Australian Government
Department of Industry, Science and Resources

National Measurement Institute

Proficiency Test Final Report AQA 26-03 MDMA/Methamphetamine

September 2026

Our purpose is to help the government build a better future for all Australians through enabling a productive, resilient and sustainable economy, enriched by science and technology.

measurement.gov.au

© Commonwealth of Australia 2026.

Unless otherwise noted, the Commonwealth owns the copyright (and any other intellectual property rights, if any) in this publication.

All material in this publication is provided under a Creative Commons Attribution 4.0 International Licence (CC BY 4.0), with the exception of:

- the Commonwealth Coat of Arms;
- the logo of the Department of Industry, Science and Resources;
- photographs of our staff and premises; and
- content supplied by third parties.

Creative Commons Attribution 4.0 International Licence is a standard form licence agreement that allows you to copy, distribute, transmit and adapt this publication provided you attribute the work. A summary of the licence terms is available at: creativecommons.org/licenses/by/4.0/. Further details are available on the Creative Commons website, at: creativecommons.org/licenses/by/4.0/legalcode.

You may not copy, distribute, transmit or adapt any material in this publication in any way that suggests that this department or the Commonwealth endorses you or any of your services or products.

Attribution

Material contained in this publication is to be attributed to this department as:

© Commonwealth of Australia, Department of Industry, Science and Resources, Proficiency Test Final Report AQA 26-03 MDMA/Methamphetamine, 2026.

Third party copyright

Wherever a third party holds copyright in material contained in this publication, the copyright remains with that party. Their permission may be required to use the material.

This department has made all reasonable efforts to:

- clearly label material where the copyright is owned by a third party;
- ensure that the copyright owner has consented to this material being contained in this publication.

Using the Commonwealth Coat of Arms

The terms of use for the Coat of Arms are available on the Department of Prime Minister and Cabinet's website, at https://www.pmc.gov.au/resources/commonwealth-coat-arms-information-and-guidelines

ACKNOWLEDGMENTS

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

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

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

Jenny Xu

Aaron Mamo

Zoë Baker-Moss

Lesley Johnston

Raluca Iavetz

Manager, Chemical Reference Values

105 Delhi Rd, North Ryde, NSW 2113, Australia

Phone: +61 2 9449 0234

Email: proficiency@measurement.gov.au

Accredited for compliance with ISO/IEC 17043

Accreditation No. 198; Site No. 14485

SUMMARY

This report presents the results of proficiency study AQA 26-03 MDMA/Methamphetamine.

The sample set consisted of 2 samples containing 3,4-methylenedioxymethamphetamine (MDMA) and 2 samples containing methamphetamine.

The assigned value for Sample S1 was the robust average of participants' results. The associated uncertainty was evaluated from the robust standard deviation of the participants' results. The consensus of participants' results is not traceable to any external reference, so although expressed in SI units, metrological traceability of the assigned value for Sample S1 has not been established.

The assigned values for Samples S2, S3, and S4 were the reference values as determined by quantitative nuclear magnetic resonance (qNMR) spectroscopy, with maleic acid as the internal standard. The reference values are traceable to the SI through Australian Standards for mass via balance calibration certificates and the purity of the NMIA maleic acid CRM QNMR010 (Batch No.: 10-Q-02).

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

i. Assess participants' capabilities to measure MDMA and methamphetamine in samples typical of a routine seizure.

Of 116 z-scores, 103 (89%) returned a z-score of $|z| \leq 2.0$, indicating an acceptable performance.

Of 116 E_n-scores, 105 (91%) returned a E_n-score of $|E_n| < 1.0$, indicating agreement of the participant's results with the assigned value within their respective expanded uncertainties.

Laboratories 1, 6, 8, 9, 10, 11, 13, 14, 15, 19, 20, 21, 22, 23, 24, 27, 29, and 30 returned acceptable z-scores and E_n-scores for all four samples.

ii. Develop the practical application of measurement uncertainty, and provide participants with information that will assist uncertainty evaluations.

Of 116 numeric results, 112 (97%) were reported with an associated measurement uncertainty. Participants used a variety of methods to evaluate their measurement uncertainty. These methods produced relative uncertainties ranging from 0.14% to 23% of the reported results.

iii. Assess participants' ability to identify cutting agents commonly found in controlled drug preparation.

Sample S1 was cut with cellulose and sucrose, Sample S2 was left uncut, Sample S3 was cut with sucrose, and Sample S4 was cut with dimethyl sulfone.

Nineteen participants (59%) reported on the identity of at least one cutting agent in the samples.

Laboratory 8 correctly identified all cutting agents added.

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

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

TABLE OF CONTENTS

1 INTRODUCTION 1
1.1 NMIA Proficiency Testing Program 1
1.2 Study Aims 1
1.3 Study Conduct 1
2 STUDY INFORMATION 2
2.1 Study Timetable 2
2.2 Participation and Laboratory Code 2
2.3 Test Material Specification 2
2.4 Test Sample Homogeneity and Stability 2
2.5 Sample Dispatch and Receipt 3
2.6 Instructions to Participants 3
2.7 Interim Report and Preliminary Report 3
3 PARTICIPANT LABORATORY INFORMATION 4
3.1 Test Methods Reported by Participants 4
3.2 Details of Participants' Calibration Standards 7
3.3 Reported Basis of Participants' Measurement Uncertainty Evaluations 8
3.4 Participants' Comments 11
4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS 12
4.1 Results Summary 12
4.2 Outliers and Extreme Outliers 12
4.3 Assigned Value 12
4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation 12
4.5 Performance Coefficient of Variation 12
4.6 Standard Deviation for Proficiency Assessment 13
4.7 z-Score 13
4.8 E_n-Score 13
4.9 Traceability and Measurement Uncertainty 13
5 TABLES AND FIGURES 14
6 DISCUSSION OF RESULTS 23
6.1 Assigned Value 23
6.2 Measurement Uncertainty Reported by Participants 23
6.3 z-Score 24
6.4 E_n-Score 26
6.5 Summary of Participants' Results and Performances 26
6.6 Identification of Cutting Agents 28
6.7 Participants' Analytical Methods 29
6.8 Comparison of Results and Date of Analysis 31
6.9 Comparison with Previous PT Studies 32
7 REFERENCES 35
APPENDIX 1 REFERENCE VALUES 36
APPENDIX 2 ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE, AND E_n-SCORE CALCULATIONS 38
A2.1 Robust Average and Associated Uncertainty 38
A2.2 z-Score and E_n-Score Calculations 38

APPENDIX 3 ACRONYMS AND ABBREVIATIONS 39

1 INTRODUCTION

1.1 NMIA Proficiency Testing Program

The National Measurement Institute Australia (NMIA) 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 comparisons'.[1] NMIA PT studies target chemical testing in areas of high public significance such as trade, environment, law enforcement, and food safety. NMIA offers studies in:

- per- and polyfluoroalkyl substances in soil, biosolids, compost, water, biota, food, and consumer goods;
- hydrocarbons, phenols, and other organic compounds in soil and water;
- pesticide residues in soil, compost, water, fruit, vegetables, and herbs;
- metals in soil, water, food, filters, and paint;
- nutrients, anions, and physical tests in water and soil;
- chlorophyll a in water; and
- controlled drug assay, drugs in wipes, and clandestine laboratory.

1.2 Study Aims

The aims of the study were to:

- assess participants' capabilities to measure 3,4-methylenedioxymethamphetamine (MDMA) and methamphetamine in samples typical of a routine seizure;
- develop the practical application of measurement uncertainty, and provide participants with information that will assist uncertainty evaluations;
- assess participants' ability to identify cutting agents commonly found in controlled drug preparation; and
- produce materials that can be used in method validation and as control samples.

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

1.3 Study Conduct

The conduct of NMIA PT studies is described in the NMIA Study Protocol for Proficiency Testing.[2] The statistical methods used are described in the NMIA Chemical Proficiency Testing Statistical Manual.[3] These documents have been prepared with reference to ISO/IEC 17043 and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[1,4]

NMIA is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of proficiency testing schemes.[1] This study falls within the scope of NMIA's accreditation (Accreditation No. 198; Site No. 14485).

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitations sent	19/01/2026
Samples sent	30/03/2026
Results due	13/07/2026
Interim Report	15/07/2026
Preliminary Report	29/07/2026

Due to sample delivery delays to some participants, the project timeline was extended.

2.2 Participation and Laboratory Code

Thirty-one laboratories registered to participate, with 2 laboratories each requesting 2 sets of samples to be analysed independently (total of 33 participants). Each participant was assigned a confidential laboratory code number for this study. Thirty-two participants submitted results by the due date.

2.3 Test Material Specification

Four test samples were prepared in March 2026. Samples S1 and S2 contained MDMA hydrochloride, and Samples S3 and S4 contained methamphetamine hydrochloride. The starting materials were supplied by the Australian Federal Police.

Cellulose, sucrose, and dimethyl sulfone purchased from Sigma-Aldrich were used as cutting agents. Sample S1 was cut with cellulose and sucrose, Sample S2 was left uncut, Sample S3 was cut with sucrose, and Sample S4 was cut with dimethyl sulfone.

The MDMA and methamphetamine were ground and sieved through a 180 µm sieve. The cutting agents were processed similarly. Test samples were prepared by mixing a known mass of sieved drug with known amounts of sieved cutting agent(s) in a tumbler overnight. Approximately 150 mg aliquots of each test sample were weighed into labelled glass vials.

Sample S1 was prepared to contain approximately 33.8% MDMA base (m/m).

Sample S2 was estimated to be around 77.0% MDMA base (m/m).

Sample S3 was prepared to contain approximately 41.3% methamphetamine base (m/m).

Sample S4 was prepared to contain approximately 70.0% methamphetamine base (m/m).

2.4 Test Sample Homogeneity and Stability

The preparation of homogeneous test samples is an important part of a PT study. Given the small (< 150 mg) test portions normally used for controlled substance analysis, the particle size must be sufficiently small and uniformly distributed to ensure minimal influence on analytical precision.

All samples were prepared using the same procedure as previous controlled drug PT studies. This has been demonstrated to produce sufficiently homogeneous samples. No homogeneity testing was conducted in this PT study. Results returned by the participants gave no reason to question the homogeneity of the test samples.

To assess the stability of the samples, results returned by participants were compared to the dates of analysis (Section 6.8). The results gave no reason to question the samples' stability.

2.5 Sample Dispatch and Receipt

A set of 4 test samples, with each sample containing approximately 150 mg of test material, was dispatched to most participants on 30 March 2026. Dispatch was delayed for some participants due to public holidays and delays with receiving export permits.

The following items were also sent with the samples:

- a letter which included a description of the test samples and instructions for participants; and
- a form for participants 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:

- Analyse each sample for the amount of drug by your routine test method. It is recommended to thoroughly mix the contents of each vial before taking a test portion for analysis.
- For each sample report % m/m drug as base. Report this figure as if reporting to a client.
- For each result report an evaluation of your expanded uncertainty as % m/m drug as base.
- Report the identity of cutting agents in all samples if this is within your normal scope of analysis.
- Give brief details of your:
    - basis of uncertainty evaluation (e.g. uncertainty budget, repeatability precision, coverage factor)
    - analytical method (e.g. sample treatment, instrument type, calibration method)
    - reference standard (e.g. source, purity)
- A result spreadsheet will be emailed to you. Please complete this spreadsheet and return by email to jenny.xu@measurement.gov.au.
- Results are to be returned by 25 May 2026.

The results due date was later changed to 13 July 2026. This was to accommodate sample delivery delays to some participants.

2.7 Interim Report and Preliminary Report

An Interim Report was emailed to all participants on 15 July 2026.

A Preliminary Report was emailed to all participants on 29 July 2026. This report included a summary of the results reported by participants, assigned values, performance coefficients of variation, 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 Test Methods Reported by Participants

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

Lab. Code	Analyte	Extraction Solvent	Internal Standard	Calib. Points	Technique	Detector	Column	Instrument Comments
1	All	Methanol	Diazepam	6	GC	FID	128-5512 DB-5ms	
2	All	Methanol	none	5	HPLC	DAD	Phenomenex C-18-XB	
3*	MDMA	acetic acid, acetonitrile, water	No IS	4	HPLC	DAD	Poroshell 120 EC-C18	Wavelength: 234 nm
	Methamphetamine							Wavelength: 210 nm
5	All	Methanol	none	5	HPLC	DAD	Agilent ZORBAX Eclipse Plus C18 4.6 x 250mm, 5um	Wavelength: 210nm
6	MDMA	Water	Phentermine	1	UPLC	DAD	Agilent Zorbax SB-C8	Wavelength: 215 nm
	Methamphetamine		N/A				Phenomenex Luna Omega PS C18	
7	MDMA	Ethanol	Propyl paraben	8	UPLC	DAD	BEH shield RP18	
	Methamphetamine			7				
8*	All	Iso-octane	Dodecane	3	GC	FID	HP1-MS	
9*	MDMA	D2O	Maleic Acid	None	QNMR			60 MHz - 3 signals : 7.2 ppm - 6.3 ppm - 1.6 ppm
	Methamphetamine							60 MHz - 2 signals : 7.2 ppm (except for S4 due to an interference) – 1.2 ppm
10*	MDMA	methanol	NO	1	HPLC	DAD	zorbax eclipse XDB 5 microns C-18 (4.6 mm x 150 mm)	Wavelength: 230 nm
	Methamphetamine							Wavelength: 210 nm

Lab. Code	Analyte	Extraction Solvent	Internal Standard	Calib. Points	Technique	Detector	Column	Instrument Comments
11	MDMA	phosphate buffer pH 2.5	-	5	HPLC	DAD	Luna Omega 3um PS C18 100Å, 150 x 4.6 mm	Wavelength: 234 nm
	Methamphetamine							Wavelength: 210 nm
12	All	Methanol	selegiline	4	HPLC	UV/Vis	HSS C18 1.8µm 2.1x150mm	
13	All	Ethyl Acetate	Diphenylamine	5	GC	FID	HP1	
14	All	MeOH : KOH buffer	Methoxyphenamine	3	UPLC	DAD	Acquity UPLC BEH C18 1.7um (2.1x1000)	Wavelength: 254nm
15	All	Methanol	Strychnine	6	UPLC	DAD	Waters Acquity UPLC BEH phenyl 1.8 um × 2.1 mm ×100 mm	
16	MDMA	Acetonitrile		4	HPLC	UV/Vis	C18	
17	MDMA	MeOH	MDMA-D5		LC	MS/MS	C18	
	Methamphetamine		Methamphetamine-D5					
18*	Methamphetamine	Dissolution in acetonitrile/water	Methoxyphenamine HCl	3	HPLC	DAD	Alltima C-18	
19	All	Water	N/A	7	HPLC	DAD	Luna 3um PFP(2) 100A 2x100mm	Wavelength: 214nm
20	All	Methanol	2,4,6-trimethylpyridine	6	GC	FID	RTX-5-Amine	

Lab. Code	Analyte	Extraction Solvent	Internal Standard	Calib. Points	Technique	Detector	Column	Instrument Comments
21	MDMA	Eluent: Acetonitrile, ammonium acetate, diethylamine & water	None	4	HPLC	DAD	LiChrosphere RP-18 (5 um)	Wavelength: 285 nm
	Methamphetamine	Eluent: Methanol, water, pyrrolidine & acetic acid		3	UPLC		ACQUITY UPLC BEH C18 (1.7 um)	Wavelength: 254 nm
22*	All	D2O	Maleic acid	NA	QNMR		NA	
23	MDMA	Methanol	N/A	6	HPLC	DAD	Luna C-18	Wavelength: 285nm
	Methamphetamine							Wavelength: 258nm
24*	All	D2O	maleic acid	-	QNMR			400MHz shield
25	All	Methanol	Propylparaben	5	UPLC	PDA	ACQUITY C-18	
26	All	Chloroform	Nortriptyline	1	GC	FID	HP5	
27	All	water	-	1	UPLC	UV/Vis	Acquity BEH C18	
28	MDMA	acetonitrile/water (80/20)	external standard	3	HPLC	DAD	C8	
29	All	Ethanol	Eicosane	6	GC	FID	HP1	
30	MDMA	Water	Phentermine	1	UPLC	DAD	Agilent Zorbax SB-C8	Wavelength: 215 nm
	Methamphetamine		N/A				Phenomenex Luna Omega PS C18	
31	MDMA	Methanol	Methadone	5	GC	FID	Rxi-5ms	
32*	All				GC	MS		
33*	All	Toluene	Phenethylamine	5	GC	FID	HP5	

*Additional information in Table 2.

