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Science and Resources

National  
Measurement  
Institute

# Proficiency Test Final Report AQA 22-21 Cocaine

June 2023

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## SUMMARY

AQA 22-21 Cocaine commenced in September 2022. Sample sets, each containing three samples of cocaine hydrochloride, were sent to thirty-one 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, approximately 84% base (m/m) 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 96  $z$ -scores, 84 (88%) returned  $|z| \leq 2.0$ , indicating a satisfactory performance.

Of 96  $E_n$ -scores, 86 (90%) returned  $|E_n| \leq 1.0$ , indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratories **1, 2, 3, 6, 7, 8, 9, 10, 11, 12, 14, 15, 18, 19, 20, 23, 25, 27, 28, 29, 30, 31** and **32** returned satisfactory  $z$ -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 96 numeric results, 93 (97%) were reported with an associated expanded measurement uncertainty. The magnitude of reported uncertainties was within 1.8% to 89% 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 caffeine and levamisole, Sample S2 was cut with procaine, and Sample S3 was cut with paracetamol.

Twenty-nine participants (91%) reported on the identity of the cutting agent(s) in at least one sample.

Laboratories **1, 2, 3, 6, 7, 8, 9, 11, 12, 13, 14, 16, 17, 18, 19, 20, 21, 23, 24, 25, 26, 27, 29, 31** and **32** 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'.<sup>1</sup> 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, 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.<sup>2</sup> The statistical methods used are described in the NMI Chemical Proficiency Testing Statistical Manual.<sup>3</sup> These documents have been prepared with reference to ISO/IEC 17043 and The International Harmonized Protocol for Proficiency Testing of Analytical Chemistry Laboratories.<sup>1,4</sup>

NMI is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of proficiency testing schemes.<sup>1</sup> 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	12/09/2022
Samples sent	14/11/2022
Results due	27/03/2023
Interim Report	12/05/2023
Preliminary Report	15/05/2023

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

### **2.2 Participation and Laboratory Code**

Thirty-one 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 September 2022. The starting material was cocaine hydrochloride samples approximately 84% base (m/m) supplied by the Australian Federal Police.

Caffeine and 4-acetamidophenol (paracetamol) purchased from Sigma Aldrich, procaine hydrochloride purchased from Labchem, and levamisole hydrochloride purchased from ACROS were used as cutting agents. Sample S1 was cut with caffeine and levamisole, Sample S2 was cut with procaine, and Sample S3 was cut with paracetamol.

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.

Sample S1 was prepared to contain approximately 18% cocaine base (m/m).

Sample S2 was prepared to contain approximately 67% cocaine base (m/m).

Sample S3 was prepared to contain approximately 52% 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 date of analyses. The results gave no reason to question the stability of the test samples (Section 6.7).

## 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 November 2022. 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 diluent(s)/adulterant(s) in all three samples if this is within your normal scope of analysis.
- Give brief details 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)

as requested by the results sheet.

- A result spreadsheet has been emailed to you. Please complete this spreadsheet and return by email to [jenny.xu@measurement.gov.au](mailto:jenny.xu@measurement.gov.au).
- Results are to be returned by 27 February 2023.

There were significant delivery delays to some international participants, and so the results due date was extended to 27 March 2023.

## 2.7 Interim Report and Preliminary Report

An Interim Report was emailed to all participants on 12 May 2023. One participant's sample delivery was extensively delayed due to additional customs issues, and so the release of the Interim Report was delayed to allow this participant to receive their samples and report results.

A Preliminary Report was emailed to all participants on 15 May 2023. This report included a summary of the results reported by laboratories, assigned values, performance coefficient of variations,  $z$ -scores and  $E_n$ -scores for each analyte in this study. No data from the Preliminary Report has been changed in the present Final Report.



### 3 PARTICIPANT LABORATORY INFORMATION

#### 3.1 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	ACN/MeOH/H <sub>2</sub> O	Analog of cocaine	7	UPLC	MS/MS	C-18 column
2	Methanol	Tetracosane	4	GC	FID	HP5 30m x 0.32 mm x 0.25 μm
3	acetonitrile/water (80/20)	none	3	HPLC	DAD	C8
4	Ethanol	Triphenylacetophenone (TPAP)	3	GC	FID	HP1-MS
5	H <sub>2</sub> O/Acetonitrile (60/40)		5	UPLC	UV/Vis	Kromasil
6	Ethanol	Tribenzylamine	6	GC	FID	HP5
7	Acetonitrile:water 75:25	Diethyl phthalate	3	UPLC	PDA	Acquity UPLC BEH C18 1.7μm (2.1 x 100 mm)
8	HPLC Methanol	Vanillin	1	UPLC	DAD	Agilent LiChrospher 60 RP-select B
9	Methanol	Diazepam	6	GC	FID	J&W 128-5512
10	Acetonitrile		4	HPLC	UV/DAD	Interchrom C8 5UM 150x4.6MM
11	methanol	NO	7	HPLC	DAD	zorbax eclipse XDB-C18 (5 microns,4.6mm X150mm)
12	Methanol	-	5	HPLC	UV	Luna Omega 3um PS C18 100Å, 150 x 4.6 mm
13	acetonitrile/water	none	1	HPLC	DAD	Kromasil
14	water/acetonitrile/ 2.5M sulphuric acid 90:10:1	None	3	HPLC	Diode Array	Shimpack XR-ODS
15	Chloroform: Methanol	Tetracosane	1	GC	FID	HP5
16	acetonitrile/water (80/20)	external standard	2	HPLC	DAD	C8
17	Water/ACN	N/A	5	HPLC	UV	Kinetex 5u C18

Lab. Code	Extraction Solvent	Internal Standard	Calib. Points	Technique	Detector	Column
18	Methanol	-	4	HPLC	DAD	ECLIPSE XDB-C18
19	Methanol	none	5	HPLC	DAD	Kinetex 2.6 $\mu$ XB-C18
20	Methanol	N/A	7	HPLC	DAD	Luna 3 $\mu$ m C8(2) 100A, 100 x 2mm
21	S1 & S2: Mobile Phase S3: Chloroform	S1 & S2: none S3: benzopinacolone	S1 & S2: 4 S3: 1	S1&S2: HPLC S3: GC	S1&S2: PDA S3: FID	S1 & S2: C18 ubondpak S3: HP-1
22	Chloroform/ Methanol	n-Tetracosane	1	GC	FID	HP5
23	d-chloroform	1,4-bis(trimethylsilyl) benzene		QNMR	NA	500MHz
24	Ethanol	Propyl Paraben	8	UPLC	DAD	BEH Shield RP18
25	Acetonitrile/Water (25:75)	N/A	3	HPLC	DAD	ODS2 Inertpak Column (25cmx4.6mm)
26	Chloroform	Octacosane	5	GC	MS	Zebron ZB-5MSplus
27	Acetonitrile/ Methanol	Pholcodine 1mg/ml	3	UPLC	PDA	ACQUITY C-18
28	Sodium Phosphate (pH 4.5)	No	4	HPLC	DAD	Hypersil GOLD C8
29	HPLC Methanol	Vanillin	1	UPLC	DAD	Agilent LiChrospher 60 RP-select B
30	Methanol	-	3	GC	FID	CP-sil5CB
31	Ethanol	Tetracosane	6	GC	FID	HP5
32	Acetonitrile	Strychnine	6	GC	FID	HP-1-MS

### 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	Unikem	100
2	Lipomed	99.58 $\pm$ 0.016
3	lipomed	99.199
4	NMI	99.8 $\pm$ 0.8
5	Lipomed (EUROMEDEX)	99.5

Lab. Code	Reference Standard	Purity (%)
6	Lipomed	99.503
7	NMI	99.8
8	Lipomed	99.199 ± 0.006
9	Lipomed	99.3
10	Lipomed	99.9
11	Lipomed	99.9
12	Chiron	99.5
13	Sigma	99.9
14	LGC (Mikromol)	99.9
15	TRC	99.88
16	NMI	99.8
17	Sigma	98.7
18		
19	Lipomed	>98.5
20	NMI	99.8
21	MacFarlan Smith	100.2
22	TRC	99.88
23	NMI	99.7
24	NMI	96.1
25	Toronto Research Chemicals	98
26	lipomed	99.199
27	NMI	99.8
28	CocaineHCL (C5776), Sigma Aldrich	100
29	Lipomed	99.199 ± 0.006
30	Duchefa	>99
31	Alcaliber	100.7
32	NMI	99.8

### 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	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM		
2	Top Down - precision and estimates of the method and laboratory bias	Control samples	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
3	Top Down - precision and estimates of the method and laboratory bias	Control samples	Laboratory bias from PT studies	NF V03-110
4	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Control samples - CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Matrix effects Recoveries of SS Standard purity	Eurachem/ CITAC Guide
5	Top Down - precision and estimates of the method and laboratory bias	Standard deviation from PT studies only		ISO/GUM
6	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM	Standard purity	
7	Standard deviation of replicate analyses multiplied by 2 or 3	Duplicate analysis	Homogeneity of sample Standard purity	Eurachem/ CITAC Guide
8	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Laboratory bias from PT studies	Eurachem/ CITAC Guide
9	Estimating Measurement Uncertainty by black box with pairs of values	Standard deviation from PT studies only		ISO/GUM ENAC G 09 or ISO 21748
10	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - RM	Laboratory bias from PT studies	
11	Top Down-precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Laboratory bias from PT studies Standard purity	Eurachem/ CITAC Guide
12	Reproducibility, bias, sample heterogeneity	Control samples - RM Duplicate analysis	Instrument calibration Homogeneity of sample Standard purity	ENFSI Best Practise manual, Eurachem/ CITAC guide

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
13	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - RM		ISO/GUM
14	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - CRM Duplicate analysis	Instrument calibration Standard purity	ISO/GUM
15	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Matrix effects Standard purity	ASCLD/LAB Guidance on the Estimation of Measurement Uncertainty, AL-PD-3061
16	Top Down - reproducibility (standard deviation) from PT studies used directly	Control samples - RM Duplicate analysis	Instrument calibration Laboratory bias from PT studies	Eurachem/ CITAC Guide
17	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Matrix effects Standard purity	Eurachem/ CITAC Guide
18	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM	Recoveries of SS Standard purity	Eurolab Technical Report No1/2007
19	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Recoveries of SS	Eurachem/ CITAC Guide
20	Top Down - precision and estimates of the method and laboratory bias	Control samples - In House Control Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Matrix effects Standard purity	ISO/GUM
21	Validation (k = 2)			
22	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Recoveries of SS Standard purity	Eurachem/ CITAC Guide
23	Top Down - precision and estimates of the method and laboratory bias	Control samples - previously analysed real seizure samples Duplicate analysis	Instrument calibration Matrix effects	Eurachem/ CITAC Guide
24				
25	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Instrument calibration Masses and volumes Recoveries of SS	ISO/GUM
26	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis	Laboratory bias from PT studies	ISO/GUM

