Australian Government
Department of Industry, Science and Resources

National Measurement Institute

Proficiency Test Final Report
AQA 23-22
Cocaine

June 2024

ACKNOWLEDGMENTS

This study was conducted by the National Measurement Institute (NMI). 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 NMI staff members in the planning, conduct and reporting of the study is acknowledged.

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SUMMARY

AQA 23-22 Cocaine commenced in September 2023. Sample sets, each containing three samples of cocaine hydrochloride, were sent to twenty-nine laboratories, with one laboratory requesting two sets of samples to be analysed by different analysts. All participants returned results.

Samples were prepared at the National Measurement Institute (NMI) laboratory in Sydney using seizures of cocaine hydrochloride supplied by the Australian Federal Police.

The assigned values were the robust averages of 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 the study were assessed against the aims as follows:

- *Assess the proficiency of laboratories measuring cocaine in samples typical of a routine seizure.*

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

Of 88 *z*-scores, 70 (80%) returned $|z| \leq 2.0$, indicating an acceptable performance.

Of 88 E_n-scores, 75 (85%) returned $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratories **3**, **4**, **6**, **8**, **10**, **12**, **13**, **21**, **22**, **24**, **26** and **27** returned acceptable *z*-scores and E_n-scores for all three samples.

- *Develop a practical application of traceability and measurement uncertainty, and provide participants with information that will assist uncertainty estimates.*

Of 88 numeric results, 85 (97%) were reported with an associated expanded measurement uncertainty. The magnitude of reported uncertainties was within 0.7% to 67% relative.

The metrological traceability of the assigned values has not been established as they were the consensus of participants' results.

- *Test the ability of participants to identify cutting agents commonly found in controlled drug preparation.*

Sample S1 was cut with paracetamol, Sample S3 was cut with glucose, and no cutting agents were added to Sample S2.

Twenty-six participants (87%) reported on the identity of the cutting agent(s) in at least one sample.

Laboratories **2**, **4**, **6**, **8**, **13**, **16**, **23**, **24**, **25**, **27** and **29** correctly identified all cutting agents in the samples.

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

The test samples of this study are homogeneous and are well characterised. Samples are available for purchase from NMI and can be used for quality control and 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 comparisons'.[1] NMI PT studies target chemical testing in areas of high public significance such as trade, environment, law enforcement and food safety. NMI offers studies in:

- pesticide residues in fruit, vegetables and herbs, soil and water;
- petroleum hydrocarbons in soil and water;
- per- and polyfluoroalkyl substances in water, soil, biosolid, food and biota;
- inorganic analytes in soil, water, filters, food and pharmaceuticals;
- controlled drug assay, drugs in wipes and clandestine laboratory; and
- allergens in food.

1.2 Study Aims

The aims of the study were to:

- assess the proficiency of laboratories measuring cocaine in samples typical of a routine seizure;
- develop a practical application of traceability and measurement uncertainty, and provide participants with information that will assist uncertainty estimates;
- test the ability of participants 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 test method was left to the participating laboratories.

1.3 Study Conduct

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

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

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitations sent	18/09/2023
Samples sent	11/12/2023
Results due	10/05/2024
Interim report	16/05/2024
Preliminary report	20/05/2024

There were substantial delivery delays to some participants, and so the project timeline was extended significantly.

2.2 Participation and Laboratory Code

Twenty-nine laboratories enrolled to participate in this study. One laboratory requested two sets of test samples to be analysed by different analysts. Each participant was randomly assigned a confidential laboratory code for this study. All participants returned results.

2.3 Test Material Specification

Three test samples were prepared in October 2023. The starting material was two batches of cocaine hydrochloride samples supplied by the Australian Federal Police. One batch was used to prepare Sample S1, and the other batch was used to prepare Samples S2 and S3.

4-Acetamidophenol (paracetamol) purchased from Sigma Aldrich and glucose purchased from a local pharmacy were used as cutting agents. Sample S1 was cut with paracetamol, Sample S3 was cut with glucose, and no cutting agents were added to Sample S2.

The cocaine was ground and sieved through a 180 µm sieve. The cutting agents were processed similarly. Test samples were then prepared by mixing a known mass of sieved drug material with a known mass of sieved cutting agent in a tumbler overnight. Portions of 150 mg of each of the test samples were then weighed out into labelled glass vials.

There are no preparation values provided for Samples S2 and S3 as the mass fraction of the cocaine hydrochloride used for these samples was not accurately known.

Sample S1 was prepared to contain approximately 60% cocaine 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 substances analysis, the particle size must be sufficiently small and uniformly distributed to ensure minimal influence on analytical precision.

No homogeneity testing was conducted in this PT study. Samples were prepared using the same procedure as previous controlled drug PT studies, which has been demonstrated to produce sufficiently homogeneous samples. 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 6.7). The results gave no reason to question the stability of the test samples.

2.5 Sample Dispatch

A set of three test samples, with each sample containing approximately 150 mg of test material, was dispatched to each participant in December 2023. The following items were also packaged with the samples:

- a covering letter with instructions for participants; and
- a form for participants to confirm the receipt of the test 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 amount of drug by your normal test method. It is recommended to thoroughly mix the content of each vial before taking a test portion for analysis.
- For each sample report % m/m cocaine as base. Report this figure as if reporting to a client.
- For each result report an estimate of the expanded uncertainty as % m/m cocaine as base.
- Report the identity of cutting agent(s) in all three samples if this is within your normal scope of analysis.
- A result spreadsheet has been emailed to you. Please complete this spreadsheet and return by email to jenny.xu@measurement.gov.au.
- Give brief details in the results sheet of your:
    - Basis of uncertainty estimate (e.g. uncertainty budget method, repeatability precision).
    - Analytical method (e.g. sample treatment, instrument type, calibration method).
    - Reference standard (e.g. source, purity)
- Results are to be returned by 12 February 2024.

There were significant delivery delays to some international participants due to delays with receiving export permits, and so the results due date was extended to 10 May 2024.

2.7 Interim Report and Preliminary Report

An Interim Report was emailed to all participants on 16 May 2024.

A Preliminary Report was emailed to all participants on 20 May 2024. This report included a summary of the results reported by participants, assigned values, performance coefficient of variations (PCVs), *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 are presented in Table 1. Some responses may be modified so that the participant cannot be identified.

Table 1 Summary of Participants' Test Methods

Lab. Code	Extraction Solvent	Internal Standard	Calib. Points	Technique	Detector	Column
1	Methanol	Diazepam	6	GC	FID	J&W 128-5512
2	Deuterium oxide	Maleic acid		NMR	Bruker AVIII 400 with BBO Prodigy cryoprobe	N/A
3	Acetonitrile/Deionised water (25:75) + 0.1% (by volume) Trifluoroacetic Acid	N/A	3	HPLC	DAD	OD52 Interpack column (25cm x 5.4mm)
4	acetonitrile/water (80/20)	external standard	3	HPLC	DAD	C8
5	Ethanol	Propyl Paraben	7	UPLC	DAD	BEH Shield RP18
6	CDCl3	TMSB		QNMR		
7	Acetonitrile/Methanol	Pholcodine 1mg/ml	3	UPLC	PDA	ACQUITY C-18
8	Ethanol	Triphenylacetophenone (TPAP)	3	GC	FID	HP1 MS
9	Methanol		4	HPLC	DAD	ECLIPSE XDB-C18
10	Acetonitrile	NA	4	HPLC	UV/Vis	PROTECOL C8 H 5UM 150X4.6MM
11	Methanol		7	HPLC	DAD	ZORBAX XDB-C18 (4.6x150 mm, 5µm)
12	Methanol	Tetracosane	4	GC	FID	HP5 30m x 0.32 mm x 0.25 µm
13	Ethanol	Tribenzylamine	6	GC	FID	HP5
14	Acetonitrile:Water (75:25)	Dibutylphthalate	3	UPLC	PDA	Acquity UPLC BEH C18 1.7µm (2.1x100mm)

Lab. Code		Extraction Solvent	Internal Standard	Calib. Points	Technique	Detector	Column
15		H2O/Acetonitrile (60/40)		5	UPLC	DAD	KROMASIL
16		Methanol	Methadone	4	GC	FID	RXI-5MS
17		HPLC Methanol	Vanillin	1	UPLC	DAD	Agilent Lichrospher 60 RP-select B
18		Methanol	none	5	HPLC	DAD	Kinetex 2.6 µ XB-C18
19		ACN/MeOH/H2O	Analog of cocaine	7	UPLC	MS/MS	C-18 Column
20		Chloroform	Octacosane	5	GC	MS	Zebron ZB-5MSplus
21		Ethanol	Tetracosane	3	GC	FID	BPX-5
22		Methanol	- External calibration	3	GC	FID	CP-sil5CB
23		Methanol	nil	7	HPLC	PDA	Waters HPLC 1260 Phenomenex C8 Luna 3u Narrow Bore 100 mm
24		Acetonitrile	Strychnine	6	GC	FID	Phenyl
25	S1, S3	Chloroform	benzopinacolone	1	GC	FID	HP-1
	S2	Mobile phase	none	4	HPLC	PDA	S2: C18 ubondpak
26		Ethanol	Tetracosane	6	GC	FID	HP5
27		acetonitrile/water (80/20)	none	3	HPLC	DAD	C8
28		acetonitrile/water	none	5	HPLC	DAD	Kromasil
29		water/acetonitrile/2.5M sulfuric acid 90:10:1	None	3	HPLC	Diode Array	Shimpack XR-ODS
30		HPLC Methanol	Vanillin	1	UPLC	DAD	Agilent LiChrospher 60 RP-select B

3.2 Details of Participant Calibration Standard

Participants were requested to provide information about their calibration standard. Responses are presented in Table 2. Some responses may be modified so that the participant cannot be identified.

Table 2 Participant Calibration Standard

Lab. Code	Reference Standard	Purity (%)
1	Lipomed	99.3
2	Sigma Aldrich	99.94 ± 0.15
3	TRC	98
4	EUROMEDEX	86.7
5	NMI	99.8
6	NMI	99.7
7	NMI	99.8
8	NMI	99.8
9		
10	Lipomed (cocaine HCl)	99.004
11	Lipomed	99.9
12	Chiron	99.5 +/- 1.5
13	Lipomed	99.199
14	NMI	99.8
15	Lipomed (EUROMEDEX)	99.5

Lab. Code	Reference Standard	Purity (%)
16	LGC	99.8
17	Lipomed	99.199 ± 0.006
18	Lipomed	>98.6
19	Unikem	100
20	lipomed	99.199
21	NMI	99.8
22	Duchefa	>99
23	NMI	99.8
24	NMI	99.8
25	MacFarlan Smith	99.6
26	Alcaliber	100.7
27	Lipomed	99.004
28	Lipomed	99
29	LGC (Mikromol)	99.9
30	Lipomed	99.199 ± 0.006

3.3 Reported Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about their basis of measurement uncertainty (MU). Responses are presented in Table 3. Some responses may be modified so that the participant cannot be identified.