Table 2 Participants' Test Methods Additional Comments

Lab. Code	Analyte	Participant Comments
3	All	5,20,60,100
8	MDMA	400uL of ammonia solution is added per 10mL ISS For S2, methylamphetamine was below our Limit of Quantification, therefore has been reported as "(trace amount)" rather than a specific purity result per our SOP.
	Methamphetamine	200uL of ammonia solution is added per 5mL ISS
9	All	Internal Standard used: Maleic Acid - Sigma Aldrich - 92816 - 99.93 % - BCCK2148.
10	All	external standard
18	Methamphetamine	Linear regression
22	All	No reference standard involved
	MDMA	Methamphetamine present in S2 - insufficient sample to determine purity
24	All	1H-q-NMR
32	All	We do not have the ability to complete a quant on these samples, previous quants have been completed by NMR, but have no access at this time.
33	MDMA	Methamphetamine present in S2 - insufficient sample to determine purity

3.2 Details of Participants' Calibration Standards

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

Table 3 Participant Calibration Standards

Lab. Code	MDMA		Methamphetamine	
	Reference Standard	Purity (%)	Reference Standard	Purity (%)
1	Lipomed HCl MDMA	83.7	Lipomed HCl Methamphetamine	79.5
2	Chiron	99.4	Chiron	99.7
3	Lipomed	99.95	Lipomed	99.95
5	NMIA	99.7±0.3	NMIA	99.8±0.3
6	Lipomed	99.8151 ± 0.0288	Lipomed	99.950 ± 0.05
7	NMIA	97.5	NMIA	99.8
8	NMIA	99.7±0.3	NMIA	99.5±1.2
11	Chiron	98.9	Chiron	99.7
13	Lipomed	99.8151	Lipomed	99.38
14	NMI	99.7±0.3	NMI	99.8±0.3
15	NMI	99.8	NMI	99.8
16	LIPOMED	99.5		
18			Sigma-Aldrich	100
19	NMI	99.8	NMI	99.8

Lab. Code	MDMA		Methamphetamine	
	Reference Standard	Purity (%)	Reference Standard	Purity (%)
20	Lipomed	99.8151	Lipomed	99.987
21	NMIA	99.8	Chiron	99.7
23	NMI	99.8	in house	100
24	maleic acid	99.0	maleic acid	99.0
25	NMI	99.7	NMI	99.8
26	Cayman	99.65	in house RM	99.8
27	NMI	99.7	NMI	99.8
28	LGC	83.3		
29	Lipomed	99.81	Lipomed	99.005
30	Lipomed	99.8151 ± 0.0288	Lipomed	99.950 ± 0.05
31	LGC	1.016		
33	NMIA	99.8	NMIA	99.8

3.3 Reported Basis of Participants' Measurement Uncertainty Evaluations

Participants were requested to provide information about the basis of their measurement uncertainty (MU) evaluations. Responses are presented in Tables 4 and 5. Some responses may be modified so that the participant cannot be identified.

Table 4 Basis of Measurement Uncertainty Evaluation

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
1	Evaluating Measurement Uncertainty by black box with pairs of values k = 2	Standard deviation from PT studies only		ISO/GUM ENAC G 09 or ISO 21748
2	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples - RM / Ex PT Sample Duplicate analysis	Instrument calibration	
3**	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS	Recoveries of SS	
5	Bottom Up (ISO/GUM, fish bone/cause and effect diagram) Coverage factor not reported			ISO/GUM
6	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis	Laboratory bias from PT studies Recoveries of SS	Eurachem/CITAC Guide

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
8	Bottom Up (ISO/GUM, fish bone/cause and effect diagram) k = 2	Control samples - CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Matrix effects Recoveries of SS Standard purity	Eurachem/CITAC Guide
9	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples - RM / Ex PT Sample	Homogeneity of sample	ISO/GUM
10	Coverage factor not reported	Control samples - CRM Duplicate analysis	Instrument calibration Laboratory bias from PT studies	
11**	Reproducibility, bias, sample heterogeneity k = 2	Control samples - RM Duplicate analysis	Instrument calibration Homogeneity of sample Standard purity	ENFSI Best Practise Manual, Eurachem/CITAC Guide
12	Top Down - precision and evaluations of the method and laboratory bias k = 1	Control samples - RM / Ex PT Sample Duplicate analysis		EA-04/16, 'EA guidelines on the expression of uncertainty in quantitative testing'.
13	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Control samples - RM / Ex PT Sample	Standard purity	ISO/GUM
14	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples Duplicate analysis	Homogeneity of sample Standard purity	Eurachem/CITAC Guide
15	Bottom Up (ISO/GUM, fish bone/cause and effect diagram) k = 2	Control samples - CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Laboratory bias from PT studies	ISO/GUM
16	Coverage factor not reported	Control samples - SS	Laboratory bias from PT studies	
18	Bottom Up (ISO/GUM, fish bone/cause and effect diagram) k = 2	Duplicate analysis	Instrument calibration Masses and volumes Matrix effects Recoveries of SS Standard purity	ISO/GUM
19	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - In-house control, CRM-based QC Duplicate analysis	Instrument calibration Masses and volumes	ISO/GUM

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
20	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Standard deviation from PT studies only		ISO/GUM
21	MDMA: Bottom Up (ISO/GUM, fish bone/cause and effect diagram) k = 3 Methamphetamine: Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - RM / Ex PT Sample Duplicate analysis	Instrument calibration Masses and volumes Standard purity	Internal Document
22	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - previously analysed real seizure samples Duplicate analysis	Homogeneity of sample Masses and volumes Matrix effects Standard purity	Nordtest Report TR537
23	Top Down - precision and evaluations of the method and laboratory bias k = 3	Control samples - RM / Ex PT Sample Duplicate analysis		Eurachem/CITAC Guide
24	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Standard deviation from PT studies only		
25	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Standard purity	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
26	Top Down - precision and evaluations of the method and laboratory bias k = 3	Control samples - RM / Ex PT Sample	Matrix effects	Eurachem/CITAC Guide
27	validation study k = 2			
28	Top Down - reproducibility (standard deviation) from PT studies used directly Coverage factor not reported	Control samples - CRM Duplicate analysis	Instrument calibration	Eurachem/CITAC Guide
29	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - RM / Ex PT Sample Duplicate analysis	Laboratory bias from PT studies	ISO/GUM

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
30	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis	Laboratory bias from PT studies Recoveries of SS	Eurachem/CITAC Guide
31	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2	Duplicate analysis	Masses and volumes	ISO/GUM
33	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - previously analysed real seizure samples Duplicate analysis	Homogeneity of sample Masses and volumes Matrix effects Standard purity	Nordtest Report TR537

*CRM = Certified Reference Material; PT = Proficiency Testing; RM = Reference Material; SS = Spiked Samples.
**Additional information in Table 5.

Table 5 Measurement Uncertainty Evaluation Additional Comments

Lab. Code	Participant Comments
3	accuracy profile - based on intermediate precision and repeatability
11	Relative expanded measurement uncertainty is used, K=2

3.4 Participants' Comments

Participants were invited to comment on the samples, the PT study in general, and make suggestions for future PT studies. Such feedback may be useful in improving future studies. Participants' comments are presented in Table 6, along with the study coordinator's response. Some responses may be modified so that the participant cannot be identified.

Table 6 Participants' Comments

Lab. Code	Participants' Comments	Study Coordinator's Response
11	Alpha-PVP and amphetamine proficiency tests would be highly appreciated.	Thank you for your suggestions. If there is sufficient interest from participating laboratories and if we can source suitable material, then we will run these PT studies in future.
13	We see a lot of seized ketamine. I know this is not an internationally controlled substance but would it maybe be possible to set up a proficiency test for this compound? Our lab performs an identification and quantification analysis for ketamine and 2C-B.	
31	No method for Methamphetamine quantification, however we do have an Amphetamine method for quantitation if this was ever possible to receive as a sample for analysis	
14	It would be beneficial to use containers without an inner lip as some material was caught under the lip and difficult to remove	Thank you for your comment. Our preliminary investigations have not yet identified a suitable container that also satisfies our sample storage and transport requirements. We welcome container suggestions from other participants.

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 7 to 11 with resultant summary statistics. 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.

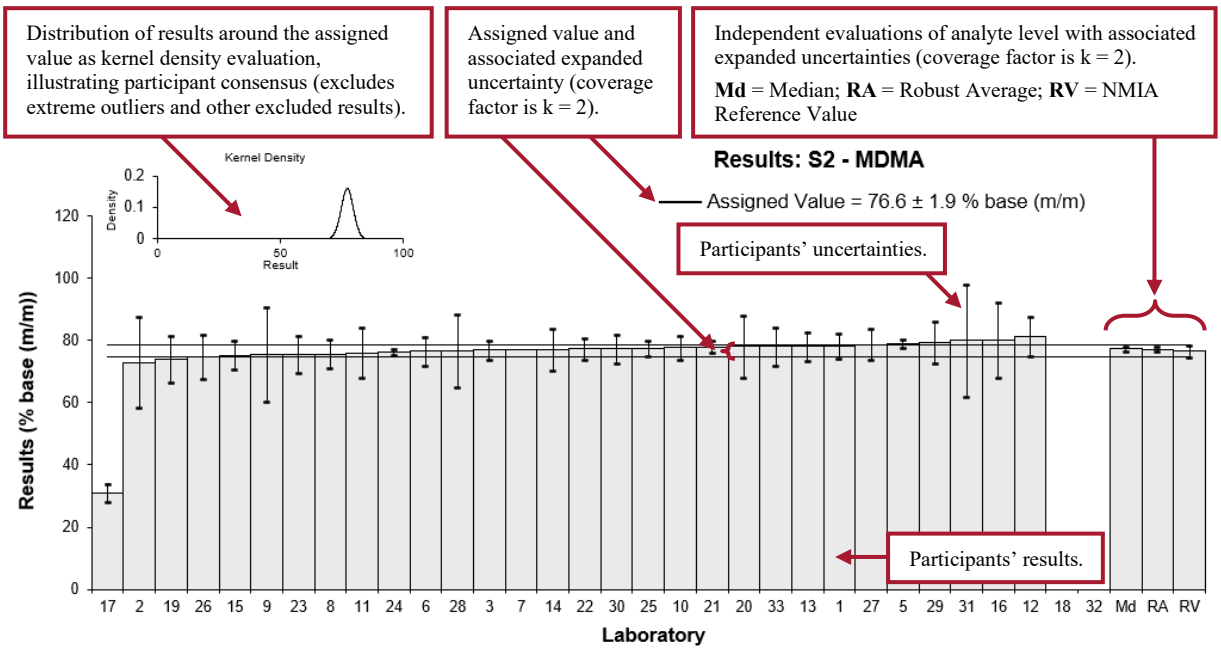


Figure 1 Guide to Presentation of Results

4.2 Outliers and Extreme Outliers

Outliers were results less than 50% and greater than 150% of the robust average, and these were removed before the calculation of the assigned values.[3,4] Extreme outliers, if applicable, were obvious blunders, e.g. results reported with incorrect units, or for a different analyte or sample, and such results were also removed before the calculation of all summary statistics.[3,4]

4.3 Assigned Value

Assigned value is defined as the 'value attributed to a particular property or characteristic of a proficiency testing item'.[1] In this PT study, the property is the % drug base (m/m) in the test samples. The assigned value for Sample S1 was the robust average of participants' results with all outliers removed, and the expanded uncertainty was evaluated from the associated robust standard deviation (robust SD) (Appendix 2). Assigned values for Samples S2, S3, and S4 were reference values determined by quantitative nuclear magnetic resonance (qNMR) spectroscopy (Appendix 1).

4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation

The robust averages and associated expanded MUs, and robust coefficients of variation (robust CVs, a measure of the variability of participants' results) were calculated as described in ISO 13528.[5] The robust CVs as presented in the Section 5 statistics tables are the robust between-laboratory CVs including outliers but excluding extreme outliers. Everywhere else in the report, the robust between-laboratory CV refers to the robust CV calculated with both outliers and extreme outliers removed.

4.5 Performance Coefficient of Variation

The performance coefficient of variation (PCV) is a set measure of the between-laboratory variation that in the judgement of the study coordinator would be expected from participants

given the analyte levels present. The PCV is not the CV of participants' results. It is set by the study coordinator and is based on the analyte levels and experience from previous studies. It is supported by mathematical models such as the Thompson-Horwitz equation.[6] By setting a realistic value for the PCV, a participant's performance does not depend on other participants' performances and can be compared from study to study when the PCV remains the same.

4.6 Standard Deviation for Proficiency Assessment

The standard deviation for proficiency assessment (SDPA, σ) is the product of the assigned value (X) and the PCV, as presented in Equation 1.

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

4.7 z-Score

For each participant's result, a z-score is calculated according to Equation 2. An example of z-score calculation is provided in Appendix 2.

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

where:

z is z-score

χ is a participant's result

X is the assigned value

σ is the SDPA from Equation 1

For the absolute value of a z-score: $|z| \leq 2.0$ is acceptable; $2.0 < |z| < 3.0$ is questionable; and $|z| \geq 3.0$ is not acceptable.

4.8 E_n-Score

The E_n-score is complementary to the z-score in assessment of laboratory performance. The E_n-score includes measurement uncertainty and is calculated according to Equation 3. An example of E_n-score calculation is provided in Appendix 2.

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

where:

E_n is E_n-score

χ is a participant's result

X is the assigned value

U_χ is the expanded uncertainty of the participant's result

U_X is the expanded uncertainty of the assigned value

For the absolute value of an E_n-score: $|E_n| < 1.0$ is acceptable; and $|E_n| \geq 1.0$ is not acceptable.

