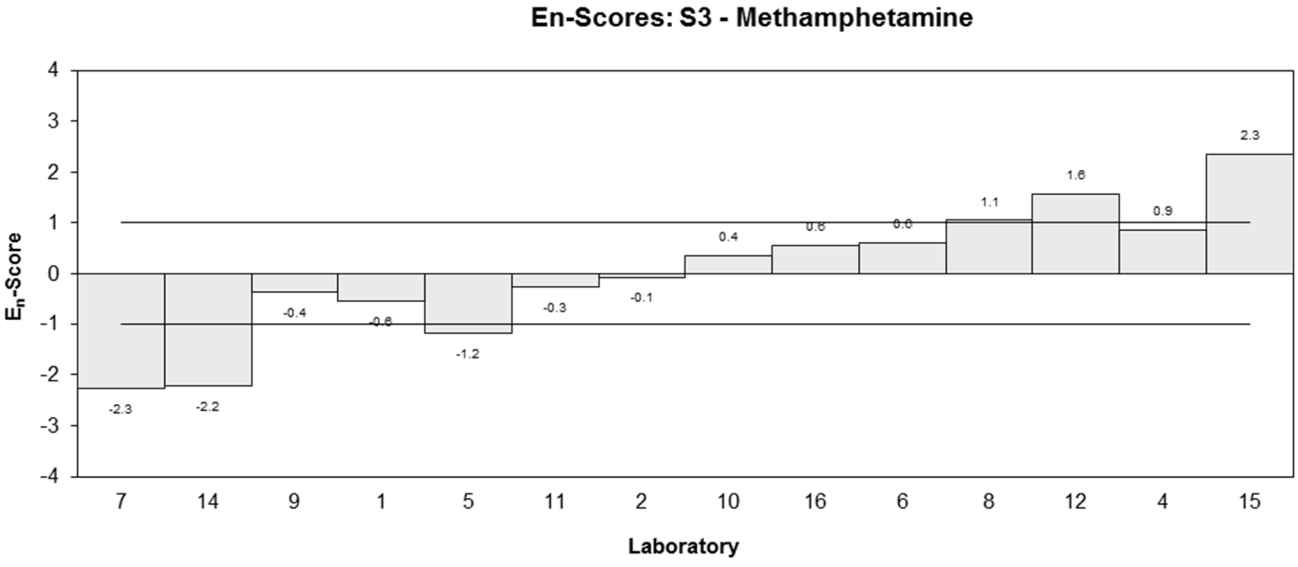

En-Scores: S3 - Methamphetamine
En-Score
Laboratory
7 14 9 1 5 11 2 10 16 6 8 12 4 15
-2.3 -2.2 -0.4 -0.6 -1.2 -0.3 -0.1 0.4 0.6 0.6 1.1 1.6 0.9 2.3

Figure 7

Table 11

Sample Details

Sample No.	S4
Matrix.	Wipe
Analyte.	MDMA
Units	ug/wipe as base

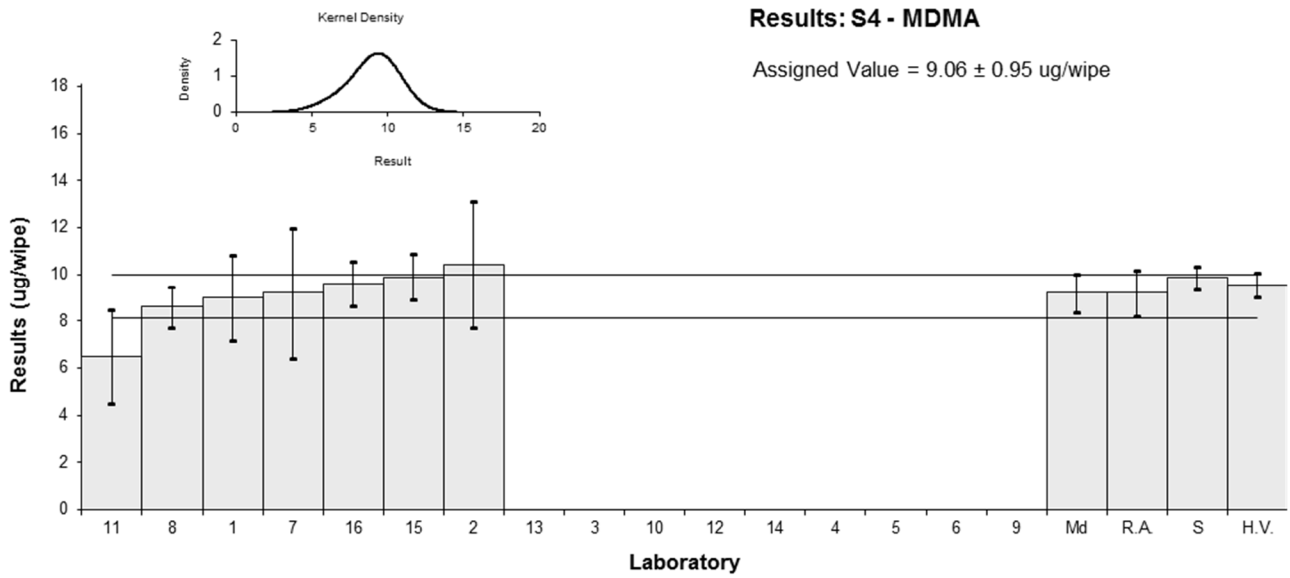
Participant Results

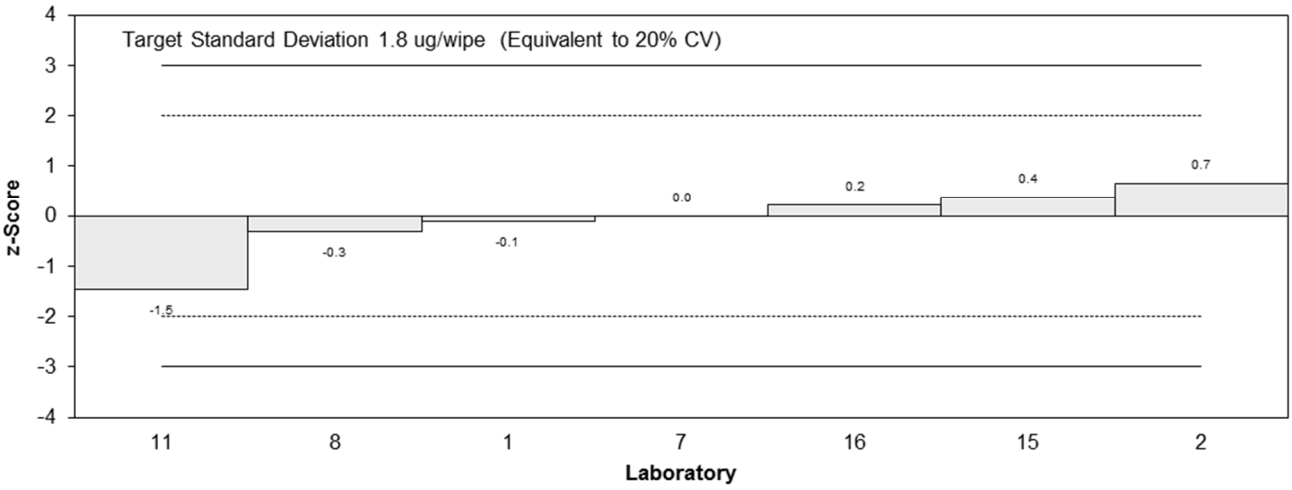
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	9.007	1.809	NR	-0.03	-0.03
2	10.4	2.7	109	0.74	0.47
3	NR	NR	NR		
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	9.2	2.8	NR	0.08	0.05
8	8.6	0.86	59	-0.25	-0.36
9	NT	NT	NT		
10	NT	NT	NT		
11	6.5	2	108	-1.41	-1.16
12	NT	NT	NT		
13	NR	NR	NR		
14	NT	NT	NT		
15	9.9	0.99	60	0.46	0.61
16	9.61	0.96	71	0.30	0.41

Statistics

Assigned Value[a]	9.06	0.95
Spike	9.86	0.49
NMI Homogeneity Value	9.56	0.52
Robust Average[a]	9.20	0.96
Median	9.20	0.82
Mean	9.03	
N	7	
Max.	10.4	
Min.	6.5	
Robust SD	1	
Robust CV	11%	

[a]The assigned value was calculated as the robust average of the combined results of duplicate pair Samples S3 and S4.