Table 3 Reported Basis of Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
1	Estimating Measurement Uncertainty by black box with pair of values k = 2	Standard deviation from PT studies only		ISO/GUM ENAC G 09 or ISO 21748
2	Bottom Up (ISO/GUM, fish bone/cause and effect diagram) Coverage factor not reported	Control samples - CRM Duplicate analysis	Instrument calibration Masses and volumes Standard purity	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
3	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples - SS	Instrument calibration Masses and volumes Recoveries of SS	ISO/GUM
4	Top Down - reproducibility (standard deviation) from PT studies used directly Coverage factor not reported	Control samples - RM Duplicate analysis	Instrument calibration Laboratory bias from PT studies	Eurachem/CITAC Guide
5	Coverage factor not reported			
6	Top Down - precision and estimates of the method and laboratory bias k = 2	Control samples - check sample cocaine seizure Duplicate analysis	Standard purity	NMI Uncertainty Course
7	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control samples - CRM Duplicate analysis	Instrument calibration Masses and volumes	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
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	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control samples - RM	Recoveries of SS	Eurolab Technical Report No1/2007

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
10	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2	Control samples - RM	Laboratory bias from PT studies	
11	VALIDATION DATA Coverage factor not reported	Control samples - CRM Duplicate analysis	Instrument calibration Masses and volumes Standard purity	Eurachem/CITAC Guide
12	Top Down - precision and estimates of the method and laboratory bias k = 1	Control samples - authentic samples Duplicate analysis	Instrument calibration Matrix effects Laboratory bias from PT studies Recoveries of SS Standard purity	EA-04/16, EA guidelines on the expression of uncertainty in quantitative testing
13	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control samples - RM	Standard purity	
14	Top Down - precision and estimates of the method and laboratory bias k = 2	Duplicate analysis	Homogeneity of sample Standard purity	Eurachem/CITAC Guide
15	Top Down - precision and estimates of the method and laboratory bias k = 2	Control samples - RM	Laboratory bias from PT studies	ISO/GUM
16	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2	Duplicate analysis	Masses and volumes	ISO/GUM
17	Top Down - precision and estimates of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis	Laboratory bias from PT studies Recoveries of SS	Eurachem/CITAC Guide
18	Top Down - precision and estimates of the method and laboratory bias k = 3	Control samples Duplicate analysis	Instrument calibration Masses and volumes Recoveries of SS	Eurachem/CITAC Guide

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
19	Top Down - precision and estimates of the method and laboratory bias k = 0.95	Control samples - RM		Eurachem/CITAC Guide ILAC-G17 and EA 4/16 (2003)
20	Top Down - precision and estimates of the method and laboratory bias k = 2	Control samples - CRM Duplicate analysis	Laboratory bias from PT studies	ISO/GUM
21	Uncertainty Budget Method k = 3	Control samples - RM Duplicate analysis	Instrument calibration Masses and volumes Standard purity	Internal SOP Document
22	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control samples - SS Duplicate analysis	Instrument calibration Recoveries of SS	ISO/GUM
23	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control samples - in-house controls Duplicate analysis	Standard purity	ISO/GUM
24	Top Down - precision and estimates of the method and laboratory bias k = 2	Control samples - CRM Duplicate analysis	Homogeneity of sample Masses and volumes Laboratory bias from PT studies	ISO/GUM
25	validation k = 2			
26	Top Down - precision and estimates of the method and laboratory bias k = 2	Control samples Duplicate analysis	Matrix effects Standard purity	ISO/GUM
27	Top Down - precision and estimates of the method and laboratory bias k = 2	Control samples	Laboratory bias from PT studies	NF V03-110
28	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples - RM		ISO/GUM

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
29	Standard deviation of replicate analyses multiplied by 2 or 3 k = 3	Control samples - CRM Duplicate analysis	Instrument calibration Standard purity	ISO/GUM
30	Top Down - precision and estimates of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis	Laboratory bias from PT studies Recoveries of SS	Eurachem/CITAC Guide

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

3.4 Participant Comments

The study coordinator welcomes comments or suggestions from participants that may improve future studies. Comments received in this study are presented in Table 4. Some responses may be modified so that the participant cannot be identified.

Table 4 Participant Comments

Lab. Code	Participants' Comments	Study Co-ordinator's Response
2	Methodology: Simultaneous observation of analyte and IS peaks in 1H NMR spectrum acquired using QNMR conditions	
3	Methodology: Less than 20mg used as analysis carried out as per the laboratory procedure (approx 10mg to be sampled)	
8	Sample S3 may contain additional sugars in addition to glucose at a low level. Methodology: A small amount of dichloromethane is used to dissolve the TPAP.	
27	Is it possible to put a sample around 5% and another around 80% or more? Indicate the measurement wavelength used by other labs for HPLC. Allow indication of the form of cocaine identified as HCl or base for all 3 samples. For this test, we identified cocaine HCl in the 3 samples (S1, S2 and S3) by IR. Methodology: results at 230nm	We aim to select a range of purities to cater for the needs of different laboratories, and previous NMI Cocaine PT studies have included samples of similar levels as those suggested here. For this study, the assigned values ranged from 14.1 % base (m/m) to 80.9 % base (m/m). We will take into consideration your other suggestions when preparing results sheets in future.
29	Uncertainty: MuM determined from multiple injections of reference material. 3x(Std Dev/mean)x100.	

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 5 to 7 with the 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 4. An example chart with interpretation guide is shown in Figure 1.

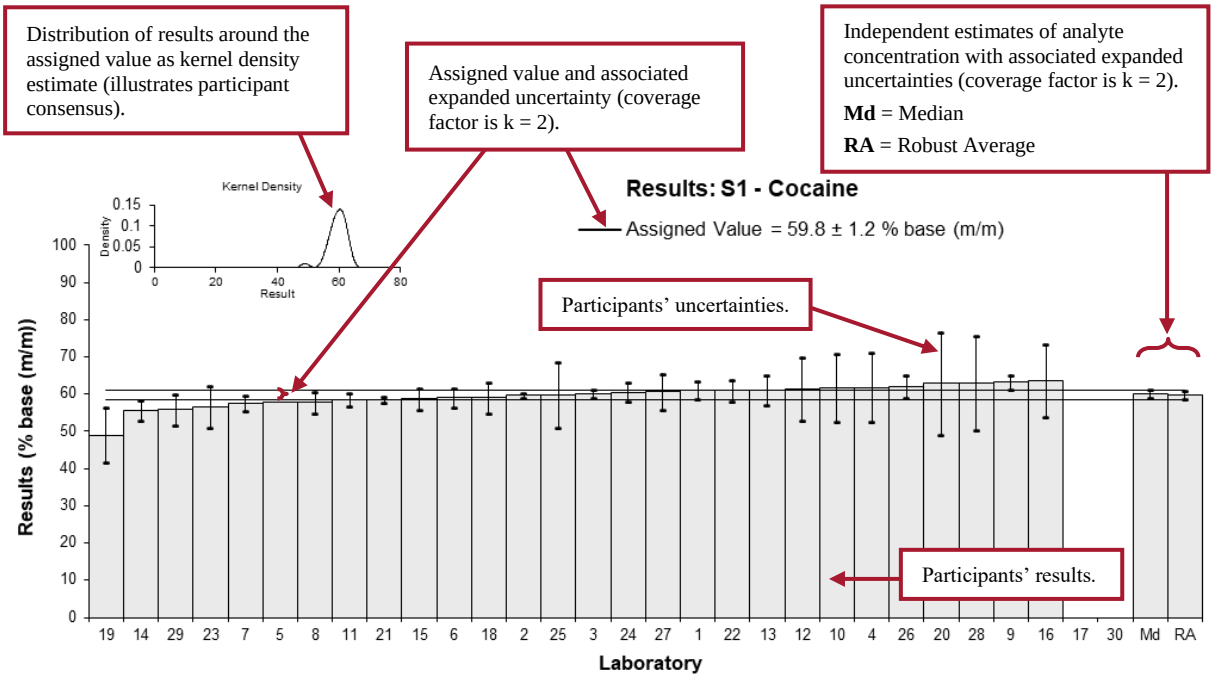


Figure 1 Guide to Presentation of Results

4.2 Assigned Value

The assigned value is defined as the 'value attributed to a particular property of a proficiency test item'.[1] In this study, the property is the % cocaine base (m/m) in the test samples. The assigned values were the robust averages of participants' results, and the expanded uncertainties were estimated from the associated robust SDs (Appendix 1).

4.3 Robust Average and Robust Between-Laboratory Coefficient of Variation

The robust averages and associated expanded MUs, and robust CVs (a measure of the variability of participants' results) were calculated using the procedure described in ISO 13528.[5]

4.4 Performance Coefficient of Variation

The performance coefficient of variation (PCV) is a measure of the between-laboratory variation that in the judgement of the study coordinator would be expected from participants, given the levels of analytes present. The PCV is set by the study coordinator, and it is not the CV of participants' results. The PCV is based on the mass fraction of the analytes and experience from previous studies, and is also supported by mathematical models such as the Thompson-Horwitz equation.[6] By setting a fixed and realistic value for the PCV, a participant's performance does not depend on other participants' performances, and can be compared from study to study.

4.5 Target Standard Deviation for Proficiency Assessment

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

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

4.6 z-Score

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

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

where:

z is *z*-score

χ is a participant's result

X is the assigned value

σ is the target standard deviation for proficiency assessment 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 unacceptable.

4.7 E_n-Score

The E_n-score is complementary to the *z*-score in assessment of laboratory performance. The E_n-score includes uncertainty and is calculated according to Equation 3.

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

where:

E_n is E_n-score

χ is a participant's result

X is the assigned value

U_χ is the expanded uncertainty of the participant's result

U_X is the expanded uncertainty of the assigned value

For the absolute value of an E_n-score:

- $|E_n| \leq 1.0$ is acceptable; and
- $|E_n| > 1.0$ is unacceptable.

4.8 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 5

Sample Details

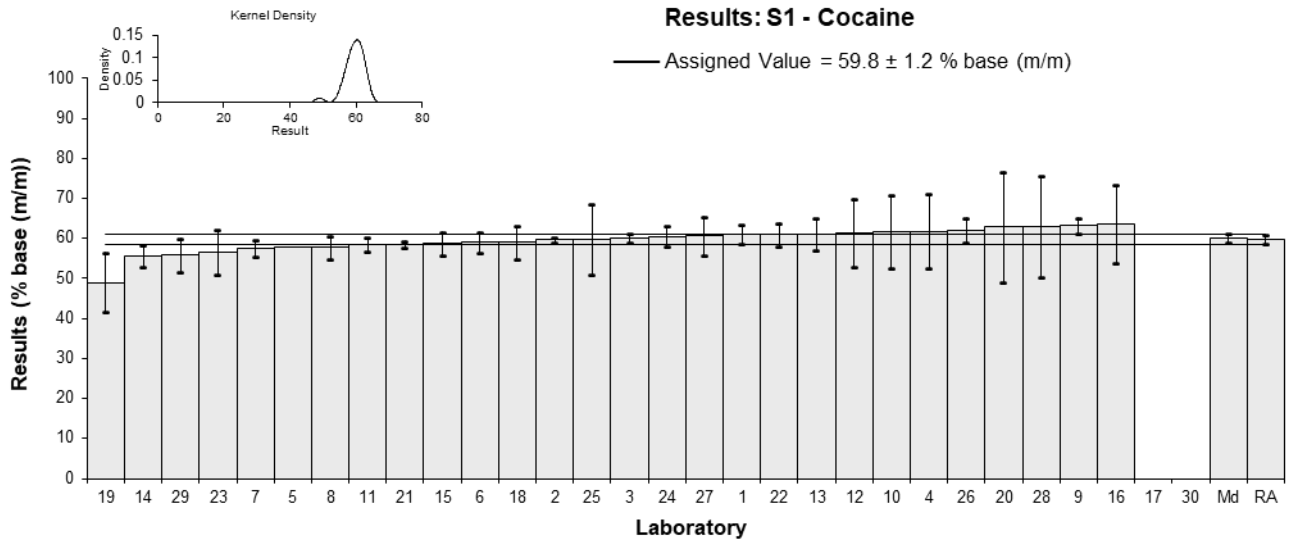
Sample No.	S1
Matrix	Powder
Analyte	Cocaine
Unit	% base (m/m)

