# Proficiency Test Final Report AQA 23-08 Methamphetamine/MDMA in Wipes

November 2023

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#### **SUMMARY**

AQA 23-08 Methamphetamine/MDMA in Wipes commenced in March 2023. Sixteen laboratories enrolled to participate, and fourteen participants submitted results.

Four test samples were prepared by spiking wipes with varying amounts of methamphetamine (Sample S1, S2 and S3) and 3,4-methylenedioxymethamphetamine (MDMA) (Sample S4).

The assigned values for all scored analytes 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 and MDMA in wipes;

No assigned value was set for the sample containing MDMA, so only the proficiency of laboratories measuring methamphetamine was assessed using both z-score and  $E_n$ -score.

Of 41 z-scores, 39 (95%) returned a z-score of  $|z| \le 2.0$ , indicating a satisfactory performance.

Of 41  $E_n$ -scores, 34 (83%) returned an  $E_n$ -score of  $|E_n| \le 1.0$ , indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratories 2, 3, 7, 8, 9, 10, 11, 15 and 17 returned satisfactory z-scores and  $E_n$ -scores for all scored analytes.

• Evaluate the participants' methods for the measurement of methamphetamine and MDMA in wipes;

Participants used various methods in this study, though many were based on the NIOSH 9111 method.

Most methodologies produced compatible results; however, one participant used a significantly different methodology (different sample treatment and instrument) and their results were not compatible with other participants' results or the spiked values.

• Develop the practical application of traceability and measurement uncertainty, and provide participants with information that will be useful in assessing their uncertainty estimates.

Of 46 numeric results, 43 (93%) 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 4.9% to 36% 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 the 'evaluation of participant performance against pre-established criteria by means of interlaboratory comparison'. 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 soil, water, fruit, vegetables and herbs;
- petroleum hydrocarbons in soil and water;
- per- and polyfluoroalkyl substances in water, soil, food and biota;
- inorganic analytes in soil, water, food and pharmaceuticals; and
- controlled drug assay, drugs in wipes, and clandestine laboratory.

## 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 controlled substances in wipes at levels specified in the Clandestine Drug Laboratory Remediation Guidelines 2011.<sup>2</sup>

## 1.3 Study Aims

The aims of the study were to:

- assess the proficiency of laboratories measuring methamphetamine and 3,4-methylenedioxymethamphetamine (MDMA) in wipes;
- evaluate the participants' methods for the measurement of methamphetamine and MDMA in wipes;
- 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.<sup>3</sup> The statistical methods used are described in the NMI Chemical Proficiency Testing Statistical Manual.<sup>4</sup> These documents have been prepared with reference to ISO/IEC 17043 and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.<sup>1,5</sup> NMI is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of PT schemes,<sup>1</sup> and this study is within the scope of NMI's accreditation.

#### **2 STUDY INFORMATION**

## 2.1 Study Timetable

The timetable of the study was:

 Invitations sent
 13/03/2023

 Samples sent
 22/05/2023

 Results due
 1/09/2023

 Interim Report
 6/09/2023

 Preliminary Report
 13/09/2023

The timeline of this PT study was extended due to delays with participants' permits.

## 2.2 Participation and Laboratory Code

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

## 2.3 Test Material Preparation and Specification

Four test samples were prepared, each containing one wipe. A 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. A MDMA spiking solution was prepared by dissolving a known mass of MDMA hydrochloride (approximately 84% 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 or MDMA spiking solution was then spiked onto the wipes using a calibrated dispenser. After spiking, the methanol solvent was allowed to evaporate off 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 3.09 µg methamphetamine base/wipe.

Sample S2 was prepared to contain 1.54 µg methamphetamine base/wipe.

Sample S3 was prepared to contain 0.774 µg methamphetamine base/wipe.

Sample S4 was prepared to contain 6.01 µg MDMA base/wipe.

#### 2.4 Homogeneity and Stability of Test Materials

No homogeneity or stability testing was conducted for this PT study's samples. The process used to prepare, store and dispatch the test samples has been demonstrated to produce sufficiently homogeneous and stable samples in previous NMI PT studies.

To assess possible instability, the assigned values were compared to the spiked values. The assigned values were 93% to 102% of the spiked values, providing good support for the stability of the test materials.

Participants' results also gave no reason to question the homogeneity or stability of the samples (Appendix 1).

## 2.5 Sample Storage, Dispatch and Receipt

The test samples were stored at 4 °C after preparation and prior to dispatch. Samples were packed with ice blanket cells or cooler bricks and sent by courier on 22 May 2023. 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.

## 2.6 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 or MDMA 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. For Sample S4, report a single result in units of µg MDMA as base/wipe. Results should be 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 or MDMA as base/wipe.
- 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.
- You do not need to test all samples. Please 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 3 July 2023 by email to proficiency@measurement.gov.au.

The results due date was extended to 1 September 2023. This was because of delays with some participants' permits, which resulted in delayed sample dispatches to those participants.

## 2.7 Interim Report and Preliminary Report

An Interim Report was emailed to all participants on 6 September 2023.

A Preliminary Report was emailed to all participants on 13 September 2023. This report included a summary of the results reported by participants, assigned values, performance coefficient of variations, z-scores and  $E_n$ -scores for each analyte in this study. No data from the Preliminary Report has been changed in the present Final Report.

## 3 PARTICIPANT LABORATORY INFORMATION

## 3.1 Participants' Test Methods

Participants were requested to provide information about their test methods. Responses received are presented in Tables 1 and 2. Some responses may be modified so that the participant cannot be identified.

Table 1 Summary of Participants' Test Methods for Methamphetamine in Wipes

Lab. Code	Desorption Solution	Sample Treatment	Filtration	Technique	Detector	Method Reference
2	0.1 M sulfuric acid	1 hr on rotary mixer, pH adjustment	none	UPLC	MS/MS	In house (based off of NIOSH9111)
3	0.1M Sulfuric Acid	1 hr on rotary mixer, pH adjustment	0.22 um Nylon Filter	UPLC	MS/MS	NIOSH 9111
5	purified water	extraction 16 hours	0.45 μm PVDF	HPLC	DAD	In house
6	0.1M sulfuric acid	1 hr on rotary mixer	Agilent PES 0.45 um, 25 mm	HPLC	MS	Based on NIOSH 9111
7	0.1M Sulfuric Acid	1Hr on Rotary Mixer	Centrifugation	HPLC	MS/MS	In-House
8	0.1 M sulfuric acid	1 hr on rotary mixer, pH adjustment	0.45 µm PES filters	HPLC	MS/MS	In house method; referencing NIOSH 9111
9	0.1 M Sulfuric acid	1 hr on rotary mixer	0.2 um Acrodisc filter	HPLC	MS/MS	NIOSH 9111
10	0.1M sulfuric acid	1 hr on rotary mixer	Centrifugation	HPLC	MS/MS	In-house (based on NIOSH 9111)
11	0.1 M sulfuric acid	1 hr on linear shaker	N/A	HPLC	MS/MS	NIOSH9111 Modified
13	0.1M Sulfuric Acid	1 hr on rotary mixer	No Filtration	HPLC	MS	NIOSH 9111
14	0.2 N sulfuric acid	1 hour on rotary mixer, pH adjustment, derivatization		GC	MS	NIOSH9106
15	0.1 M sulfuric acid	1 hr on rotary mixer	0.2 μm 15 mm RC filter	UPLC	MS/MS	NIOSH 9111
16	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	based on NIOSH 9111
17	0.1M sulfuric acid	30min on orbital shaker	Nil	UPLC	MS/MS	NIOSH 9111