Kernel Density
Density
Result
Results: S4 - MDMA
Assigned Value = 9.06 ± 0.95 ug/wipe
Results (ug/wipe)
Laboratory
11 8 1 7 16 15 2 13 3 10 12 14 4 5 6 9 Md R.A. S H.V.


z-Scores: S4 - MDMA
Target Standard Deviation 1.8 ug/wipe (Equivalent to 20% CV)
z-Score
-1.5
-0.3
-0.1
0.0
0.2
0.4
0.7
Laboratory
11 8 1 7 16 15 2

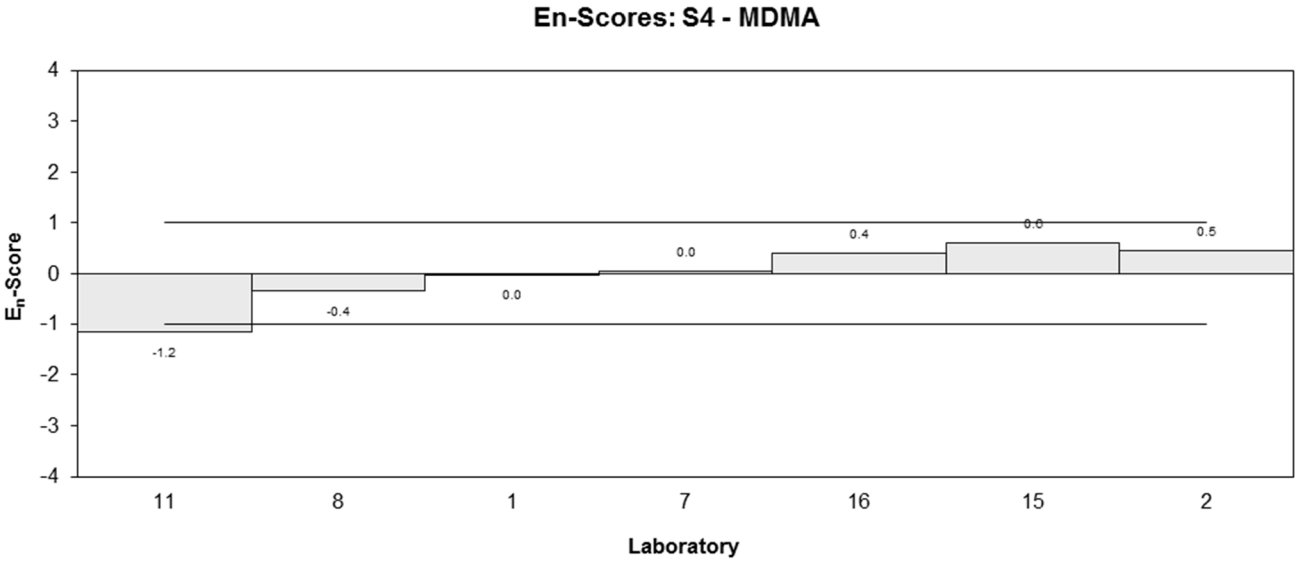

En-Scores: S4 - MDMA
En-Score
-1.2
-0.4
0.0
0.0
0.4
0.0
0.5
Laboratory
11 8 1 7 16 15 2

Figure 8

Table 12

Sample Details

Sample No.	S4
Matrix.	Wipe
Analyte.	Methamphetamine
Units	ug/wipe as base

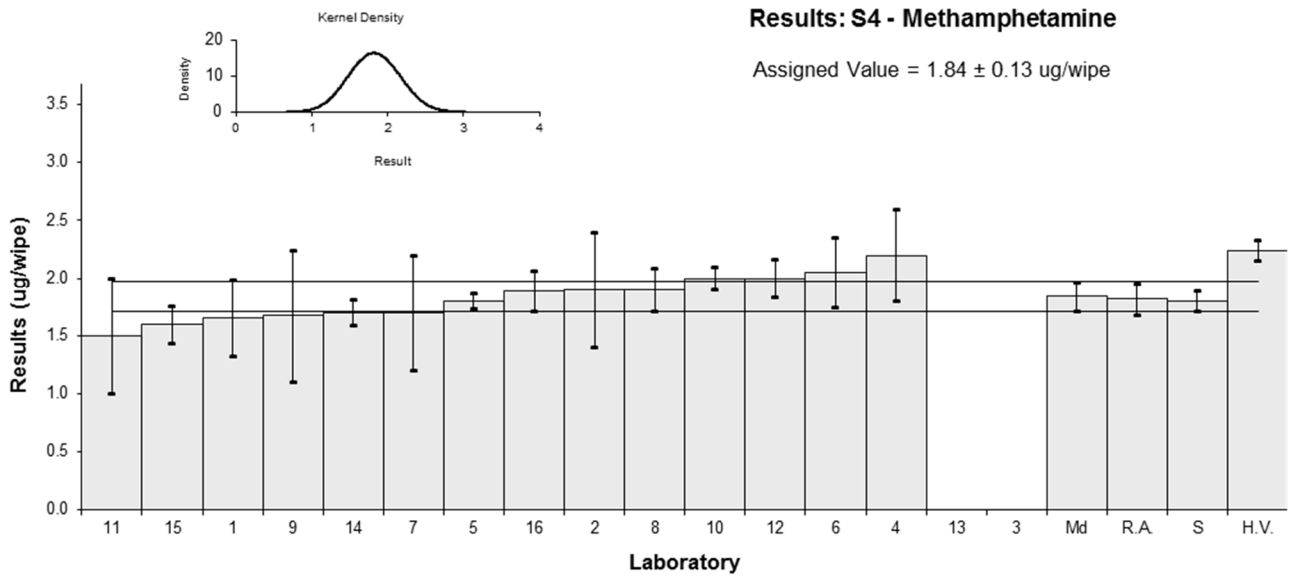
Participant Results

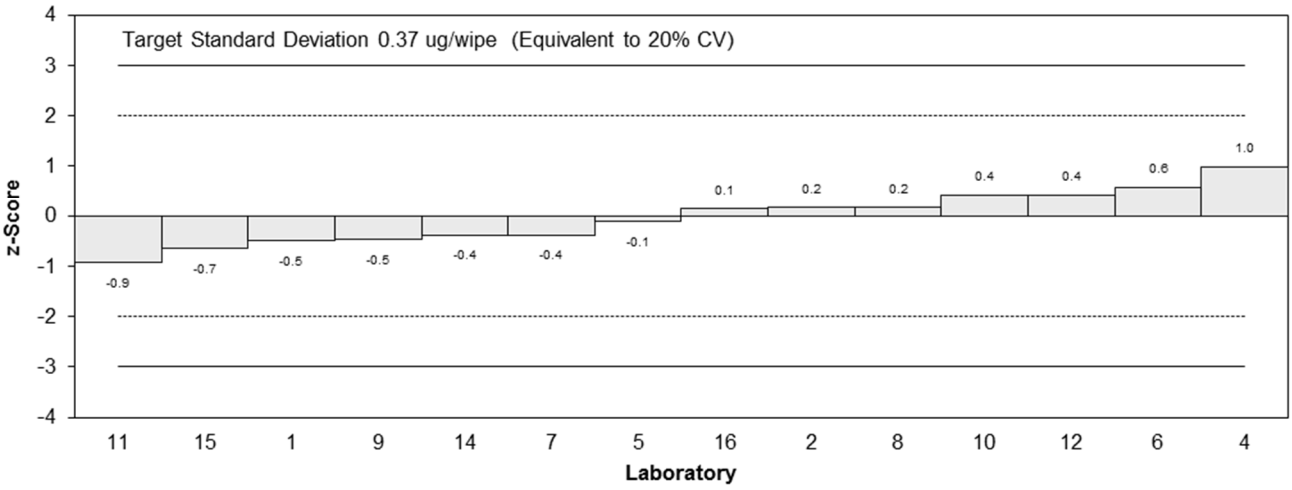
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.655	0.331	NR	-0.50	-0.52
2	1.9	0.5	121	0.16	0.12
3	NR	NR	NR		
4	2.2	0.4	114	0.98	0.86
5	1.80	0.0642	NR	-0.11	-0.28
6	2.05	0.30	NR	0.57	0.64
7	1.7	0.5	NR	-0.38	-0.27
8	1.9	0.19	61	0.16	0.26
9	1.67	0.57	104	-0.46	-0.29
10	2.0	0.1	101	0.43	0.98
11	1.5	0.5	102	-0.92	-0.66
12	2.0	0.17	97	0.43	0.75
13	NR	NR	NR		
14	1.70	0.11	>98	-0.38	-0.82
15	1.6	0.16	62	-0.65	-1.16
16	1.89	0.18	71	0.14	0.23

Statistics

Assigned Value[a]	1.84	0.13
Spike	1.80	0.09
NMI Homogeneity Value	2.24	0.09
Robust Average[a]	1.82	0.14
Median	1.84	0.13
Mean	1.83	
N	14	
Max.	2.2	
Min.	1.5	
Robust SD	0.22	
Robust CV	12%	

[a]The assigned value was calculated as the robust average of the combined results of duplicate pair Samples S3 and S4.


Kernel Density
Density
Result
Results: S4 - Methamphetamine
Assigned Value = 1.84 ± 0.13 ug/wipe
Results (ug/wipe)
Laboratory
11 15 1 9 14 7 5 16 2 8 10 12 6 4 13 3 Md R.A. S H.V.


z-Scores: S4 - Methamphetamine
Target Standard Deviation 0.37 ug/wipe (Equivalent to 20% CV)
z-Score
Laboratory
11 15 1 9 14 7 5 16 2 8 10 12 6 4
-0.9 -0.7 -0.5 -0.5 -0.4 -0.4 -0.1 0.1 0.2 0.2 0.4 0.4 0.6 1.0