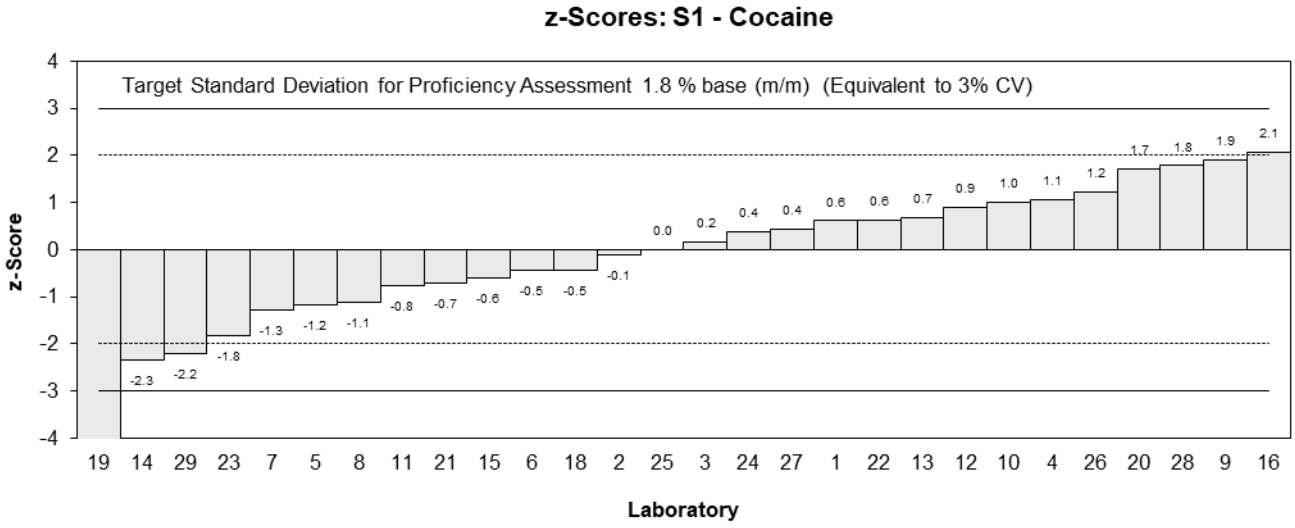
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	60.9	2.4	0.61	0.41
2	59.6	0.63	-0.11	-0.15
3	60.10	1	0.17	0.19
4	61.7	9.3	1.06	0.20
5	57.7	NR	-1.17	-1.75
6	59	2.66	-0.45	-0.27
7	57.49	2.10	-1.29	-0.96
8	57.8	2.9	-1.11	-0.64
9	63.2	2	1.90	1.46
10	61.6	9.2	1.00	0.19
11	58.4	1.8	-0.78	-0.65
12	61.4	8.6	0.89	0.18
13	61	3.9	0.67	0.29
14	55.6	2.6	-2.34	-1.47
15	58.7	2.9	-0.61	-0.35
16	63.52	9.76	2.07	0.38
17	NR	NR		
18	59	4.1	-0.45	-0.19
19	49	7.4	-6.02	-1.44
20	62.87	13.83	1.71	0.22
21	58.5	0.9	-0.72	-0.87
22	60.92	3	0.62	0.35
23	56.5	5.6	-1.84	-0.58
24	60.5	2.6	0.39	0.24
25	59.8	8.7	0.00	0.00
26	62	3.2	1.23	0.64
27	60.59	4.85	0.44	0.16
28	63.00	12.60	1.78	0.25
29	55.81	4.14	-2.22	-0.93
30	NR	NR		

Statistics

Assigned Value	59.8	1.2
Robust Average	59.8	1.2
Median	60.0	1.1
Mean	59.5	
N	28	
Max	63.52	
Min	49	
Robust SD	2.6	
Robust CV	4.3%	


Results: S1 - Cocaine
Kernel Density
Density
Result
Assigned Value = 59.8 ± 1.2 % base (m/m)
Results (% base (m/m))
Laboratory
19 14 29 23 7 5 8 11 21 15 6 18 2 25 3 24 27 1 22 13 12 10 4 26 20 28 9 16 17 30 Md RA


z-Scores: S1 - Cocaine
Target Standard Deviation for Proficiency Assessment 1.8 % base (m/m) (Equivalent to 3% CV)
z-Score
Laboratory
19 14 29 23 7 5 8 11 21 15 6 18 2 25 3 24 27 1 22 13 12 10 4 26 20 28 9 16
-2.3 -2.2 -1.8 -1.3 -1.2 -1.1 -0.8 -0.7 -0.6 -0.5 -0.5 -0.1 0.0 0.2 0.4 0.4 0.6 0.6 0.7 0.9 1.0 1.1 1.2 1.7 1.8 1.9 2.1

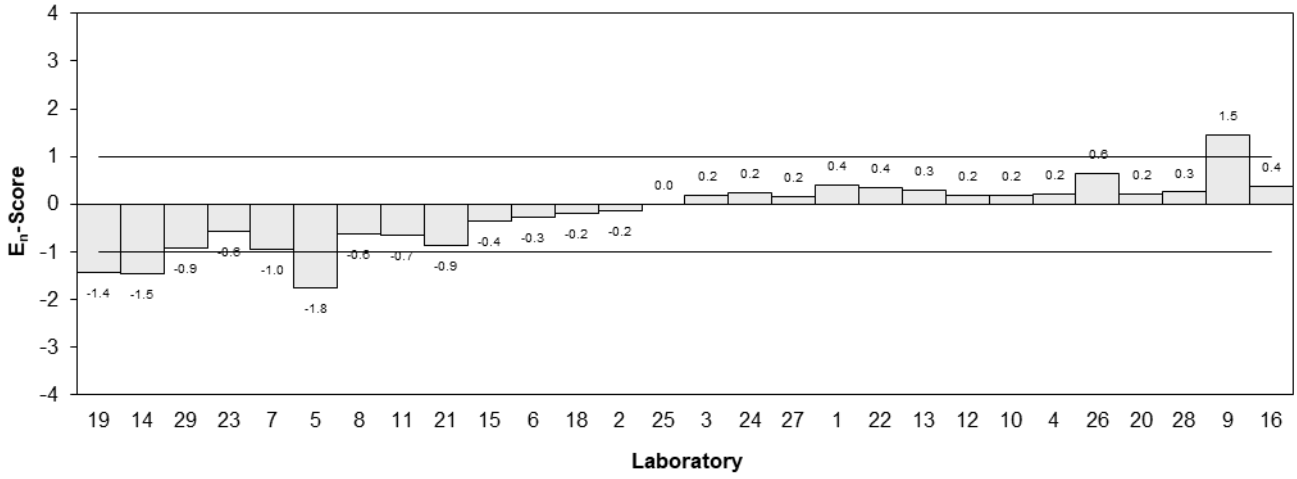

En-Scores: S1 - Cocaine
En-Score
Laboratory
19 14 29 23 7 5 8 11 21 15 6 18 2 25 3 24 27 1 22 13 12 10 4 26 20 28 9 16
-1.4 -1.5 -0.9 -0.6 -1.0 -1.8 -0.6 -0.7 -0.9 -0.4 -0.3 -0.2 -0.2 0.0 0.2 0.2 0.2 0.4 0.4 0.3 0.2 0.2 0.2 0.6 0.2 0.3 1.5 0.4

Figure 2

Table 6

Sample Details

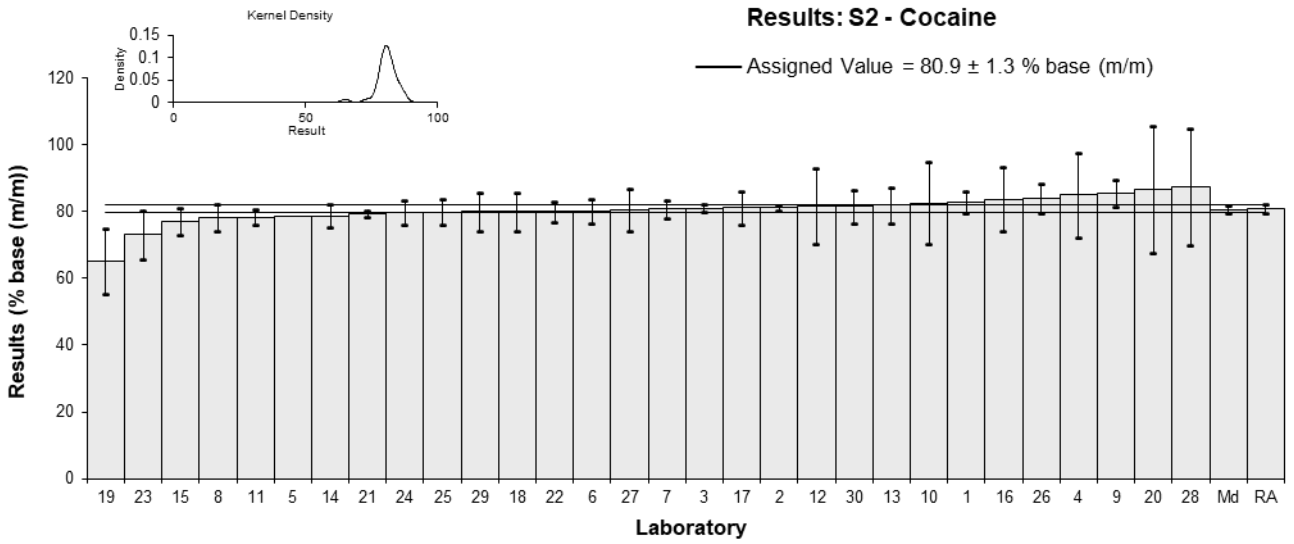
Sample No.	S2
Matrix	Powder
Analyte	Cocaine
Unit	% base (m/m)