Table 2 Summary of Participants' Test Methods for MDMA in Wipes

Lab. Code	Desorption Solution	Sample Treatment	Filtration	Technique	Detector	Method Reference
2	NS					
3	NT					
5	purified water	extraction 16 hours	0.45 μm PVDF	HPLC	DAD	In house
6			NS			
7			NT			
8	0.1 M sulfuric acid  1 hr on rotary mixer, pH adjustment		0.45 µm PES filters	HPLC	MS/MS	In house method; referencing NIOSH 9111
9	0.1 M Sulfuric acid 1 hr on rotary mixer		0.2 um Acrodisc filter	HPLC	MS/MS	NIOSH 9111
10	NS					
11			NS			
13			NS			
14	0.2 N sulfuric acid  1 hour on rotary mixer, pH adjustment, derivatization			GC	MS	NIOSH9106
15	NS					
16	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	based on NIOSH 9111
17			NS			

## 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 3. Some responses may be modified so that the participant cannot be identified.

Table 3 Reported Basis of Uncertainty Estimates

Lab. Code	Annual to Estimating MII	Information Sources	s for MU Estimation*	Cuide Decument for Estimating MII	
Lab. Code	Approach to Estimating MU	Precision	Method Bias	Guide Document for Estimating MU	
2	Top Down - precision and estimates of the method and laboratory bias		Instrument calibration CRM Recoveries of SS	ISO/GUM	
3	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	Recoveries of SS	NMI Uncertainty Course	
5	Top Down - precision and estimates of the method and laboratory bias	Control samples	Laboratory bias from PT studies	Nordtest Report TR537	
6	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Instrument calibration CRM Recoveries of SS Standard purity	ISO/GUM	
7	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Instrument calibration CRM Recoveries of SS Standard purity	NMI Uncertainty Course	
8	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Instrument calibration CRM Recoveries of SS	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results	
9	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Instrument calibration CRM Recoveries of SS Standard purity	Nata Technical Note 33	
10	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Instrument calibration CRM Recoveries of SS Standard purity	NMI Uncertainty Course  NPAAC Requirements for the Estimation of Measurement Uncertainty	

Lab Cada	Annual to Estimation MII	Information Sources	s for MU Estimation*	Cuida Decument for Estimating MII
Lab. Code	Approach to Estimating MU	Precision	Method Bias	Guide Document for Estimating MU
11	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Instrument calibration Laboratory bias from PT studies Standard purity	Eurachem/CITAC Guide
13	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)  Control samples - CR Duplicate analysis		Instrument calibration CRM Standard purity	NMI Uncertainty Course
14	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Instrument calibration Recoveries of SS	Eurachem/CITAC Guide
15	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM	Instrument calibration CRM Standard purity	Eurachem/CITAC Guide
16	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
17	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

<sup>\*</sup> 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 4. Some responses may be modified so that that participant cannot be identified.

Table 4 Participants' Standards

	Methamphetamine					MDMA			
Lab. Code	Calibration Standard		Internal Standard		Calibration Standard		Internal Standard		
Code	Origin	Purity (%)	Origin	Standard	Origin	Purity (%)	Origin	Standard	
2	Cerilliant	1000ug/ml	Cerilliant	1000ug/ml			NS		
3	Calibration Standard	100	Calibration Standard	Methamphetamine-D5			NT		
5	Sigma	100			Internal	100			
6	Cerilliant M-009, 1mg/mL		Cerilliant M-093, 1mg/mL	Methamphetamine-D14	NS				
7	Lipomed	>99	Lipomed	Methamphetamine-D5			NT		
8	PM Separations	99.6	PM Separations	Methamphetamine.D5	PM Separations	100	PM Separations	MDMA.D5	
9	NMI	99.8	CERILLIANT	Methamphetamine-D14	NMI	99.4	SUPELCO	MDMA-D5	
10	Lipomed	Lipomed 98.5 Supelco Methamphetamin		Methamphetamine-D5	NS				
11	NMI	99.8	LGC	Methamphetamine-D9			NS		
13	Lipomed	99.95	Supelco	D,1 - methylamphetamine-d5			NS		
14	NMI	99.8	Cerilliant	Methamphetamine-D14	NMI	99.8	Cerilliant	MDMA-D5	
15	Cerilliant	Cerilliant 1.000 +-0.006 mg/mL Cerilliant Methampheta		Methamphetamine-D14	NS				
16	Lipomed	>98.5	Chiron	Methamphetamine-D5	Lipomed	>98.5	Chiron	MDMA-D5	
17	Lipomed d,l- Methamphetamine. HCI 1mg/mL calibrated in methanol 1mL	99.9	Lipomed d,l- Methamphetamine- D14.HCl	Methamphetamine-D14			NS		

## 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 5, along with the study coordinator's response where appropriate. Some responses may be modified so that the participant cannot be identified.

Table 5 Participants' Comments

Lab. Code	Participant Comments	Study Coordinator's Response
	Small isopropyl alcohol wipes used; while in line with NIOSH 9111 we typically encounter (95% of samples) much larger wipes ~15x15cm. Real world samples also arrive in PP centrifuge tubes and are	Thank you for your suggestions, we will take these into consideration for future studies.
2	analysed there-in resulting in less handling (***we acknowledge that samples most likely cannot be prepared this way)	For this study, samples were packaged as per the Clandestine Drug Laboratory Remediation Guidelines 2011. <sup>2</sup>
	The country are halous are greatered as a few management area. The test is used for a consideration of	Samples were prepared to have analyte levels near those specified in the Clandestine Drug Laboratory Remediation Guidelines 2011. <sup>2</sup>
5	The samples are below our normal scope for measurement area. The test is used for examination of suitability of our GCMS and HPLC-DAD methods for detection and quantification of traces. The method uncertainty is not established in this concentration area.	This participants' results were significantly lower than the spiked value (which may be due to a methodology bias), and this may be why the samples were below their normal scope for measurement.
6	Blank provided was analysed with samples, no methamphetamine was detected.	Thank you for your suggestion, we will take this into consideration for future studies.
	Ideally use wipes that are commonly used for sampling - these were too small.	into consideration for future studies.
7	The laboratory is not currently testing for MDMA, but is looking to add in the future	
8	In-house lab procedure recommendation of samples to be chilled during transport, however COC states this did not occur (this was reported to NMI on the SRA. Samples were dispatched on 22nd May 2023, Samples received 23rd May 2023.	All samples were packed with ice blanket cells or cooler bricks for dispatch. We did not receive this participant's sample receipt notification until their results were submitted, and had not been made aware of the reported shipping conditions.
		Nevertheless, a short period at ambient temperatures would not invalidate results (Appendix 1).