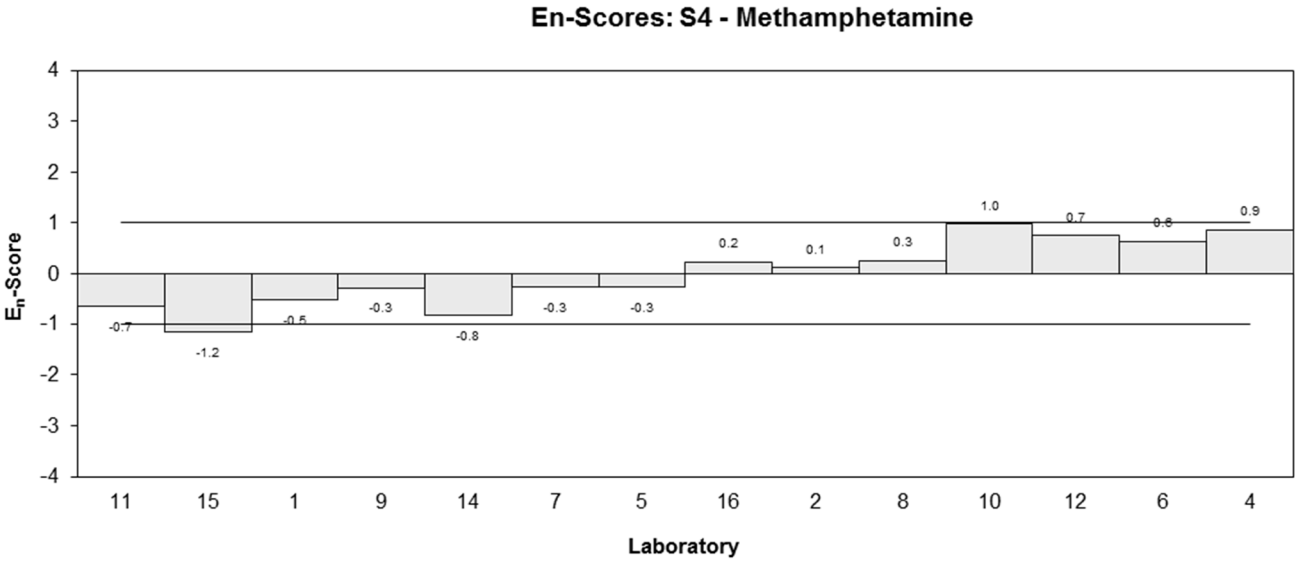

En-Scores: S4 - Methamphetamine
En-Score
Laboratory
11 15 1 9 14 7 5 16 2 8 10 12 6 4
-0.7 -1.2 -0.5 -0.3 -0.8 -0.3 -0.3 0.2 0.1 0.3 1.0 0.7 0.6 0.9

Figure 9

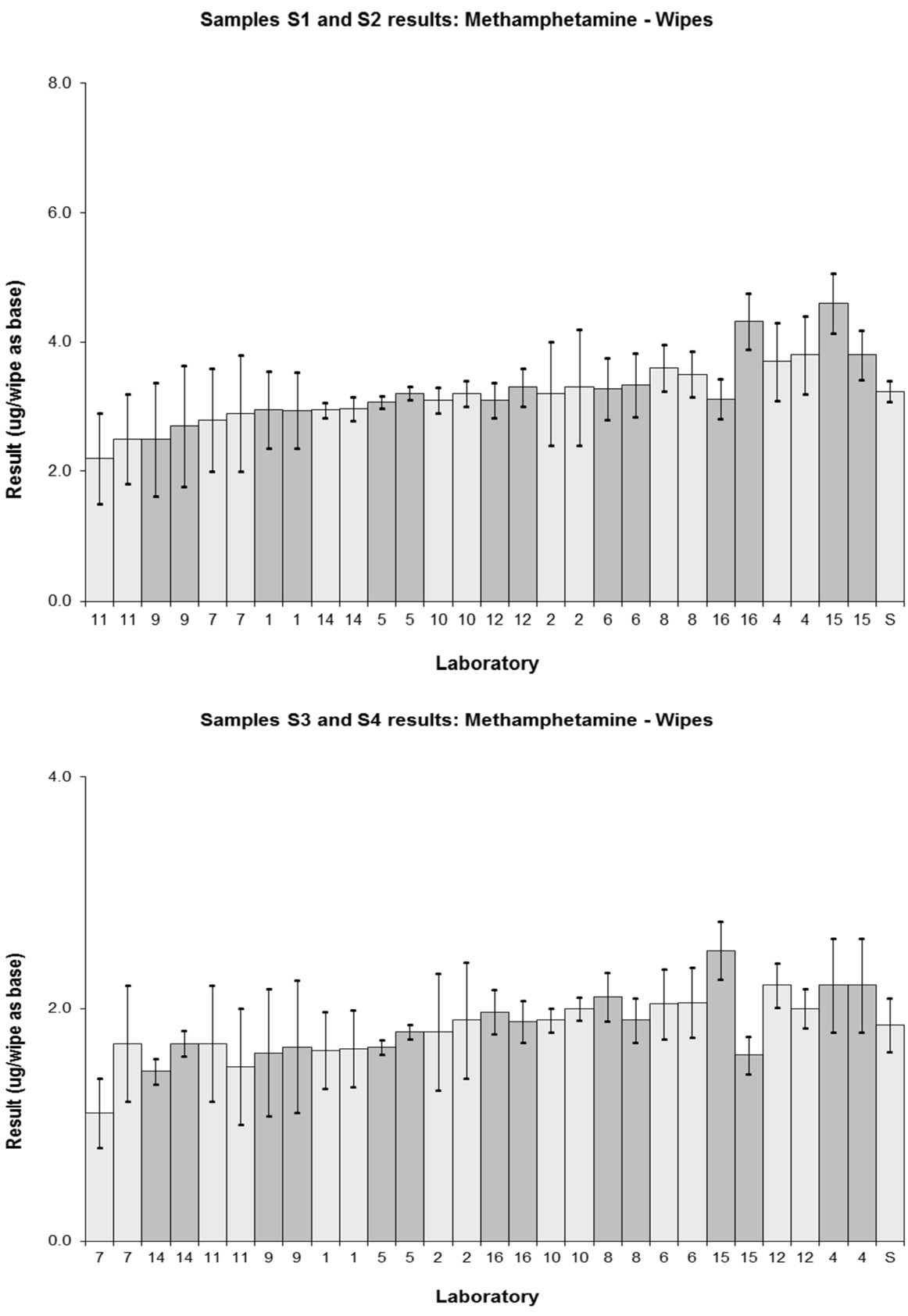
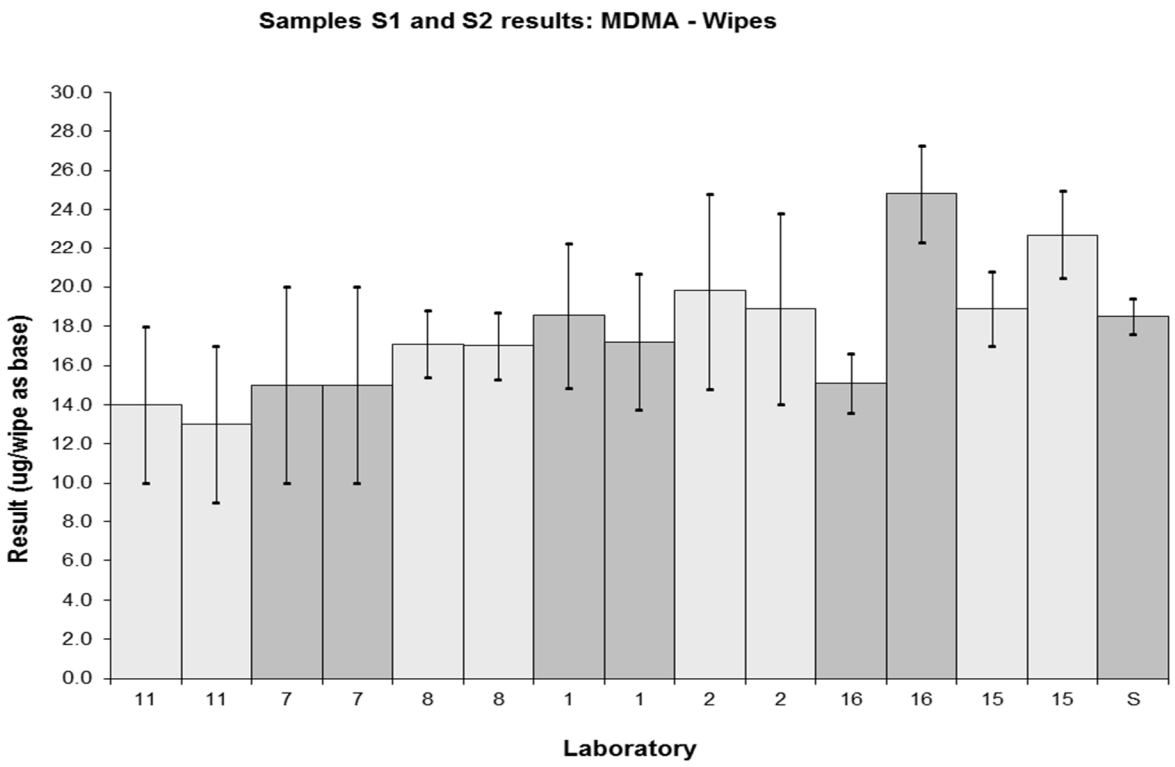