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
27	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
28	Top Down - precision and estimates of the method and laboratory bias	Control samples - Samples from police case Duplicate analysis	Laboratory bias from PT studies	Nordtest Report TR537
29	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Laboratory bias from PT studies	Eurachem/ CITAC Guide
30	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Instrument calibration Recoveries of SS	ISO/GUM
31	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis	Matrix effects Standard purity	ISO/GUM
32	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Laboratory bias from PT studies	ISO/GUM

\* 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 Coordinator's Response
3	is it possible to put a sample around 5% and another around 80% or more?	Thank you for your suggestion. 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, samples were prepared at around 18%, 52% and 67% cocaine base (m/m).
4	Extra sample was used for S1 determination to ensure analysis would fall within calibrated range. Methodology: A small amount of dichloromethane was used to dissolve the TPAP prior to the addition of Ethanol	
11	Qualitative analysis was carried out by GC-MS	

Lab. Code	Participants' Comments	Study Coordinator's Response
13	Methodology: 1 point, 5 injections	
14	Analysis for Inert cutting agents not undertaken as part of standard analytical procedure for S1 and S2 samples Uncertainty: MuM determined from multiple injections of reference material. $3 \times (\text{Std Dev}/\text{mean}) \times 100$ . no analysis undertaken for inert bulking agents for S1 and S2 samples	
15	Methodology: Quantitation of Cocaine by GC	
21	Methodology: Different methods used due to interfering substance - paracetamol	
23	Methodology: No reference standard involved	
25	Routine case samples would always be round down i.e. -1.6% for example $18.2 \times 0.9840 = 17.9$ Methodology: Less than 20mg used as following lab SOP which states approximately 10mg Uncertainty: The reported result (in routine case samples) is defined as the mean of the individual results multiplied by the uncertainty correction factor and is rounded down to the nearest whole number (unless <1% w/w). e.g a mean result of 18.2% with an uncertainty correction value of 98.40% would give a reported result of $18.2 \times 0.9840 = 17.9$ therefore rounded down to 17%.	
30	Methodology: external calibration	

## 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.

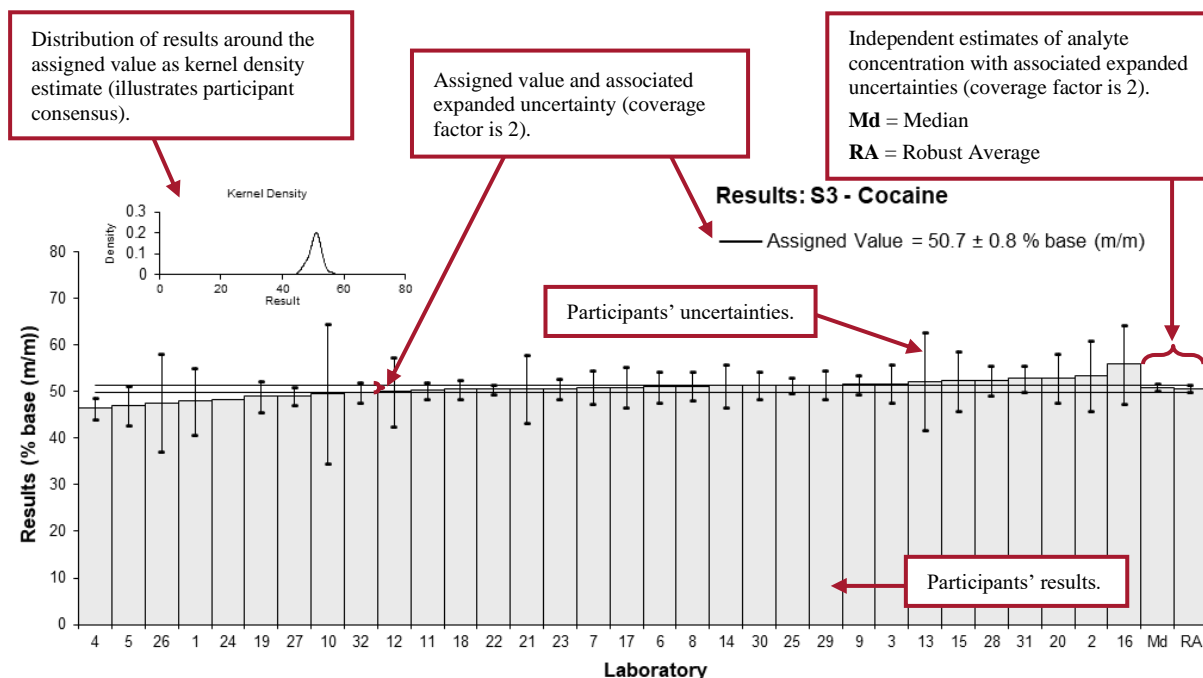


Figure 1 Guide to Presentation of Results

### 4.2 Assigned Value

The assigned value is defined as the ‘value attributed to a particular property or characteristic of a proficiency testing item’.<sup>1</sup> 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.<sup>5</sup>

### 4.4 Performance Coefficient of Variation (PCV)

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.<sup>6</sup> 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 ( $\sigma$ ) is the product of the assigned value ( $X$ ) and the PCV, as presented in Equation 1.

$$\sigma = X \times \text{PCV} \quad \text{Equation 1}$$

#### 4.6 z-Score

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

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

where:

- $z$  is z-score
- $\chi$  is a participant's result
- $X$  is the assigned value
- $\sigma$  is the target standard deviation for proficiency assessment from Equation 1

For the absolute value of a z-score:

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

#### 4.7 $E_n$ -Score

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

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

where:

- $E_n$  is  $E_n$ -score
- $\chi$  is a participant's result
- $X$  is the assigned value
- $U_\chi$  is the expanded uncertainty of the participant's result
- $U_X$  is the expanded uncertainty of the assigned value

For the absolute value of an  $E_n$ -score:

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

#### 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.<sup>7</sup>

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

## 5 TABLES AND FIGURES

Table 5

### Sample Details

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

### Participant Results

Lab. Code	Result	Uncertainty	z	E <sub>n</sub>
1	18	2.7	0.95	0.18
2	18.0	2.5	0.95	0.20
3	17.30	1.38	-0.38	-0.14
4	16.5	0.9	-1.90	-1.05
5	15	1.35	-4.76	-1.81
6	17.9	1.1	0.76	0.35
7	17.3	1.2	-0.38	-0.16
8	16.6	1.0	-1.71	-0.86
9	17.7	1.4	0.38	0.14
10	16.9	15	-1.14	-0.04
11	17.8	1.6	0.57	0.18
12	17.8	2.6	0.57	0.11
13	18.56	3.7	2.02	0.29
14	18.12	1.64	1.18	0.37
15	17.7	2.1	0.38	0.09
16	19.1	2.9	3.05	0.55
17	19	1.62	2.86	0.91
18	17.5	1	0.00	0.00
19	17	1.2	-0.95	-0.40
20	18	3	0.95	0.17
21	16.5	0.8	-1.90	-1.17
22	16.7	0.3	-1.52	-1.89
23	17.2	0.77	-0.57	-0.36
24	16.1	NR	-2.67	-4.67
25	18.2	1.6	1.33	0.43
26	17.07	3.76	-0.82	-0.11
27	17.20	1.30	-0.57	-0.22
28	18.2	1.1	1.33	0.61
29	17.2	1.1	-0.57	-0.26
30	17.66	3	0.30	0.05
31	17.4	1.5	-0.19	-0.07
32	17.5	0.8	0.00	0.00

### Statistics

<b>Assigned Value</b>	17.5	0.3
<b>Robust Average</b>	17.5	0.3
<b>Median</b>	17.5	0.3
<b>Mean</b>	17.5	
<b>N</b>	32	
<b>Max</b>	19.1	
<b>Min</b>	15	
<b>Robust SD</b>	0.74	
<b>Robust CV</b>	4.2%	