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.[7]

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

5 TABLES AND FIGURES

Table 7

Sample Details

Sample No.	S1
Matrix	Powder
Analyte	MDMA
Unit	% base (m/m)

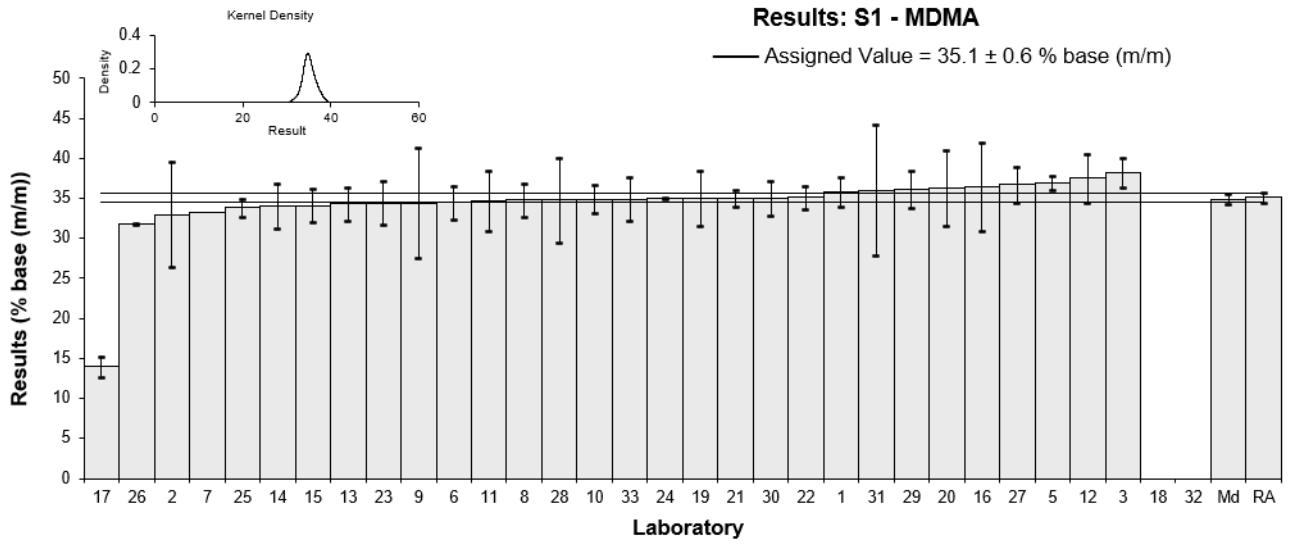
Participant Results

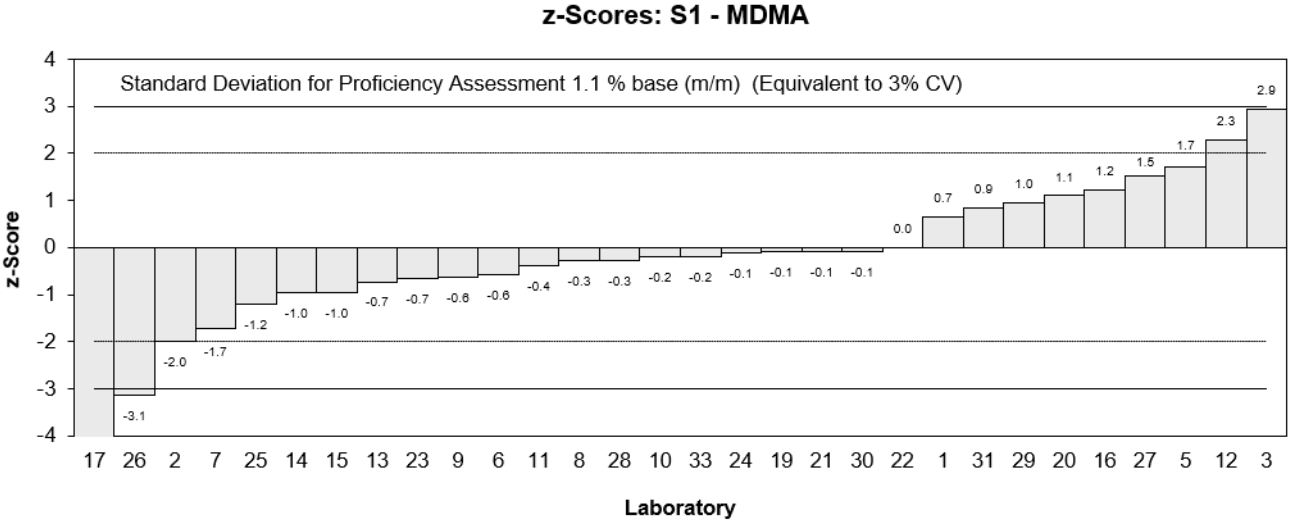
Lab. Code	Result	Uncertainty	z	E_n
1	35.8	1.8	0.66	0.37
2	33	6.6	-1.99	-0.32
3	38.2	1.8	2.94	1.63
5	36.9	0.9	1.71	1.66
6	34.5	2.1	-0.57	-0.27
7	33.3	NR	-1.71	-3.00
8	34.8	2.1	-0.28	-0.14
9	34.43	6.9	-0.64	-0.10
10	34.9	1.75	-0.19	-0.11
11	34.7	3.8	-0.38	-0.10
12	37.5	3.0	2.28	0.78
13	34.33	2.1	-0.73	-0.35
14	34.1	2.8	-0.95	-0.35
15	34.1	2.1	-0.95	-0.46
16	36.4	5.5	1.23	0.23
17**	14	1.3	-20.04	-14.74
18	NR	NR		
19	35	3.5	-0.09	-0.03
20	36.26	4.71	1.10	0.24
21	35	1	-0.09	-0.09
22	35.1	1.5	0.00	0.00
23	34.4	2.7	-0.66	-0.25
24	34.98	0.05	-0.11	-0.20
25	33.83	1.1	-1.21	-1.01
26	31.8	0.1	-3.13	-5.43
27	36.7	2.3	1.52	0.67
28	34.8	5.3	-0.28	-0.06
29	36.1	2.3	0.95	0.42
30	35.0	2.1	-0.09	-0.05
31	36	8.2	0.85	0.11
32	NR	NR		
33	34.9	2.7	-0.19	-0.07

** Excluded Result, see Section 6.1

Statistics

Assigned Value	35.1	0.6
Robust Average	35.1	0.6
Median	34.9	0.6
Mean	35.1	
N	29	
Max	38.2	
Min	31.8	
Robust SD	1.3	
Robust CV	3.6%	


Results: S1 - MDMA
Assigned Value = 35.1 ± 0.6 % base (m/m)
Kernel Density
Density
Result
Results (% base (m/m))
Laboratory
17 26 2 7 25 14 15 13 23 9 6 11 8 28 10 33 24 19 21 30 22 1 31 29 20 16 27 5 12 3 18 32 Md RA


z-Scores: S1 - MDMA
Standard Deviation for Proficiency Assessment 1.1 % base (m/m) (Equivalent to 3% CV)
z-Score
Laboratory
17 26 2 7 25 14 15 13 23 9 6 11 8 28 10 33 24 19 21 30 22 1 31 29 20 16 27 5 12 3

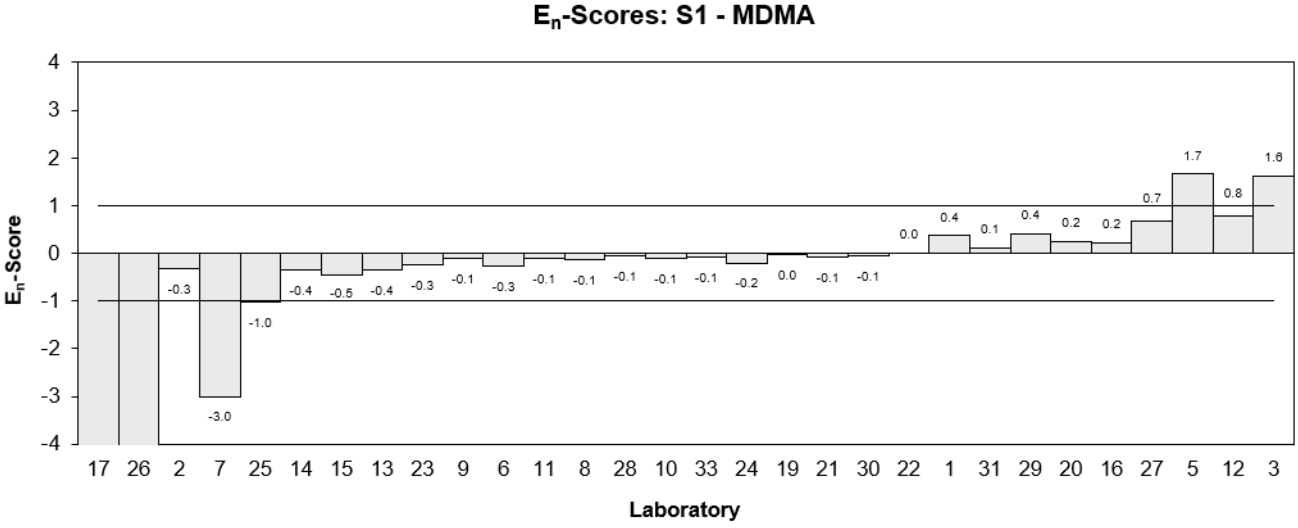

En-Scores: S1 - MDMA
En-Score
Laboratory
17 26 2 7 25 14 15 13 23 9 6 11 8 28 10 33 24 19 21 30 22 1 31 29 20 16 27 5 12 3

Figure 2

Table 8

Sample Details

Sample No.	S2
Matrix	Powder
Analyte	MDMA
Unit	% base (m/m)

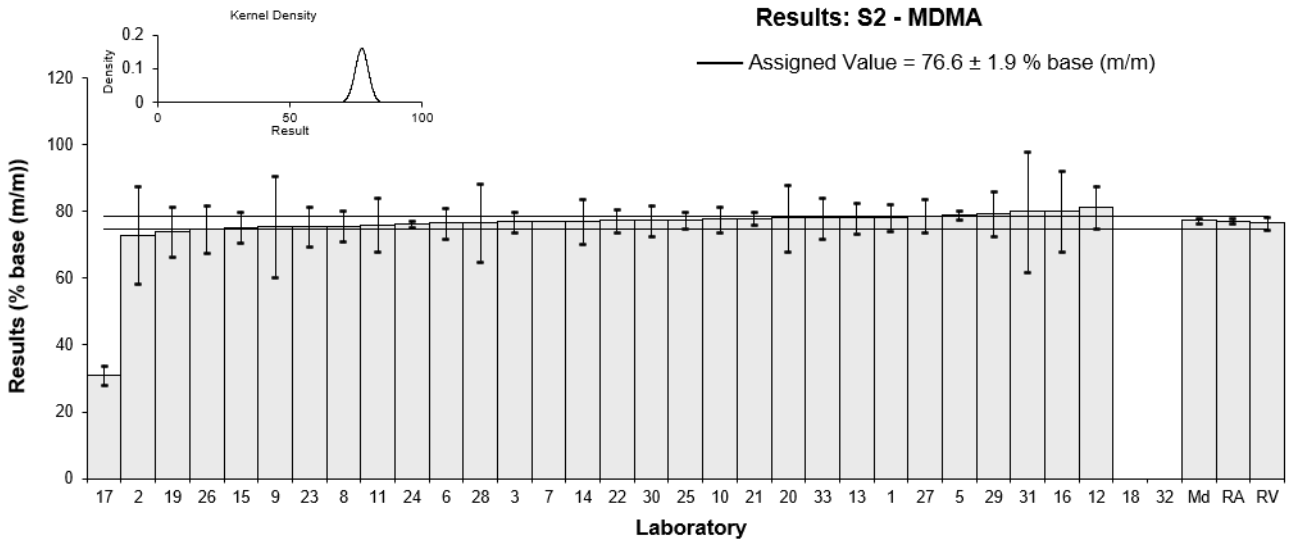
Participant Results

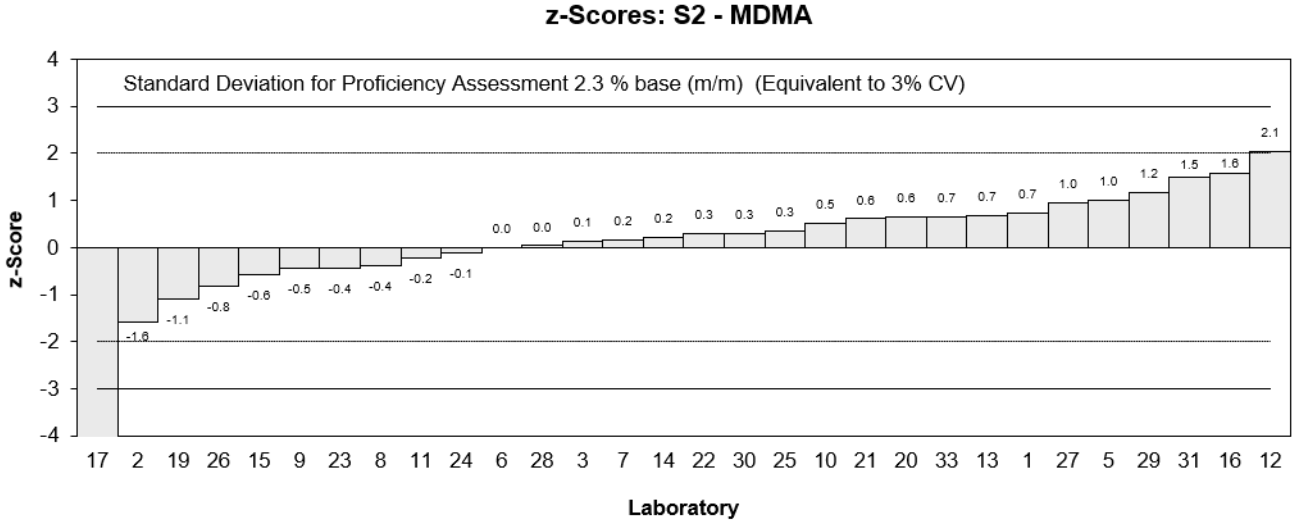
Lab. Code	Result	Uncertainty	z	E_n
1	78.3	3.9	0.74	0.39
2	73	14.6	-1.57	-0.24
3	76.9	3.2	0.13	0.08
5	78.9	1.4	1.00	0.97
6	76.6	4.6	0.00	0.00
7	77.0	NR	0.17	0.21
8	75.7	4.6	-0.39	-0.18
9	75.57	15.1	-0.45	-0.07
10	77.8	3.89	0.52	0.28
11	76.1	8.2	-0.22	-0.06
12	81.3	6.5	2.05	0.69
13	78.15	4.7	0.67	0.31
14	77.1	6.7	0.22	0.07
15	75.3	4.7	-0.57	-0.26
16	80.2	12	1.57	0.30
17**	31	3	-19.84	-12.84
18	NR	NR		
19	74.1	7.41	-1.09	-0.33
20	78.08	10.15	0.64	0.14
21	78	2	0.61	0.51
22	77.3	3.3	0.30	0.18
23	75.6	6	-0.44	-0.16
24	76.33	0.91	-0.12	-0.13
25	77.39	2.4	0.34	0.26
26	74.7	7.2	-0.83	-0.26
27	78.8	5.0	0.96	0.41
28	76.7	11.6	0.04	0.01
29	79.3	6.8	1.17	0.38
30	77.3	4.7	0.30	0.14
31	80	18.02	1.48	0.19
32	NR	NR		
33	78.1	6.2	0.65	0.23