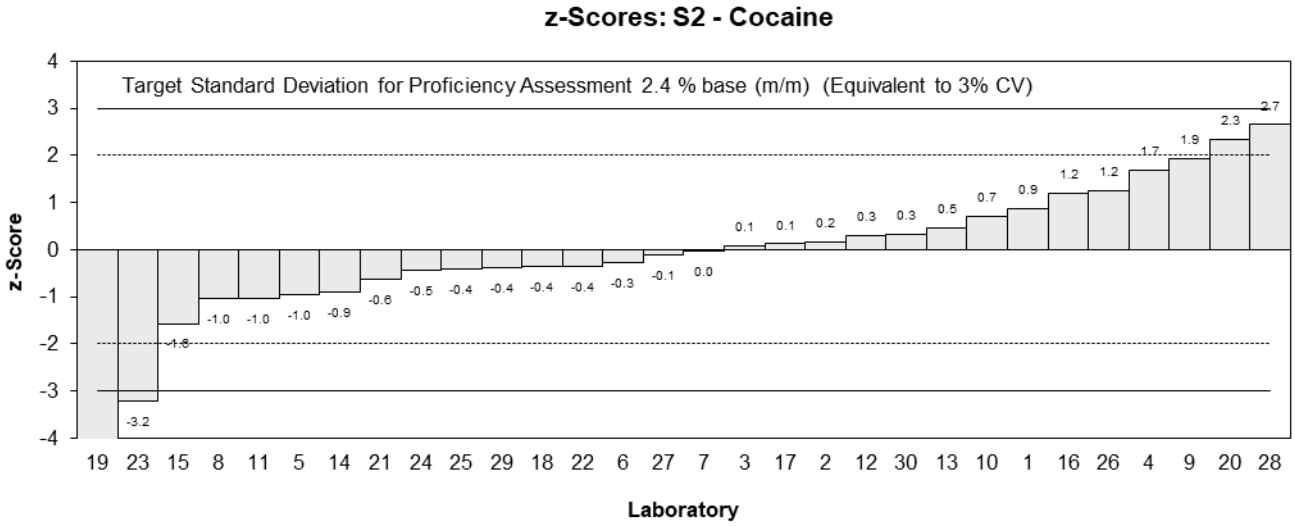
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	83	3.3	0.87	0.59
2	81.3	0.77	0.16	0.26
3	81.10	1.30	0.08	0.11
4	85	12.8	1.69	0.32
5	78.6	NR	-0.95	-1.77
6	80.2	3.61	-0.29	-0.18
7	80.83	2.70	-0.03	-0.02
8	78.4	4.0	-1.03	-0.59
9	85.6	4	1.94	1.12
10	82.6	12.4	0.70	0.14
11	78.4	2.2	-1.03	-0.98
12	81.6	11.4	0.29	0.06
13	82	5.3	0.45	0.20
14	78.7	3.4	-0.91	-0.60
15	77.1	3.9	-1.57	-0.92
16	83.78	9.76	1.19	0.29
17	81.2	4.9	0.12	0.06
18	80	5.6	-0.37	-0.16
19	65	9.8	-6.55	-1.61
20	86.55	19.04	2.33	0.30
21	79.4	1.1	-0.62	-0.88
22	80.01	3	-0.37	-0.27
23	73.1	7.3	-3.21	-1.05
24	79.8	3.5	-0.45	-0.29
25	79.9	3.8	-0.41	-0.25
26	83.9	4.4	1.24	0.65
27	80.60	6.45	-0.12	-0.05
28	87.32	17.46	2.65	0.37
29	79.98	5.93	-0.38	-0.15
30	81.7	5.0	0.33	0.15

Statistics

Assigned Value	80.9	1.3
Robust Average	80.9	1.3
Median	80.7	1.1
Mean	80.6	
N	30	
Max	87.32	
Min	65	
Robust SD	2.8	
Robust CV	3.5%	


Results: S2 - Cocaine
Kernel Density
Density
Result
Assigned Value = 80.9 ± 1.3 % base (m/m)
Results (% base (m/m))
Laboratory
19 23 15 8 11 5 14 21 24 25 29 18 22 6 27 7 3 17 2 12 30 13 10 1 16 26 4 9 20 28 Md RA


z-Scores: S2 - Cocaine
Target Standard Deviation for Proficiency Assessment 2.4 % base (m/m) (Equivalent to 3% CV)
z-Score
Laboratory
19 23 15 8 11 5 14 21 24 25 29 18 22 6 27 7 3 17 2 12 30 13 10 1 16 26 4 9 20 28

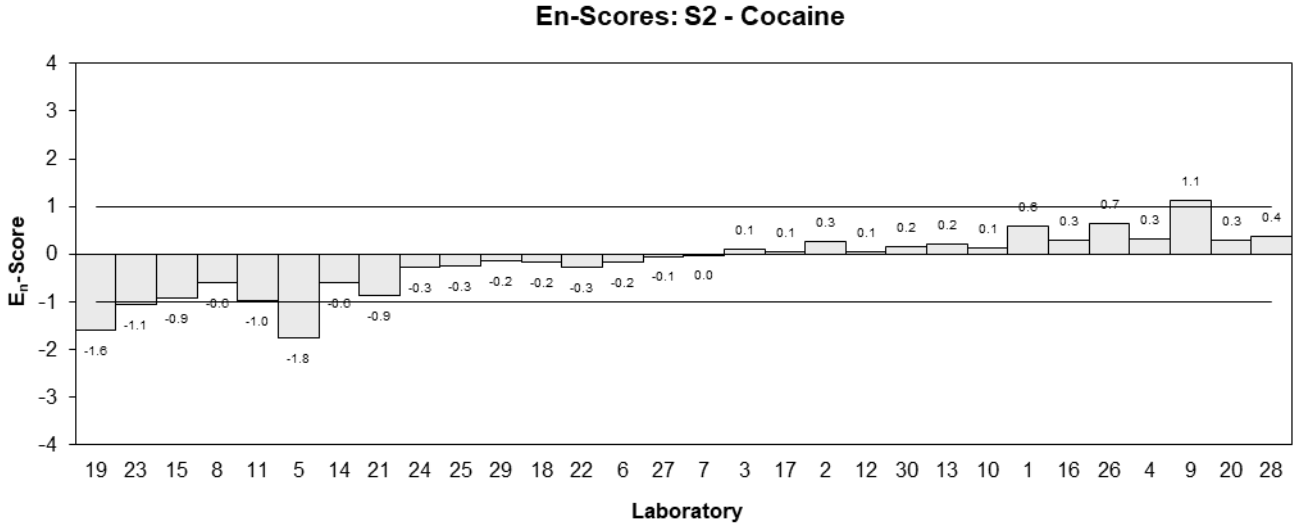

En-Scores: S2 - Cocaine
En-Score
Laboratory
19 23 15 8 11 5 14 21 24 25 29 18 22 6 27 7 3 17 2 12 30 13 10 1 16 26 4 9 20 28

Figure 3

Table 7

Sample Details

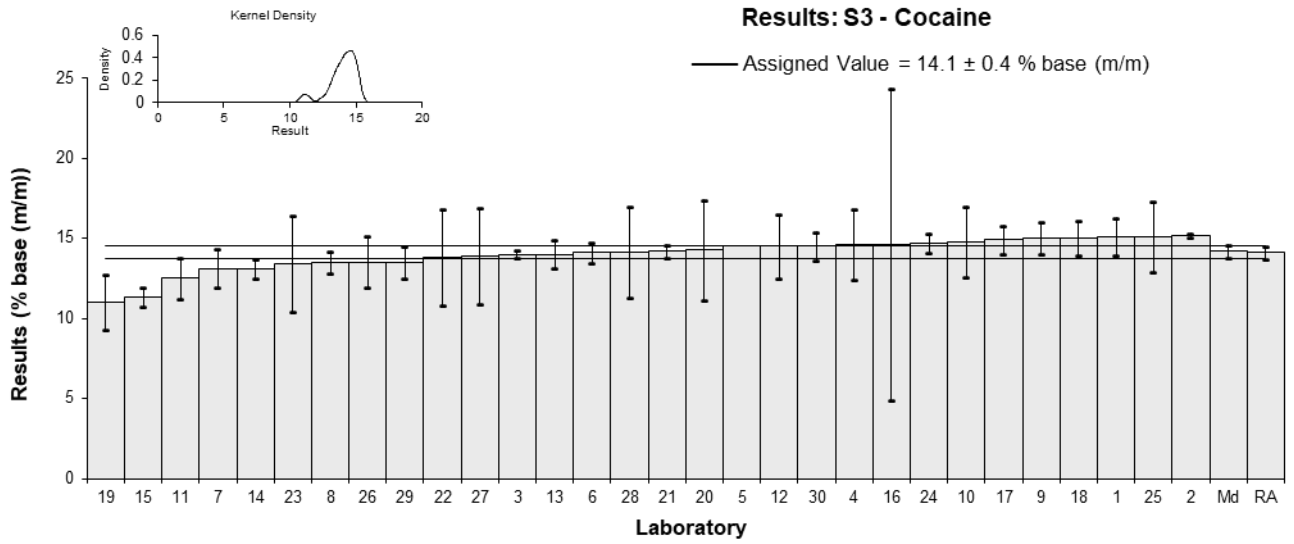
Sample No.	S3
Matrix	Powder
Analyte	Cocaine
Unit	% base (m/m)

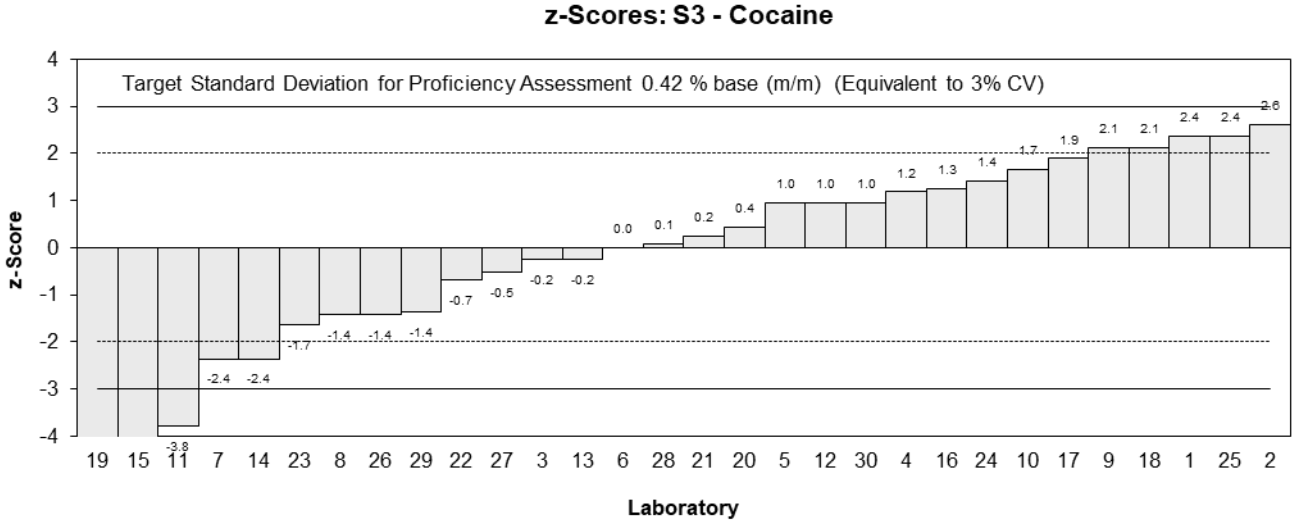
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	15.1	1.2	2.36	0.79
2	15.2	0.11	2.60	2.65
3	14	0.22	-0.24	-0.22
4	14.6	2.2	1.18	0.22
5	14.5	NR	0.95	1.00
6	14.1	0.63	0.00	0.00
7	13.10	1.20	-2.36	-0.79
8	13.5	0.7	-1.42	-0.74
9	15	1	2.13	0.84
10	14.8	2.2	1.65	0.31
11	12.5	1.3	-3.78	-1.18
12	14.5	2.0	0.95	0.20
13	14	0.9	-0.24	-0.10
14	13.1	0.6	-2.36	-1.39
15	11.3	0.6	-6.62	-3.88
16	14.63	9.76	1.25	0.05
17	14.9	0.9	1.89	0.81
18	15	1.1	2.13	0.77
19	11	1.7	-7.33	-1.78
20	14.28	3.14	0.43	0.06
21	14.2	0.4	0.24	0.18
22	13.81	3	-0.69	-0.10
23	13.4	3	-1.65	-0.23
24	14.7	0.6	1.42	0.83
25	15.1	2.2	2.36	0.45
26	13.5	1.6	-1.42	-0.36
27	13.88	3.00	-0.52	-0.07
28	14.13	2.83	0.07	0.01
29	13.52	1	-1.37	-0.54
30	14.5	0.9	0.95	0.41