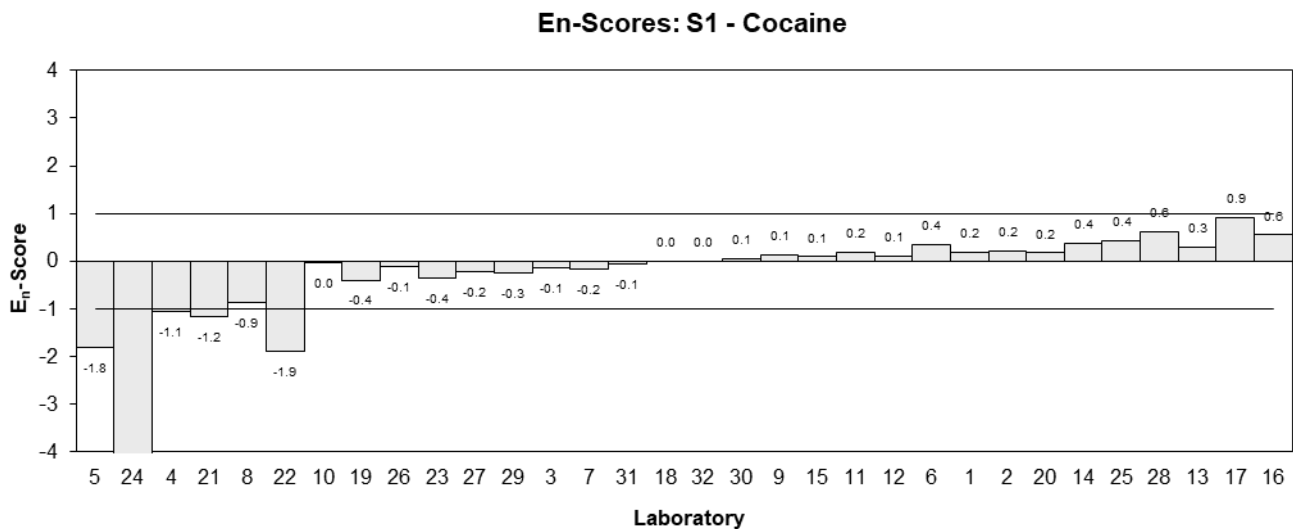
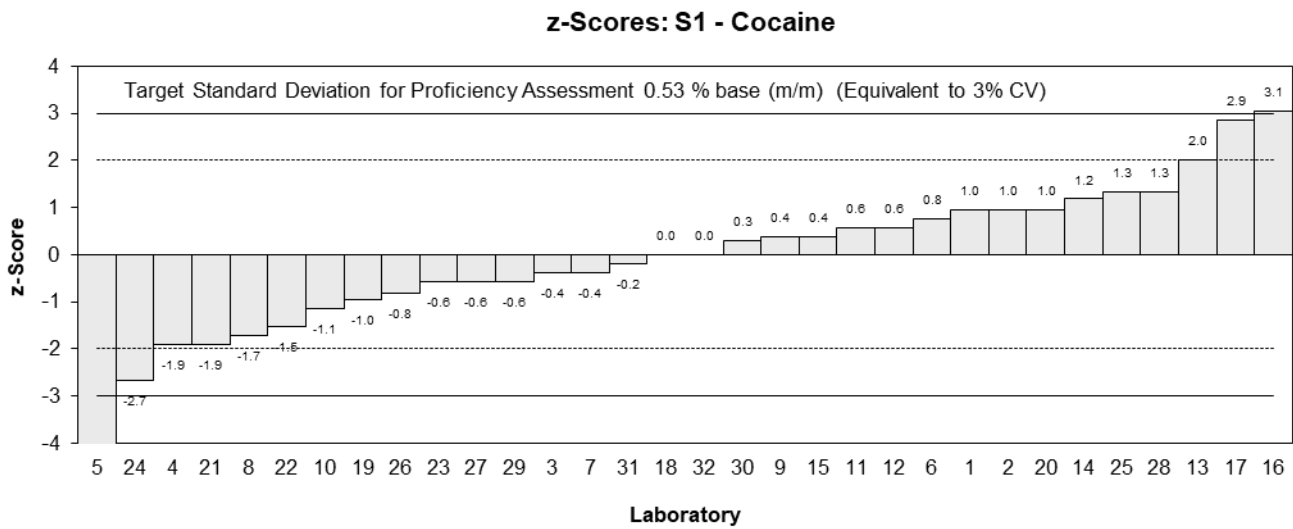
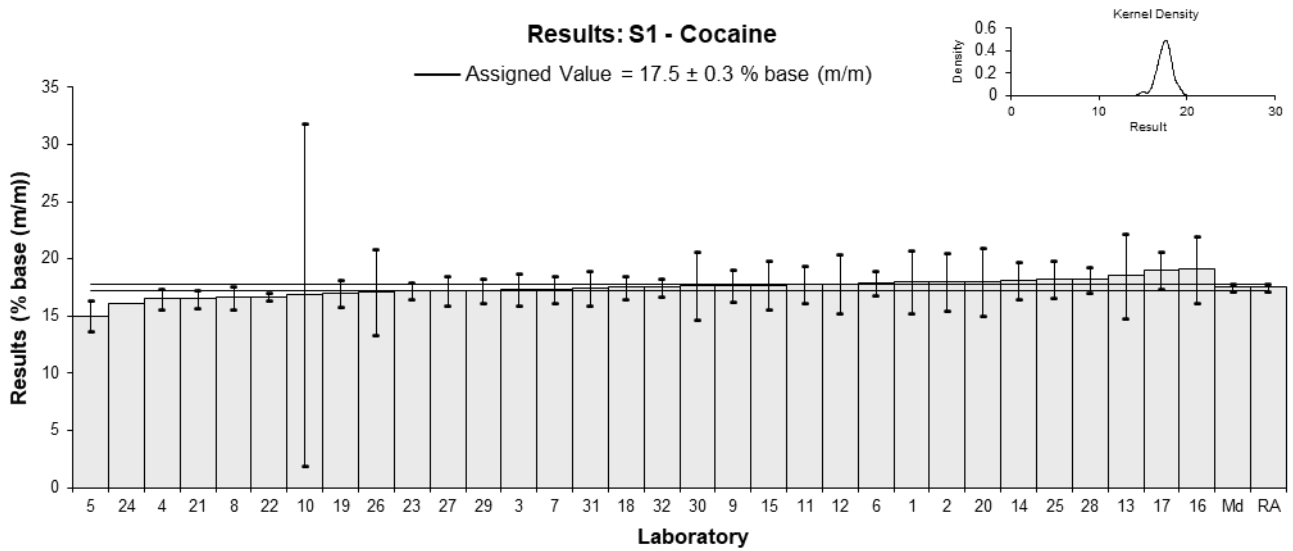


Figure 2

Table 6

## Sample Details

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

## Participant Results

Lab. Code	Result	Uncertainty	z	E <sub>n</sub>
1	65	9.8	-0.80	-0.16
2	69.0	9.7	1.20	0.25
3	67.88	5.43	0.64	0.23
4	63.1	3.2	-1.75	-1.05
5	52	4.68	-7.31	-3.06
6	67.0	4.3	0.20	0.09
7	67.0	4.8	0.20	0.08
8	67.1	4.1	0.25	0.12
9	67.1	2.7	0.25	0.18
10	65.2	15	-0.70	-0.09
11	65.5	2.2	-0.55	-0.46
12	65.4	9.7	-0.60	-0.12
13	70.5	14.1	1.95	0.28
14	68.41	6.19	0.91	0.29
15	65.6	8.1	-0.50	-0.12
16	73.4	11.1	3.40	0.61
17	68	5.78	0.70	0.24
18	67.5	4	0.45	0.22
19	65	4.6	-0.80	-0.34
20	70	7	1.70	0.48
21	67.4	3.2	0.40	0.24
22	65.7	1.3	-0.45	-0.57
23	65.3	2.94	-0.65	-0.42
24	62.5	NR	-2.05	-4.56
25	67.2	1.6	0.30	0.33
26	63.5	13.97	-1.55	-0.22
27	67.70	2.40	0.55	0.43
28	67.3	4.0	0.35	0.17
29	67.2	4.1	0.30	0.14
30	67.39	3	0.40	0.25
31	66.6	3.5	0.00	0.00
32	65.1	2.8	-0.75	-0.51

## Statistics

<b>Assigned Value</b>	66.6	0.9
<b>Robust Average</b>	66.6	0.9
<b>Median</b>	67.1	0.9
<b>Mean</b>	66.3	
<b>N</b>	32	
<b>Max</b>	73.4	
<b>Min</b>	52	
<b>Robust SD</b>	2.0	
<b>Robust CV</b>	3%	

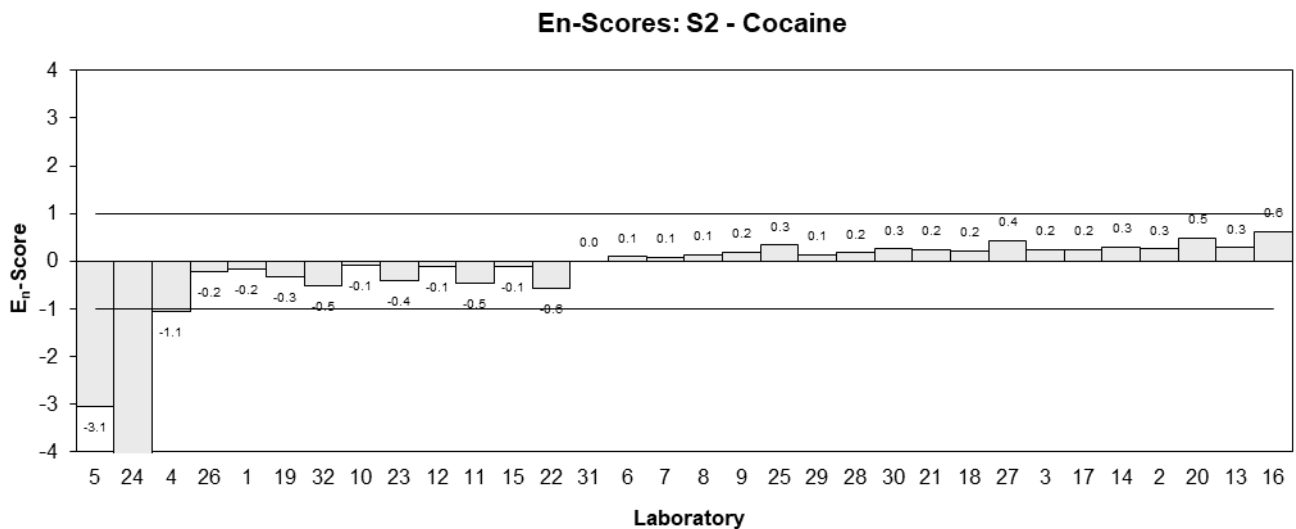
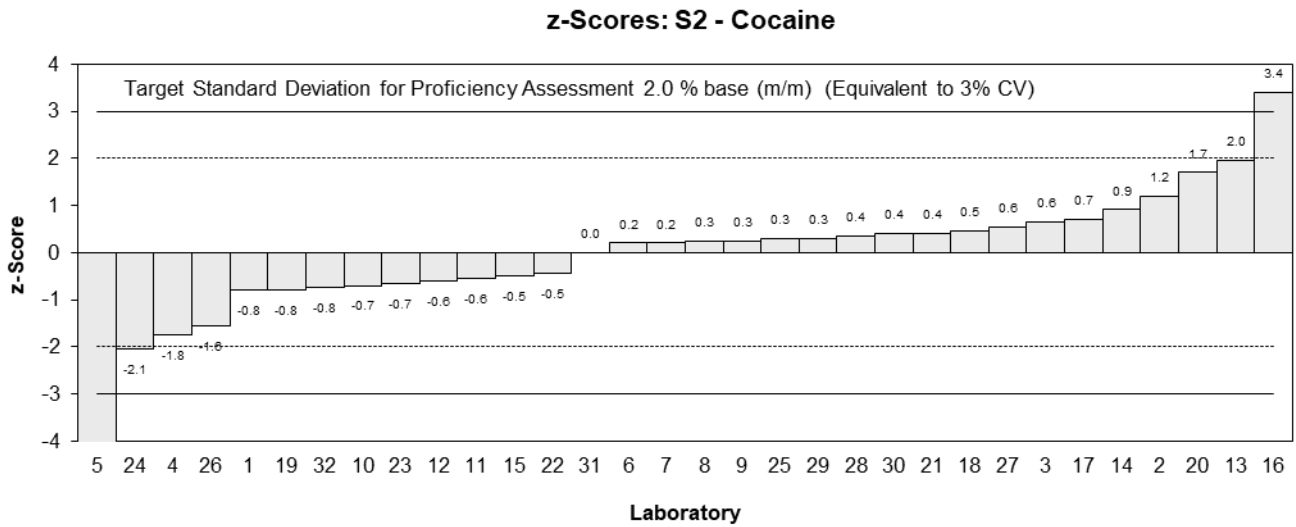
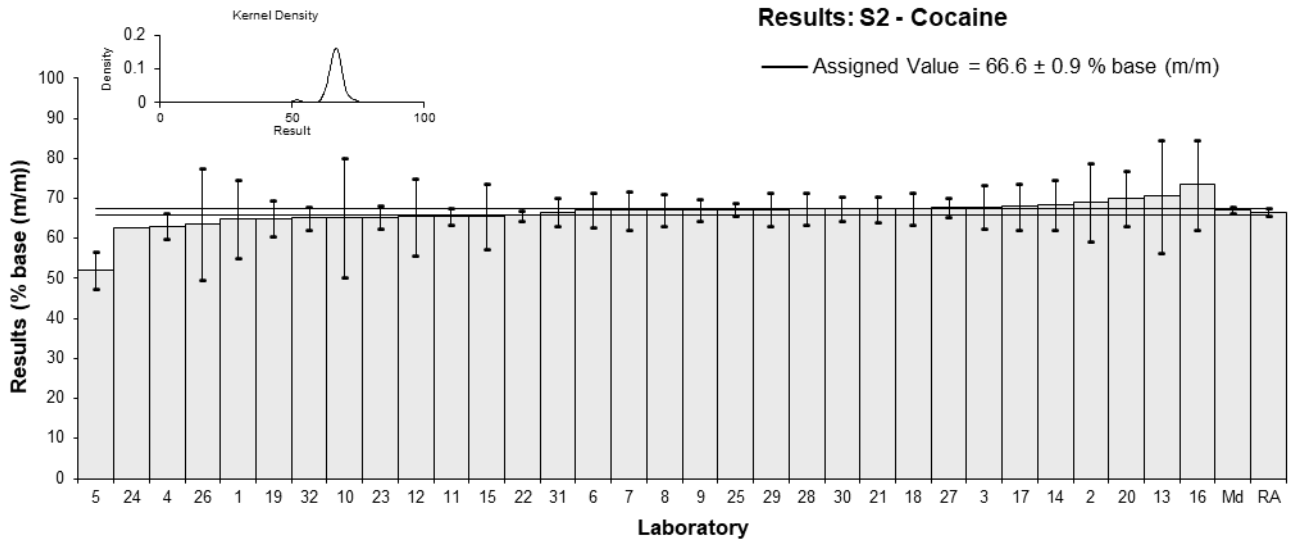