** Excluded Result, see Section 6.1

Statistics

Assigned Value	76.6	1.9
Reference Value	76.6	1.9
Robust Average	77.2	0.8
Median	77.3	0.7
Mean	77.2	
N	29	
Max	81.3	
Min	73	
Robust SD	1.8	
Robust CV	2.4%	


Kernel Density
Density
Result
Results: S2 - MDMA
Assigned Value = 76.6 ± 1.9 % base (m/m)
Results (% base (m/m))
Laboratory
17 2 19 26 15 9 23 8 11 24 6 28 3 7 14 22 30 25 10 21 20 33 13 1 27 5 29 31 16 12 18 32 Md RA RV


z-Scores: S2 - MDMA
Standard Deviation for Proficiency Assessment 2.3 % base (m/m) (Equivalent to 3% CV)
z-Score
Laboratory
17 2 19 26 15 9 23 8 11 24 6 28 3 7 14 22 30 25 10 21 20 33 13 1 27 5 29 31 16 12

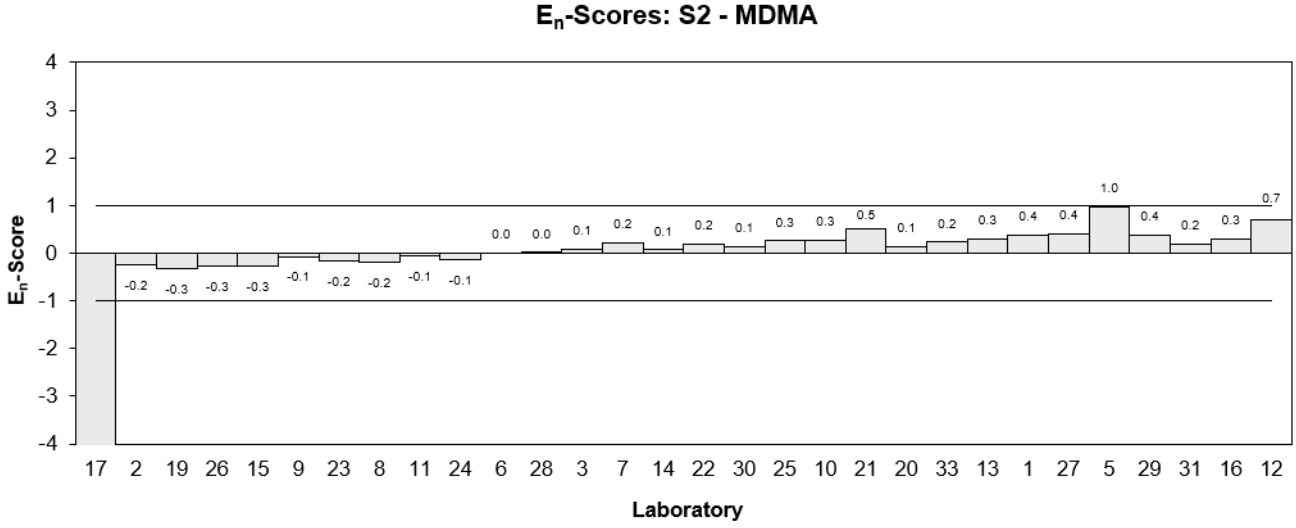

En-Scores: S2 - MDMA
En-Score
Laboratory
17 2 19 26 15 9 23 8 11 24 6 28 3 7 14 22 30 25 10 21 20 33 13 1 27 5 29 31 16 12

Figure 3

Table 9

Sample Details

Sample No.	S3
Matrix	Powder
Analyte	Methamphetamine
Unit	% base (m/m)

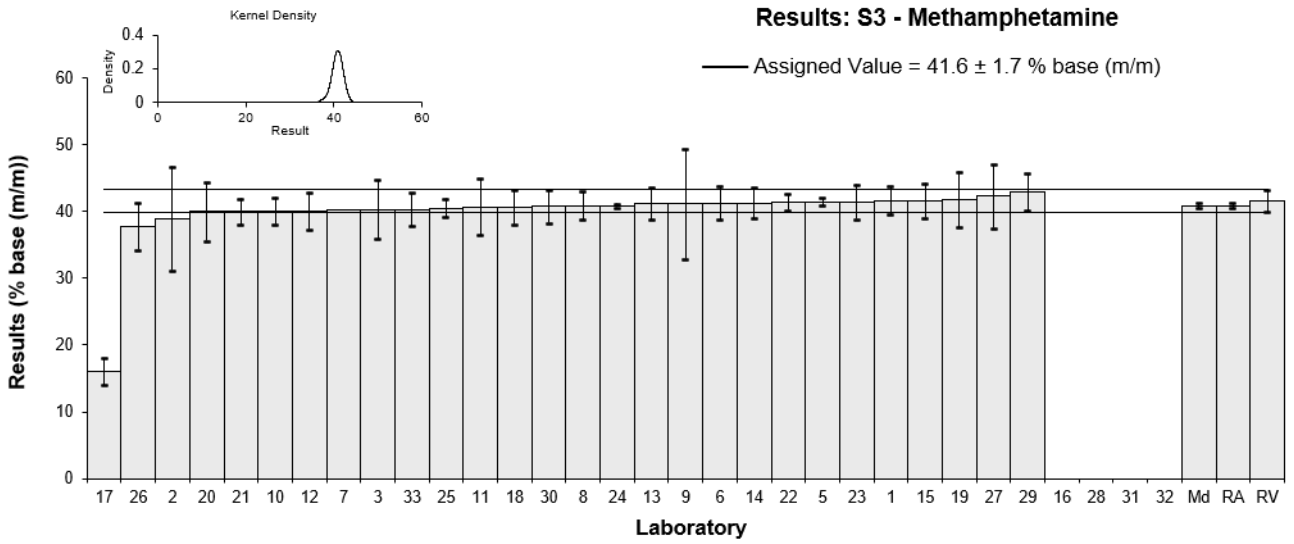
Participant Results

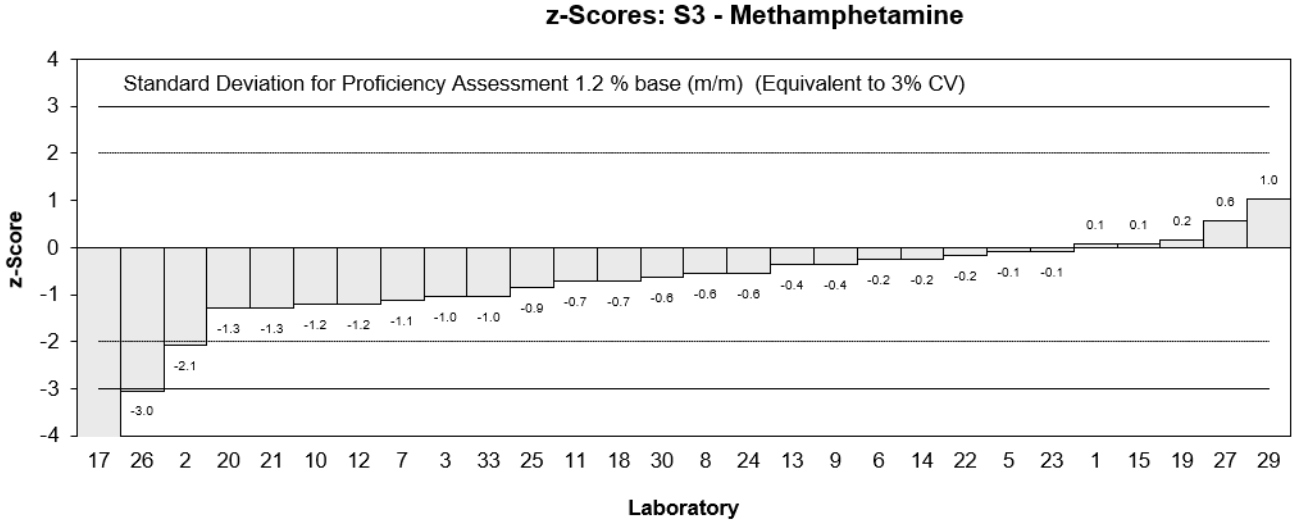
Lab. Code	Result	Uncertainty	z	E_n
1	41.7	2.1	0.08	0.04
2	39	7.8	-2.08	-0.33
3	40.3	4.4	-1.04	-0.28
5	41.5	0.6	-0.08	-0.06
6	41.3	2.5	-0.24	-0.10
7	40.2	NR	-1.12	-0.82
8	40.9	2.1	-0.56	-0.26
9	41.16	8.2	-0.35	-0.05
10	40.1	2.01	-1.20	-0.57
11	40.7	4.2	-0.72	-0.20
12	40.1	2.8	-1.20	-0.46
13	41.15	2.4	-0.36	-0.15
14	41.3	2.3	-0.24	-0.10
15	41.7	2.6	0.08	0.03
16	NR	NR		
17**	16	2	-20.51	-9.75
18	40.7	2.6	-0.72	-0.29
19	41.8	4.18	0.16	0.04
20	40	4.4	-1.28	-0.34
21	40	2	-1.28	-0.61
22	41.4	1.2	-0.16	-0.10
23	41.5	2.6	-0.08	-0.03
24	40.9	0.25	-0.56	-0.41
25	40.53	1.4	-0.86	-0.49
26	37.8	3.5	-3.04	-0.98
27	42.3	4.8	0.56	0.14
28	NR	NR		
29	42.9	2.8	1.04	0.40
30	40.8	2.5	-0.64	-0.26
31	NR	NR		
32	NR	NR		
33	40.3	2.5	-1.04	-0.43

** Excluded Result, see Section 6.1

Statistics

Assigned Value	41.6	1.7
Reference Value	41.6	1.7
Robust Average	40.9	0.4
Median	40.9	0.4
Mean	40.8	
N	27	
Max	42.9	
Min	37.8	
Robust SD	0.84	
Robust CV	2%	


Kernel Density
Density
Result
Results: S3 - Methamphetamine
Assigned Value = 41.6 ± 1.7 % base (m/m)
Results (% base (m/m))
Laboratory
17 26 2 20 21 10 12 7 3 33 25 11 18 30 8 24 13 9 6 14 22 5 23 1 15 19 27 29 16 28 31 32 Md RA RV


z-Scores: S3 - Methamphetamine
Standard Deviation for Proficiency Assessment 1.2 % base (m/m) (Equivalent to 3% CV)
z-Score
Laboratory
17 26 2 20 21 10 12 7 3 33 25 11 18 30 8 24 13 9 6 14 22 5 23 1 15 19 27 29

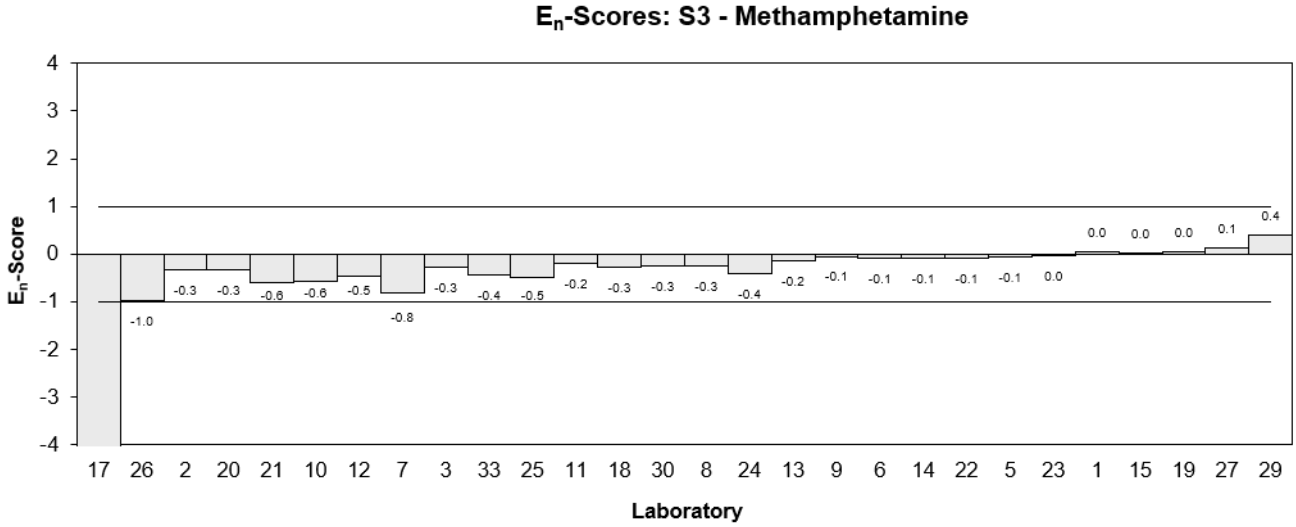

En-Scores: S3 - Methamphetamine
En-Score
Laboratory
17 26 2 20 21 10 12 7 3 33 25 11 18 30 8 24 13 9 6 14 22 5 23 1 15 19 27 29

Figure 4

Table 10

Sample Details

Sample No.	S4
Matrix	Powder
Analyte	Methamphetamine
Unit	% base (m/m)

