



Australian Government  
Department of Industry,  
Science and Resources

National  
Measurement  
Institute

# Proficiency Test Final Report AQA 24-20 Cocaine

June 2025

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## ACKNOWLEDGMENTS

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

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

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

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## SUMMARY

AQA 24-20 Cocaine commenced in September 2024. 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 Australia (NMIA) 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 participants' capabilities to measure cocaine in samples typical of a routine seizure.*

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

Of 90  $z$ -scores, 77 (86%) returned  $|z| \leq 2.0$ , indicating an acceptable performance.

Of 90  $E_n$ -scores, 75 (83%) returned  $|E_n| < 1.0$ , indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratories **5, 7, 10, 11, 12, 15, 16, 18, 19, 20, 22, 23, 25, 26, 29** and **30** returned acceptable  $z$ -scores and  $E_n$ -scores for all three samples.

- *Develop the practical application of measurement uncertainty.*

Of 90 numeric results, 84 (93%) were reported with an associated expanded measurement uncertainty. The magnitude of reported uncertainties was within 1.8% to 20% relative.

The most common coverage factor used by participants was  $k = 2$ .

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

Samples S1 and S2 were cut with niacinamide, and Sample S3 was cut with sucrose.

Twenty-five participants (83%) reported on the identity of the cutting agent(s) in at least one sample.

Laboratories **1, 11** and **15** correctly identified all cutting agents in the samples.

Significantly more participants were able to correctly identify niacinamide in Samples S1 and S2 than sucrose in Sample S3.

- *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 NMIA and can be used for quality control and method validation purposes.

## **1 INTRODUCTION**

### **1.1 NMIA Proficiency Testing Program**

The National Measurement Institute Australia (NMIA) is responsible for Australia's national measurement infrastructure, providing a range of services including a chemical proficiency testing program.

Proficiency testing (PT) is the 'evaluation of participant performance against pre-established criteria by means of interlaboratory comparisons'.<sup>1</sup> NMIA PT studies target chemical testing in areas of high public significance such as trade, environment, law enforcement and food safety. NMIA offers studies in:

- pesticide residues in soil, water, fruit, vegetables and herbs;
- hydrocarbons, phenols and volatile organic compounds in soil and water;
- inorganic analytes in soil, water, filters, food and pharmaceuticals;
- per- and polyfluoroalkyl substances in soil, water, biosolid, biota and food;
- 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 participants' capabilities to measure cocaine in samples typical of a routine seizure;
- develop the practical application of measurement uncertainty;
- assess participants' ability to identify cutting agents commonly found in controlled drug preparation; and
- produce materials that can be used in method validation and as control samples.

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

### **1.3 Study Conduct**

The conduct of NMIA proficiency tests is described in the NMIA Study Protocol for Proficiency Testing.<sup>2</sup> The statistical methods used are described in the NMIA 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>

NMIA is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043:2023 as a provider of proficiency testing schemes.<sup>1</sup> This controlled drug PT study is within the scope of NMIA's accreditation.



## **2 STUDY INFORMATION**

### **2.1 Study Timetable**

The timetable of the study was:

Invitations sent	16/09/2024
Samples sent	25/11/2024
Results due	7/04/2025
Interim Report	27/05/2025
Preliminary Report	28/05/2025

There was a substantial delivery delay to an international participant due to permit issues. The release of the Interim Report was delayed to allow this participant to receive and analyse the samples.

### **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 2024. The starting material was cocaine hydrochloride supplied by the Australian Federal Police.

Niacinamide (nicotinamide) and sucrose purchased from Sigma Aldrich were used as cutting agents. Samples S1 and S2 were blind duplicates, cut with niacinamide, and Sample S3 was cut with sucrose.

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.

Samples S1 and S2 were prepared to contain approximately 39.1% cocaine base (m/m).

Sample S3 was prepared to contain approximately 73.7% 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 November 2024. 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, and to use at least 20 mg for analysis. Samples should be covered when not being used.
- For each sample report % m/m cocaine as base. Report this figure as if reporting to a client.
- For each result report an evaluation 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](mailto:jenny.xu@measurement.gov.au).
- Give brief details in the results sheet of your:
  - Basis of uncertainty evaluation (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 3 February 2025.

The results due date was later extended to 7 April 2025 due to general delays with sample delivery to multiple international participants.

## 2.7 Interim Report and Preliminary Report

An Interim Report was emailed to all participants on 27 May 2025. The delivery of the samples to one international participant was significantly delayed due to permit issues, and so to allow this participant to report results, the release of the Interim Report was delayed.