Figure 3

Table 7

## Sample Details

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

## Participant Results

Lab. Code	Result	Uncertainty	z	E <sub>n</sub>
1	48	7.2	-1.78	-0.37
2	53.4	7.5	1.78	0.36
3	51.76	4.14	0.70	0.25
4	46.4	2.4	-2.83	-1.70
5	47	4.23	-2.43	-0.86
6	51.1	3.3	0.26	0.12
7	51.0	3.6	0.20	0.08
8	51.2	3.1	0.33	0.16
9	51.6	2.1	0.59	0.40
10	49.5	15	-0.79	-0.08
11	50.3	1.8	-0.26	-0.20
12	50.0	7.4	-0.46	-0.09
13	52.26	10.5	1.03	0.15
14	51.31	4.64	0.40	0.13
15	52.3	6.4	1.05	0.25
16	55.9	8.4	3.42	0.62
17	51	4.34	0.20	0.07
18	50.5	2	-0.13	-0.09
19	49	3.4	-1.12	-0.49
20	53	5.3	1.51	0.43
21	50.6	7.4	-0.07	-0.01
22	50.5	1.0	-0.13	-0.16
23	50.6	2.28	-0.07	-0.04
24	48.4	NR	-1.51	-2.88
25	51.4	1.6	0.46	0.39
26	47.61	10.47	-2.03	-0.29
27	49.10	1.90	-1.05	-0.78
28	52.4	3.1	1.12	0.53
29	51.5	3.1	0.53	0.25
30	51.36	3	0.43	0.21
31	52.9	2.8	1.45	0.76
32	49.9	2.2	-0.53	-0.34

## Statistics

<b>Assigned Value</b>	50.7	0.8
<b>Robust Average</b>	50.7	0.8
<b>Median</b>	51.0	0.7
<b>Mean</b>	50.7	
<b>N</b>	32	
<b>Max</b>	55.9	
<b>Min</b>	46.4	
<b>Robust SD</b>	1.8	
<b>Robust CV</b>	3.6%	

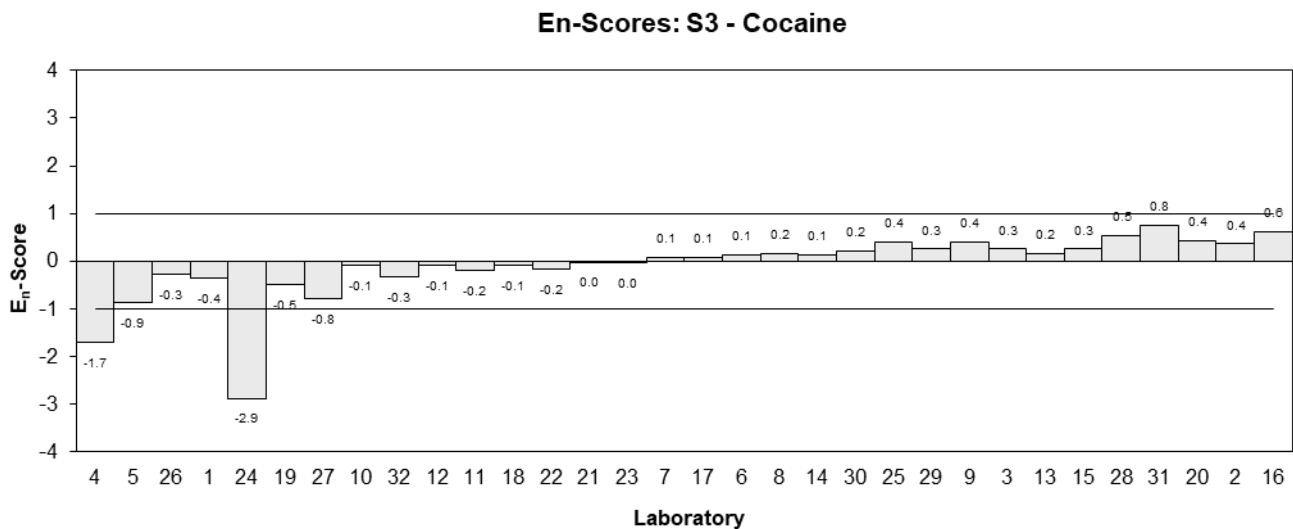
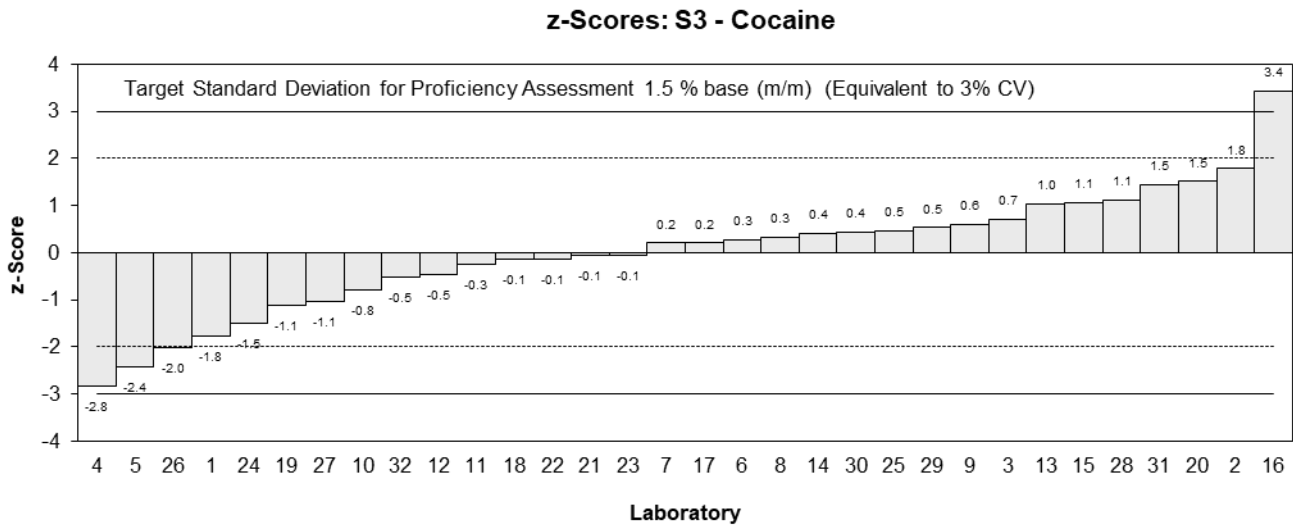
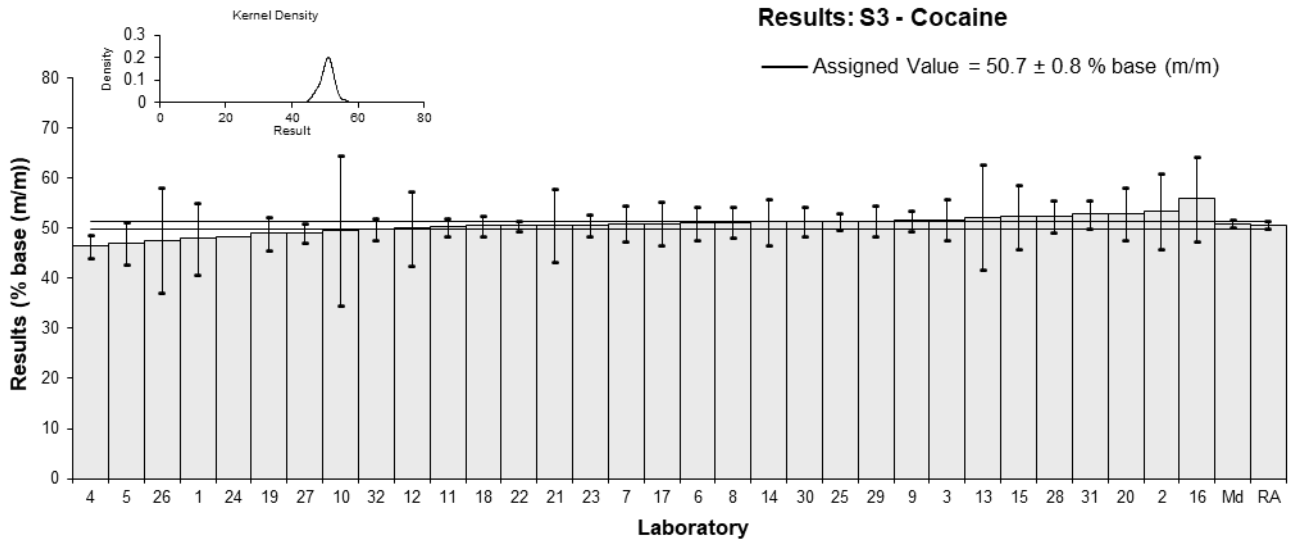