Statistics

Assigned Value	14.1	0.4
Robust Average	14.1	0.4
Median	14.2	0.4
Mean	14.0	
N	30	
Max	15.2	
Min	11	
Robust SD	0.83	
Robust CV	5.9%	


Results: S3 - Cocaine
Kernel Density
Density
Result
Assigned Value = 14.1 ± 0.4 % base (m/m)
Results (% base (m/m))
Laboratory
19 15 11 7 14 23 8 26 29 22 27 3 13 6 28 21 20 5 12 30 4 16 24 10 17 9 18 1 25 2 Md RA


z-Scores: S3 - Cocaine
Target Standard Deviation for Proficiency Assessment 0.42 % base (m/m) (Equivalent to 3% CV)
z-Score
Laboratory
19 15 11 7 14 23 8 26 29 22 27 3 13 6 28 21 20 5 12 30 4 16 24 10 17 9 18 1 25 2

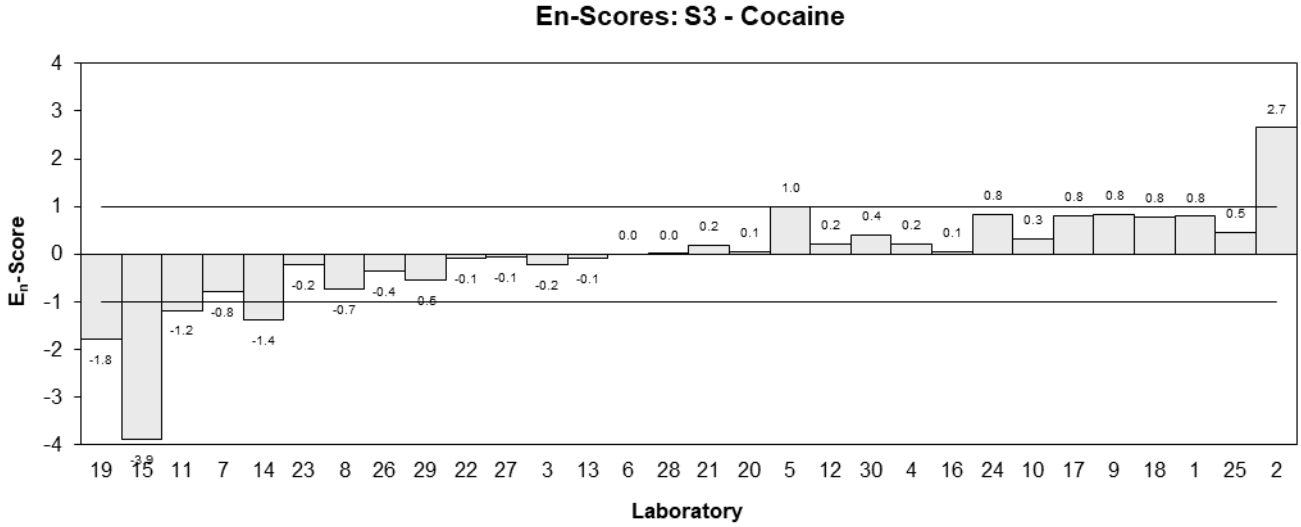

En-Scores: S3 - Cocaine
En-Score
Laboratory
19 15 11 7 14 23 8 26 29 22 27 3 13 6 28 21 20 5 12 30 4 16 24 10 17 9 18 1 25 2

Figure 4

Table 8 Participants' Identification of Cutting Agents*

Lab. Code	Cutting Agents		
	S1	S2	S3
Preparation	4-Acetamidophenol (paracetamol)	-	Glucose
1	Acetaminophen	-	-
2	Paracetamol	no cutting agent	Glucose/Glucodin
3	Paracetamol	N/A	N/A
4	Acetaminophen	/	Dextrose
5	Acetaminophen: 32.7 %		
6	Paracetamol		Glucose
7	paracetamol		
8	Paracetamol	-	Glucose
9	paracetamol		
10			
11	Paracetamol		
12	Paracetamol		
13	paracetamol		sugar (dextrose)
14	Paracetamol		
15			
16	Paracetamol	None	Glucose
17	-	-	-
18	paracetamol	N/A	N/A
19	paracetamol	none	none
20	acetaminophen	/	/
21	Acetaminophen	N/A	N/A
22	Paracetamol		
23	Paracetamol	insufficient sample	Glucose
24	Paracetamol		glucose
25	Paracetamol	-	Glucose
26	Paracetamol		
27	paracetamol		dextrose
28	Paracetamol	None	Sucralose
29	Paracetamol	none detected	Glucose
30	-	-	-

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

6 DISCUSSION OF RESULTS

6.1 Assigned Value

The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528.[5] 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.[3,4] The calculation of the expanded uncertainty for a robust average is presented in Appendix 1, using Sample S1 as an example.

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

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded MU associated with their results and the basis of this uncertainty estimate (Table 3). Two participants reported using the NATA GAG Estimating and Reporting MU as their guide; NATA no longer publishes this document.[9]

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.[7]

Of 88 numeric results, 85 (97%) were reported with an associated expanded MU. Laboratory **5** did not report any uncertainties; this laboratory was not accredited.

The magnitude of reported uncertainties was within the range 0.7% to 67% relative. In general, an expanded uncertainty of less than 3% may be unrealistically small for the routine measurement of illicit drugs, while over 10% may be too large and not fit for purpose. Of the 85 MUs, 47 (55%) were between 3% and 10% relative to the result, ten were less than 3% and 28 were greater than 10%.

Laboratories **16** and **22** reported the same uncertainty across all three samples. Participants were requested to report their uncertainties in units of % base (m/m), however these participants may have reported their uncertainties as relative uncertainties.

Participants were also requested to report the coverage factor associated with their uncertainties (Table 3). Fourteen participants reported a coverage factor of k = 2, three participants reported a coverage factor of k = 3, one participant reported a coverage factor of k = 1 and one participant reported a coverage factor of k = 0.95.

Uncertainties associated with results returning an acceptable *z*-score but an unacceptable E_n-score may have been underestimated.

In some cases, the 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 63.52 ± 9.76% base (m/m), it is better to report 63.5 ± 9.8% base (m/m).[8]

6.3 z-Score

A target SD equivalent to 3% PCV was used to calculate *z*-scores. The CVs predicted by the Thompson-Horwitz equation,[6] between-laboratory CVs (as robust CV), and target SDs (as PCV) obtained in this study are presented in Table 9.

Table 9 Comparison of Thompson-Horwitz CVs, Between-Laboratory CVs and Target SDs

Sample	Analyte	Assigned Value (% base (m/m))	Thompson-Horwitz CV[a] (%)	Between-Laboratory CV[b] (%)	Target SD (as PCV) (%)
S1	Cocaine	59.8	1.3	4.3	3
S2	Cocaine	80.9	1.1	3.5	3
S3	Cocaine	14.1	2.7	5.9	3

[a] Calculated from the assigned value.

[b] Robust between-laboratory CV with outliers removed, if applicable.

Of 88 results for which *z*-scores were calculated, 70 (80%) returned a *z*-score of $|z| \leq 2.0$, indicating an acceptable performance.

Thirteen participants received acceptable *z*-scores across all three samples: **3**, **4**, **5**, **6**, **8**, **10**, **12**, **13**, **21**, **22**, **24**, **26** and **27**. Laboratories **17** and **30** reported results for two samples, and received acceptable *z*-scores across both samples.

Fifteen participants returned at least one questionable or unacceptable *z*-score. Laboratory **19** returned unacceptable *z*-scores for all three samples, with all results being lower than the assigned value (negative bias); this participant should check their methodology for the cause of this bias.

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

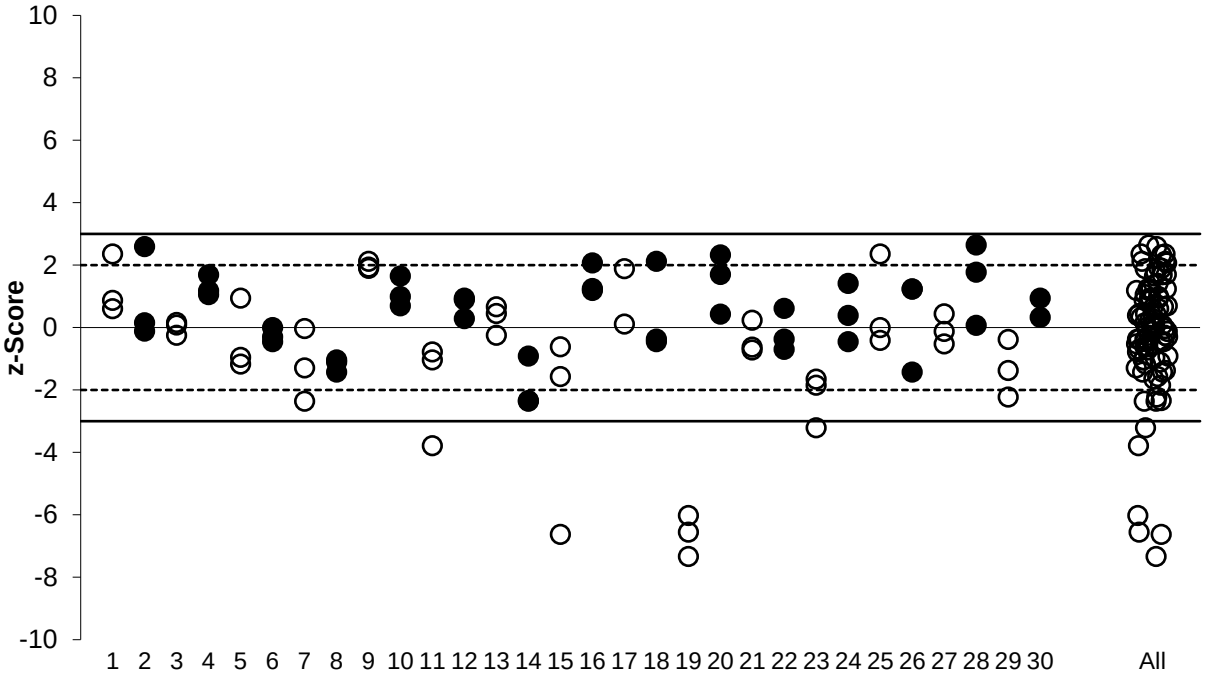


Figure 5 *z*-Score Dispersal by Laboratory

6.4 E_n-Score

E_n-Scores can be interpreted in conjunction with *z*-scores, as an unacceptable E_n-score can be caused by an inappropriate 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 88 results for which E_n-scores were calculated, 75 (85%) returned an acceptable E_n-score of $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Twenty participants received acceptable E_n-scores across all three samples: **1**, **3**, **4**, **6**, **7**, **8**, **10**, **12**, **13**, **16**, **18**, **20**, **21**, **22**, **24**, **25**, **26**, **27**, **28** and **29**. Laboratories **17** and **30** reported results for two samples, and received acceptable E_n-scores across both samples.

Eight participants returned at least one unacceptable E_n-score. Laboratory **19** returned unacceptable E_n-scores for all three samples.