Lab. Code	Participant Comments	Study Coordinator's Response
9	DO not write on the swabs that they are MA or MDMA as this leads to an element of bias.  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.  Uncertainty: Methamphetamine 10% @ 99.7% confidence; MDMA 10% @ 95% confidence  Sample S1: Methylamphetamine was detected in Sample S1 at levels above those specified in the Clandestine Drug Laboratory Remediation Guidelines.  Sample S2: Methylamphetamine was detected in Sample S2 at levels above those specified in the Clandestine Drug Laboratory Remediation Guidelines.  Sample S3: Methylamphetamine was detected in Sample S3 at levels above those specified in the guidelines.	This PT study is not a qualitative study, and participants are informed what analytes have been spiked in each sample, including on the sample dispatch letter provided with the samples.
	Sample S4: MDMA was detected in Sample S4 at levels below those specified in the Clandestine Drug Laboratory Remediation Guidelines.	
15	Samples S1, S2 and S3: Reported as to commercial clients	
16	Corrected on instrument but not for extraction i.e. instrument internal standard correction used NOT extracted internal standard correction.	

#### 4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

## 4.1 Results Summary

Participant results are listed in Tables 6 to 9 with resultant summary statistics: robust average, median, mean, number of 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 5.

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

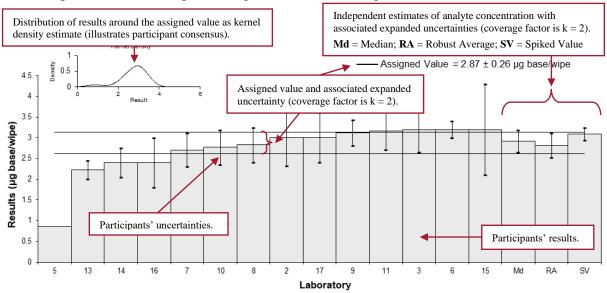


Figure 1 Guide to Presentation of Results

#### 4.2 Outliers and Extreme Outliers

Outliers were any result less than 50% and greater than 150% of the robust average, and these were removed before the calculation of the assigned value.<sup>4,5</sup> Extreme outliers were any obvious blunders, e.g. results with incorrect units, or for a different analyte or sample, and such results were removed before the calculation of all summary statistics.<sup>4</sup>

#### 4.3 Assigned Value

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

## 4.4 Robust Average and Robust Between Laboratories Coefficient of Variation

The robust averages and expanded MUs, and robust CVs (a measure of the variability of participants' results) were calculated using the procedure described in ISO 13528.<sup>6</sup>

#### 4.5 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.

## 4.6 Target Standard Deviation for Proficiency Assessment

The target SD for proficiency assessment ( $\sigma$ ) 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.

$$\sigma = X \times PCV$$

#### 4.7 *z*-Score

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

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

Equation 2

where:

z is z-score

 $\chi$  is a participant's result

X is the assigned value

 $\sigma$  is the target standard deviation from Equation 1

For the absolute value of a *z*-score:

- $|z| \le 2.0$  is satisfactory;
- 2.0 < |z| < 3.0 is questionable;
- $|z| \ge 3.0$  is unsatisfactory.

#### 4.8 $E_n$ -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.

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

Equation 3

where:

 $E_n$  is  $E_n$ -score

 $\chi$  is a participant's result

X is the assigned value

 $U_{\chi}$  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:

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

## 4.9 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC 17025 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.<sup>7</sup>

Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.<sup>8</sup>

## 5 TABLES AND FIGURES

Table 6

## Sample Details

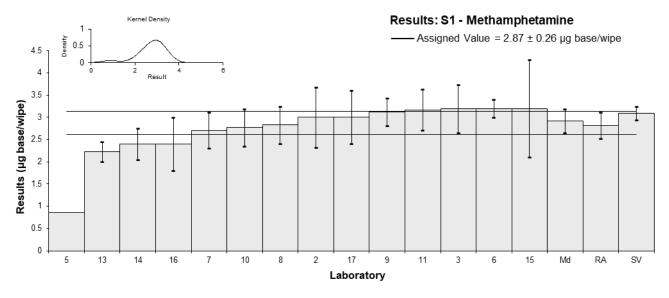
Sample No.	S1
Matrix	Wipe
Analyte	Methamphetamine
Unit	μg base/wipe

## **Participant Results**

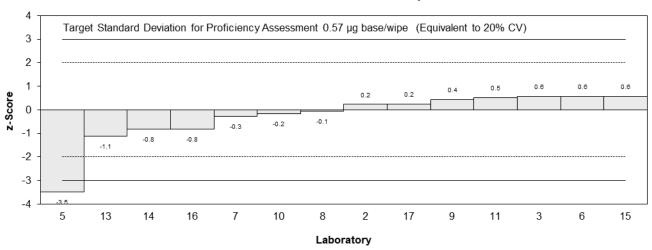
Lab. Code	Result	Uncertainty	Rec	z	En
2	3.0	0.68	106	0.23	0.18
3	3.2	0.54	NR	0.57	0.55
5*	0.86	NR	NR	-3.50	-7.73
6	3.2	0.2	102.6	0.57	1.01
7	2.709	0.406	NR	-0.28	-0.33
8	2.83	0.42	126	-0.07	-0.08
9	3.12	0.31	NR	0.44	0.62
10	2.77	0.42	NR	-0.17	-0.20
11	3.17	0.46	NR	0.52	0.57
13	2.23	0.22	NR	-1.11	-1.88
14	2.4	0.35	125	-0.82	-1.08
15	3.2	1.1	NR	0.57	0.29
16	2.4	0.6	100	-0.82	-0.72
17	3.01	0.60	106	0.24	0.21