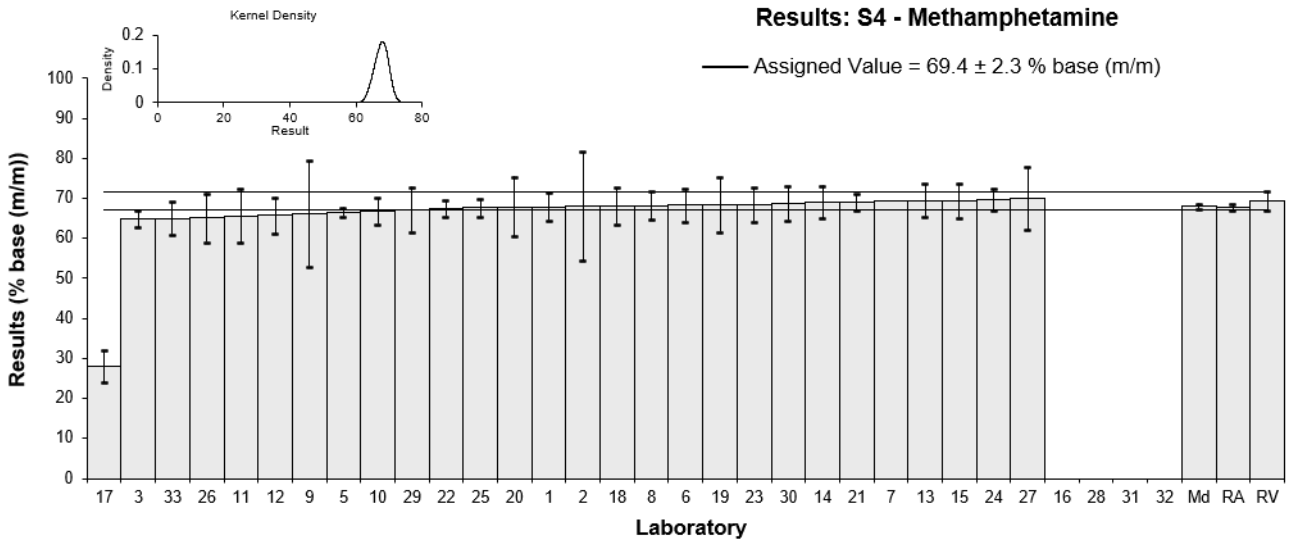
Participant Results

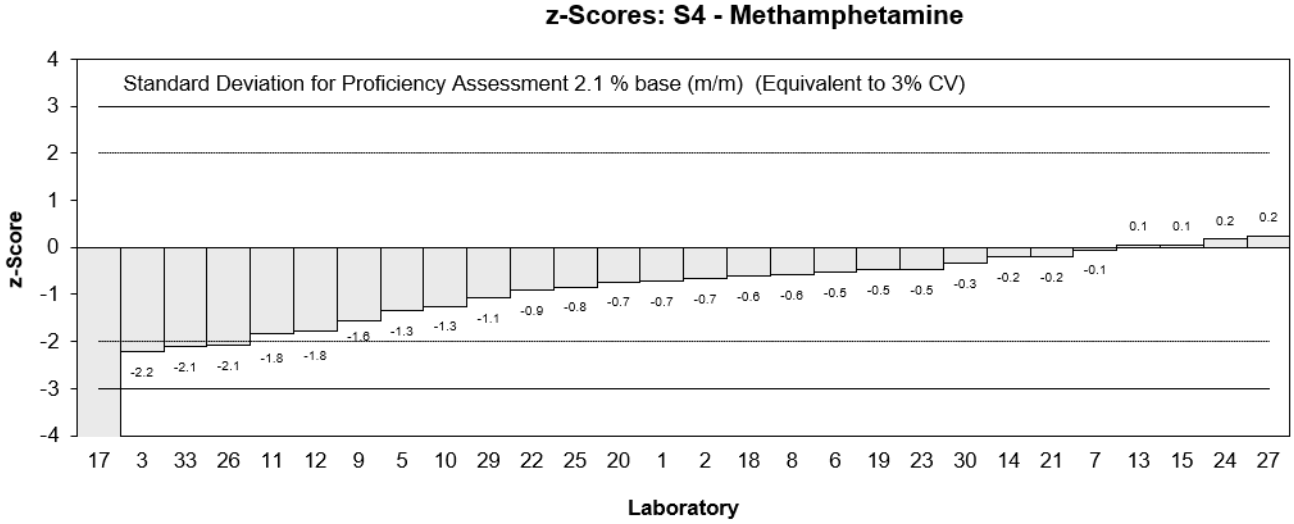
Lab. Code	Result	Uncertainty	z	E_n
1	67.9	3.4	-0.72	-0.37
2	68	13.6	-0.67	-0.10
3	64.8	2.1	-2.21	-1.48
5	66.6	1.1	-1.34	-1.10
6	68.3	4.1	-0.53	-0.23
7	69.3	NR	-0.05	-0.04
8	68.2	3.5	-0.58	-0.29
9	66.15	13.2	-1.56	-0.24
10	66.8	3.34	-1.25	-0.64
11	65.6	6.8	-1.83	-0.53
12	65.7	4.6	-1.78	-0.72
13	69.5	4.1	0.05	0.02
14	69.0	4.1	-0.19	-0.09
15	69.5	4.3	0.05	0.02
16	NR	NR		
17**	28	4	-19.88	-8.97
18	68.1	4.5	-0.62	-0.26
19	68.4	6.84	-0.48	-0.14
20	67.87	7.47	-0.73	-0.20
21	69	2	-0.19	-0.13
22	67.5	2.0	-0.91	-0.62
23	68.4	4.3	-0.48	-0.21
24	69.75	2.63	0.17	0.10
25	67.65	2.2	-0.84	-0.55
26	65.1	6.1	-2.07	-0.66
27	69.9	7.8	0.24	0.06
28	NR	NR		
29	67.2	5.6	-1.06	-0.36
30	68.7	4.2	-0.34	-0.15
31	NR	NR		
32	NR	NR		
33	65.0	4.1	-2.11	-0.94

** Excluded Result, see Section 6.1

Statistics

Assigned Value	69.4	2.3
Reference Value	69.4	2.3
Robust Average	67.7	0.8
Median	68.0	0.7
Mean	67.7	
N	27	
Max	69.9	
Min	64.8	
Robust SD	1.7	
Robust CV	2.4%	


Kernel Density
Density
Result
Results: S4 - Methamphetamine
Assigned Value = 69.4 ± 2.3 % base (m/m)
Results (% base (m/m))
17 3 33 26 11 12 9 5 10 29 22 25 20 1 2 18 8 6 19 23 30 14 21 7 13 15 24 27 16 28 31 32 Md RA RV
Laboratory


z-Scores: S4 - Methamphetamine
Standard Deviation for Proficiency Assessment 2.1 % base (m/m) (Equivalent to 3% CV)
z-Score
17 3 33 26 11 12 9 5 10 29 22 25 20 1 2 18 8 6 19 23 30 14 21 7 13 15 24 27
Laboratory

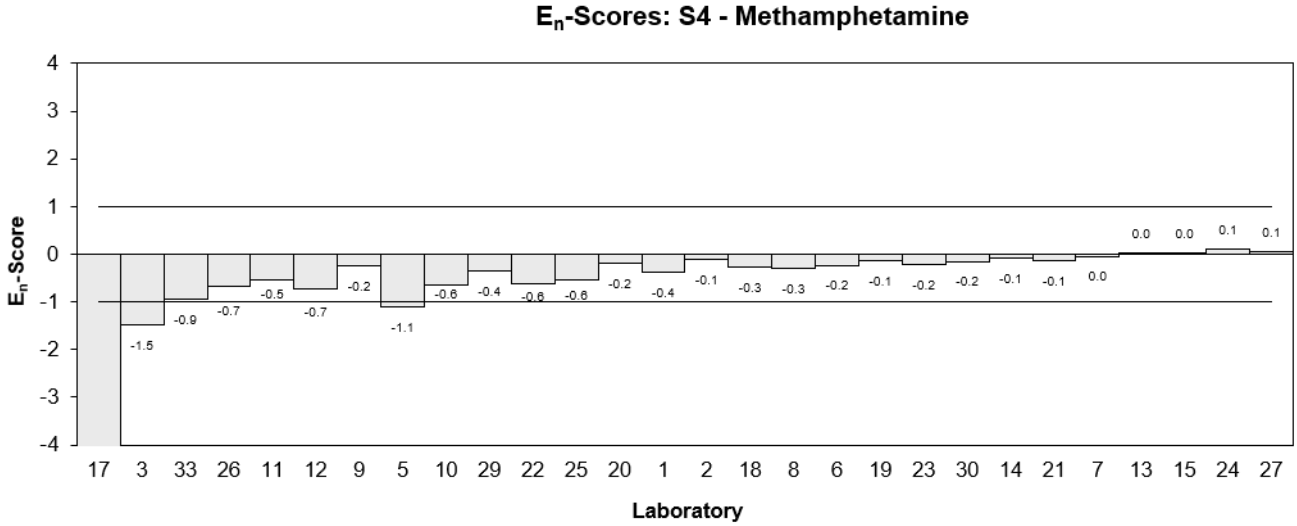

En-Scores: S4 - Methamphetamine
En-Score
17 3 33 26 11 12 9 5 10 29 22 25 20 1 2 18 8 6 19 23 30 14 21 7 13 15 24 27
Laboratory

Figure 5

Table 11 Participants' Identification of Cutting Agents*

Lab. Code	Cutting Agents			
	S1	S2	S3	S4
Preparation	Cellulose and Sucrose	N/A	Sucrose	Dimethyl sulfone
1				Dimethylsulfone
2			Phenacetin	Phenacetin
3	Sucrose		Sucrose	
5				
6		Methamphetamine		
7				
8	Cellulose, Sucrose	Methylamphetamine (trace amount)	Sucrose	Dimethylsulfone
9			dimethylsulfone	dimethylsulfone
10				
11				
12				
13			Sugars	Dimethyl sulfone
14				
15	Sucrose		Glucose, Sucrose	Dimethylsulfone
16				
17				
18				
19			Sucrose	Dimethyl sulfone
20				
21				Dimethylsulfone
22	sucrose (indicated)	methamphetamine	DMS, sucrose (indicated)	DMS
23				
24	Sucrose		Sucrose	
25				Dimethylsulfone (DMS)
26				dimethyl sulfone
27			sucrose	
28	cellulose		sucrose/dimethylsulfone	dimethylsulfone
29				
30		Methamphetamine		
31	Cellulose			
32				
33	sucrose (indicated)	methamphetamine	DMS, sucrose (indicated)	DMS

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

6 DISCUSSION OF RESULTS

6.1 Assigned Value

The assigned value for Sample S1 was the robust average of participants' results. The robust average and associated expanded uncertainty were calculated using the procedure described in ISO 13528.[5] The calculation of the expanded uncertainty for a robust average is presented in Appendix 2.

The reference values obtained using qNMR spectroscopy were used as the assigned values for Samples S2, S3, and S4. Maleic acid was used as the internal standard. The uncertainties of the reference values were evaluated in accordance with the ISO GUM.[9] Additional details are given in Appendix 1.

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 measurements of the reference values were made using qNMR and are traceable to the SI through Australian Standards for mass via balance calibration certificates and the purity of the NMIA maleic acid CRM (QNMR010, Batch No.: 10-Q-02).

The results reported by Laboratory 17 were consistently approximately 2 times lower than the consensus value across all samples. To avoid unfair scoring, these results were excluded from the robust average calculations as they would bias the assigned value (for Sample S1); they were also excluded from the calculation of all summary statistics for all samples.

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an evaluation of the expanded MU associated with their results and the basis of this evaluation. It is a requirement of ISO/IEC 17025 that laboratories have procedures to evaluate and report the uncertainty of chemical measurements.[7]

Of 116 numeric results, 112 (97%) were reported with an associated measurement uncertainty. Participants used a wide variety of procedures to evaluate their reported uncertainties (Tables 4 and 5). One participant reported using the NATA GAG Estimating and Reporting MU as their guide; this document has been removed from the NATA website and is considered obsolete.[10]

Laboratory 7 did not report any measurement uncertainties. This participant was not accredited to ISO/IEC 17025.

The magnitude of reported relative uncertainties was within the range of 0.14% to 23%. In general, an expanded relative uncertainty of less than 3% may be unrealistically small for routine analysis, while over 10% may be too large to be fit-for-purpose. In this study, 72 (64%) relative uncertainties were between 3% and 10%.

The following participants may need to review the procedure they used to evaluate their uncertainties, or review if an error was made when reporting:

- Laboratories 2, 9, 11, 16, 20, 28, and 31 reported relative uncertainties all greater than 10%, which may be too large to be fit-for-purpose.
- Laboratory 5 reported relative uncertainties all less than 3%, which may be unrealistically small for routine analysis.
- Laboratories 24 and 26 reported some relative uncertainties less than 1%, which is unrealistically small for routine analysis.

Participants were also requested to report the coverage factor associated with their uncertainties (Table 4). Most participants reported k = 2, with a few reporting k = 3. One

participant, Laboratory 12, reported k = 1; therefore, this participant's uncertainties were reported as standard uncertainty rather than expanded uncertainty as requested for this PT study.

Uncertainties associated with results returning an acceptable z-score but a not acceptable 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 2 significant figures and then to write the result with the corresponding number of decimal places. For example, instead of 78.08 ± 10.15%, report this as 78 ± 10%.[8]

6.3 z-Score

The z-score compares the participants' deviation from the assigned value based on the standard deviation for proficiency assessment (SDPA). The SDPA defines acceptable performance in a PT study. Unlike the standard deviation based on between-laboratory CV, setting the SDPA as a realistic value enables z-scores to be used as reference points for the assessment of laboratory performance, independent of the group's performance.

SDPAs equivalent to 3% PCV were used to calculated z-scores in this study.

The CVs predicted by the Thompson-Horwitz equation,[6] between-laboratory CVs, and the SDPAs (as PCVs) are presented for comparison in Table 12.

Table 12 Comparison of Between-Laboratory CVs, Thompson/Horwitz CVs, and SDPAs

Sample	Analyte	Numeric Results Reported*	Assigned Value (% base (m/m))	Between-Laboratory CV* (%)	Thompson-Horwitz CV (%)	SDPA (as PCV, %)
S1	MDMA	29	35.1	3.6	1.7	3
S2	MDMA	29	76.6	2.4	1.1	3
S3	Methamphetamine	27	41.6	2	1.6	3
S4	Methamphetamine	27	69.4	2.4	1.2	3

*Values exclude outliers if applicable. Shaded cells represent between-laboratory CVs higher than both the SDPA and the Thompson-Horwitz CV.

Of 116 results for which z-scores were calculated, 103 (89%) returned a z-score of $|z| \leq 2.0$, indicating an acceptable performance.

Laboratories 1, 5, 6, 7, 8, 9, 10, 11, 13, 14, 15, 19, 20, 21, 22, 23, 24, 25, 27, 29, and 30 returned acceptable z-scores for all 4 reported results.

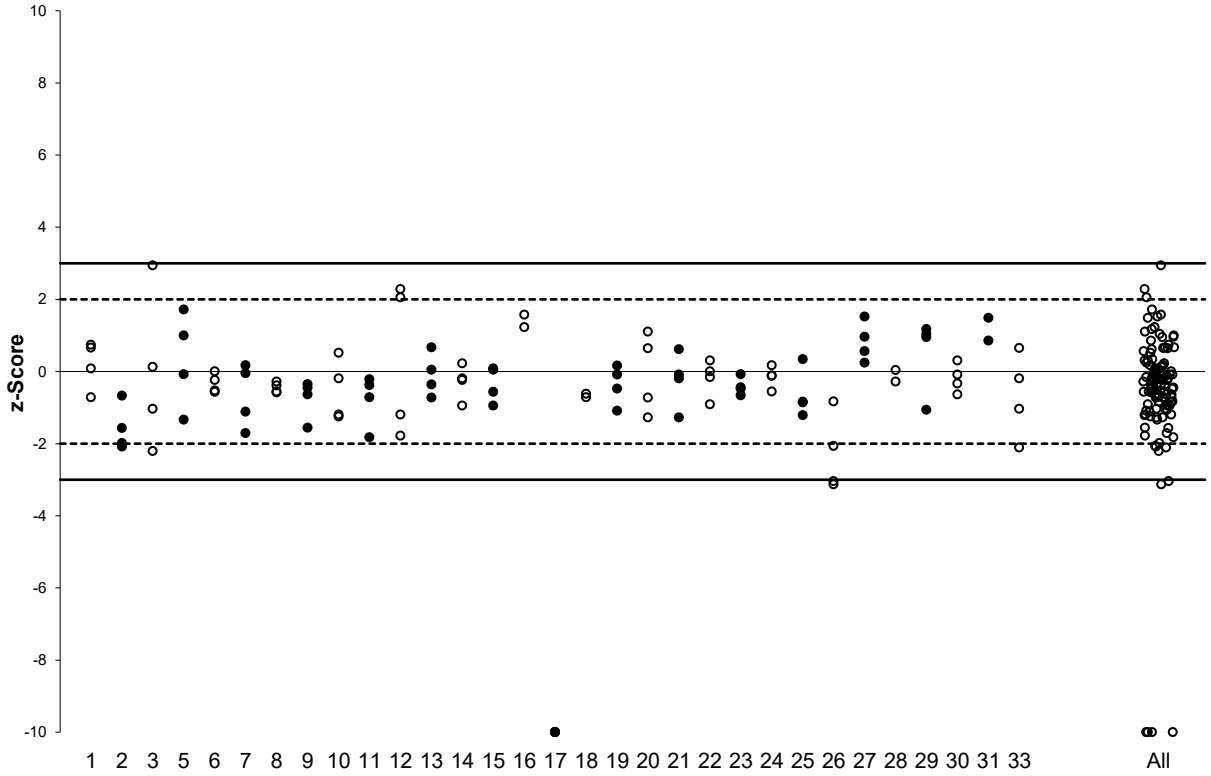
Laboratories 16, 18, 28, and 31 reported results for only one of the analytes (MDMA or methamphetamine) and received acceptable z-scores for all their reported results.