A Preliminary Report was emailed to all participants on 28 May 2025. 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	Comments
1	Methanol	nil	7	HPLC	DAD	Waters HPLC 1260 Phenomenex C8 Luna 3u Narrow Bore 100 mm	Wavelength: 214 nm
2	MeOH	N/A	7	HPLC	DAD	ZORBAX XDB-C18 (4.6x150mm,5µm)	
3			5	HPLC			
4	Water/ACN	N/A	5	HPLC	UV	Kinetex 5u C18	Wavelength: 233nm
5	Acetonitrile:Water (75:25)	Dibutylphthalate	3	UPLC	PDA	Acquity UPLC BEH C18 1.7µm (2.1x100mm)	Wavelength: 275nm
6	Acetonitrile	N.A	4	HPLC	UV/Vis	PROTECOL C8 H 5UM 150X4.6MM	
7	Methanol	Tetracosane	4	GC	FID	HP5	
8	Methanol	Diazepam	6	GC	FID	J&W 128-5512	
9	methanol		4	HPLC	DAD	ECLIPSE XDB-25	Wavelength: 230nm
10	acetonitrile/water (80/20)	none	3	HPLC	DAD	C8	Wavelength: 230 nm Eluent: acetonitrile/phosphate buffer (pH4)/water (277/413/310)
11	Ethanol	triphenylacetylphenone (TPAP)	3	GC	FID	HP-1MS	A small amount of dichloromethane was used to dissolve the TPAP.

Lab. Code	Extraction Solvent	Internal Standard	Calib. Points	Technique	Detector	Column	Comments
12	H2O/Acetonitrile (60/40)	/	5	UPLC	DAD	C8	
13	Ethanol	Tribenzylamine	6	GC	FID	HP5	
14	Methanol	N/A	7	HPLC	UV/Vis	Hypersil Gold 150x3mm	Test method for cocaine quantification
15	Acetonitrile	Strychnine	6	GC	FID	HP1	
16	HPLC Methanol	Vanillin	1	UPLC	DAD	Agilent LiChrospher 60 RP-select B	Wavelength: 230 nm
17	Ethanol	Propylparaben	7	UPLC	DAD	BEH Shield RP18	
18	1:1 chloroform:methanol + ethyl acetate	Isopropylcocaine	5	GC	FID	HP5	
19	Ethanol	TBA	4	GC	FID	HP1	
20	72% ultra pure water + 28% acetonitrile		5	HPLC	UV/Vis	Kromasil C8	Internal method HPLC-UV
21	ACN/MeOH/H2O	Analog of cocaine	7	UPLC	MS/MS	C-18 Column	
22	water/acetonitrile/2.5M sulphuric acid 90:10:1	None	3	HPLC	Diode Array	Shimpack XR-ODS	Wavelength: 230nm
23	Methanol	none	5	HPLC	DAD	Kinetex 2.6 µ XB-C18	
24	Chloroform	2,2,2-triphenylacetophenone	1	GC	FID	HP1	
25	Methanol	Methadone	5	GC	FID	RXI-5MS	
26	Methanol	-	3	GC	FID	CP-sil5CB	External calibration
27	acetonitrile/water	none	5	HPLC	DAD	Kromasil	
28	Acetonitrile/Methanol	Pholcodine 1mg/ml	3	UPLC	PAD	ACQUITY C-18	
29	acetonitrile/water (80/20)	external standard	3	HPLC	DAD	C8	
30	HPLC Methanol	Vanillin	1	UPLC	DAD	Agilent LiChrospher 60 RP-select B	Wavelength: 230 nm

### 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	NMI	99.8
2	Lipomed	99.9
3		
4	British Pharmacopoeia	99.30
5	NMIA	99.8+/- 1.0
6	Lipomed (cocaine HCl)	99.004
7		
8	Lipomed	99.3
9		
10	Lipomed	99.004
11	NMI	99.8
12	Lipomed (EUROMEDEX)	99
13	Lipomed	99.199
14	Sigma-Aldrich	99.9
15	NMI	99.6
16	Lipomed	99.199 ± 0.006
17	NMIA	99.8
18	NMIA D826d	99.6
19	Fagron	99.7
20	LIPOMED	99.199 ± 0.006
21	Unikem	100
22	Cayman	>98
23	Lipomed	>98.6
24	Macfarlan Smith Ltd	100.4
25	LGC	100.7
26	Duchefa	>99
27	Lipomed	99.0004
28	NMI	99.6
29	LIPOMED	85.5
30	Lipomed	99.199 ± 0.006

### 3.3 Reported Basis of Participants' Measurement Uncertainty Evaluation

Participants were requested to provide information about their basis of measurement uncertainty (MU). Responses are presented in Tables 3 to 4. Some responses may be modified so that the participant cannot be identified.