Figure 10 Results for methamphetamine in duplicate Samples S1/S2 and S3/S4



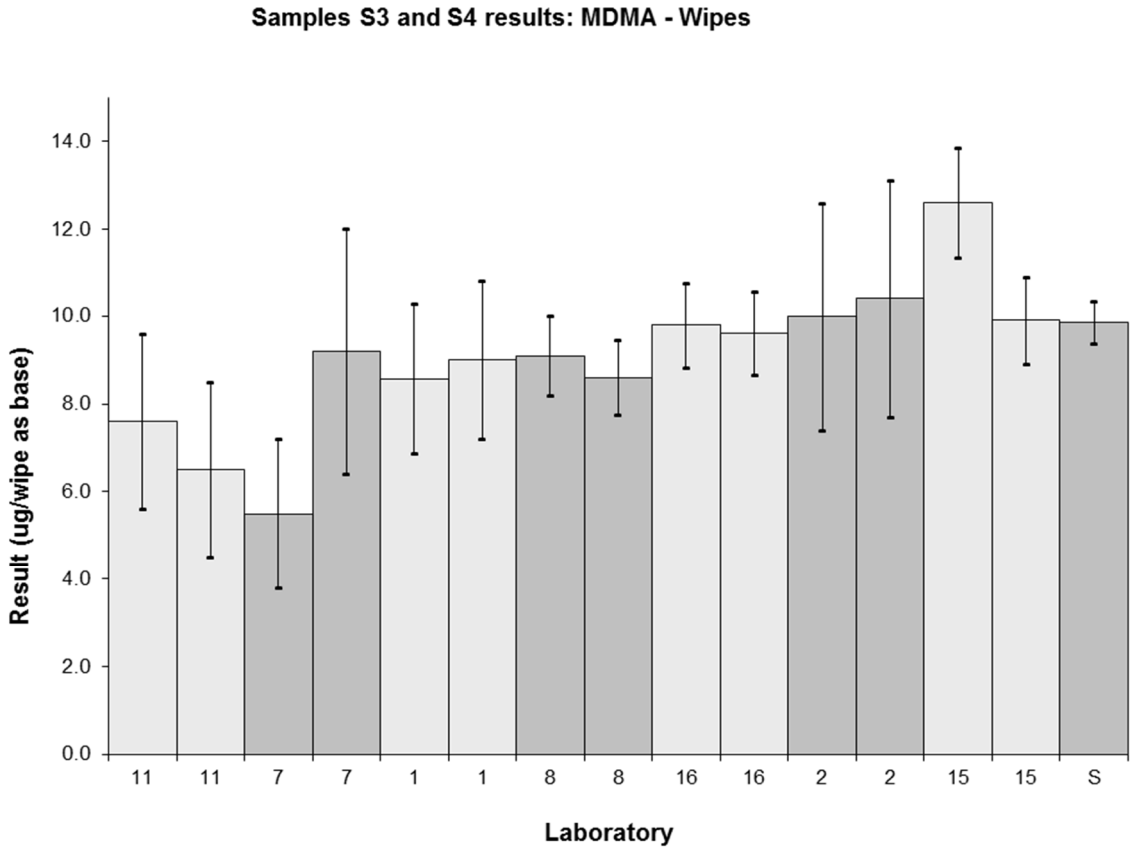


Figure 11 Results for MDMA in duplicate Samples S1/S2 and S3/S4

6 DISCUSSION OF RESULTS

6.1 Assigned Value

Assigned values for methamphetamine and MDMA in the four wipes samples were the robust averages of participants' results and were in good agreement with the spike and homogeneity values.

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 measurement uncertainty associated with their results. All results were reported with an expanded measurement uncertainty, indicating that laboratories have addressed this requirement of ISO 17025.[6] The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Table 2.

The magnitude of reported uncertainties was within the range of 3% to 35% relative.

Laboratories with a satisfactory z-score and an unsatisfactory E_n-score are likely to have underestimated the expanded uncertainty associated with the result.

In some cases the results were reported with an inappropriate number of significant figures. 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 1.655 ± 0.331 µg/wipe, the recommended format is 1.66 ± 0.33 µg/wipe).[7]

6.3 z-Score

A target standard deviation equivalent to 20% PCV was used to calculate z-scores. Target SDs, the between-laboratory coefficient of variation predicted by the Thomson - Horwitz equation[8] and participants' coefficient of variation obtained in this study are presented in Table 13.

A summary of z-scores by laboratory is presented in Figure 12.

81 of 84 numeric results (96%) returned a satisfactory z-score with $|z| \leq 2$.

- Participants with the lab code number **1, 2, 4, 5, 6, 8, 9, 10, 11, 12** and **14** returned satisfactory scores for all analytes for which they tested and reported results;
- Three participants each returned one questionable z-score;
- Two participants did not submit numeric results for any analyte in any sample;
- There were no reported results that returned an unsatisfactory z-score of greater or equal to 3.

Table 13 Target SD (as PCV), Thompson Horwitz CV and Participants CV

Sample	Analyte	Assigned Value (µg/wipe as base)	Target SD (as PCV)	Thompson Horwitz CV	Participants CV
S1	Methamphetamine	3.18	20%	22%	15%
S1	MDMA	17.4	20%	22%	15%
S2	Methamphetamine	3.18	20%	22%	15%
S2	MDMA	17.4	20%	22%	26%
S3	Methamphetamine	1.84	20%	22%	18%
S3	MDMA	9.06	20%	22%	28%
S4	Methamphetamine	1.84	20%	22%	12%
S4	MDMA	9.06	20%	22%	11%

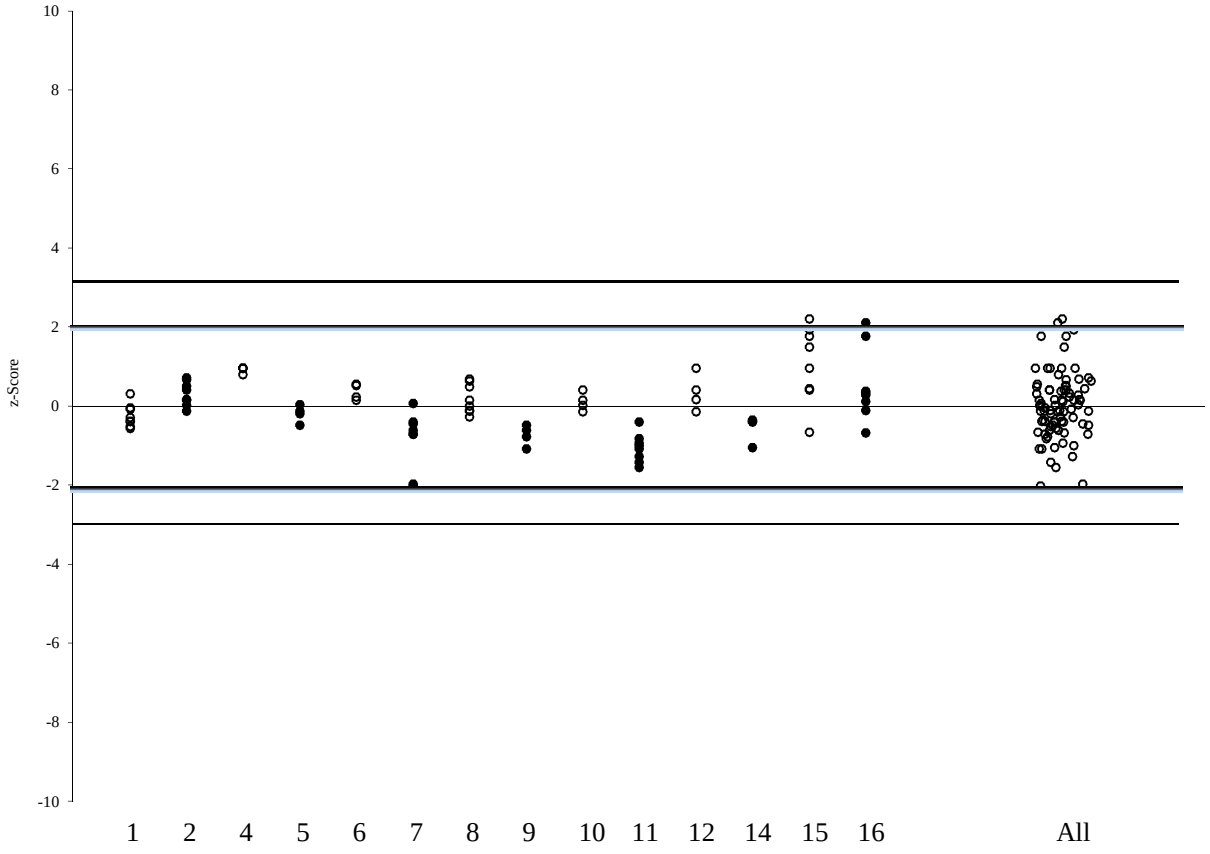


Figure 12 Summary of participants' z-score.

6.4 E_n-Score

The dispersal of participants' E_n-scores is graphically presented in Figure 13. Where a laboratory did not report an expanded uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score.