Figure 4

Table 8 Participants' Identification of Cutting Agents\*

Lab. Code	Cutting Agents		
	S1	S2	S3
Preparation	Caffeine, Levamisole	Procaine	Paracetamol
1	caffeine, levamisole	procaine	paracetamol
2	Caffeine, levamisole	Procaine	Paracetamol
3	caffeine, levamisole	procaine	paracetamol
4	Caffeine	Procaine	Paracetamol
5			
6	Caffeine (73.6%), Levamisole (base: 4.9%)	Procaine (base: 16.0%)	Paracetamol
7	Caffeine, Levamisole	Procaine	Paracetamol
8	caffeine, levamisole (specific isomer not determined)	Procaine	Paracetamol
9	caffeine, levamisole	procaine	acetaminophen
10			
11	Levamisole, caffeine	Procaine	p-acetamidophenol
12	caffeine, levamisole	procaine	paracetamol
13	Caffeine, Levamisole	Procaine	Acetaminophen
14	caffeine; Levamisole	Procaine; cinnamoylcocaine	Paracetamol, benzoylecgonine
15	CAFFEINE	PROCAINE	ACETAMINOPHEN
16	Caffeine/Levamisole	Procaine	Acetaminophen
17	Caffeine, Levamisole	Procaine	Paracetamol
18	Caffeine, Tetramisole	Procaine	Paracetamol
19	caffeine, levamisole	procaine	paracetamol
20	Caffeine, Levamisole/Dexamisole	Procaine	Paracetamol
21	caffeine, phenylimidothiazole	procaine	paracetamol
22	Caffeine	Procaine	Acetaminophen
23	Caffeine, levamisole	Procaine	Paracetamol
24	levamisole: 4.3% caffeine: 75.5%	procaine: 16.3%	acetaminophen: 42.2%
25	levamisole, caffeine	Procaine	paracetamol
26	caffeine + levamisole	procaine	acetaminophen
27	caffeine,levamisole	procaine	paracetamol
28	Tetramisole/Levamisole, Caffeine	Procaine	
29	Caffeine, Levamisole (specific isomer not determined)	Procaine	Paracetamol
30			
31	Caffeine, levamisole	Procaine	Paracetamol
32	caffeine, levamisole	procaine	paracetamol

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



## 6 DISCUSSION OF RESULTS

### 6.1 Assigned Value

The assigned values were the robust averages of the results reported by participants. The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528.<sup>5</sup> Results less than 50% and greater than 150% of the robust average were removed before calculation of the assigned value, if applicable.<sup>3,4</sup> The calculation of the expanded uncertainty for robust averages is presented in Appendix 1, with Sample S3 as an example.

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

### 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 (Section 3.3). One participant reported using the NATA GAG Estimating and Reporting MU as their guide; NATA no longer publishes this document.<sup>9</sup>

It is a requirement of ISO/IEC 17025 that laboratories have procedures to estimate the uncertainty of chemical measurements and to report this uncertainty in specific circumstances, including when the client's instruction so requires.<sup>7</sup> From July 2012 this is also a requirement of the ANAB-ASCLD/LAB international accreditation program.

Of 96 numeric results, 93 (97%) were reported with an associated expanded MU. Laboratory **24** did not report any uncertainties; this laboratory did not report whether they were accredited or not.

The magnitude of reported uncertainties was within the range 1.8% to 89% relative. Of the 93 expanded MUs, 62 (67%) were between 3% and 10% relative to the result. Laboratories reporting uncertainties smaller than 3% or larger than 10% relative may wish to consider whether these estimates are realistic or fit for purpose.

Laboratory **10** reported the same uncertainty for all three samples; participants were requested to report their uncertainties in units of % base (m/m), however this participant may have reported their relative uncertainties instead of absolute uncertainties.

Uncertainties associated with results returning a satisfactory  $z$ -score but an unsatisfactory  $E_n$ -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  $68.41 \pm 6.19\%$  base (m/m), it is better to report  $68.4 \pm 6.2\%$  base (m/m).<sup>8</sup>

### 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,<sup>6</sup> 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 (%)	Between-Laboratory CV (%)	Target SD (as PCV) (%)
S1	Cocaine	17.5	2.4	4.2	3
S2	Cocaine	66.6	1.2	3.0	3
S3	Cocaine	50.7	1.4	3.6	3

Of 96 results for which  $z$ -scores were calculated, 84 (88%) returned a  $z$ -score of  $|z| \leq 2.0$ , indicating a satisfactory performance.

Twenty-five participants: **1, 2, 3, 6, 7, 8, 9, 10, 11, 12, 14, 15, 18, 19, 20, 21, 22, 23, 25, 27, 28, 29, 30, 31** and **32** returned satisfactory  $z$ -scores for all three samples.

Seven participants returned at least one questionable or unsatisfactory  $z$ -score. Laboratories **5** and **16** returned questionable or unsatisfactory  $z$ -scores for all samples.

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

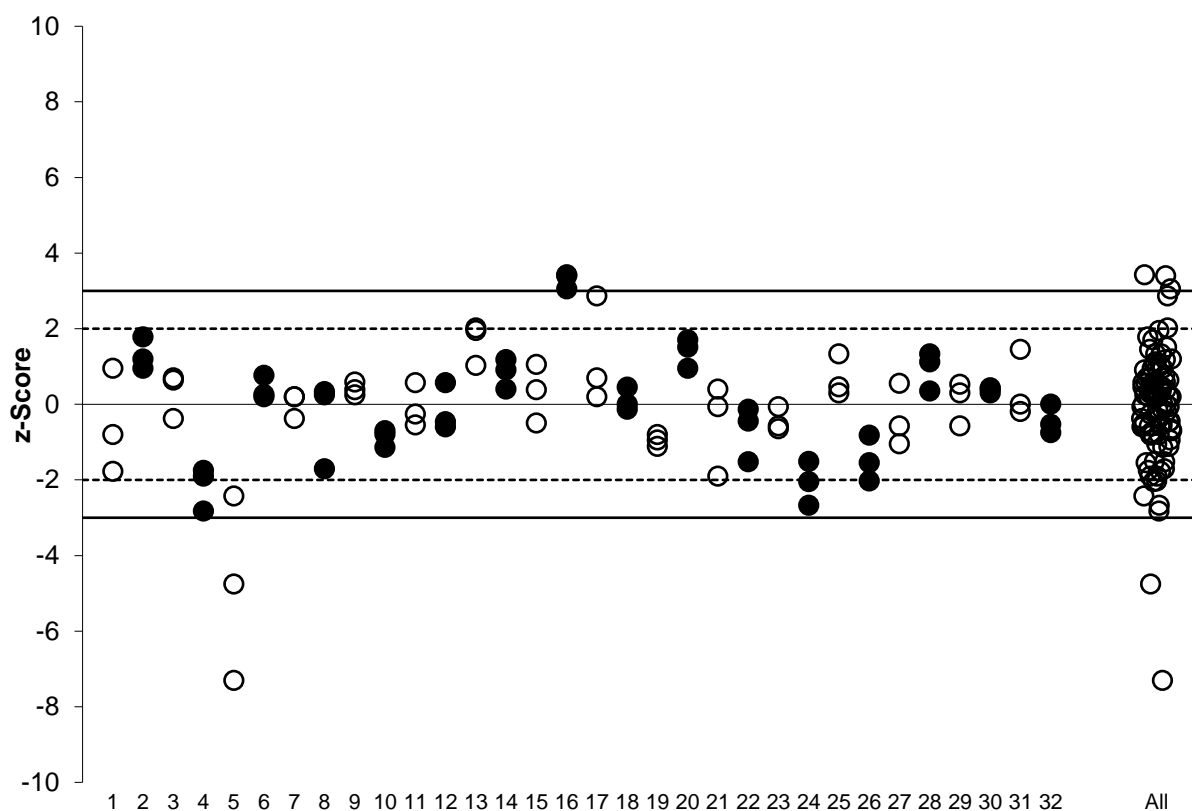


Figure 5  $z$ -Score Dispersal by Laboratory

#### 6.4 $E_n$ -Score

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

Of 96 results for which  $E_n$ -scores were calculated, 86 (90%) returned a satisfactory  $E_n$ -score of  $|E_n| \leq 1.0$ , indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Twenty-seven participants: **1, 2, 3, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 23, 25, 26, 27, 28, 29, 30, 31** and **32** returned satisfactory  $E_n$ -scores for all three samples.

Five participants returned at least one unsatisfactory  $E_n$ -score. Laboratories **4** and **24** returned unsatisfactory  $E_n$ -scores for all three samples.

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

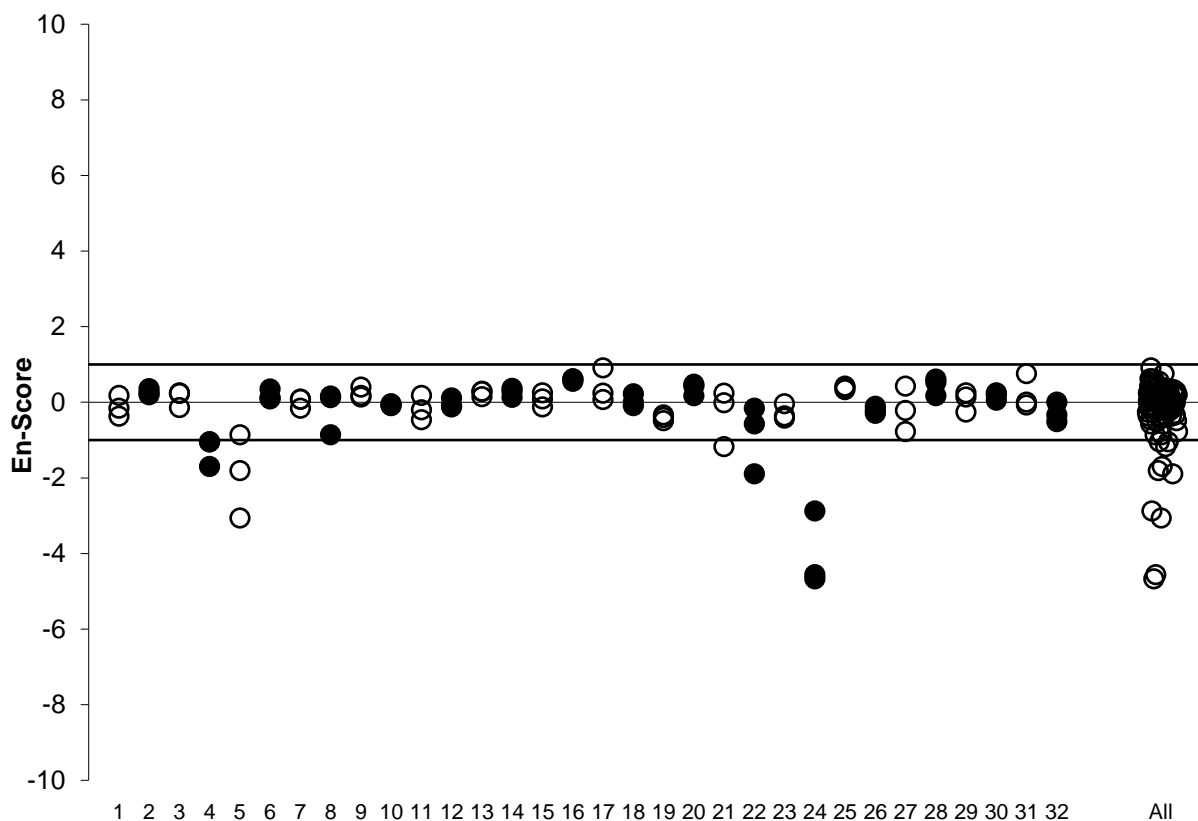


Figure 6  $E_n$ -score Dispersal by Laboratory

### 6.5 Identification of Cutting Agent

The test samples were prepared using seizures of cocaine hydrochloride approximately 84% base (m/m), supplied by the Australian Federal Police. The study coordinator added caffeine and levamisole to Sample S1, procaine to Sample S2, and paracetamol to Sample S3.