<sup>\*</sup> Outlier, see Section 4.2

Assigned Value	2.87	0.26
Spike Value	3.09	0.15
Robust Average	2.82	0.29
Median	2.92	0.27
Mean	2.72	
N	14	
Max	3.2	
Min	0.86	
Robust SD	0.43	
Robust CV	15%	



z-Scores: S1 - Methamphetamine



En-Scores: S1 - Methamphetamine

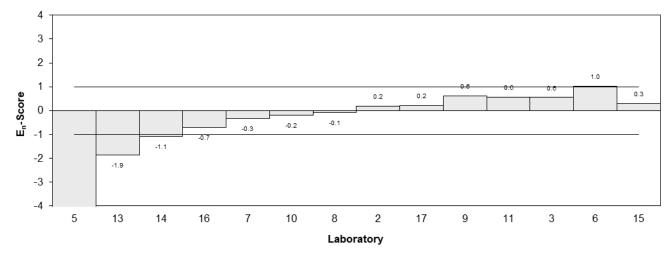


Figure 2

Table 7

## Sample Details

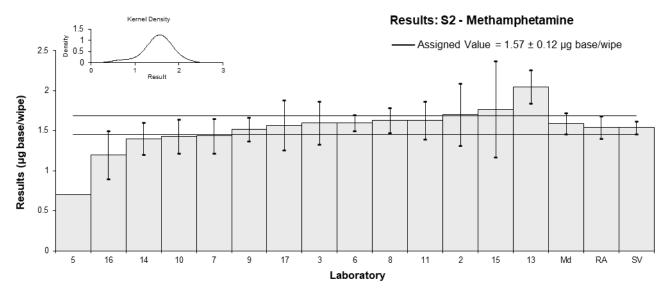
Sample No.	S2
Matrix	Wipe
Analyte	Methamphetamine
Unit	μg base/wipe

## **Participant Results**

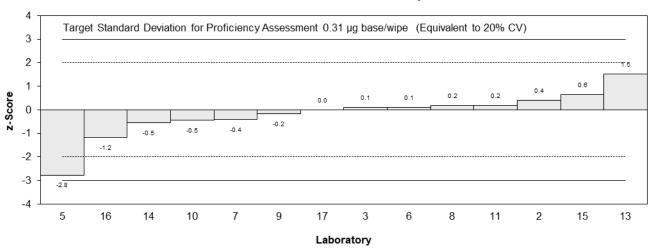
Lab. Code	Result	Uncertainty	Rec	z	En
2	1.7	0.39	106	0.41	0.32
3	1.6	0.27	NR	0.10	0.10
5*	0.7	NR	NR	-2.77	-7.25
6	1.6	0.1	102.6	0.10	0.19
7	1.437	0.216	NR	-0.42	-0.54
8	1.63	0.16	107	0.19	0.30
9	1.52	0.15	NR	-0.16	-0.26
10	1.43	0.21	NR	-0.45	-0.58
11	1.63	0.24	NR	0.19	0.22
13	2.05	0.21	NR	1.53	1.98
14	1.4	0.20	88	-0.54	-0.73
15	1.77	0.6	NR	0.64	0.33
16	1.2	0.3	97	-1.18	-1.15
17	1.57	0.31	106	0.00	0.00

<sup>\*</sup> Outlier, see Section 4.2

Assigned Value	1.57	0.12
Spike Value	1.54	0.08
Robust Average	1.54	0.14
Median	1.59	0.13
Mean	1.52	
N	14	
Max	2.05	
Min	0.7	
Robust SD	0.21	
Robust CV	13%	



z-Scores: S2 - Methamphetamine



En-Scores: S2 - Methamphetamine

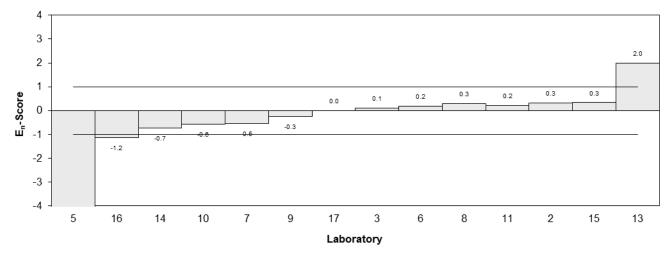


Figure 3

Table 8

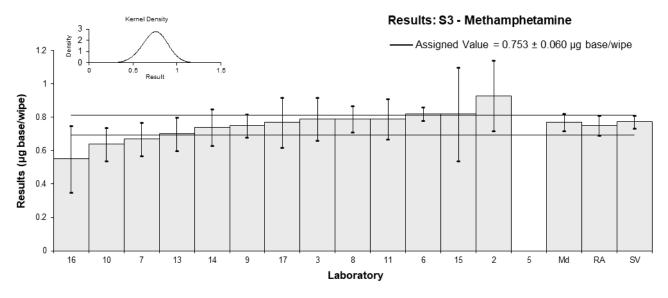
## Sample Details

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

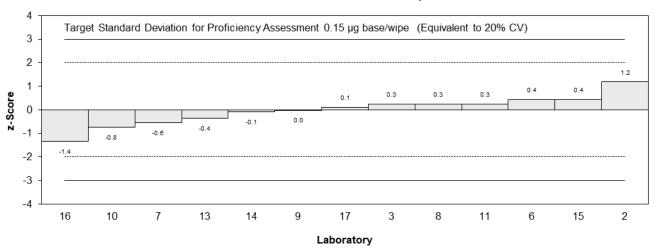
## **Participant Results**

Lab. Code	Result	Uncertainty	Rec	z	En
2	0.93	0.21	106	1.18	0.81
3	0.79	0.13	NR	0.25	0.26
5	< 0.6	NR	NR		
6	0.82	0.04	102.6	0.44	0.93
7	0.67	0.10	NR	-0.55	-0.71
8	0.79	0.08	107	0.25	0.37
9	0.75	0.07	NR	-0.02	-0.03
10	0.64	0.10	NR	-0.75	-0.97
11	0.79	0.12	NR	0.25	0.28
13	0.7	0.1	NR	-0.35	-0.45
14	0.74	0.11	102	-0.09	-0.10
15	0.82	0.28	NR	0.44	0.23
16	0.55	0.2	97	-1.35	-0.97
17	0.77	0.15	106	0.11	0.11