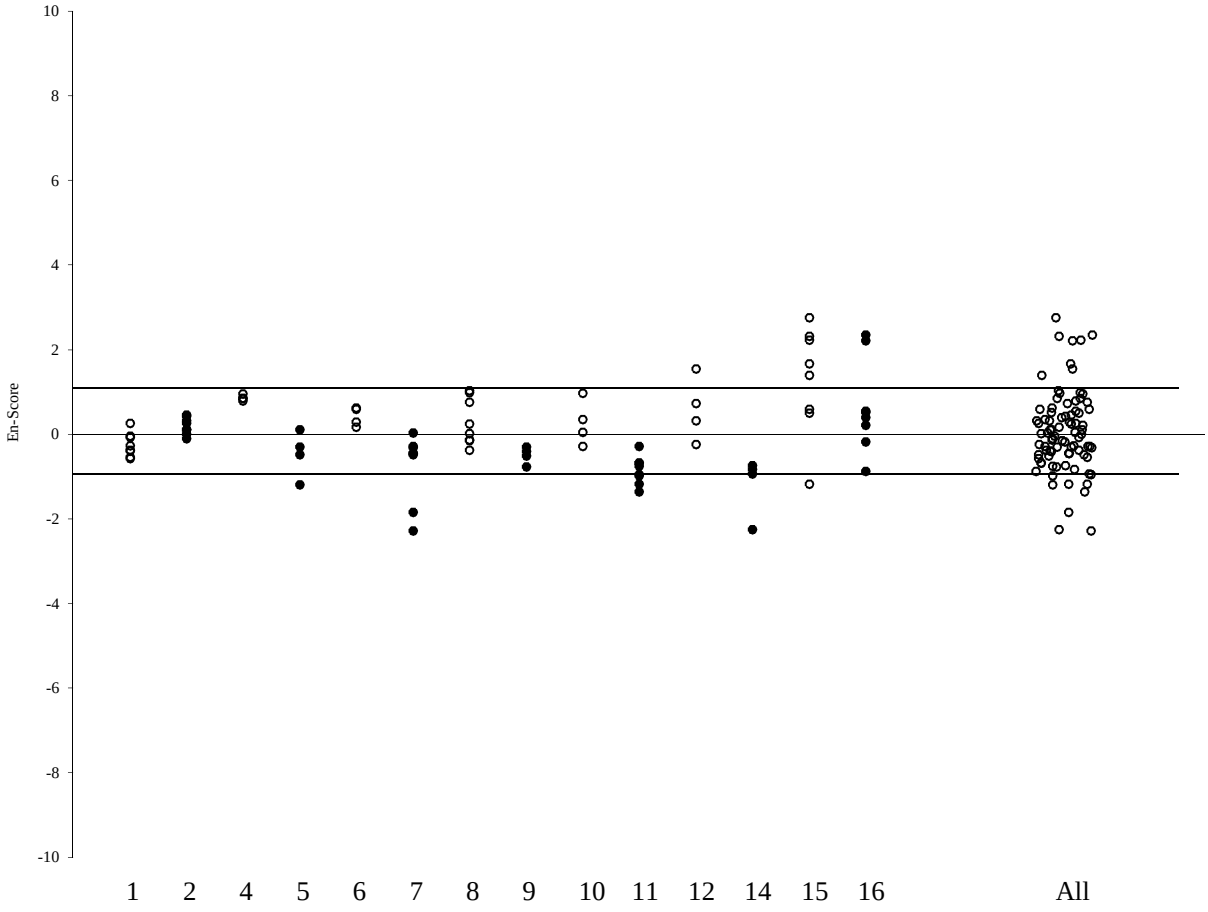


Figure 13 Summary of participants' E_n-Score

68 of 84 numeric results (81%) returned a satisfactory E_n-score with $|E_n| \leq 1$.

- Six participants – **1, 2, 4, 6, 9** and **10** returned satisfactory E_n-scores for all analytes for which they tested and reported results;
- Eight laboratories returned at least one questionable E_n-score; and
- There were no participants who returned an unsatisfactory E_n-score for all samples.

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 accreditation status, participants' methods and reference standards is presented Table 14 and Figures 14 to 17.

No trend with any particular sample preparation method or instrumental technique was evident.

Table 14 Summary of Participants, Accreditation Status, Methods and Reference Standards Used

Accreditation status	**Laboratory Code**
Yes to ISO 17025	1 2 4 5 6 7 9 10 11 12 13*
Other/Unspecified	8 14 15
No	3* 16

Sample Treatment	**Laboratory Code**
Rotary mixer/shaking/tumbling	1 2 4 6 7 9 10 11 12 14 16
Vortexing	8 15
Sonication	11
pH adjustment	5 7 9 11
Other/None	3* 5 13*

Desorption Solution	**Laboratory Code**
Water	8 15 16
Sulfuric acid	1 2 4 5 6 7 9 10 12 14
Methanol then hexane/triethylamine	13*
0.1 M formic acid	11
Methanol	3*

Instrumental technique	**Laboratory Code**
LC/LCMS/LCMSMS/LCMS QQQ	1 2 4 6 8 9 10 11 14 15 16
UPLC/UPLC-MSMS	8 12 15
GCMS	3* 5 7 13*

Sources of Calibration Standard	**Laboratory Code**
NMI Australia	6 8 15 16
Lipomed	12 14
Cerilliant	4 5 9 10
Chiron	11
Other/none/unspecified	1 2 3* 7 13*

*Laboratories 3 and 13 only performed qualitative analysis.

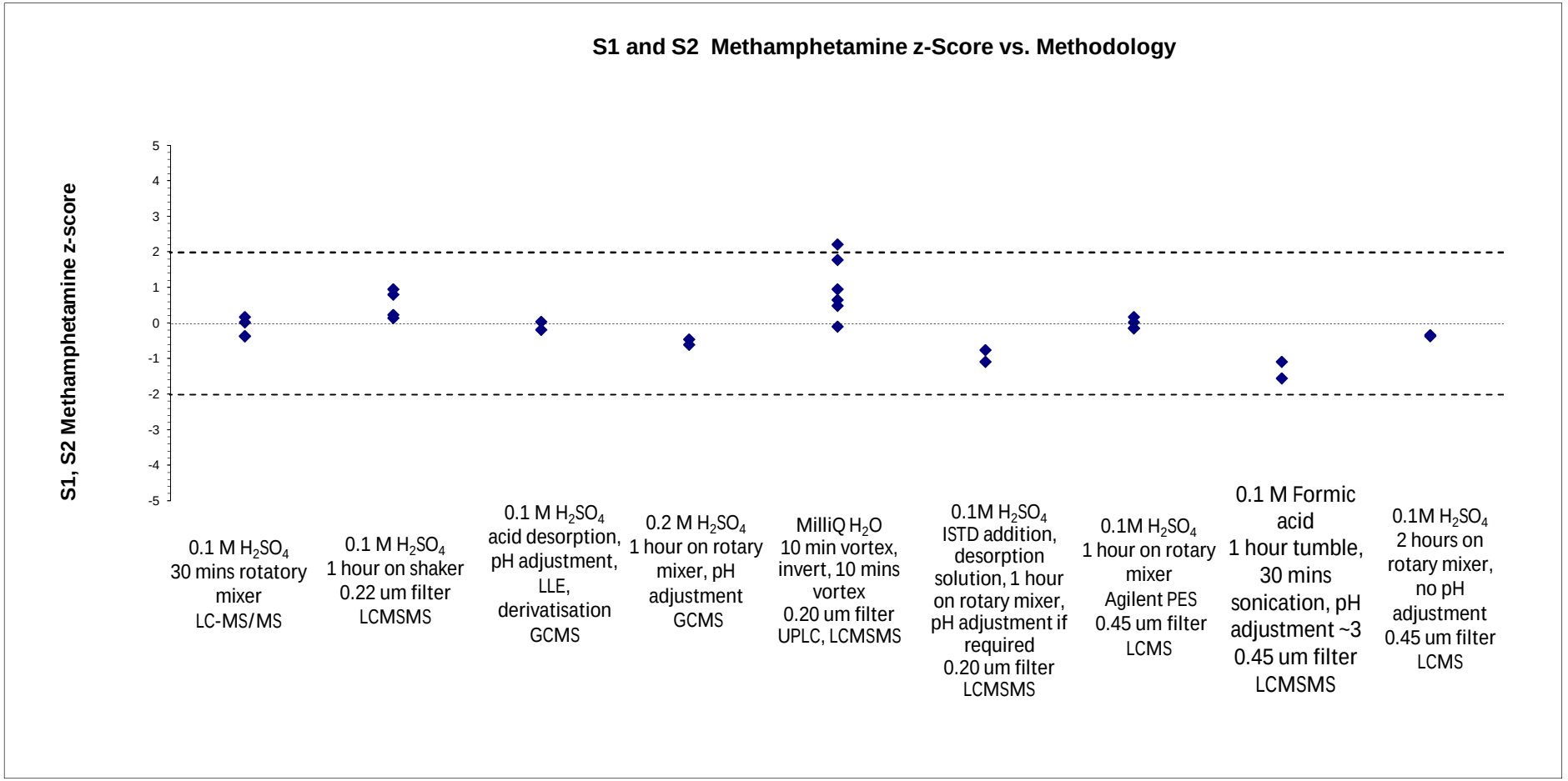


Figure 14 Participants' Performance for Methamphetamine in S1 and S2 versus Methodology