Twenty-nine participants (91%) reported on the identity of the cutting agent(s) in at least one sample (Table 8).

Laboratories **1, 2, 3, 6, 7, 8, 9, 11, 12, 13, 14, 16, 17, 18, 19, 20, 21, 23, 24, 25, 26, 27, 29, 31** and **32** correctly identified all cutting agents in the samples.

Sample S1 was spiked with caffeine and levamisole, with caffeine being added in a significantly greater proportion. In addition to the laboratories listed above, Laboratory **28** correctly identified both caffeine and levamisole, while Laboratories **4, 15** and **22** reported the presence of caffeine only in this sample.

For Sample S2, all participants reporting for cutting agents correctly identified procaine as the cutting agent. Laboratory **14** also reported cinnamoylcocaine, an alkaloidal impurity found in cocaine.<sup>10</sup>

For Sample S3, all participants reporting for cutting agents correctly identified paracetamol as the cutting agent, except Laboratory **28** who did not report any cutting agents in this sample. Laboratory **14** also reported benzoylecgonine, an alkaloidal impurity found in cocaine.<sup>10</sup>

## 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.

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

Laboratory **21** reported using two different methodologies, because of the interference from the paracetamol cutting agent in Sample S3.

Table 10 Summary of Participants' Analyses

		Lab. Code
Accreditation	Yes to ISO/IEC 17025	1, 2, 4, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 17, 19, 20, 21, 22, 23, 25, 26, 27, 28, 29, 30, 31, 32
	Not Accredited / Not Reported	3, 5, 16, 18, 24
Average Sample Mass Used per Analysis (mg)	5 – 20	3, 5, 7, 9, 10, 13, 16, 17, 18, 23, 25, 26, 28, 31
	21 – 30	6, 8, 12, 14, 15, 19, 20, 22, 27, 29, 30
	31 – 50	1, 2, 4, 11, 24
	51 – 100	32
	101 – 150	21
Conversion to Base?	Yes	2, 6, 7, 9, 11, 13, 15, 17, 20, 21, 22, 23, 25, 26, 27, 28, 30, 31
	No	3, 4, 5, 8, 12, 14, 16, 18, 19, 29, 32
	Not Reported	1, 10, 24
Instrument Used for Quantification	HPLC-DAD	3, 10, 11, 13, 14, 16, 18, 19, 20, 21 (S1, S2), 25, 28
	HPLC-UV/Vis	12, 17
	UPLC-DAD	7, 8, 24, 27, 29
	UPLC-UV/Vis	5
	UPLC-MS/MS	1
	GC-FID	2, 4, 6, 9, 15, 21 (S3), 22, 30, 31, 32
	GC-MS	26
	QNMR	23
Solvent	Acetonitrile	10, 32
	Acetonitrile/Water(/Acid)	3, 5, 7, 13, 14, 16, 17, 25
	Acetonitrile/Methanol(/Water)	1, 27
	Chloroform	21 (S3), 26
	Chloroform/Methanol	15, 22
	Ethanol	4, 6, 24, 31
	Methanol	2, 8, 9, 11, 12, 18, 19, 20, 29, 30
	Other	21 (S1, S2), 23, 28

		Lab. Code
Sources of Calibration Standard	NMI Australia	4, 7, 16, 20, 23, 24, 27, 32
	Lipomed	2, 3, 5, 6, 8, 9, 10, 11, 19, 26, 29
	Sigma Aldrich / Merck	13, 17, 28
	Toronto Research Chemicals	15, 22, 25
	Other	1, 12, 14, 21, 30, 31
	Not Reported	18

Plots of the z-score versus various methodology parameters are presented in Figures 7 to 11. No significant trend was observed.