Assigned Value	0.753	0.060
Spike Value	0.774	0.039
Robust Average	0.753	0.060
Median	0.770	0.051
Mean	0.751	
N	13	
Max	0.93	
Min	0.55	
Robust SD	0.086	
Robust CV	11%	



z-Scores: S3 - Methamphetamine



En-Scores: S3 - Methamphetamine

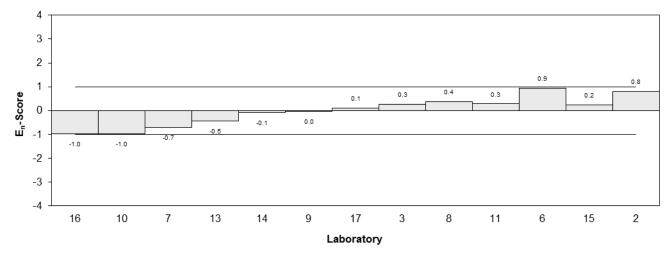


Figure 4

Table 9

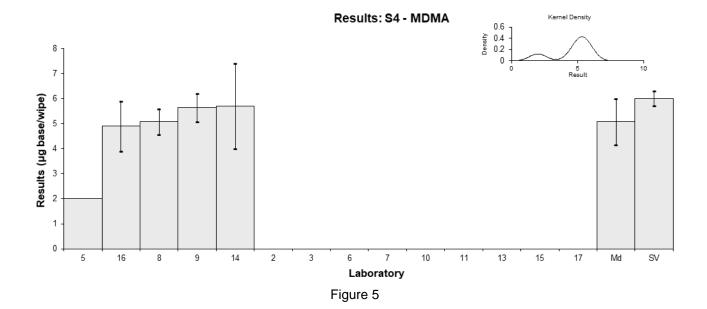
## Sample Details

Sample No.	S4
Matrix	Wipe
Analyte	MDMA
Unit	μg base/wipe

## **Participant Results**

Lab. Code	Result	Uncertainty	Rec
2	NS	NS	NS
3	NT	NT	NT
5	2	NR	NR
6	NS	NS	NS
7	NT	NT	NT
8	5.08	0.51	110
9	5.64	0.56	NR
10	NS	NS	NS
11	NS	NS	NS
13	NS	NS	NS
14	5.7	1.7	110
15	NS	NS	NS
16	4.9	1	103
17	NS	NS	NS

Assigned Value	Not Set	
Spike Value	6.01	0.30
Robust Average	NA (N<6)	
Median	5.08	0.93
Mean	4.7	
N	5	
Max	5.7	
Min	2	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	



#### 6 DISCUSSION OF RESULTS

## 6.1 Assigned Value

The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528.<sup>6</sup> The assigned values for all scored analytes were the robust averages of participants' results, after results less than 50% and greater than 150% of the robust average had been removed.<sup>4,5</sup> The calculation of the expanded uncertainty for a robust average is presented in Appendix 2, using Sample S3 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.

No assigned value was set for Sample S4 MDMA as there were too few numeric results reported. However, participants can still compare their result with the descriptive statistics and spiked value, as presented in Section 5.

A comparison of the assigned values and the spiked values for scored analytes is presented in Table 10. Assigned values were between 93% and 102% of the spiked values, providing good support for the assigned values and as well as evidence for the stability of these analytes in the test samples.

Sample	Analyte	Assigned Value (µg base/wipe)	Spiked Value (µg base/wipe)	Assigned Value / Spiked Value (%)
S1	Methamphetamine	2.87	3.09	93
S2	Methamphetamine	1.57	1.54	102
S3	Methamphetamine	0.753	0.774	97

Table 10 Comparison of Assigned Values and Spiked Values

## 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 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.<sup>7</sup>

Laboratories 7, 10 and 13 reported their uncertainties as a relative uncertainty (i.e. x %) rather than as  $\mu$ g base/wipe. These uncertainty values were modified accordingly by the study coordinator for this report.

Of 46 numeric results, 43 (93%) were reported with an associated MU. Participants used a wide variety of procedures to estimate their uncertainty (Table 3). A few participants reported using NATA MU guidance documents as their guide; NATA no longer publishes these documents.<sup>9</sup>

Laboratory **5** did not report uncertainties for any of their results. This participant was not accredited to ISO/IEC 17025.

The magnitude of reported uncertainties were within the range of 4.9% to 36% relative.

Uncertainties associated with results returning a satisfactory z-score but an unsatisfactory  $E_n$ -score may have been underestimated.

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  $2.709 \pm 0.406~\mu g$  base/wipe, it is recommended to report this as  $2.71 \pm 0.41~\mu g/wipe.^8$ 

#### 6.3 *z*-Score

Target SDs equivalent to 20% PCV were used to calculate *z*-scores. CVs predicted by the Thompson-Horwitz equation,<sup>10</sup> between-laboratory CVs and target SDs (as PCV) for scored analytes in this study are presented for comparison in Table 11.

Table 11 Thompson-Horwitz CVs, Between-Laboratory CVs and Target SDs (as PCV)

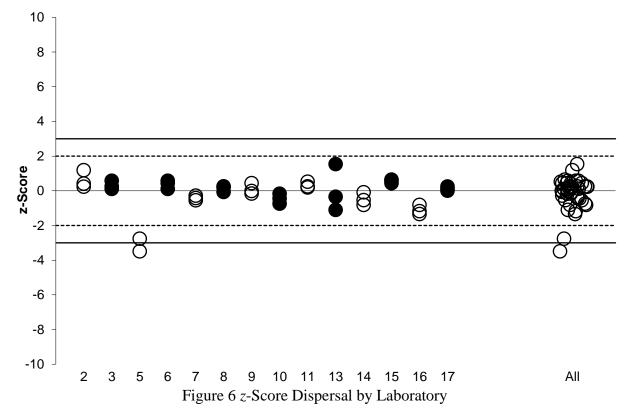
Sample	Analyte	Assigned Value (µg base/wipe)	Thompson-Horwitz CV <sup>a</sup> (%)	Between-Laboratory CV <sup>b</sup> (%)	Target SD (as PCV) (%)
S1	Methamphetamine	2.87	22	13	20
S2	Methamphetamine	1.57	22	11	20
S3	Methamphetamine	0.753	22	11	20

<sup>&</sup>lt;sup>a</sup> Calculated from the assigned value.

Of 41 results for which z-scores were calculated, 39 (95%) returned a z-score of  $|z| \le 2.0$ , indicating satisfactory performance.

All laboratories except Laboratory **5** returned satisfactory *z*-scores for all reported numeric results. Laboratory **5** reported results lower than the assigned value or robust average across all samples; this participant may need to investigate the source of this negative bias.

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



<sup>&</sup>lt;sup>b</sup> Robust between-laboratory CV with outliers removed, if applicable.