Table 3 Reported Basis of Uncertainty Evaluation

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
1	Top Down – precision and estimates of the method and laboratory bias k = 2	Control samples – RM / PT Sample Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Matrix effects Standard purity	SWGDRUG Supplemental Document SD-4: Measurement Uncertainty for Purity Determinations
2	validation data Coverage factor not reported	Control samples – CRM Duplicate analysis	Instrument calibration Masses and volumes Standard purity	Eurachem/CITAC Guide
3	Coverage factor not reported			
4	Top Down – precision and estimates of the method and laboratory bias k = 2	Control samples – RM / PT Sample Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Matrix effects Standard purity	Eurachem/CITAC Guide
5	Top Down – precision and estimates of the method and laboratory bias k = 2	Duplicate analysis		Eurachem/CITAC Guide
6	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2	Control samples – RM / PT Sample	Laboratory bias from PT studies	
7	k = 1	Control samples – RM / PT Sample Duplicate analysis		EA-04/16 EA guidelines on the expression of uncertainty in quantitative testing
8	Estimating Measurement Uncertainty by black box with pairs of values k = 2	Standard deviation from PT studies only		ISO/GUM ENAC G 09 or ISO 21748
9	Top Down – precision and estimates of the method and laboratory bias Coverage factor not reported	Control samples – RM / PT Sample	Recoveries of SS	
10	Top Down – precision and estimates of the method and laboratory bias k = 2	Control samples	Laboratory bias from PT studies	NF V03-110

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
11	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
12	Top Down – precision and estimates of the method and laboratory bias k = 2	Control samples – RM / PT Sample	Laboratory bias from PT studies	ISO/GUM
13	Top Down – precision and estimates of the method and laboratory bias Coverage factor not reported	Control samples – RM / PT Sample	Standard purity	
14	Top Down – precision and estimates of the method and laboratory bias Coverage factor not reported	Duplicate analysis	Instrument calibration Standard purity	UKAS
15	Top Down – precision and estimates of the method and laboratory bias Coverage factor not reported	Control samples – CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes	ISO/GUM
16	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
17	Coverage factor not reported			
18	Top Down – precision and estimates of the method and laboratory bias k = 2	Control samples – RM / PT Sample Duplicate analysis	Standard purity	NMI Uncertainty Course
19	Top Down – precision and estimates of the method and laboratory bias Coverage factor not reported	Control samples – RM / PT Sample Duplicate analysis		Internal document based on Eurachem/CITAC, ISO/GUM
20	Top Down – precision and estimates of the method and laboratory bias k = 2	Control samples – CRM Duplicate analysis	Laboratory bias from PT studies	
21	Top Down – precision and estimates of the method and laboratory bias k = 0.95	Control samples – RM / PT Sample		ILAC-G17 and EA 4/16 (2003)

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
22	Standard deviation of replicate analyses multiplied by 2 or 3 k = 3	Control samples – CRM Duplicate analysis	Instrument calibration Standard purity	ISO/GUM
23	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
24	validation k = 2			
25	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2	Duplicate analysis	Masses and volumes	ISO/GUM
26	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
27	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples – RM / PT Sample		
28	Top Down – precision and estimates of the method and laboratory bias Coverage factor not reported	Control samples – CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
29	Top Down – reproducibility (standard deviation) from PT studies used directly Coverage factor not reported	Control samples – RM / PT Sample Duplicate analysis	Instrument calibration Laboratory bias from PT studies	Eurachem/CITAC Guide
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

Table 4 Additional Information for Measurement Uncertainty

Lab. Code	Participants' Comments
14	MoU for this method is yet to be determined as it is still in the development stage.
22	MuM determined from multiple injections of reference material. $3 \times (\text{Std Dev}/\text{mean}) \times 100$ .