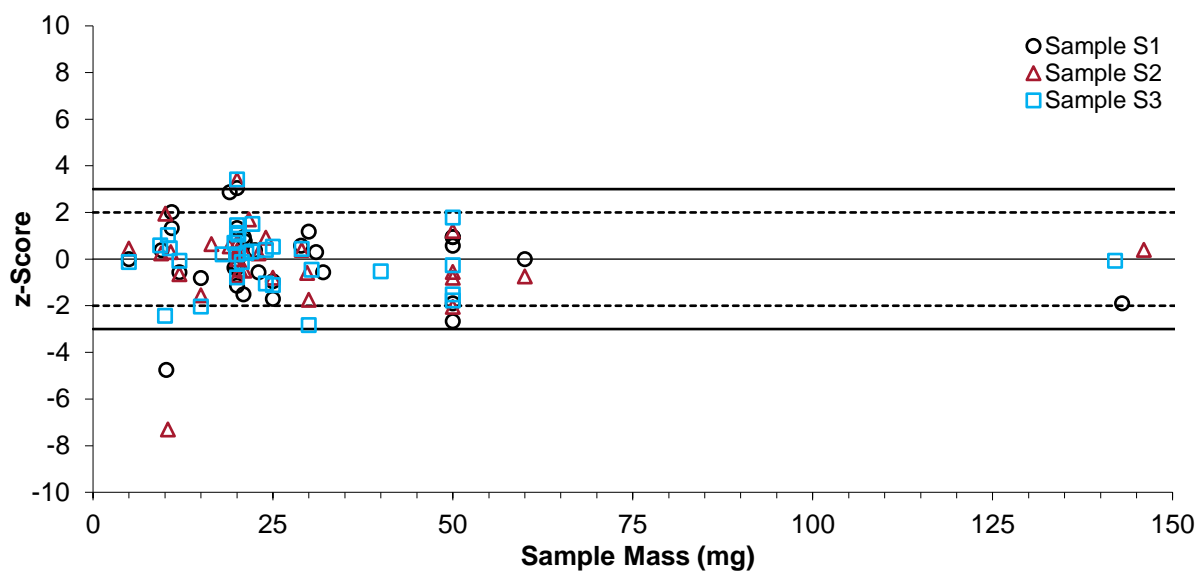


Figure 7 z-Score vs Sample Mass Used for Analysis

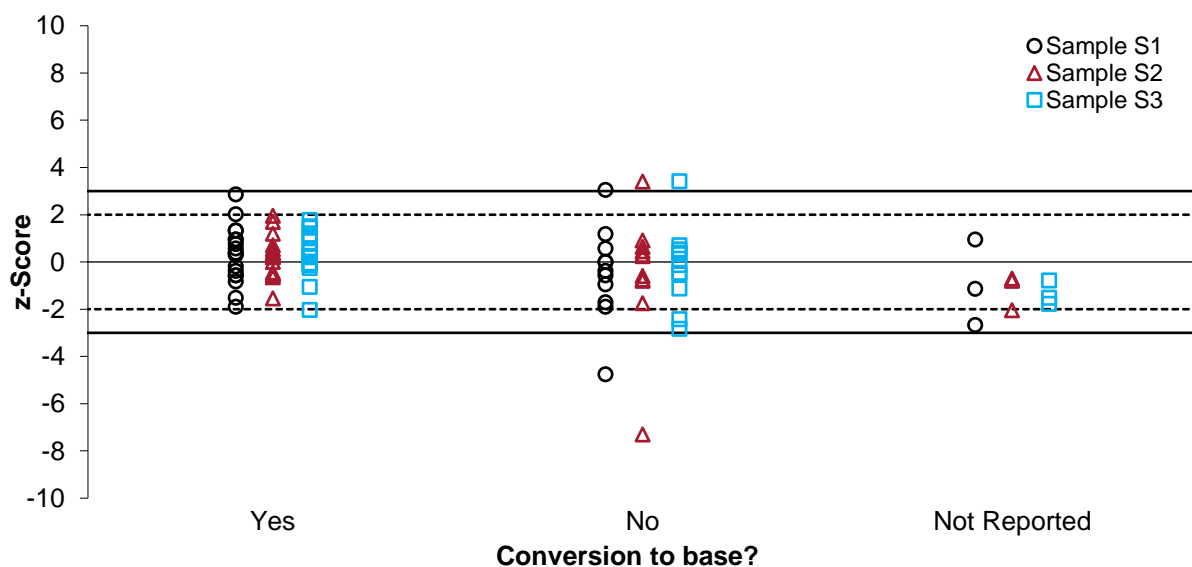


Figure 8 z-Score vs Sample Processing

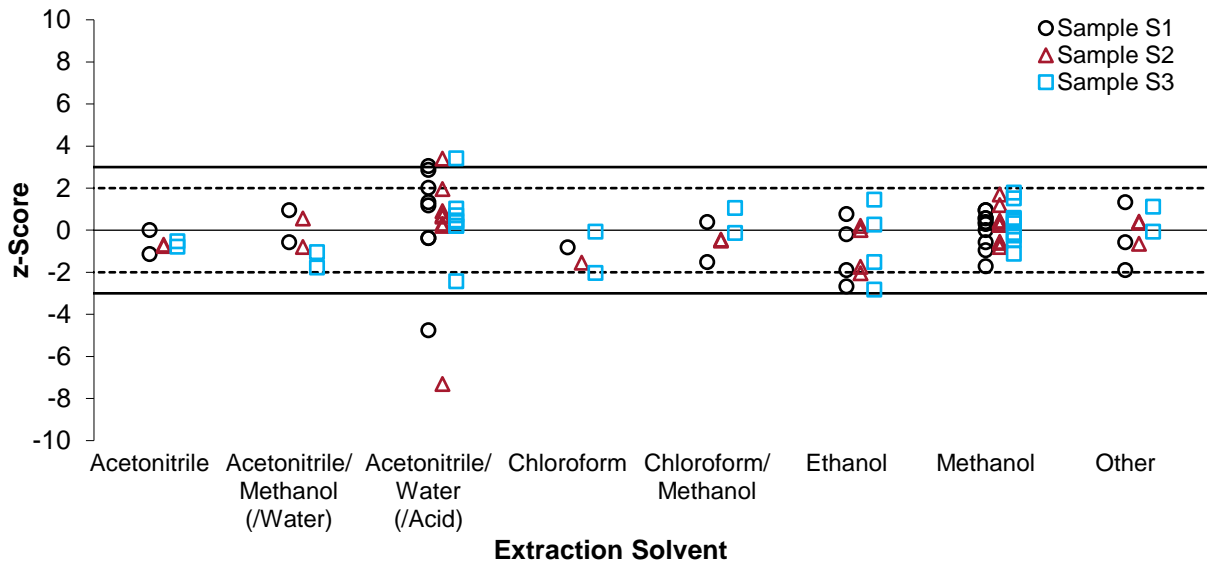


Figure 9 z-Score vs Extraction Solvent

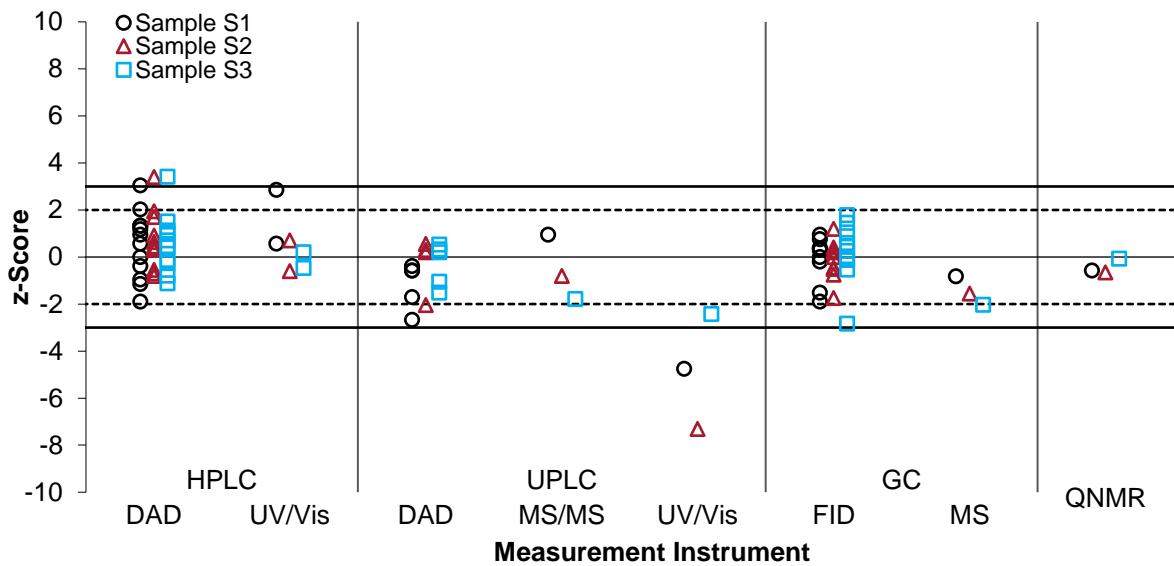


Figure 10 z-Score vs Measurement Instrument

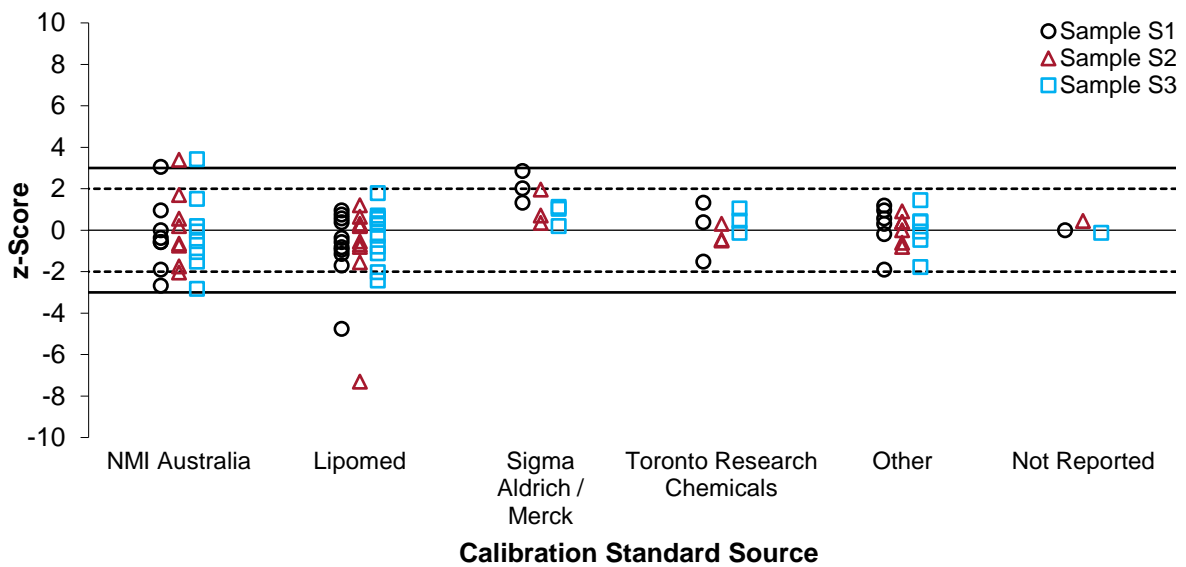


Figure 11 z-Score vs Source of Calibration Standard

## 6.7 Comparison of Results and Date of Analysis

As there were significant delays with sample delivery to some participants, caused by customs issues, the samples were analysed by participants over the course of approximately 6 months. No trend was found between when the samples were analysed and the results obtained (Figure 12).

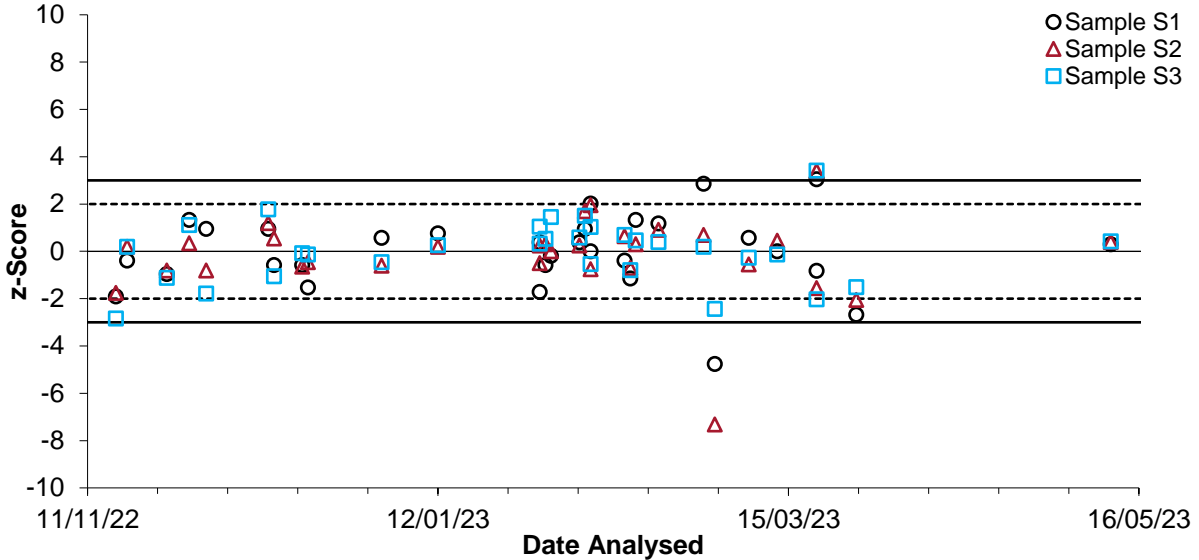


Figure 12 z-Score vs Sample Analysis Date

## 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 satisfactory performance, presented as a percentage of the total number of scores, obtained by participants from 2013 to 2022 (last 10 studies) are presented in Figure 13. The average proportion of satisfactory z-scores and  $E_n$ -scores over this period is 82% and 84% respectively. While each PT study has a different group of participants, taken as a group, the performance over this period has been improving.

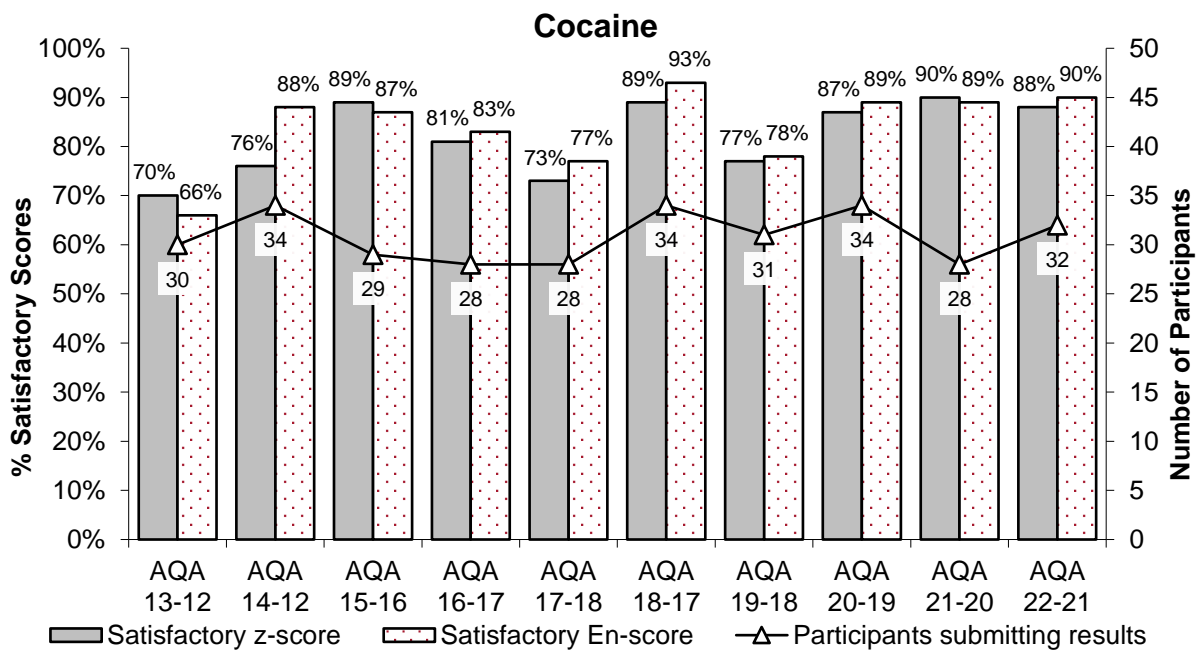


Figure 13 Summary of Participants' Performance in Cocaine PT Studies

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, achieving 83% and 82% satisfactory  $z$ -scores over this period for Australian laboratories and international laboratories respectively.