#### 6.4 $E_n$ -Score

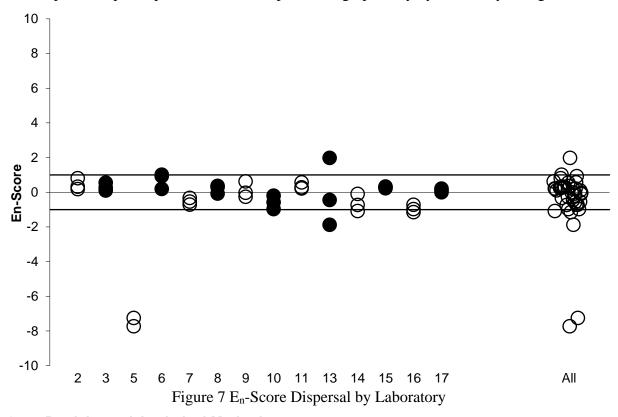
 $E_n$ -scores can be interpreted in conjunction with *z*-scores, as an unsatisfactory  $E_n$ -score can either be caused by issues with measurement, or uncertainty, or both. If a participant did not report an uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the  $E_n$ -score.

Of 41 results for which  $E_n$ -scores were calculated, 34 (83%) returned an  $E_n$ -score of  $|E_n| \le 1.0$ , indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Nine participants returned satisfactory  $E_n$ -scores for all three scored samples: Laboratories 2, 3, 7, 8, 9, 10, 11, 15 and 17.

Laboratory 5 returned unsatisfactory  $E_n$ -scores for both of their scored results.

The dispersal of participants'  $E_n$ -scores is presented graphically by laboratory in Figure 7.



#### 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 12. Most participants used methods that were based on NIOSH 9111.<sup>11</sup>

Table 12 Summary of Participants' Analyses

		Lab. Code
Accreditation	Yes to ISO/IEC 17025	2, 3, 5, 6, 7, 8, 9, 10, 11, 13, 14, 15, 16, 17
Accreditation	Not Accredited	5
	Rotary Mixer / Shaking / Tumbling	2, 3, 6, 7, 8, 9, 10, 11, 13, 14, 15, 16, 17
Canada Taraturant	Centrifuge	7, 10
Sample Treatment	Sonication	16
	pH Adjustment	2, 3, 8, 14
Pite a Head	Yes	3, 5, 6, 8, 9, 15, 16
Filter Used	No	2, 7, 10, 11, 13, 14, 17
December 9.1 days	0.1 M Sulfuric Acid	2, 3, 6, 7, 8, 9, 10, 11, 13, 14, 15, 16, 17
Desorption Solution	Purified Water	5
	GC-MS	14
	HPLC-DAD	5
Instrument Used for Quantification	HPLC-MS	6, 13
Quantification	HPLC-MS/MS	7, 8, 9, 10, 11, 16
	UPLC-MS/MS	2, 3, 15, 17
	NMI	9, 11, 14
	Cerilliant	2, 6, 15
Sources of	Lipomed	7, 10, 13, 16, 17
Calibration Standard	PM Separations	8
	Sigma Aldrich	5 (Methamphetamine)
	Other / Not Reported	3, 5 (MDMA)
	Methamphetamine-D5	3, 7, 8, 10, 13, 16
Internal Standard –	Methamphetamine-D9	11
Methamphetamine	Methamphetamine-D14	6, 9, 14, 15, 17
	Not Reported	2, 5
Internal Standard –	MDMA-D5	8, 9, 14, 16
MDMA	Not Reported	5

A comparison of *z*-scores with various methodology parameters for scored analytes is given in Figures 8 to 11.

One participant used purified water as their desorption solution, with a 16 hour extraction, and samples were analysed using HPLC-DAD. They were the only participant not to use sulfuric acid as the desorption solution, and also the only participant to use HPLC-DAD. This participant's results were consistently low as compared to the assigned values and spiked values, and this may have been due to their methodology.

There was no trend observed with the other methodologies employed.

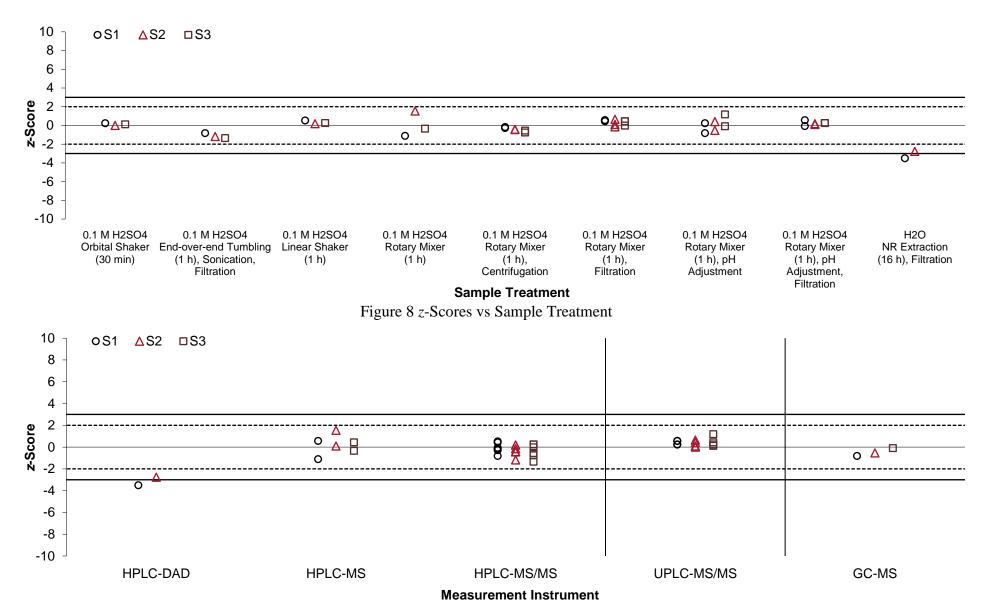


Figure 9 z-Scores vs Measurement Instrument

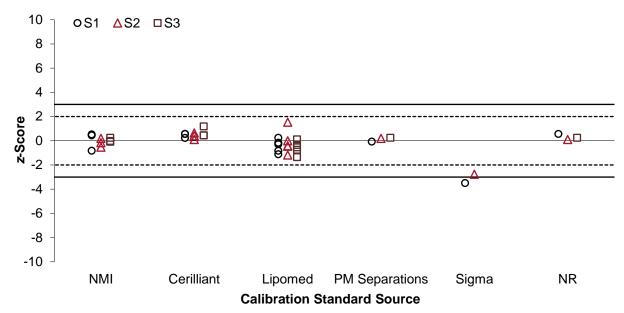


Figure 10 z-Scores vs Calibration Standard Source

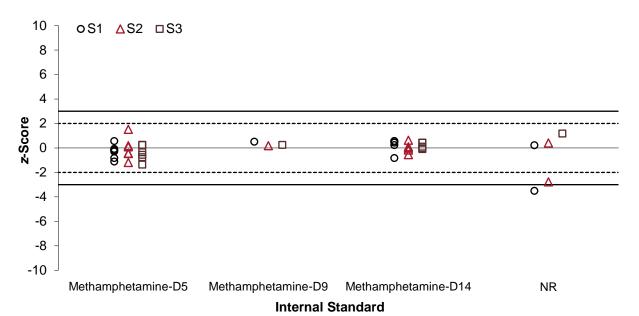


Figure 11 z-Scores vs Internal Standard

## 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.<sup>2</sup>

For methamphetamine in indoor residential surface areas, the IL criteria is  $0.5~\mu g/100~cm^2$  (corresponding in this study to  $0.5~\mu g/wipe$ ). For MDMA in indoor residential surface areas, the IL criteria is  $7~\mu g/100~cm^2$  (corresponding in this study to  $7~\mu g/wipe$ ). Laboratories should be able to identify if a sample is above or below the relevant IL.