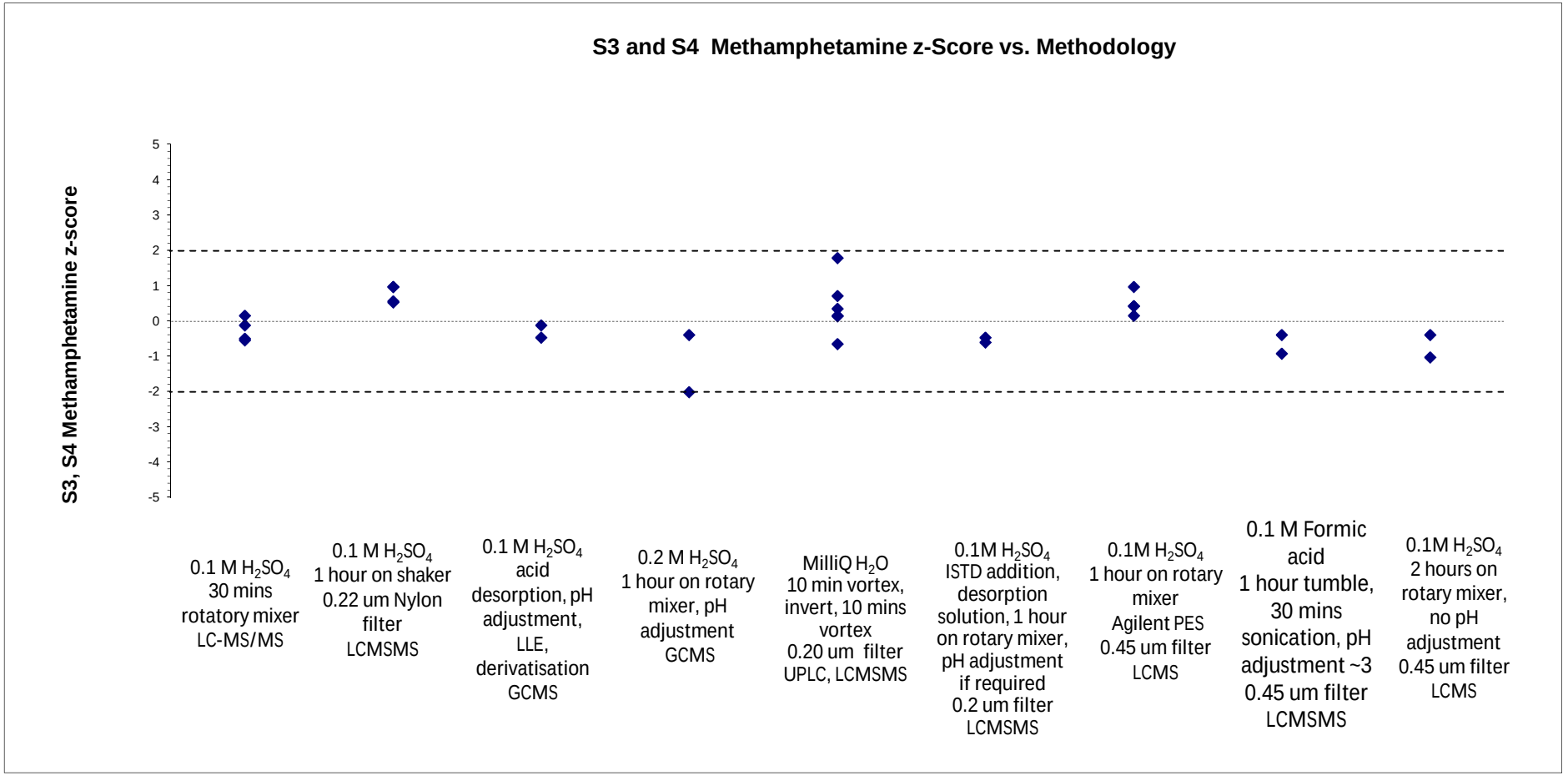


Figure 15 Participants' Performance for Methamphetamine in S3 and S4 versus Methodology

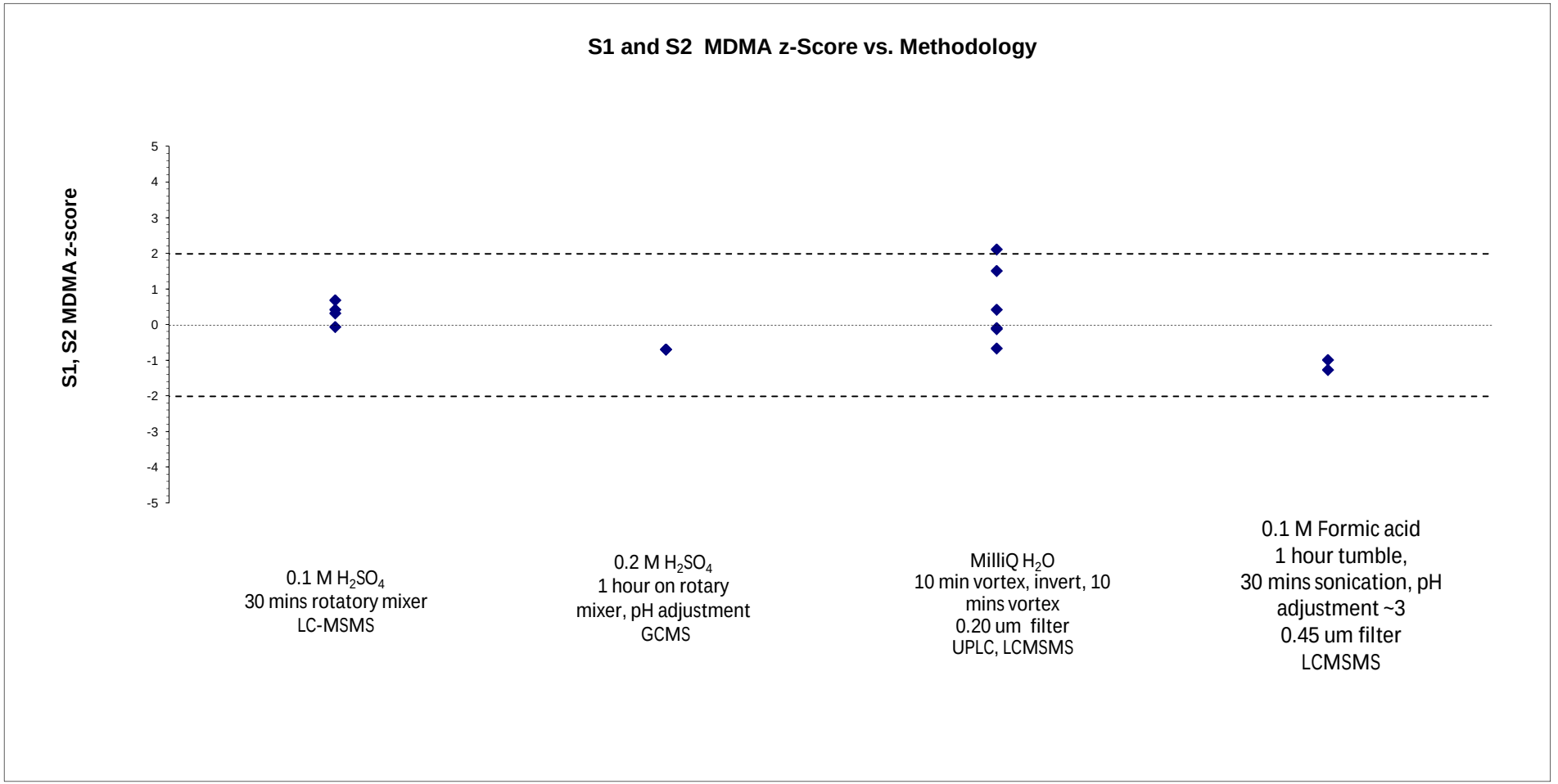


Figure 16 Participants' Performance for MDMA in S1 and S2 versus Methodology

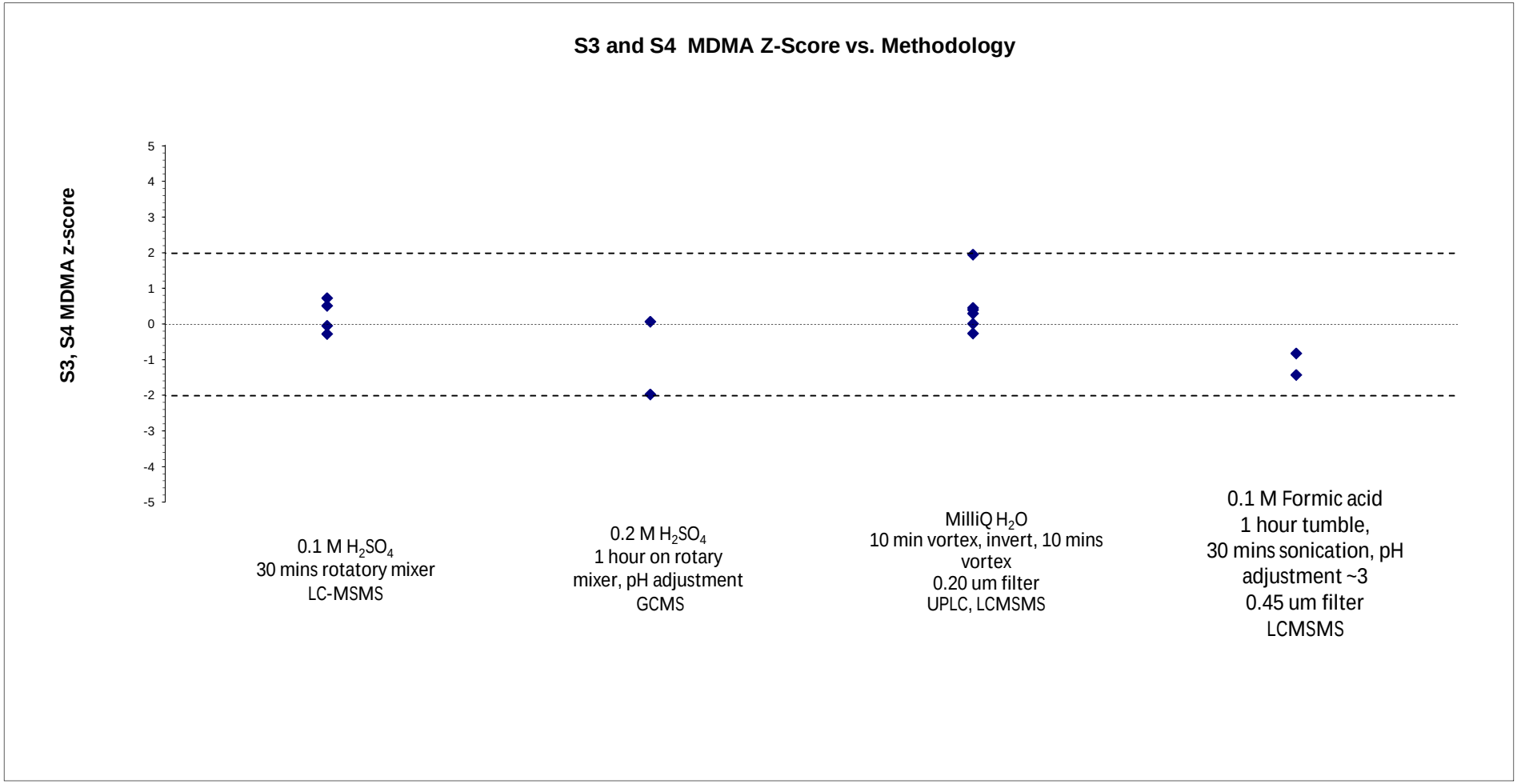


Figure 17 Participants' Performance for MDMA in S3 and S4 versus Methodology

6.6 Participants' Within – Laboratory Repeatability

The study included pair duplicate Samples S1/S2 and S3/S4. The same target standard deviation was used to calculate z-scores for analytes in both samples of each pair. This allowed evaluation of the within laboratory repeatability of laboratories.