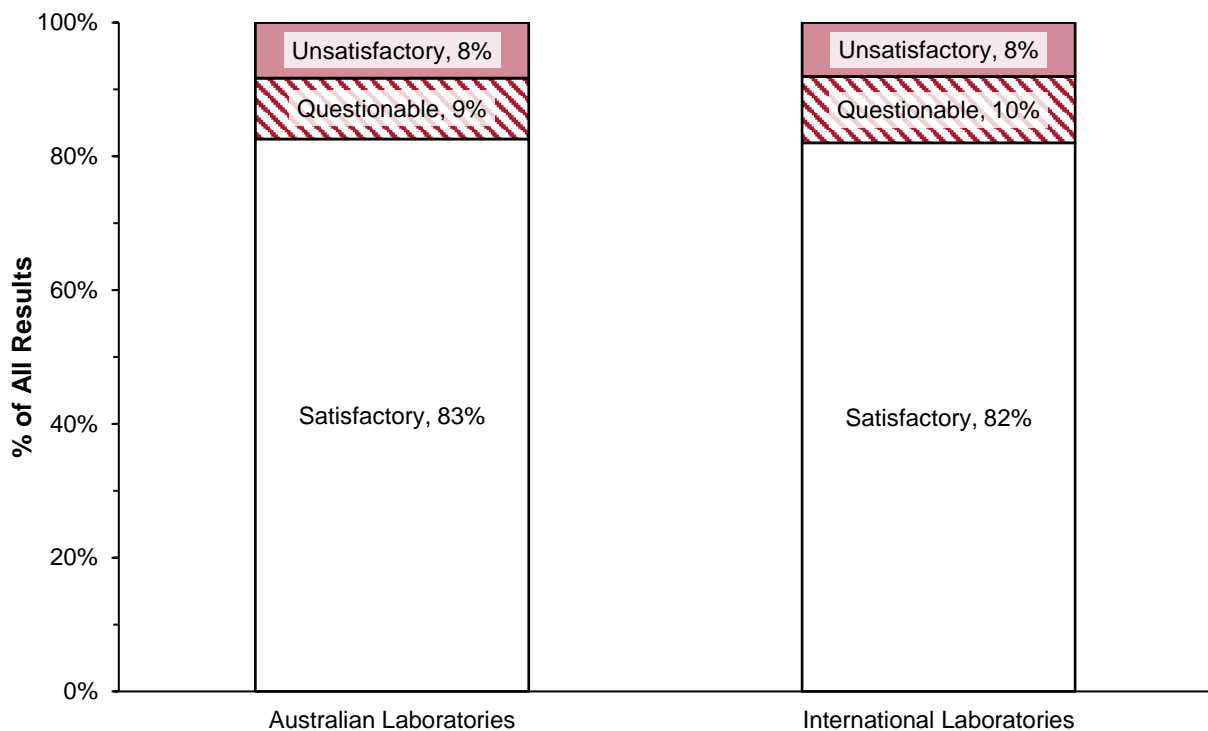


Figure 14 Comparison of Australian and International Laboratories in NMI Cocaine PT Studies

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 and for Australian and international laboratories respectively. One Australian and three international laboratories have achieved satisfactory  $z$ -scores across all samples in all NMI Cocaine PT studies participated in over this period.



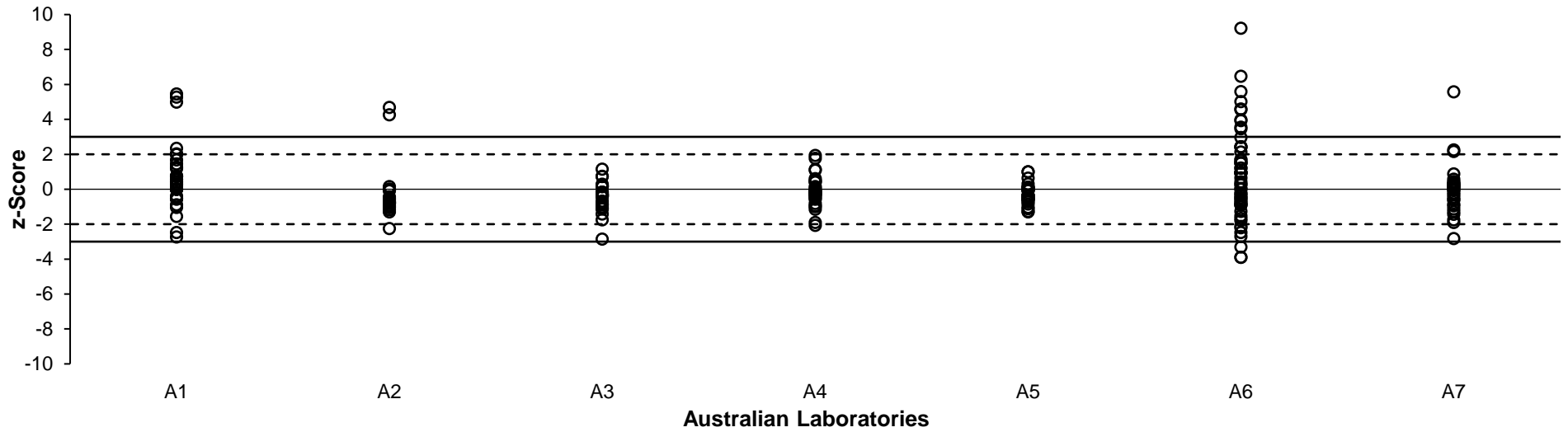


Figure 15 Summary of Australian Participants' z-Scores in NMI Cocaine PT Studies

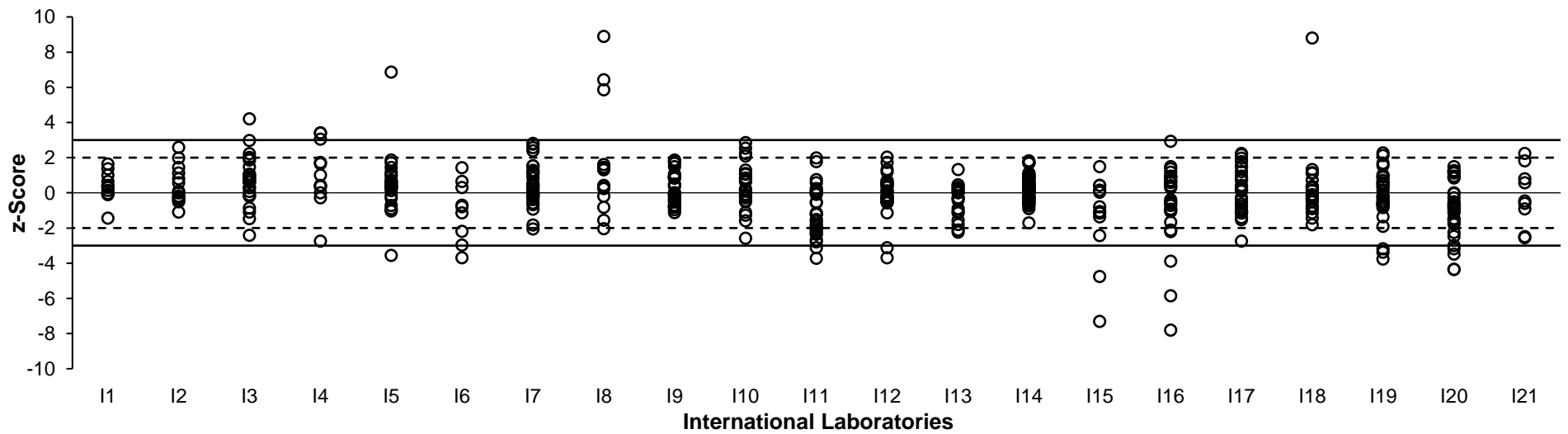


Figure 16 Summary of International Participants' z-Scores in NMI Cocaine PT Studies

## 7 REFERENCES

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

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- [7] ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*.
- [8] Eurachem/CITAC Guide CG 4, QUAM:2012.P1, *Quantifying Uncertainty in Analytical Measurement*, 3<sup>rd</sup> Edition, viewed May 2023, <[http://eurachem.org/images/stories/guides/pdf/quam2012\\_P1.pdf](http://eurachem.org/images/stories/guides/pdf/quam2012_P1.pdf)>
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## 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.<sup>5</sup> The associated uncertainties were estimated as according to Equation 4.

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

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 S3 is set out below in Table 11.

Table 11 Uncertainty of Sample S3 Robust Average

No. results ( $p$ )	32
Robust average	50.7% base (m/m)
$S_{rob\ average}$	1.8% base (m/m)
$u_{rob\ average}$	0.4% base (m/m)
$k$	2
$U_{rob\ average}$	0.8% base (m/m)

Therefore, the robust average of Sample S3 is  $50.7 \pm 0.8\%$  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
$18 \pm 2.7$	$17.5 \pm 0.3$	3% as PCV, or: $0.03 \times 17.5 =$ 0.525% base (m/m)	$z\text{-Score} = \frac{18-17.5}{0.525}$ $= 0.95$	$E_n\text{-Score} = \frac{18-17.5}{\sqrt{2.7^2+0.3^2}}$ $= 0.18$

## **APPENDIX 2 ACRONYMS AND ABBREVIATIONS**

ANAB	ANSI (American National Standards Institute) National Accreditation Board
ASCLD/LAB	American Society of Crime Laboratory Directors/Laboratory Accreditation Board
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
DAD	Diode Array Detection
EA	European Accreditation
ENFSI	European Network of Forensic Science Institutes
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
Md	Median
Max	Maximum value in a set of results
Min	Minimum value in a set of results
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
NR	Not Reported
PCV	Performance Coefficient of Variation
PDA	Photodiode Array
PT	Proficiency Testing
QNMR	Quantitative Nuclear Magnetic Resonance
RA	Robust Average
RM	Reference Material
SD	Standard Deviation
SI	International System of Units
SS	Spiked Samples
U	Expanded Uncertainty
UPLC	Ultra Performance Liquid Chromatography
UV/Vis	Ultraviolet/Visible detection

**END OF REPORT**