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

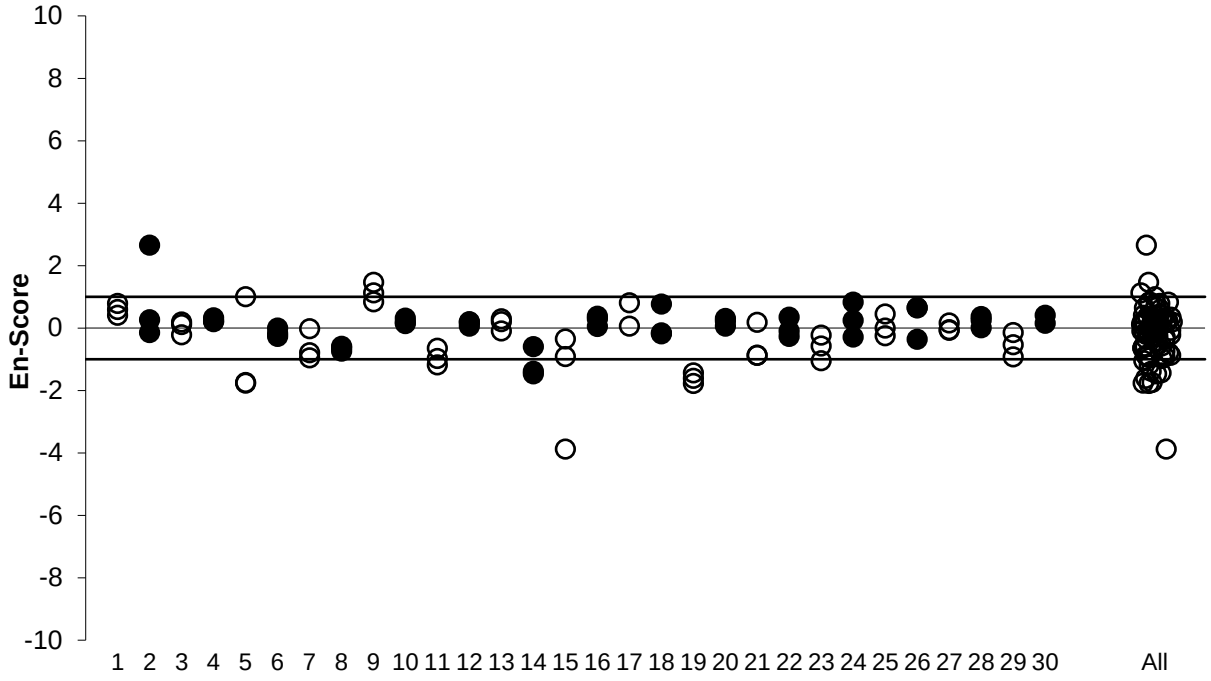


Figure 6 E_n-Score Dispersal by Laboratory

6.5 Identification of Cutting Agent

The test samples were prepared using seizures of cocaine hydrochloride supplied by the Australian Federal Police. The study coordinator added paracetamol to Sample S1, glucose to Sample S3, and no cutting agents were added to Sample S2.

Twenty-six participants (87%) reported on the identity of the cutting agent(s) in at least one sample (Table 8).

Laboratories **2**, **4**, **6**, **8**, **13**, **16**, **23**, **24**, **25**, **27** and **29** correctly identified all cutting agents in the samples.

A higher proportion of participants were able to identify paracetamol as compared to glucose; all 26 participants who reported on the identity of cutting agents correctly identified paracetamol in Sample S1, as compared to 11 participants who correctly identified glucose in Sample S3. Laboratory **28** incorrectly reported sucralose as the cutting agent in Sample S3.

6.6 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, and a summary of accreditation status, methods and reference standards used is presented in Table 10.

Table 10 Summary of Participants' Analyses

		Lab. Code
Accreditation	Yes to ISO/IEC 17025	1, 2, 3, 6, 8, 10, 11, 12, 13, 14, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25, 26, 28, 29, 30
	Not Accredited / Not Reported	4, 5, 7, 9, 15, 27
Average Sample Mass Used per Analysis (mg)	< 20	1, 2, 3, 6, 8, 9, 15, 27, 28
	20 – 30	4, 7, 10, 13, 14, 16, 17, 18, 20, 22, 23, 26, 29, 30
	31 – 50	5, 12, 19
	51 – 100	11, 21, 24
	> 100	25
Conversion to Base?	Yes	1, 3, 7, 10, 12, 13, 14, 15, 20, 22, 23, 25, 26, 28
	No	2, 5, 6, 8, 9, 11, 16, 17, 18, 21, 24, 27, 29, 30
	Not Reported	4, 19
Instrument Used for Quantification	HPLC-DAD	3, 4, 9, 11, 18, 23, 25 (S2), 27, 28, 29
	HPLC-UV/Vis	10
	UPLC-DAD	5, 7, 14, 15, 17, 30
	UPLC-MS/MS	19
	GC-FID	1, 8, 12, 13, 16, 21, 22, 24, 25 (S1, S3), 26
	GC-MS	20
	QNMR	2, 6
Solvent	Acetonitrile	10, 24
	Acetonitrile/Water	4, 14, 15, 27, 28
	Acetonitrile/Water/Acid	3, 29
	Acetonitrile/Methanol(/Water)	7, 19
	Chloroform	20, 25 (S1, S3)
	Ethanol	5, 8, 13, 21, 26
	Methanol	1, 9, 11, 12, 16, 17, 18, 22, 23, 30
	Other	2, 6, 25 (S2)
Sources of Calibration Standard	NMI Australia	5, 6, 7, 8, 14, 21, 23, 24
	Lipomed	1, 4, 10, 11, 13, 15, 17, 18, 20, 27, 28, 30
	LGC	16, 29
	Other	2, 3, 12, 19, 22, 25, 26
	Not Reported	9

Plots of the *z*-score versus various methodology parameters are presented in Figures 7 to 11. Where charts refer to n = *x*, this corresponds to *x* number of participants using that methodology.

One participant used UPLC-MS/MS for their measurement; this participant returned low unacceptable results for all samples.