Scatter plots of z-scores for S1 and S2 are presented in Figure 18 and for S3 and S4 are presented in Figure 19. Most laboratories are plotted in the upper-right or lower-left quadrants. This is consistent with systematic bias being the major contributor to the observed variation in results.

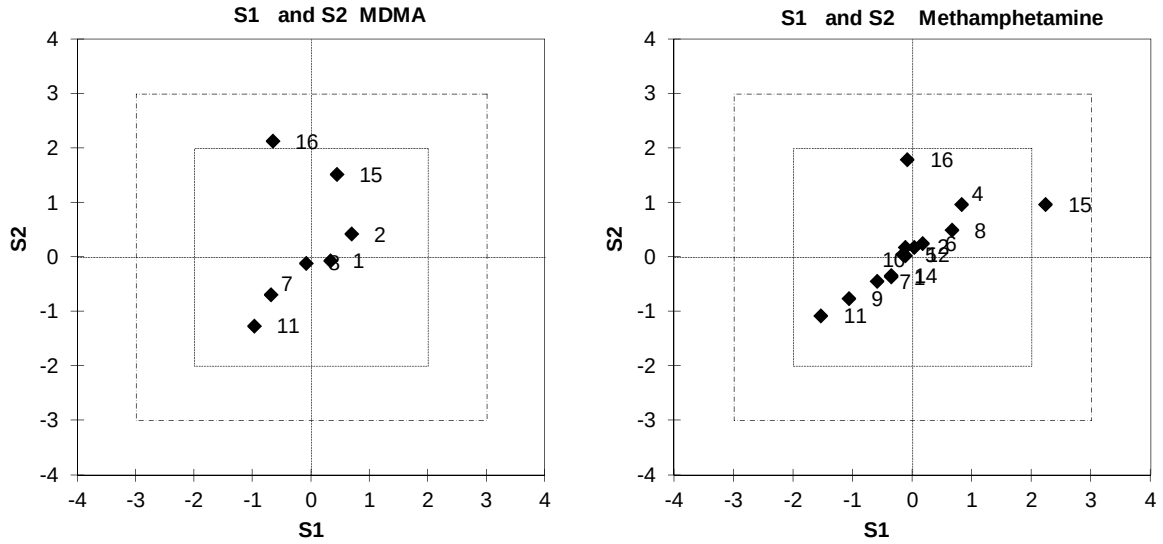


Figure 18 z-Score Scatter Plots for S1 and S2

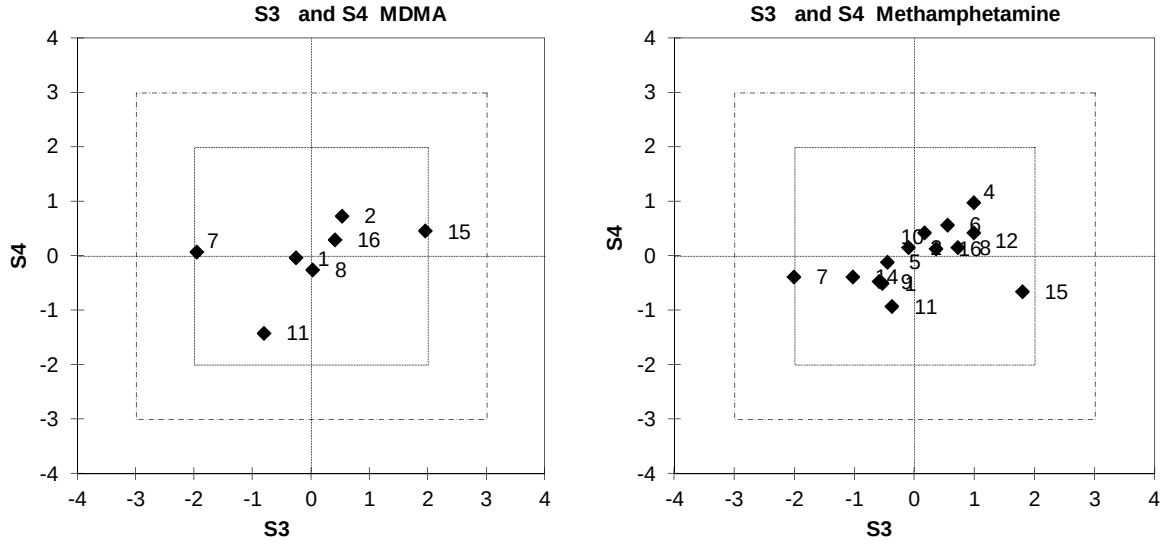


Figure 19 z-Score Scatter Plots for S3 and S4

7 REFERENCES

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

[2] NMI 2016, *Chemical Proficiency Testing Study Protocol*, viewed 3 May 2018, http://www.measurement.gov.au.

[3] NMI 2016, *Chemical Proficiency Testing Statistical Manual*, viewed 3 May 2018, <http://www.measurement.gov.au >.

[4] Thompson, M., Ellison, SLR. & Wood, R. 2006. 'The international harmonized protocol for proficiency testing of (chemical) analytical laboratories', *Pure Appl. Chem*, vol 78, pp 145-196.

[5] ISO/IEC 13528 2015, *Statistical methods for use in proficiency testing by interlaboratory comparisons.*

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

[7] Eurachem 2012, *Quantifying Uncertainty in Analytical Measurement*, 3rd edition, viewed 10 May 2017, <http://www.eurachem.org/images/stories/Guides/pdf/QUAM2012_P1.pdf>.

[8] Thompson M. and Lowthian, P.J. 1995. 'A Horwitz-like function describes precision in a proficiency test', *Analyst*, vol 120, pp 271-272

APPENDIX 1 - PARTICIPANT LABORATORIES

ACS Laboratories (Australia) VIC	AMAL Analytical VIC
Analytica Laboratories Ltd, NEW ZEALAND	CHEMCENTRE WA
ChemInspect Consultancy Ltd, NEW ZEALAND	Envirolab Services Ltd Trading as LABTEC, NEW ZEALAND
Envirolab Services NSW	Eurofins mgt, QLD
Eurofins Forensic Services Limited, UK	Forensic and Industrial Science Ltd, NEW ZEALAND
Hill Laboratories, NEW ZEALAND	National Measurement Institute NSW
Queensland Health Forensic and Scientific Services QLD	

APPENDIX 2 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

A1.1 Sample Preparation

Samples used were large Liv-Wipe alcohol wipes from Livingstone International Pty Ltd. The wipes were removed from the individual packaging using tweezers and long-nosed pliers and unfolded. The analytes were spiked onto the wipes using calibrated GILSON positive displacement pipettes. After spiking, the methanol solvent was allowed to evaporate and the wipes were placed in H056A amber glass jars, labelled and placed in a refrigerator.

The analytes of interest in S1 and S2 were at a similar level and approximately double the concentration of S3 and S4.

A1.2 Sample Analysis and Homogeneity Testing

Homogeneity testing was conducted for each analyte. 7 samples were analysed at the National Measurement Institute Sydney laboratory and the average of the results was reported as the homogeneity value. Tables 15 to 18 set out the testing of homogeneity of Methamphetamine and MDMA in duplicate pairs S1/S2 and S3/S4.

NMI holds third party (NATA) accreditation to ISO17025 for these tests. The method used involved 0.2 M H_2SO_4 as desorption solution, and GCMS measurements. Methamphetamine-D_{14} are MDMA-D_5 were used as internal standards

Since the entire sample was used in each analysis, it was not possible to apply analysis of variance (ANOVA) to determine if samples were sufficiently homogeneous. When it is not possible to conduct replicate measurements, the standard deviation of the results (sd) will be compared with the target standard deviation of the PT (σ) calculated as described in section 4.4. The proficiency test samples may be considered sufficiently homogeneous if : $sd \leq 0.3\ \sigma$.[5]

For wipe samples, the mean of the 7 measurements were used as the NMI homogeneity value. All samples were found to be sufficiently homogeneous for use in this PT study.