Six participants returned at least one questionable or not acceptable z-score.

Laboratory 17 returned not acceptable z-scores for all 4 samples. These results were significantly negatively biased by approximately the same factor. A calculation or dilution error may be the cause.

Laboratory 32 did not report any numeric results, and so did not receive any z-scores in this study.

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

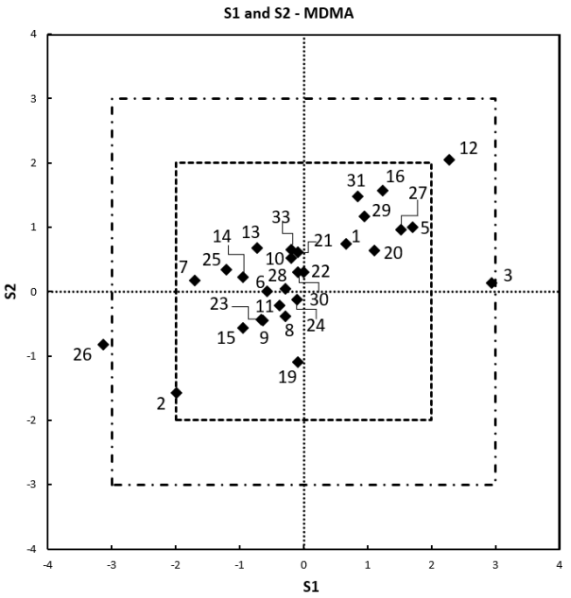


z-Scores less than -10.0 were plotted at -10.0.

Figure 6 z-Score Dispersal by Laboratory

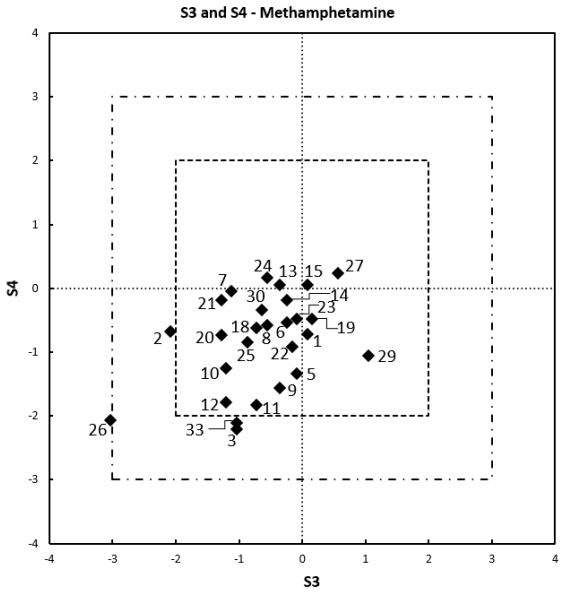
Scatter plots of z-scores for MDMA in Samples S1 and S2, and methamphetamine in Samples S3 and S4, are presented in Figures 7 and 8 respectively. Scores are predominantly in the upper right and lower left quadrants, indicating that laboratory bias is the major contributor to the variability of results. Points close to the diagonal axis demonstrate excellent repeatability, while points close to zero demonstrate excellent repeatability and accuracy.

In this study, results reported for methamphetamine were biased low compared to the reference value.



Laboratory 17 is off-scale.

Figure 7 z-Score Scatter Plot – MDMA



Laboratory 17 is off-scale.

Figure 8 z-Score Scatter Plot – Methamphetamine

6.4 E_n-Score

The E_n-score indicates how closely a result agrees with the assigned value, taking into consideration the respective uncertainties. E_n-scores can be interpreted in conjunction with z-scores, as a not acceptable E_n-score can either be caused by issue with measurement, an inappropriate evaluation of measurement uncertainty, or both.

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

Of 116 results for which E_n-scores were calculated, 105 (91%) returned an E_n-score of $|E_n| < 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

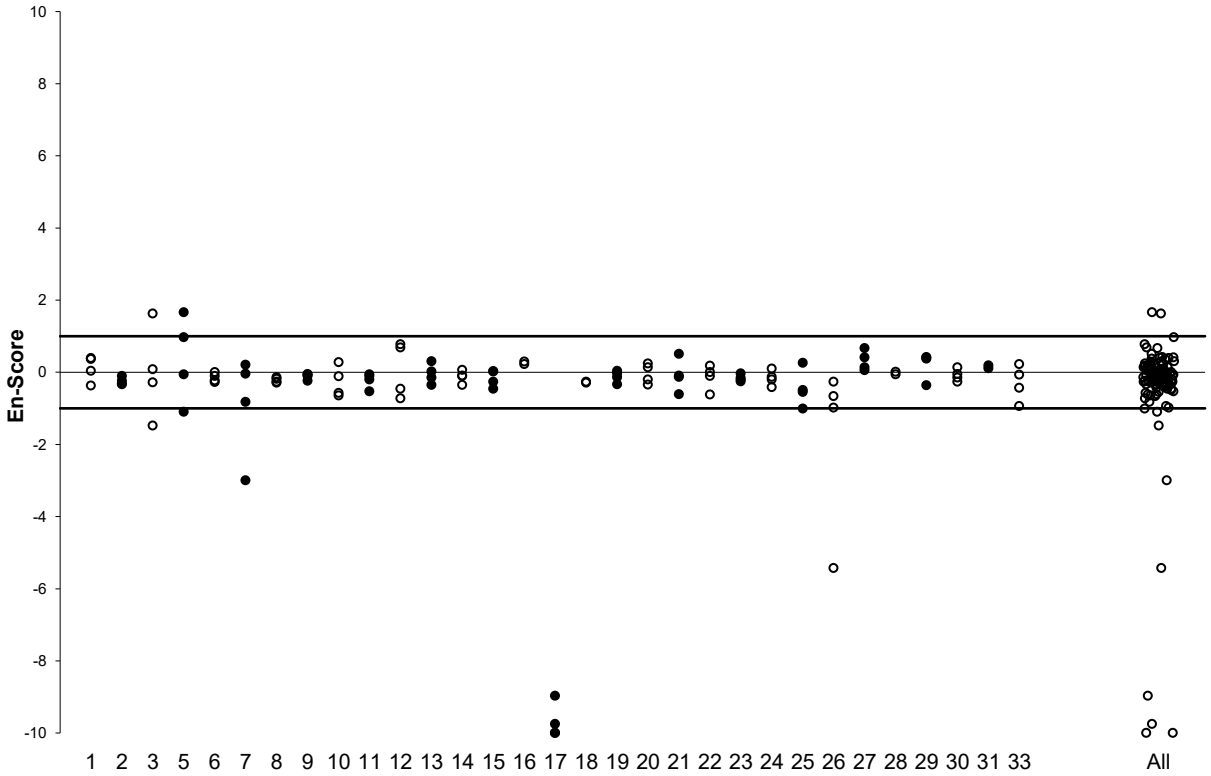
Laboratories 1, 2, 6, 8, 9, 10, 11, 12, 13, 14, 15, 19, 20, 21, 22, 23, 24, 27, 29, 30, and 33 returned acceptable E_n-scores for all 4 reported results.

Laboratories 16, 18, 28, and 31 received acceptable E_n-scores for their 2 reported results.

Six participants returned at least one not acceptable E_n-score.

Laboratory 17 returned not acceptable E_n-scores for all 4 samples, due to issues with measurement rather than due to an inappropriate evaluation of measurement uncertainty.

The dispersal of participants' E_n-scores is presented graphically in Figure 9.



E_n-Scores less than -10.0 were plotted at -10.0.

Figure 9 E_n-Score Dispersal by Laboratory

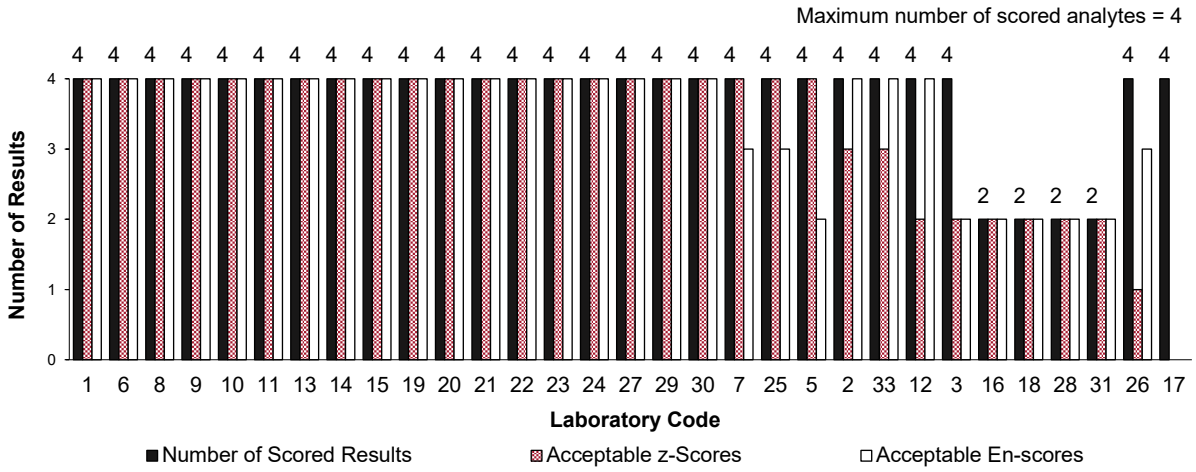
6.5 Summary of Participants' Results and Performances

Summaries of participants' results and performances in this PT study are presented in Table **13** and Figure 10.

Table 13 Summary of Participants' Results*

Lab. Code	S1 MDMA	S2 MDMA	S3 Methamphetamine	S4 Methamphetamine
AV	35.1	76.6	41.6	69.4
1	35.8	78.3	41.7	67.9
2	33	73	39	68
3	38.2	76.9	40.3	64.8
5	36.9	78.9	41.5	66.6
6	34.5	76.6	41.3	68.3
7	33.3	77.0	40.2	69.3
8	34.8	75.7	40.9	68.2
9	34.43	75.57	41.16	66.15
10	34.9	77.8	40.1	66.8
11	34.7	76.1	40.7	65.6
12	37.5	81.3	40.1	65.7
13	34.33	78.15	41.15	69.5
14	34.1	77.1	41.3	69.0
15	34.1	75.3	41.7	69.5
16	36.4	80.2	NR	NR
17	14	31	16	28
18	NR	NR	40.7	68.1
19	35	74.1	41.8	68.4
20	36.26	78.08	40	67.87
21	35	78	40	69
22	35.1	77.3	41.4	67.5
23	34.4	75.6	41.5	68.4
24	34.98	76.33	40.9	69.75
25	33.83	77.39	40.53	67.65
26	31.8	74.7	37.8	65.1
27	36.7	78.8	42.3	69.9
28	34.8	76.7	NR	NR
29	36.1	79.3	42.9	67.2
30	35.0	77.3	40.8	68.7
31	36	80	NR	NR
32	NR	NR	NR	NR
33	34.9	78.1	40.3	65.0

*All values are in % base (m/m). AV = Assigned Value, NR = Not Reported. Shaded cells are results which returned a questionable or not acceptable z-score.



Laboratories 16, 28, and 31 analysed Samples S1 and S2 only; Laboratory 18 analysed Samples S3 and S4 only (maximum of 2 scored analytes).

Figure 10 Summary of Participants' Performance

6.6 Identification of Cutting Agents

Sample S1 was prepared by adding cellulose and sucrose to MDMA hydrochloride. No cutting agent was added to the MDMA hydrochloride used for Sample S2. Samples S3 and S4 were prepared by adding sucrose and dimethyl sulfone respectively to methamphetamine hydrochloride.

Participants were requested to identify the cutting agents in the samples if part of their routine analysis, and the results reported are presented in Table 11.

Nineteen participants (59%) reported on the identity of at least one cutting agent in the samples.

Laboratory 8 correctly identified all cutting agents added in this study.

The cutting agents in Sample S1 challenged participants. Only one participant identified both sucrose and cellulose (Laboratory 8). A further 2 participants identified cellulose only (Laboratories 28 and 31) and 5 participants identified sucrose only (Laboratories 3, 15, 22, 24, and 33).

No cutting agents were added to Sample S2 by the study coordinator. However, 5 participants reported to the presence of methamphetamine (Laboratories 6, 8, 22, 30, and 33). This may have been an impurity in the original MDMA matrix.

Nine participants correctly identified the presence of sucrose in Sample S3 (Laboratories 3, 8, 15, 19, 22, 24, 27, 28, and 33). The identification of sucrose may have been less challenging in this sample as compared to Sample S1 due to the higher proportion of sucrose added. Laboratory 15 also reported glucose, which was not added to this sample. Four participants (Laboratories 9, 22, 28, and 33) reported dimethyl sulfone, which was an impurity in the original methamphetamine matrix. Laboratory 13 reported 'sugars' more generally, without specifying the specific compound.

For Sample S4, 12 participants correctly identified dimethyl sulfone as the cutting agent (Laboratories 1, 8, 9, 13, 15, 19, 21, 22, 25, 26, 28, and 33).

Laboratory 2 reported the presence of phenacetin in both Samples S3 and S4. This compound was not added to either sample.

Laboratory 4 returned their results after the Interim Report was issued, and therefore they have not been included in this study. This participant only reported on cutting agents.

Information about the cutting agents was not included in the Interim Report and therefore not publicly available before their results submission. Accordingly, it is noted that Laboratory 4 correctly identified the presence of sucrose in Samples S1 and S3.

6.7 Participants' Analytical Methods

Participants were requested to analyse the samples using their routine 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 Tables 1 and 2. Results excluded from Section 5 were also excluded from the charts in this section.

The measurement of MDMA in Sample S1 posed a greater analytical challenge for participants in comparison to the other samples, with higher variability between participants' results. This may be due to the lower analyte level.

Participants used between 9 and 100 mg of sample per analysis, with the vast majority using 50 mg or less (Figure 11). Caution should be exercised when a small sample size is taken for analysis as this may not be representative of the whole sample. PT samples are prepared to be sufficiently homogeneous, and so the homogeneity of these PT samples may not reflect the homogeneity of routine samples encountered by laboratories.