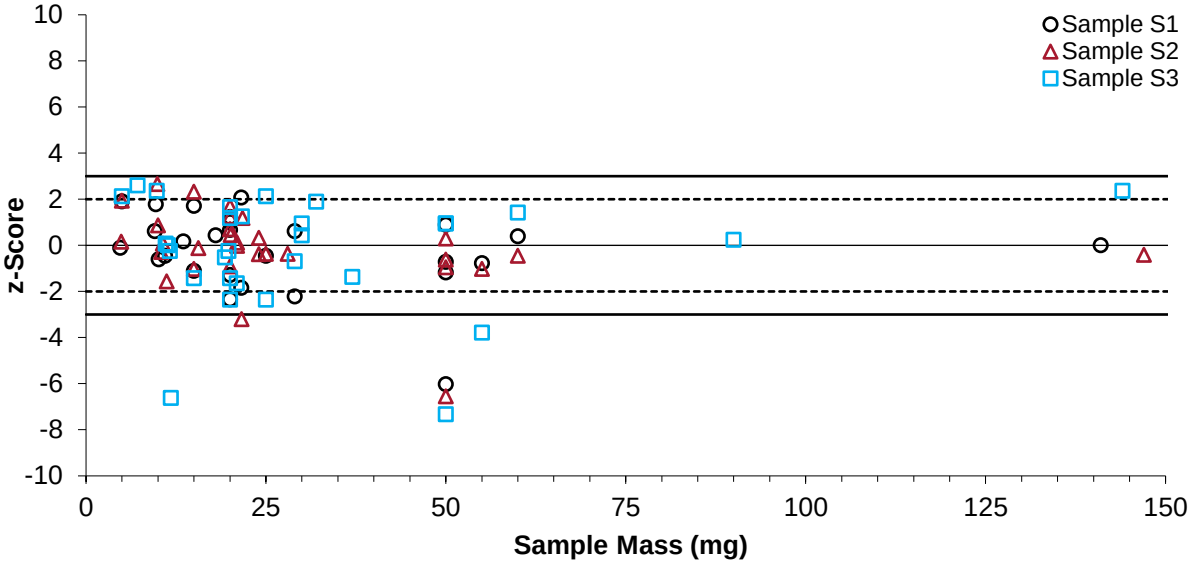


Figure 7 *z*-Score vs Sample Mass Used for Analysis

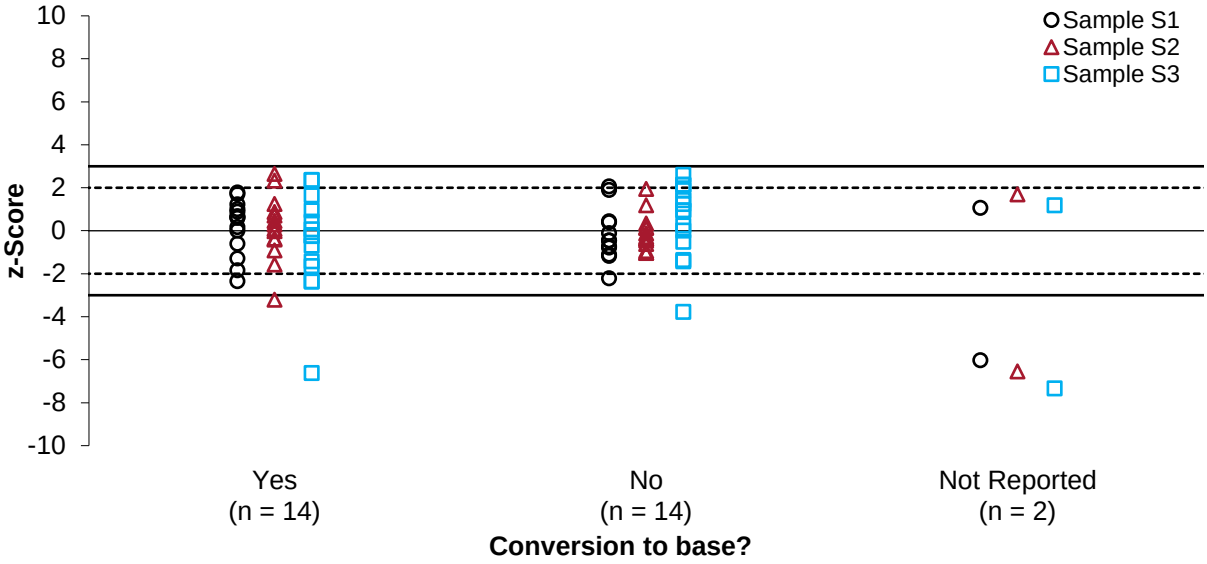


Figure 8 *z*-Score vs Sample Processing

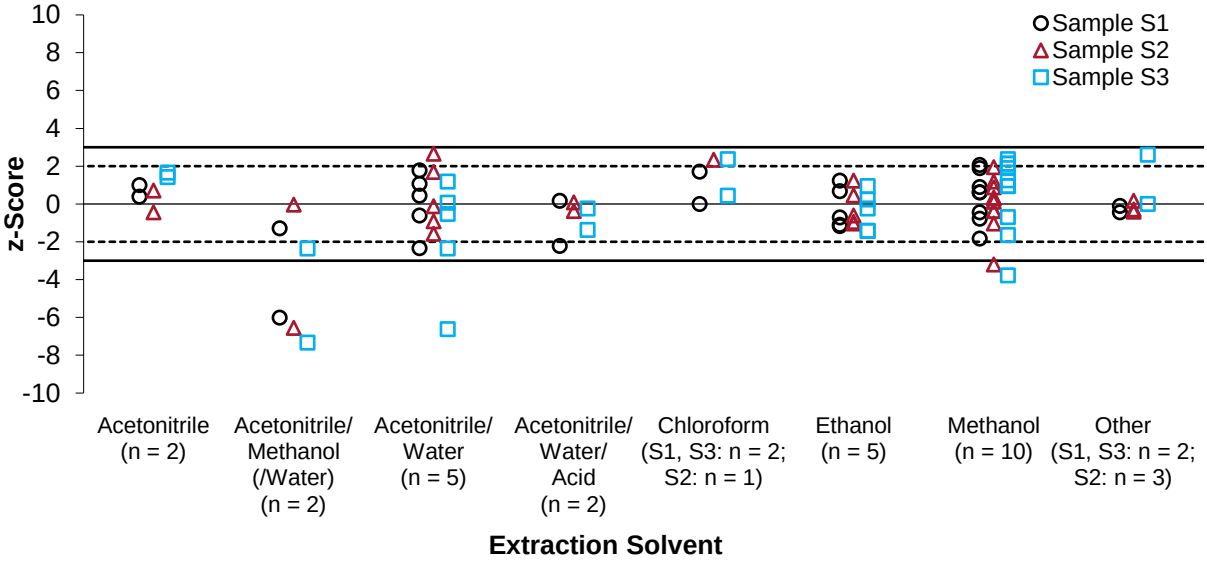


Figure 9 *z*-Score vs Extraction Solvent

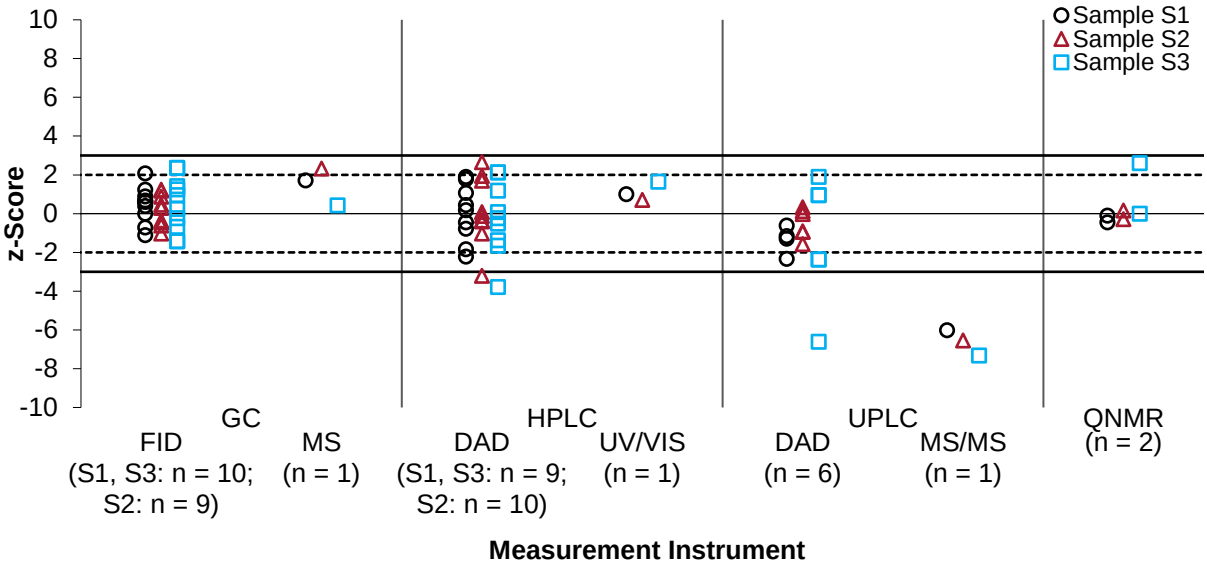


Figure 10 *z*-Score vs Measurement Instrument

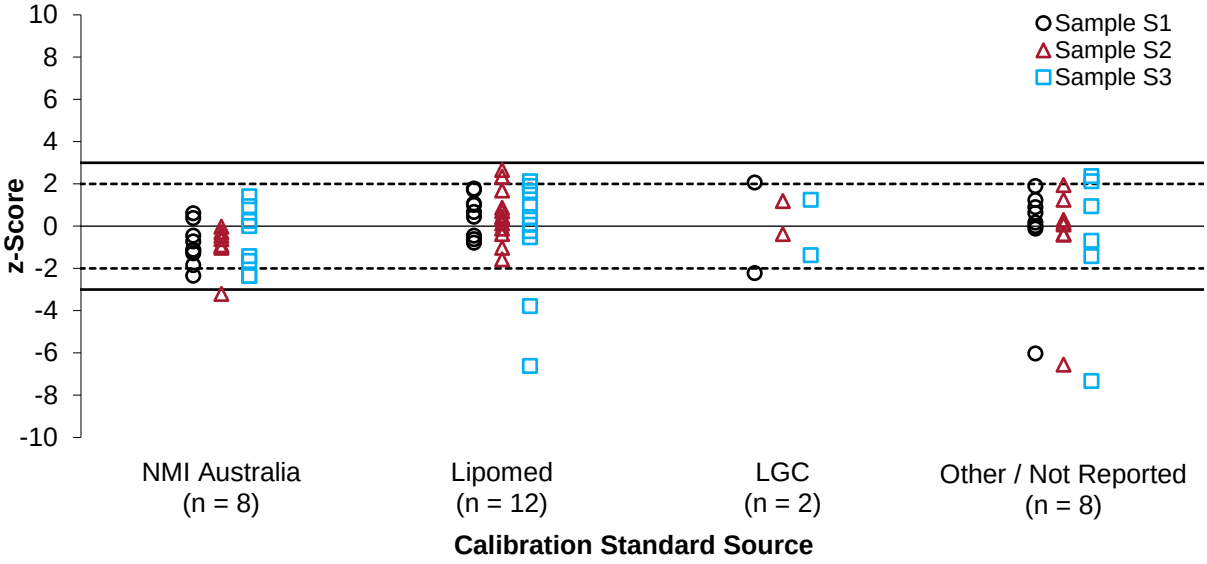


Figure 11 *z*-Score vs Source of Calibration Standard

6.7 Comparison of Results and Date of Analysis

As there were significant delays with sample delivery to some participants, the samples were analysed by participants over the course of approximately five months. No trend was found between when the samples were analysed and the results obtained (Figure 12).

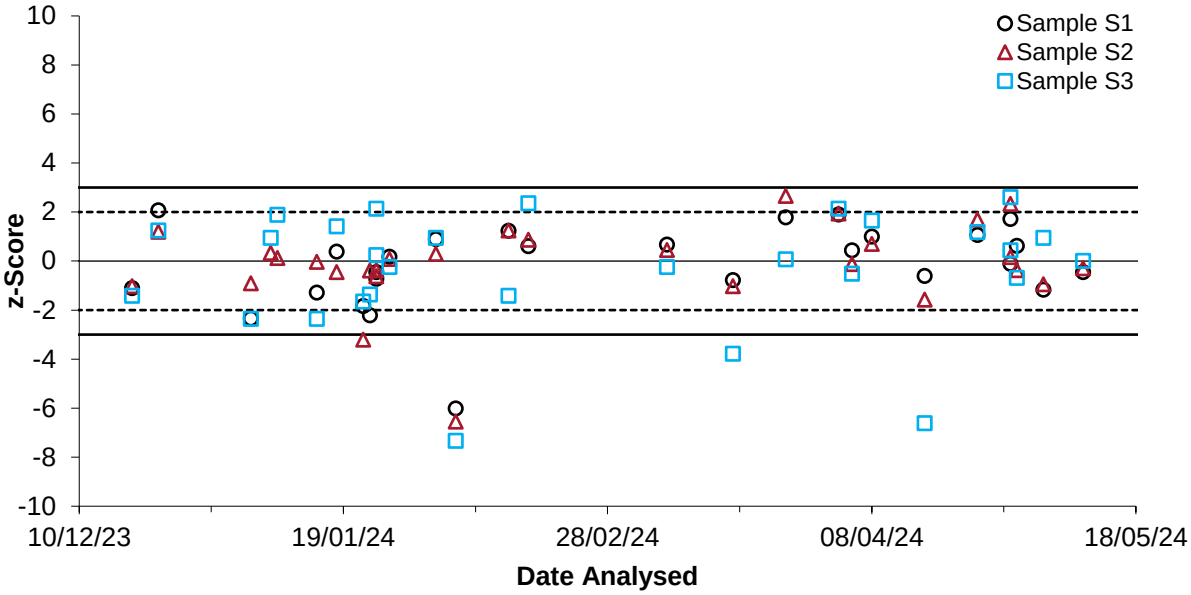


Figure 12 *z*-Score vs Sample Analysis Date

6.8 Comparison with Previous Cocaine PT Studies

To enable direct comparison with previous Cocaine PT studies, the target SD used to calculate *z*-scores has been kept constant at 3% PCV.

A summary of the acceptable performance, presented as a percentage of the total number of scores, obtained by participants from 2014 to 2023 (last 10 studies) are presented in Figure 13. The average proportion of acceptable *z*-scores and E_n-scores over this period is 83% and 86% respectively.

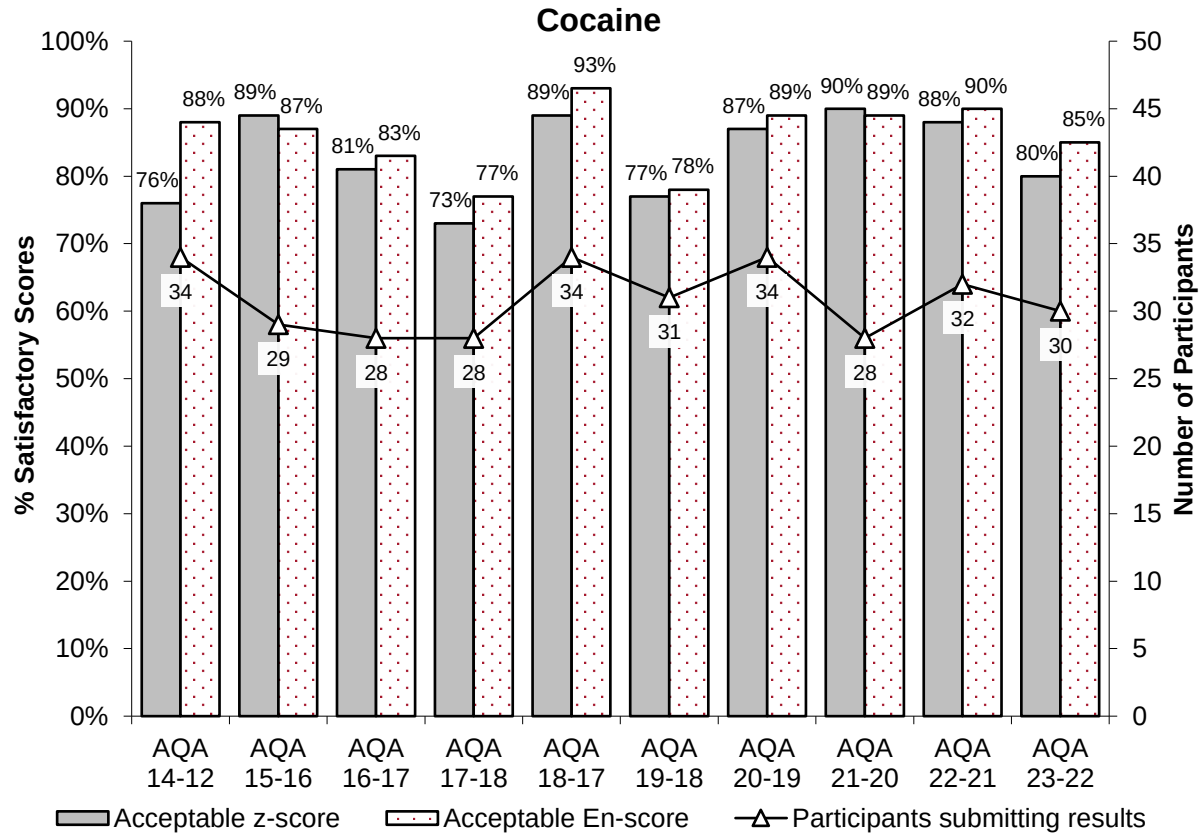


Figure 13 Summary of Participants' Performance in Cocaine PT Studies

Several participants have consistently participated in NMI Cocaine PT studies, and individual performance history reports are emailed to each participant at the end of the study. The consideration of *z*-scores for an analyte over time provides much more useful information than a single *z*-score. Over time, laboratories should expect at least 95% of their scores to lie within the range $|z| \leq 2.0$. 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 comparison of all results from Australian and international laboratories in NMI Cocaine PT studies over the last ten years is presented in Figure 14. Overall both groups have performed similarly, with both achieving 83% acceptable *z*-scores over this period.