Table 15 Methamphetamine in S1 and S2 Homogeneity Data

Sample number	Average Result
S1-05	2.74
S1-13	2.74
S1-17	3.12
S1-20	3.07
S1-24	3.21
S1-25	3.10
S2-32	3.22
Mean	**3.03**
sd	**0.197**
Target σ	**0.64**

Test	Value	Critical	Result
$sd/\sigma \leq 0.3$	0.3	0.3	**Pass**

Table 16 MDMA in S1 and S2 Homogeneity Data

Sample number	Average Result
S1-05	15.79
S1-13	16.14
S1-17	16.56
S1-20	15.35
S1-24	15.53
S1-25	15.85
S2-32	16.26
Mean	**15.93**
sd	**0.437**
Target σ	**3.5**

Test	Value	Critical	Result
$sd/\sigma \leq 0.3$	0.1	0.3	**Pass**

Table 17 Methamphetamine in S3 and S4 Homogeneity Data

Sample number	Average Result
S3-01	2.06
S3-08	2.31
S3-10	2.32
S3-18	2.34
S3-20	2.23
S3-25	2.22
S4-33	2.22
Mean	**2.24**
sd	**0.092**
Target σ	**0.37**

Test	Value	Critical	Result
$sd/\sigma \leq 0.3$	0.2	0.3	**Pass**

Table 18 MDMA in S3 and S4 Homogeneity Data

Sample number	Average Result
S3-01	10.44
S3-08	9.32
S3-10	10.02
S3-18	9.64
S3-20	9.10
S3-25	9.55
S4-33	8.86
Mean	**9.56**
sd	**0.52**
Target σ	**1.8**

Test	Value	Critical	Result
$sd/\sigma \leq 0.3$	0.3	0.3	**Pass**

APPENDIX 3 – STABILITY STUDY

Participants were advised to store the samples refrigerated if analyses cannot be commenced on the day of receipt.

Sample condition on receipt and the date when the samples were received and analysed by the participants are presented in Table 19.

Table 19 Condition on Receipt and the Date when the Samples were Received and Analysed

Lab Code	Received Date	Arrival Condition	Analysis Date
1	09.08.2018	Good	10.08.2018
2	10.07.2018	Moderate Temperature 16.4 C	24.07.2018
3	19.07.2048	Samples in screw-lid jars	7-15.08.2018
4	10.07.2018	Good Condition	12.07.2018
5	10.07.2018	Fit for analysis	03.08.2018
6	11.07.2018	Acceptable	11.07.2018
7	09.07.2018	Good	07.08.2018
8	10.07.2018		26.07.2018
9	10.07.2018	All jars intact	06.08.2018
10	10.07.2018	Good	17.07.2018
11	10.07.2018	Satisfactory	12.07.2018
12	10.07.2018	Acceptable	16.07.2018
13	20.07.2018	Sealed / Dry - Not refrigerated /refrigerated at laboratory.	09.08.2018
14	10/07/2018	Fine	12.07.2018
15	10/07/2018		27.07-03.08.2018
16	10/07/2018	Good	20.07.2018

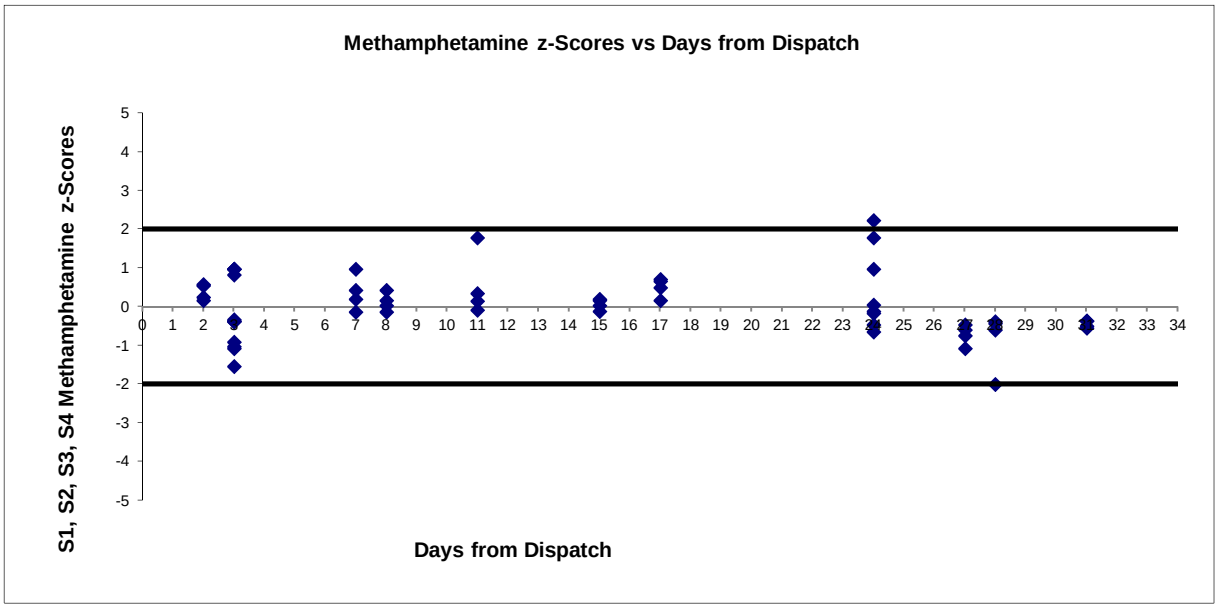


Figure 20 Methamphetamine z-Scores vs. Analysis Date

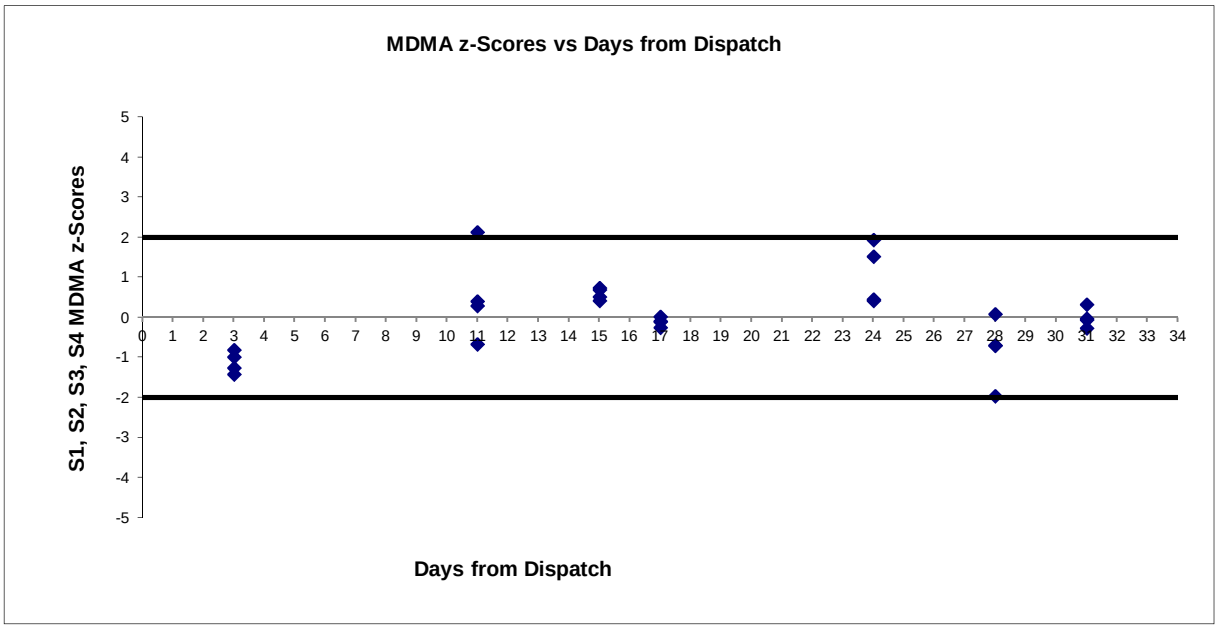


Figure 21 MDMA z-Scores vs. Analysis Date

No correlation between reported results, the received date, the analysis date or the sample condition at arrival was observed (Table 19 and Figures 20 and 21).

APPENDIX 4 - MEASUREMENT UNCERTAINTY OF THE ROBUST AVERAGE

When the robust average is calculated using the procedure described in 'ISO13528:2015, Statistical methods for use in proficiency testing by interlaboratory comparisons – Annex C'[5], the uncertainty is estimated as:

$$u_{rob\ average} = 1.25 * S_{rob\ average} / \sqrt{p} \qquad \text{Equation 4}$$

where:

$u_{rob\ average}$	robust average standard uncertainty
$S_{rob\ average}$	robust average standard deviation
p	number of results

The expanded uncertainty ($U_{rob\ average}$) 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 17.

Table 20 Uncertainty of assigned value for methamphetamine in Sample S1 as ug/wipe as base.

No. results (p)	14
Robust average	3.11
$S_{rob\ average}$	0.45
$u_{rob\ average}$	0.15
k	2
$U_{rob\ average}$	0.30

The robust average for methamphetamine in Sample S1 is 3.11 ± 0.30 ug/wipe as base.

APPENDIX 5 - ACRONYMS AND ABBREVIATIONS

ASCLD	American Society of Crime Laboratory Directors
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
$\|E_n\|$	Absolute value of an E_n-score
GC	Gas Chromatography
GC-MS	Gas Chromatography Mass Spectrometry
GUM	Guide to the expression of uncertainty in measurement
HPLC	High Performance Liquid Chromatography
ISO	International Standards Organisation
LC	Liquid Chromatography
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
NATA	National Association of Testing Authorities
NMI	National Measurement Institute Australia
NR	Not Reported
NT	Not Tested
PT	Proficiency Test
PCV	Performance Coefficient of Variation
Robust CV	Robust between-laboratory Coefficient of Variation
Robust SD	Robust Standard Deviation
SI	International System of Units
Target SD (σ)	Target standard deviation
UPLC	Ultra Performance Liquid Chromatography
UV	Ultraviolet
$\|z\|$	Absolute value of a z-score

END OF REPORT