For this study, Samples S1, S2 and S3 were prepared to contain methamphetamine greater than the relevant IL (that is, the theoretical location which was sampled would require remediation). Sample S4 was prepared to contain MDMA less than the relevant IL (that is, the theoretical location which was sampled would not require further remediation).

A summary of results and uncertainties for each sample is presented graphically in Figures 12 to 15.

In this study, most results reported were correctly above or below the relevant IL, as compared to the spiked values (SV) and/or assigned values (AV) for that sample. However, there were some inconsistencies observed, specifically for Samples S3 and S4 where the levels of methamphetamine and MDMA were closer to the ILs. For Sample S3 Laboratory 16 and Sample S4 Laboratory 14, their results were correctly above or below the IL respectively, however their uncertainties spanned the IL. For Sample S3, Laboratory 5 reported that the sample was less than their limit of reporting (LOR), however their LOR was greater than the relevant IL.

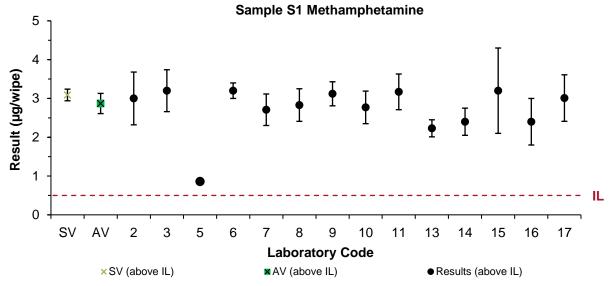


Figure 12 Sample S1 Spiked Value, Assigned Value and Participant Results

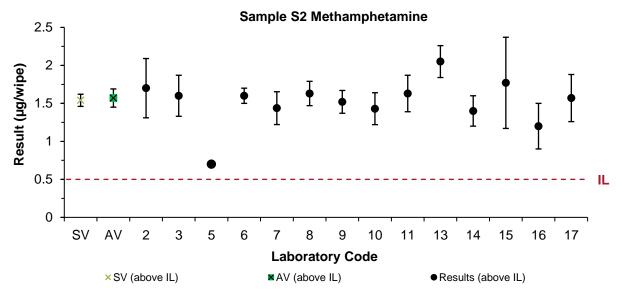


Figure 13 Sample S2 Spiked Value, Assigned Value and Participant Results

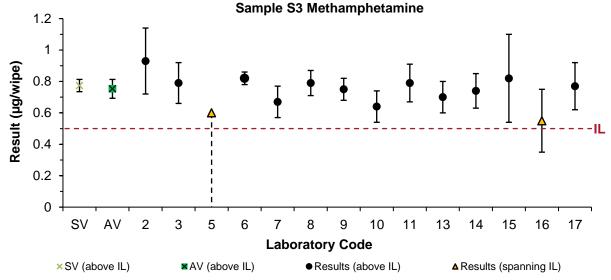


Figure 14 Sample S3 Spiked Value, Assigned Value and Participant Results

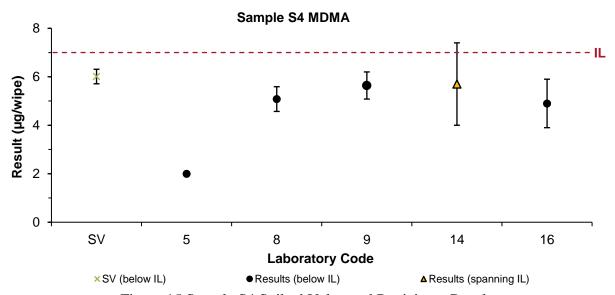


Figure 15 Sample S4 Spiked Value and Participant Results

## 6.7 Comparison with Previous Controlled Substances in Wipes PT Studies

NMI has run four 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 (2018–2023) is presented in Figure 16. 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 97% and 81% respectively.

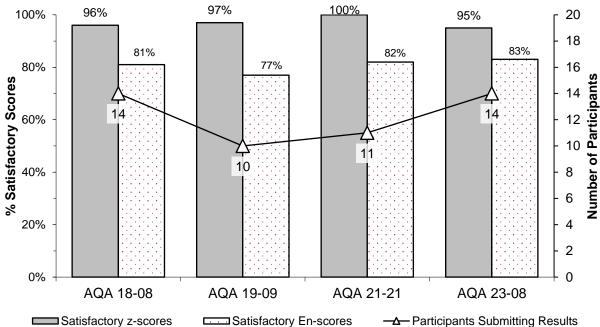


Figure 16 Summary of Participation and Satisfactory Performance in Controlled Substances in Wipes PT Studies

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  $|z| \le 2.0$ . 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

Please note that for all undated references, the latest edition of the referenced document (including any amendments) applies.

- [1] ISO/IEC 17043:2010, Conformity assessment General requirements for proficiency testing.
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- [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, Statistical methods for use in proficiency testing by interlaboratory comparison.
- [7] ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories.
- [8] Eurachem/CITAC Guide CG 4, QUAM:2012.P1, *Quantifying Uncertainty in Analytical Measurement*, 3<sup>rd</sup> edition, viewed October 2023, <a href="http://www.eurachem.org/images/stories/Guides/pdf/QUAM2012\_P1.pdf">http://www.eurachem.org/images/stories/Guides/pdf/QUAM2012\_P1.pdf</a>>.
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- [10] 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.
- [11] NIOSH, 2011, NIOSH Manual of Analytical Methods (NMAM), 5<sup>th</sup> edition, Methamphetamine on Wipes by Liquid Chromatography/Mass Spectrometry NIOSH Method 9111, viewed October 2023, <a href="https://www.cdc.gov/niosh/docs/2014-151/pdfs/methods/9111.pdf">https://www.cdc.gov/niosh/docs/2014-151/pdfs/methods/9111.pdf</a>>
- [12] NIOSH, 2011, *NIOSH Manual of Analytical Methods (NMAM)*, 5<sup>th</sup> edition, *Methamphetamine and Illicit Drugs, Precursors and Adulterants on Wipes by Liquid-Liquid Extraction NIOSH Method 9106*, viewed October 2023, <a href="https://www.cdc.gov/niosh/docs/2014-151/pdfs/methods/9106.pdf">https://www.cdc.gov/niosh/docs/2014-151/pdfs/methods/9106.pdf</a>

#### APPENDIX 1 HOMOGENEITY AND STABILITY

## **A1.1 Homogeneity**

No homogeneity testing was completed for this study as the samples were prepared using a process demonstrated in previous NMI PT studies to produce sufficiently homogeneous samples. The results of this study also gave no reason to question the samples' homogeneity. Comparisons of results for all scored analytes to sample number analysed by participants are presented in Figures 17 to 19. Results have only been included if the sample number was known (i.e. when the participant was sent only one sample set). No trend was observed.