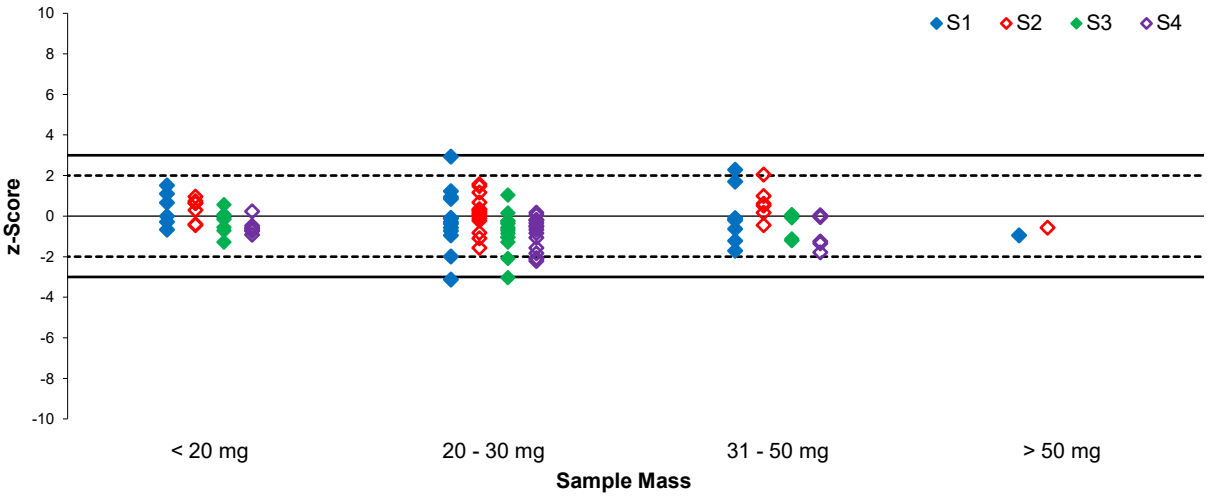


Figure 11 z-Score vs Sample Mass Used for Analysis

The most common source of calibration standards was NMIA (Figure 12).

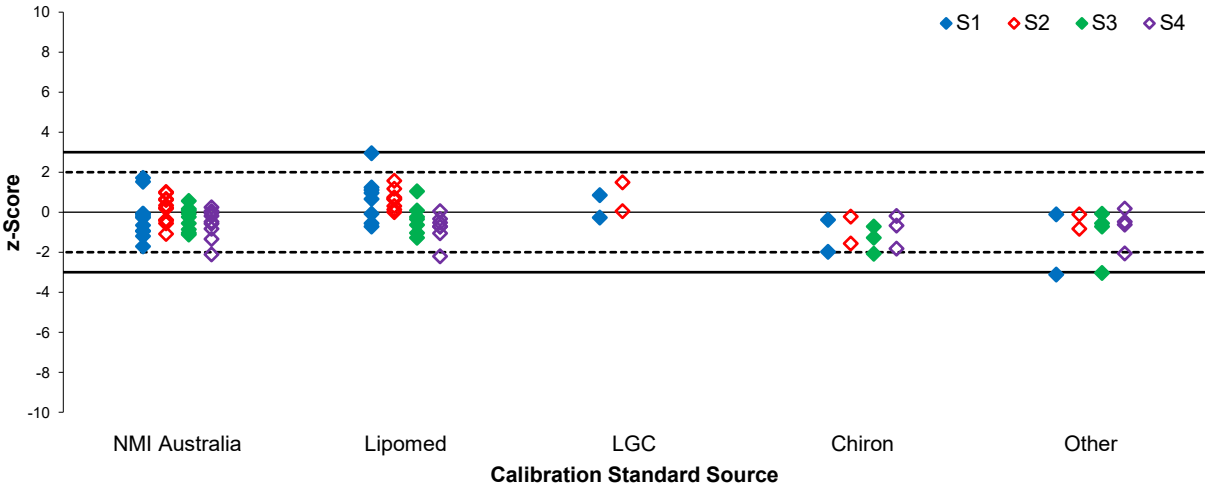


Figure 12 z-Score vs Calibration Standard Source

A wide range of extraction solvents were reported by participants.

Participants used the following instrumental techniques for both qualitative and quantitative analysis:

- gas chromatography (GC) coupled to:
  - flame ionisation detection (FID); or
  - mass spectrometry (MS);
- liquid chromatography (LC), including high performance LC (HPLC) or ultra performance LC (UPLC), coupled to:
  - diode array detection (DAD);
  - ultraviolet/visible spectroscopy (UV-Vis); or
  - tandem mass spectrometry (MS/MS)
- quantitative nuclear magnetic resonance (qNMR) spectroscopy.

Plots of z-scores versus extraction solvent or instrumental technique are presented in Figures 13 and 14 respectively.

The most common methodology employed for both MDMA and methamphetamine was sample extraction with methanol, followed by analysis using LC-DAD.

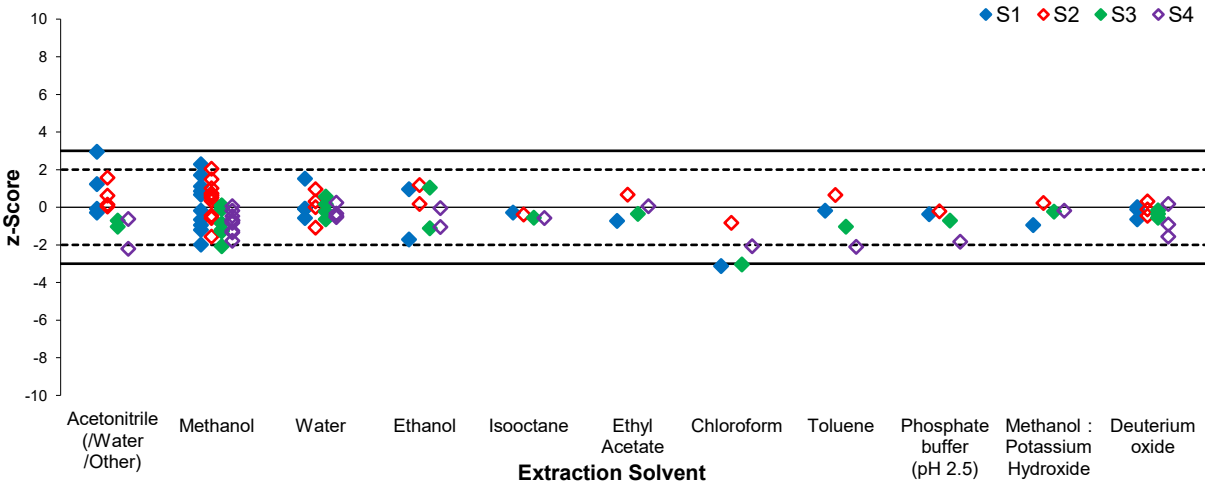


Figure 13 z-Score vs Extraction Solvent

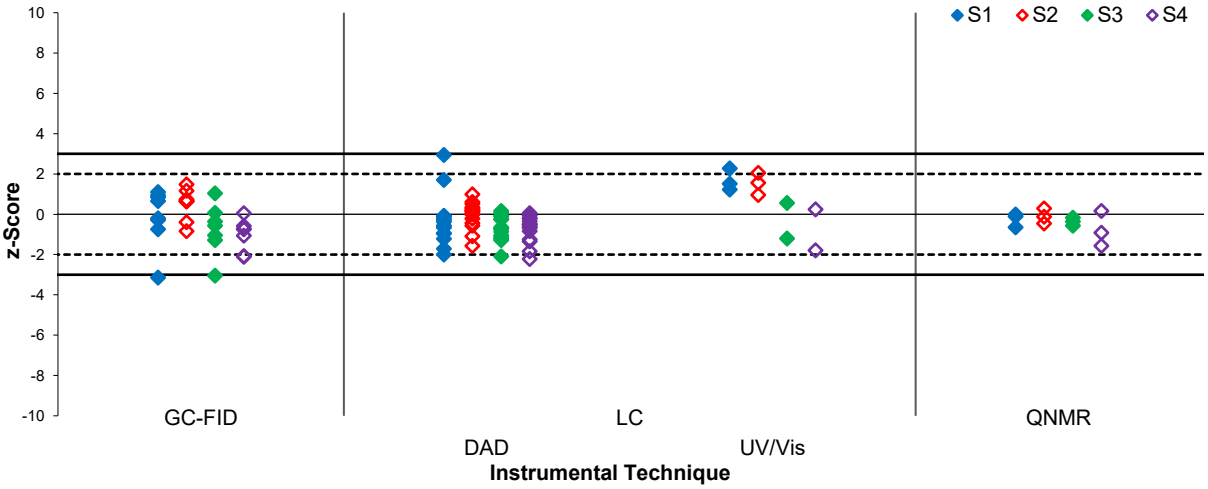


Figure 14 z-Score vs Instrumental Technique

For laboratories using qNMR techniques, NMR instruments with higher field strength provide better spectral resolution, which may assist with avoiding overlaps with impurities. Laboratory 9 reported using a 60 MHz instrument, while Laboratory 24 reported using a 400 MHz instrument. In this study, all results returned by participants using qNMR spectroscopy achieved acceptable z-scores, regardless of instrument.

For laboratories using LC-DAD, a range of different wavelengths were reported. For methamphetamine, the most common wavelength was 210 nm, while for MDMA, participants were more evenly distributed across different options. Results were mostly compatible regardless of wavelength selected (Figure 15).

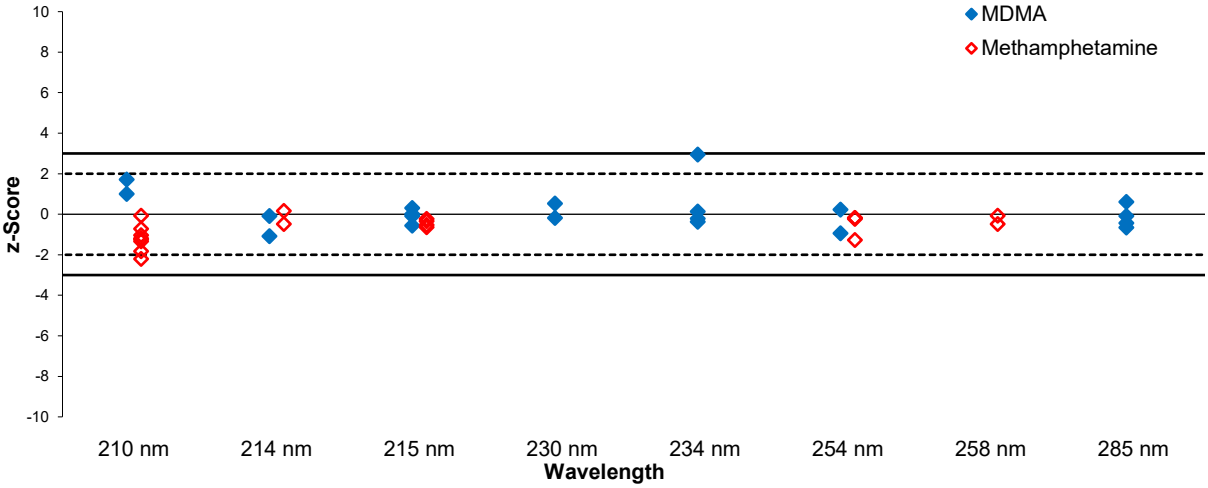


Figure 15 z-Score vs Measurement Wavelength

There may be an association between low or high bias in the reported results and the analytical methodology employed. However, where there are only a limited number of results, it may not be possible to draw definitive conclusions. Nevertheless, participants showing biased results are encouraged to review their methodologies to identify potential sources of bias.

6.8 Comparison of Results and Date of Analysis

As there were delays with sample delivery to some participants, the samples were analysed by participants over approximately 3 months. No stability issues were identified, with no trend between when the samples were analysed and the z-scores obtained (Figure 16; results excluded from Section 5 were excluded from this plot to avoid biasing the data).

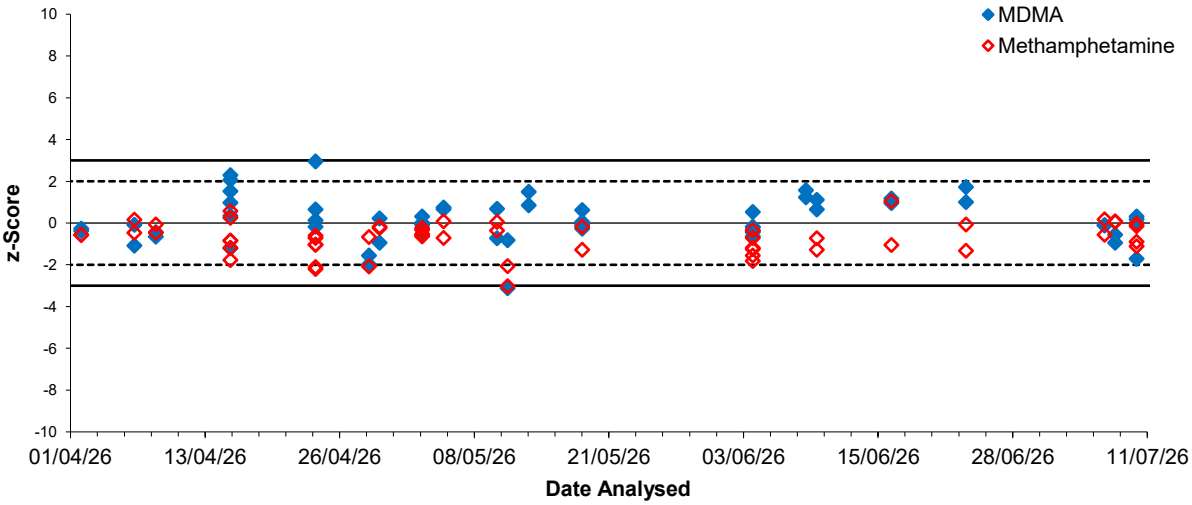
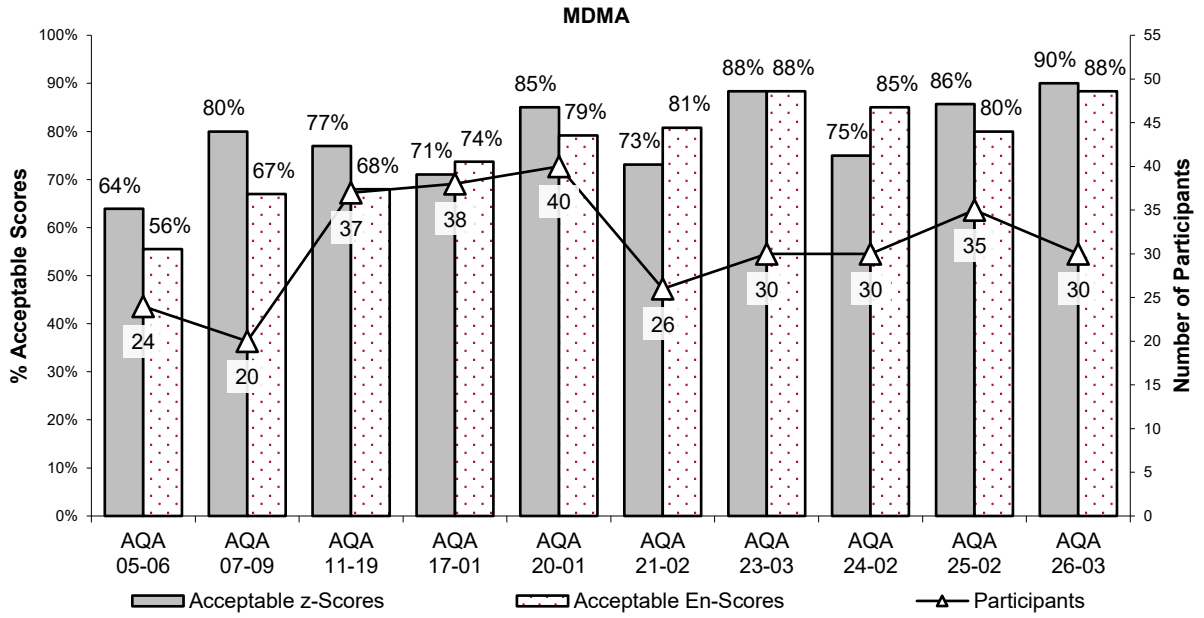