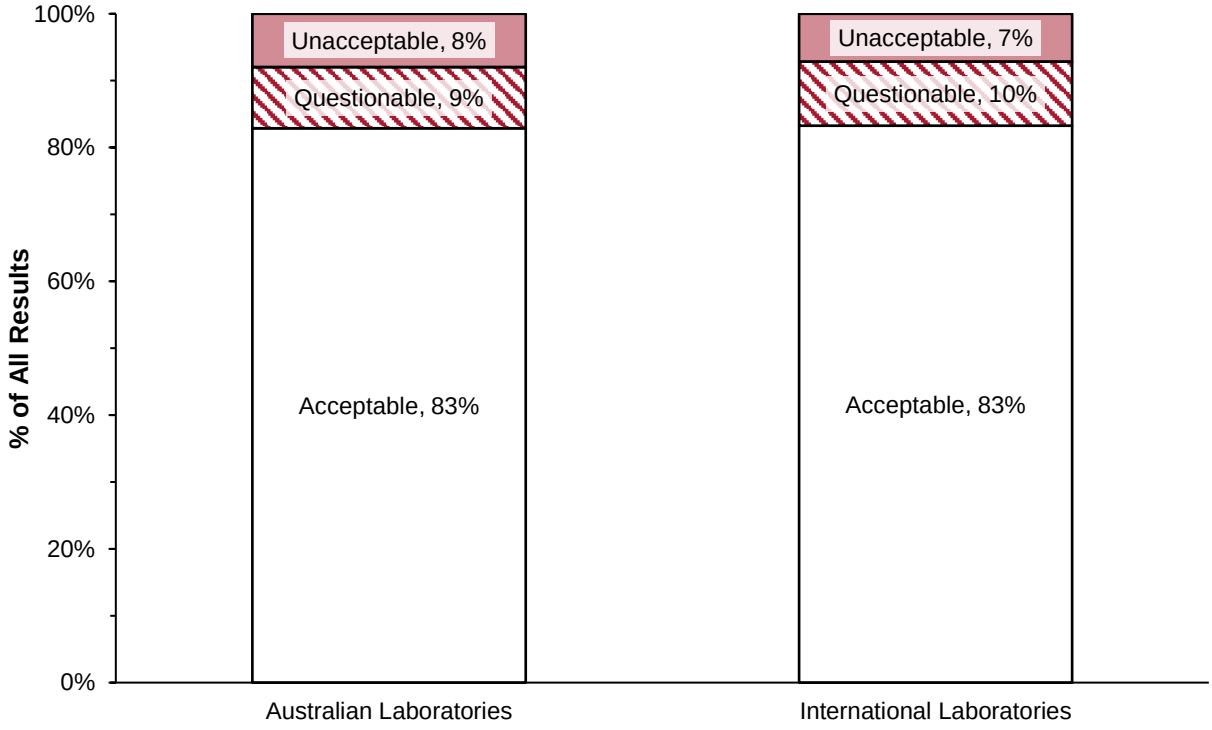


Figure 14 Comparison of Australian and International Laboratories in NMI Cocaine PT Studies

For those laboratories consistently participating in NMI Cocaine PT studies, a summary of individual laboratory's performances over the last ten studies is presented in Figures 15 and 16 for Australian and international laboratories respectively. One Australian and two international laboratories have achieved acceptable *z*-scores across all samples in all NMI Cocaine PT studies participated in over this period.

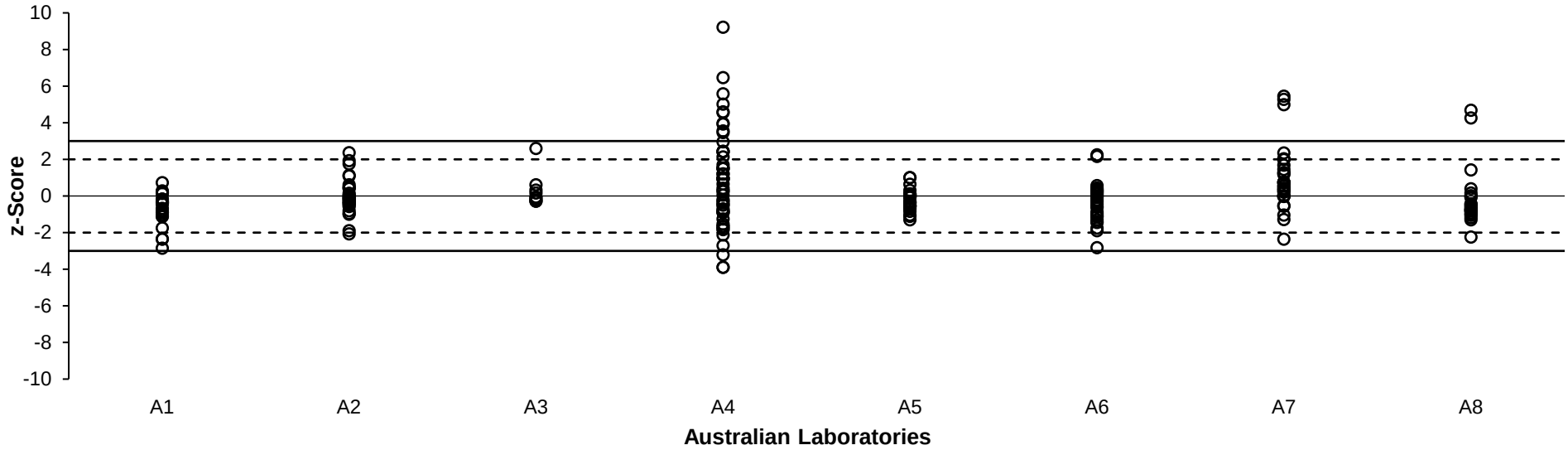


Figure 15 Summary of Australian Participants' z-Scores in NMI Cocaine PT Studies

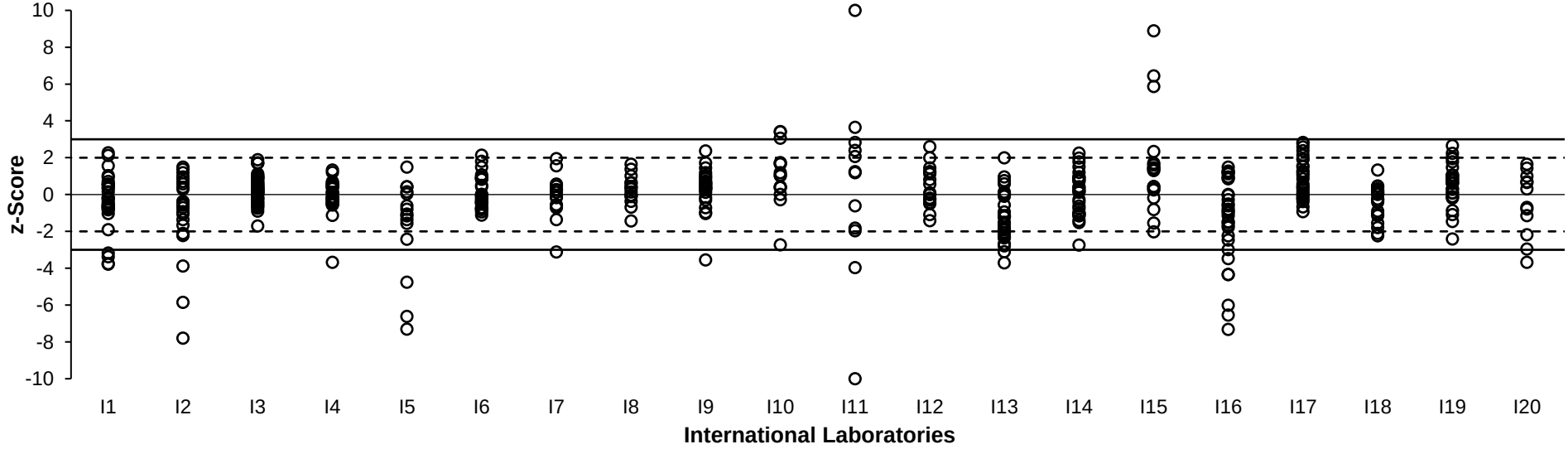


Figure 16 Summary of International Participants' z-Scores in NMI Cocaine PT Studies

7 REFERENCES

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

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

[2] NMI, 2024, *Study Protocol for Proficiency Testing*, viewed May 2024, <https://www.industry.gov.au/sites/default/files/2020-10/cpt_study_protocol.pdf>

[3] NMI, 2024, *Chemical Proficiency Testing Statistical Manual*, viewed May 2024, <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 May 2024, <http://eurachem.org/images/stories/guides/pdf/quam2012_P1.pdf>

[9] NATA, 2020, *Update to Measurement Uncertainty resources*, viewed May 2024, <https://nata.com.au/news/update-to-measurement-uncertainty-resources/>

APPENDIX 1 ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE AND E_n-SCORE CALCULATIONS

A1.1 Robust Average and Associated Uncertainty

Robust averages were calculated using the procedure described in ISO 13528.[5] The associated uncertainties were estimated as according to Equation 4.

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

where:

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

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

p is the number of results

The expanded uncertainty ($U_{rob\ average}$) 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 11.

Table 11 Uncertainty of Sample S1 Robust Average

No. results (p)	28
Robust average	59.8% base (m/m)
$S_{rob\ average}$	2.6% base (m/m)
$u_{rob\ average}$	0.6% base (m/m)
k	2
$U_{rob\ average}$	1.2% base (m/m)

Therefore, the robust average of Sample S1 is 59.8 ± 1.2% base (m/m).

A1.2 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 12.

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

Participant Result (% base (m/m))	Assigned Value (% base (m/m))	Target SD	z-Score	E_n-Score
60.9 ± 2.4	59.8 ± 1.2	3% as PCV, or: 0.03 × 59.8 = 1.794% base (m/m)	$z = \frac{60.9 - 59.8}{1.794} = 0.61$	$E_n = \frac{60.9 - 59.8}{\sqrt{2.4^2 + 1.2^2}} = 0.41$

APPENDIX 2 ACRONYMS AND ABBREVIATIONS

CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
DAD	Diode Array Detection
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
Max	Maximum
Md	Median
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
NMI	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
SD	Standard Deviation
SI	International System of Units
SS	Spiked Samples
UPLC	Ultra Performance Liquid Chromatography
UV/Vis	Ultraviolet/Visible detection

END OF REPORT