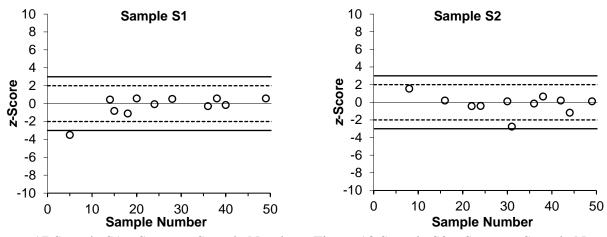


Figure 17 Sample S1 z-Score vs Sample Number Figure 18 Sample S2 z-Score vs Sample Number

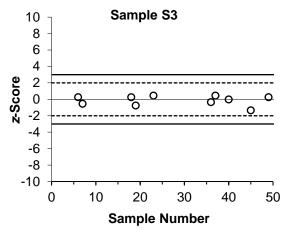


Figure 19 Sample S3 z-Score vs Sample Number

## A1.2 Stability

No stability testing was conducted for this study as the process used to prepare, store and dispatch the samples was demonstrated in previous NMI PT studies to produce sufficiently stable samples.

Samples were stored at 4°C after preparation and before dispatch. Samples were dispatched with ice blanket cells or cooler bricks on 22 May 2023, and participants were advised to store the samples refrigerated if analyses could not be commenced on the day of receipt.

Days in transit, reported sample condition on receipt and date of analysis are presented in Table 13. All participants reported that their samples arrived in at least an acceptable condition. All samples were delivered within seven days; ambient temperatures for this amount of time should not invalidate results, as supported by data from NIOSH. 11,12

Table 13 Summary of Days in Transit, Reported Arrival Condition, and Analysis Date

Lab. Code	Days in Transit	Reported Arrival Condition	Date of Analysis
2	1	Good - chilled	23/05/2023
3	1	Good	5/06/2023
5	7	ok	3/07/2023
6	2	Acceptable	7/06/2023
7	1	Acceptable	25/05/2023
8	1	GOOD	20/06/2023
9	1	Good	21/06/2023
10	1	Acceptable	23/05/2023
11	2	Acceptable	26/05/2023
13	1	Acceptable for analysis	15/08/2023
14	1	Good	30/05/2023
15	1	fine	2/06/2023
16	1	Fit for Analysis	30/06/2023
17	1	Acceptable	26, 27, 28/07/2023

A comparison of *z*-scores to the analysis date is also presented in Figure 20. No significant correlation was observed.

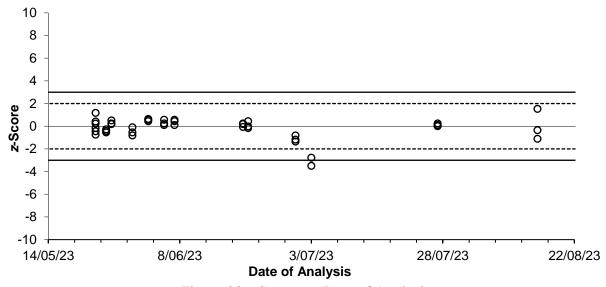


Figure 20 z-Scores vs Date of Analysis

## APPENDIX 2 ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE AND $E_{rr}$ -SCORE CALCULATIONS

## A2.1 Robust Average and Associated Uncertainty

When the robust average is calculated using the procedure described in ISO 13528,<sup>6</sup> the uncertainty is estimated as:

$$u_{rob\ av} = \frac{1.25 \times S_{rob\ av}}{\sqrt{p}}$$
 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 14.

Table 14 Uncertainty of Robust Average for Methamphetamine in Sample S3

No. results (p)	13	
Robust Average	0.753 µg base/wipe	
$S_{rob\;average}$	0.086 µg base/wipe	
$u_{rob\ average}$	0.030 µg base/wipe	
k	2	
$U_{rob\ average}$	0.060 µg base/wipe	

Hence, the robust average for methamphetamine in Sample S3 is  $0.753 \pm 0.060 \,\mu g$  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 (Section 4).

A worked example is set out below in Table 15.

Table 15 z-Score and E<sub>n</sub>-Score Calculation for Sample S1 Result Reported by Laboratory 2

Participant Result (µg base/wipe)	Assigned Value (µg base/wipe)	Target SD	z-Score	E <sub>n</sub> -Score
$3.0 \pm 0.68$	$2.87 \pm 0.26$	20% as PCV, or: 0.2 × 2.87 = 0.574 μg base/wipe	$z = \frac{3.0 - 2.87}{0.574}$ $= 0.23$	$E_n = \frac{3.0 - 2.87}{\sqrt{0.68^2 + 0.26^2}}$ $= 0.18$

#### **APPENDIX 3 ACRONYMS AND ABBREVIATIONS**

CITAC Cooperation on International Traceability in Analytical Chemistry

CRM Certified Reference Material
CV Coefficient of Variation
DAD Diode Array Detection

GAG General Accreditation Guidance (NATA)

GC Gas Chromatography

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

LOR Limit of Reporting

Max Maximum Md Median

MDMA 3,4-methylenedioxymethamphetamine

Min Minimum

MS Mass Spectrometry

MS/MS Tandem Mass Spectrometry
MU Measurement Uncertainty
N Number of numeric results

NA Not Applicable

NATA National Association of Testing Authorities, Australia NIOSH National Institute for Occupational Safety and Health

NMI National Measurement Institute, Australia

NPAAC National Pathology Accreditation Advisory Council

NR Not Reported

NS Not Supplied

NT Not Tested

PCV Performance Coefficient of Variation

PT Proficiency Testing
RA Robust Average
RM Reference Material
SD Standard Deviation

SI International System of Units

SS Spiked Samples SV Spiked Value

UPLC Ultra Performance Liquid Chromatography

#### END OF REPORT