Figure 16 z-Score vs Sample Analysis Date

6.9 Comparison with Previous PT Studies

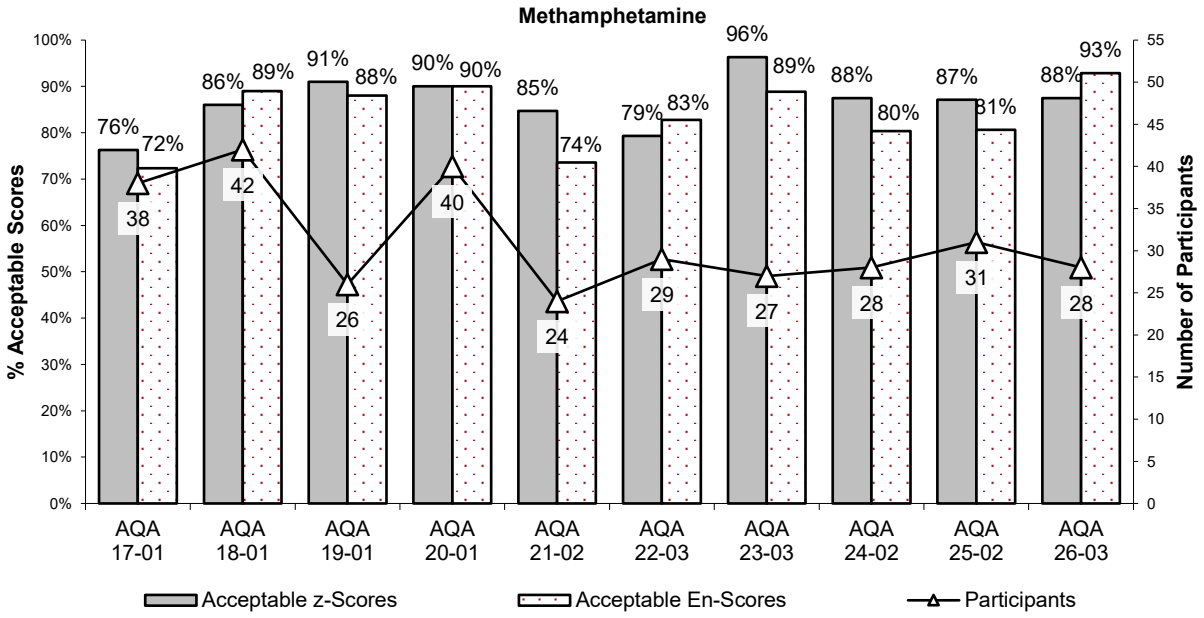
A summary of the acceptable performance for MDMA from 2005 – 2026 (last ten studies with MDMA) is presented in Figure 17. The average proportions of acceptable z-scores and E_n-scores over this period are 79% and 77% respectively. This PT study reflects the best performance from participants in MDMA analysis over this time period.



One sample in AQA 25-02 was scored using 7% PCV; all other samples included in this chart were scored using 3% PCV.

Figure 17 Summary of Participants' Performance in NMIA MDMA PT Studies

A summary of the acceptable performance for methamphetamine from 2017 – 2026 (last ten studies with methamphetamine) is presented in Figure 18. The average proportions of acceptable z-scores and E_n-scores over this period are 87% and 84% respectively.



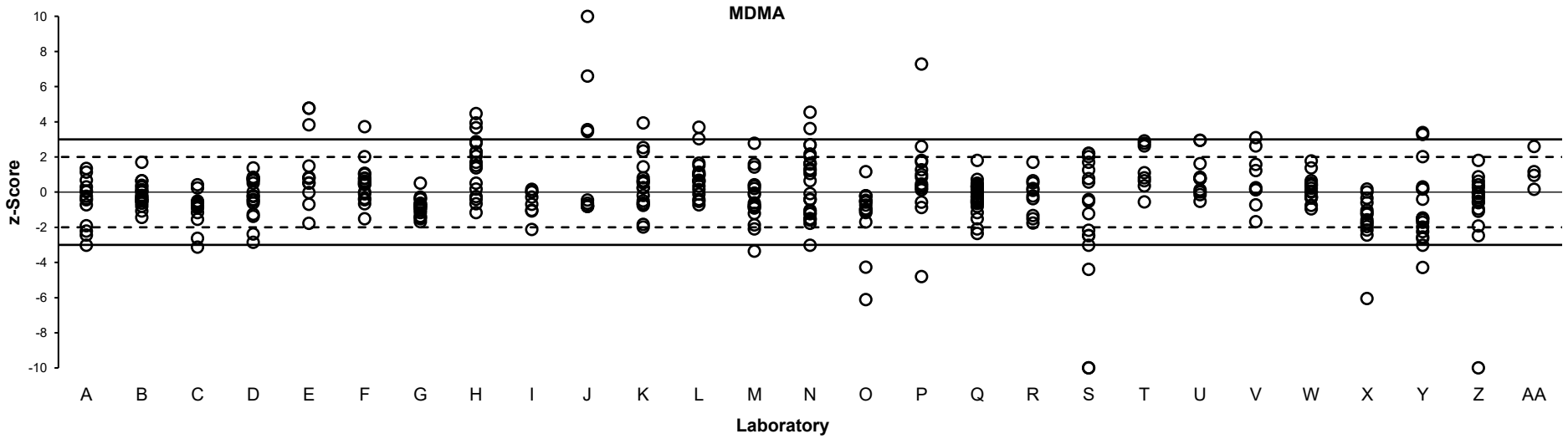
One sample in AQA 23-03 was scored using 5% PCV; all other samples included in this chart were scored using 3% PCV.

Figure 18 Summary of Participants' Performance in NMIA Methamphetamine PT Studies

A number of participants have consistently participated in NMIA MDMA and methamphetamine PT studies, and individual performance history reports are emailed to each participant at the end of every PT study. The consideration of z-scores for an analyte over time provides much more useful information than a single z-score. Over time, laboratories should expect at least 95% of their scores to lie within the range $|z| \leq 2.0$. 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.

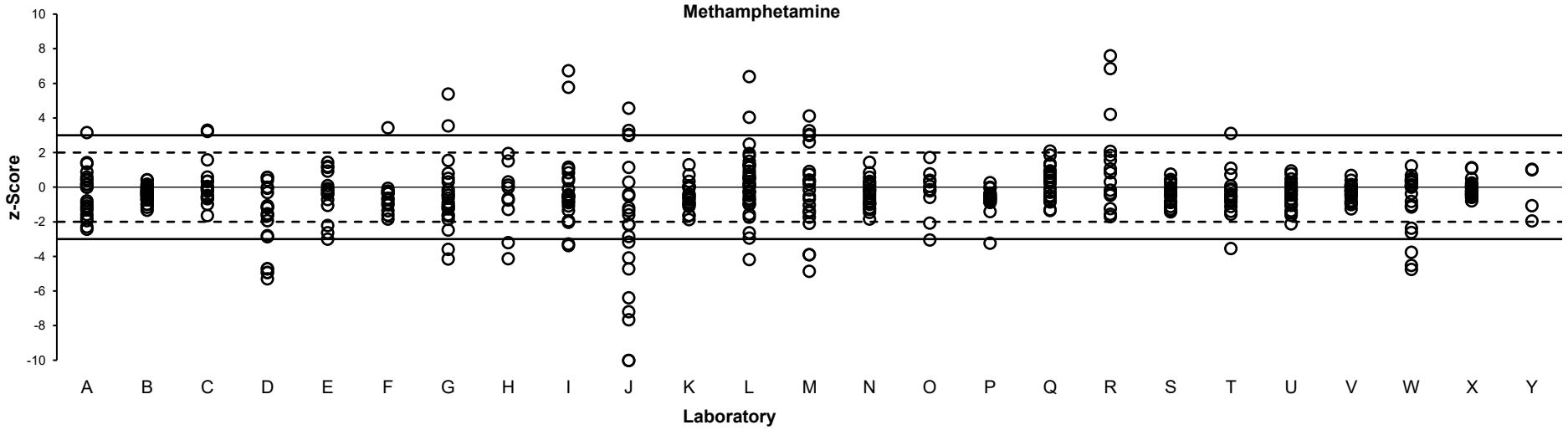
A summary of individual laboratory's performances over the last eight NMIA MDMA PT studies is presented in Figure 19. Four laboratories have achieved acceptable z-scores across all MDMA samples in PT studies participated in over this period.

A summary of individual laboratory's performances over the last ten NMIA methamphetamine PT studies is presented in Figure 20. Seven laboratories have achieved acceptable z-scores across all MDMA samples in PT studies participated in over this period.



z-Scores greater than 10.0 or less than -10.0 have been plotted at 10.0 or -10.0 respectively.

Figure 19 Summary of Participants' z-Scores in NMIA MDMA PT Studies



z-Scores less than -10.0 have been plotted at -10.0.

Figure 20 Summary of Participants' z-Scores in NMIA Methamphetamine PT Studies

7 REFERENCES

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

[1] ISO/IEC 17043, *Conformity assessment — General requirements for the competence of proficiency testing providers*.

[2] Commonwealth of Australia, Department of Industry, Science and Resources, NMIA, 2026, *Study Protocol for Proficiency Testing*, viewed August 2026, <https://www.industry.gov.au/sites/default/files/2020-10/cpt_study_protocol.pdf>.

[3] Commonwealth of Australia, Department of Industry, Science and Resources, NMIA, 2025*, Chemical Proficiency Testing Statistical Manual*, viewed August 2026, <https://www.industry.gov.au/sites/default/files/2019-07/cpt_statistical_manual.pdf>.

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

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

[6] 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.

[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*, 3rd edition, viewed August 2026, <http://www.eurachem.org/images/stories/Guides/pdf/QUAM2012_P1.pdf>.

[9] ISO/IEC Guide 98-3, *Uncertainty of measurement – Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*.

[10] NATA, 2020, *Update to Measurement Uncertainty resources*, viewed August 2026, <https://nata.com.au/news/update-to-measurement-uncertainty-resources/>

APPENDIX 1 REFERENCE VALUES

Three sample vials from each of Samples S2, S3, and S4 were analysed in duplicate under repeatability conditions and in random order. Measurements were made using qNMR spectroscopy with maleic acid as the internal standard. The data supplied with the material is shown in Table 14 and is traceable to the SI unit for mass, the kilogram (kg). The internal standard solution was prepared gravimetrically in D_2O.

Table 14 Maleic Acid CRM Details

Supplier	Catalogue No.	Batch No.	Purity % (95% confidence)
NMIA, Chemical Reference Materials	QNMR010	10-Q-02	98.8 ± 0.12

Samples were prepared gravimetrically, by accurately weighing approximately 20 mg of sample and dissolving this in 900 µL of internal standard solution which was also accurately weighed. Samples were analysed on a Bruker Ascend 500 MHz NMR spectrometer, using a qNMR relaxation time of 25 s. The mass fractions of MDMA and methamphetamine were determined from the NMR responses at around 1.24 ppm and 1.25 ppm respectively.

The averages of the mass fractions determined for the different vials of Samples S2, S3, and S4 (Tables 15 to 17) were used as the reference values and the assigned values. The standard uncertainties were evaluated in accordance with the ISO GUM by combining standard uncertainty terms for method precision, sample homogeneity, weighing of sample, preparation and addition of standard solution, purity of the internal standard, the very small uncertainties in molecular weights, an evaluation of potential interference made by comparing the results from different NMR signals, and the between-batch variation.[9] A coverage factor, k, was calculated using effective degrees of freedom derived from the Welch-Satterthwaite equation.

The measured reference values for Samples S2, S3, and S4 were in agreement with the robust averages of participants' results, within their respective associated uncertainties.

Table 15 Reference Value for Sample S2

Vial No.	MDMA (% base (m/m))	
	Replicate 1	Replicate 2
208	76.6	76.5
213	76.5	76.6
237	76.7	76.6
Average	76.6	
CV	0.086%	

Sample S2 Reference Value: 76.6 ± 1.9 % MDMA base (m/m)

The uncertainty is an expanded uncertainty at 95% confidence level (k = 2.1).[9]

Table 16 Reference Value for Sample S3

Vial No.	Methamphetamine (% base (m/m))	
	Replicate 1	Replicate 2
323	42.0	41.8
338	41.4	41.1
346	41.8	41.3
Average	41.6	
CV	0.84%	

Sample S3 Reference Value: 41.6 ± 1.7 % methamphetamine base (m/m)

The uncertainty is an expanded uncertainty at 95% confidence level (k = 2.2).[9]

Table 17 Reference Value for Sample S4

Vial No.	Methamphetamine (% base (m/m))	
	Replicate 1	Replicate 2
403	68.5	70.3
428	69.9	69.4
440	69.8	68.4
Average	69.4	
CV	1.1%	

Sample S4 Reference Value: 69.4 ± 2.3 % methamphetamine base (m/m)

The uncertainty is an expanded uncertainty at 95% confidence level (k = 2.2).[9]

APPENDIX 2 ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE, AND E_n-SCORE CALCULATIONS

A2.1 Robust Average and Associated Uncertainty

When the robust average is calculated using the procedure described in ISO 13528, the associated uncertainty is evaluated according to Equation 4.[5]

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

where:

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

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

p is the number of results

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

A worked example for Sample S1 is set out below in Table 18.

Table 18 Uncertainty of Robust Average of Sample S1 MDMA

No. results (p)	29
Robust Average	35.1% base (m/m)
$S_{rob\ average}$	1.3% base (m/m)
$u_{rob\ average}$	0.3% base (m/m)
k	2
$U_{rob\ average}$	0.6% base (m/m)

Therefore, the robust average for Sample S1 MDMA is 35.1 ± 0.6% base (m/m).

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 19.

Table 19 z-Score and E_n-Score for Sample S1 MDMA Result Reported by Laboratory 10

Participant Result (% base (m/m))	Assigned Value (% base (m/m))	SDPA	z-Score	E_n-Score
34.9 ± 1.75	35.1 ± 0.6	3% as PCV, or: 0.03 × 35.1 = 1.053% base (m/m)	$z = \frac{34.9 - 35.1}{1.053} = -0.19$	$E_n = \frac{34.9 - 35.1}{\sqrt{1.75^2 + 0.6^2}} = -0.11$

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
DMS	Dimethyl sulfone
EA	European Accreditation
FID	Flame Ionisation 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
ISO	International Organization for Standardization
k	Coverage factor
LC	Liquid Chromatography
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
NATA	National Association of Testing Authorities, Australia
NMIA	National Measurement Institute Australia
NMR	Nuclear Magnetic Resonance
NR	Not Reported
PCV	Performance Coefficient of Variation
PDA	Photodiode Array
PT	Proficiency Testing
qNMR	Quantitative NMR
RA	Robust Average
RM	Reference Material
RV	Reference Value
SD	Standard Deviation
SDPA	Standard Deviation for Proficiency Assessment
SI	International System of Units
SS	Spiked Samples
UPLC	Ultra Performance Liquid Chromatography
UV/Vis	Ultraviolet/Visible spectroscopy

END OF REPORT