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

Table 5 Participant Comments

Lab. Code	Participants' Comments	Study Coordinator's Response
10	Is it possible to put a sample around 5% and another around 80% or more? 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.	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 39.4 % base (m/m) to 73.7 % base (m/m).  We request participants to report the cocaine as base to ensure that results from all participants are consistent.
14	No diluents or adulterants were detected as we primarily use GCMS for that analysis and it was not used to screen these samples.	
18	It would be good to increase the amount of sample provided. The lower purity samples require more sample to be weighed out for response to fall within the calibration range and repeat analysis is difficult if a mistake occurred during analysis. Also, some laboratories may analyse the samples using two methods to assess performance of both methods.	Most participants reported using less than 50 mg for each analysis, so the 150 mg provided to participants allows for repeat analysis.  Participants can also request additional sample sets at enrolment if they require a higher mass for analysis, or if they wish to analyse the samples using two different methods (whether for internal investigation, or to submit an additional set of results for assessment in the PT study).  For security and accountability reasons, NMI PT studies are conducted using the minimum practical amount of controlled substance.

## 4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

### 4.1 Results Summary

Participant results are listed in Tables 6 to 8 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.

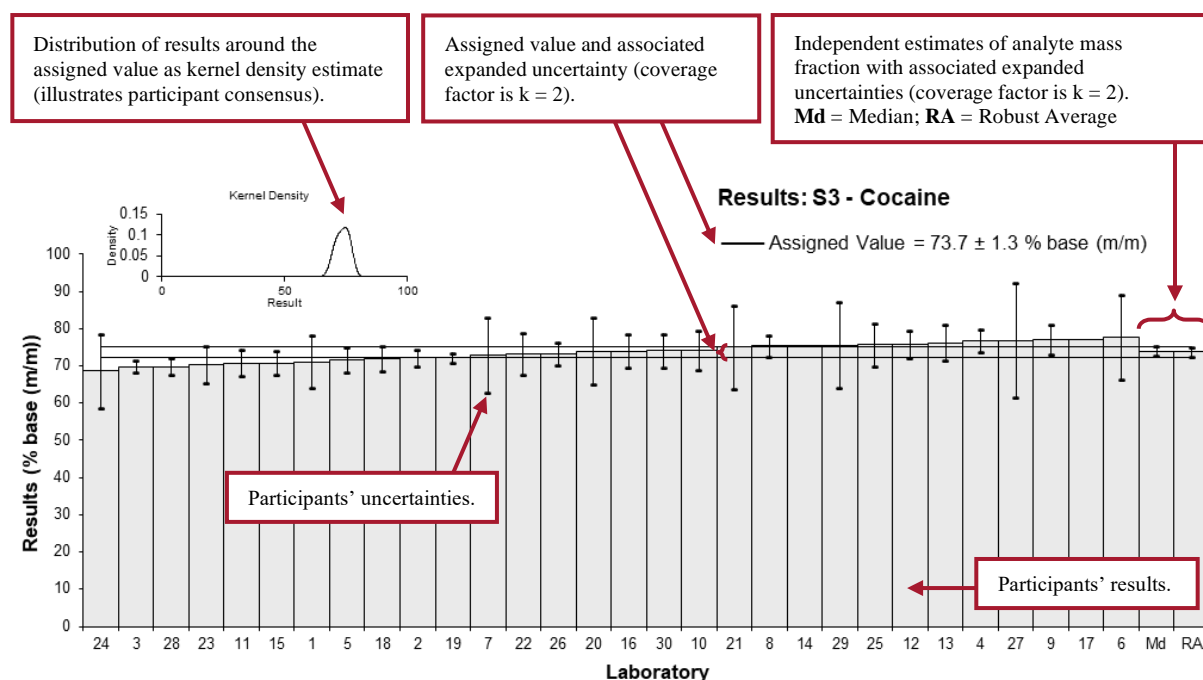


Figure 1 Guide to Presentation of Results

### 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 evaluated 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

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.

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

#### 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 acceptable;
- $2.0 < |z| < 3.0$  is questionable; and
- $|z| \geq 3.0$  is unacceptable.

#### 4.7 E<sub>n</sub>-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:

- $|E_n| < 1.0$  is acceptable; and
- $|E_n| \geq 1.0$  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.<sup>7</sup>

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

## 5 TABLES AND FIGURES

Table 6

### Sample Details

<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	36.6	3.7	-2.37	-0.75
2	40.6	1.9	1.02	0.60
3	38.6	0.9	-0.68	-0.74
4	41.75	4.59	1.99	0.51
5	38.1	1.7	-1.10	-0.72
6	35.4	5.3	-3.38	-0.75
7	39.3	6	-0.08	-0.02
8	41.4	1.7	1.69	1.11
9	39.6	2	0.17	0.10
10	40.43	2.83	0.87	0.36
11	37.6	1.88	-1.52	-0.91
12	39.3	2.0	-0.08	-0.05
13	42.5	2.7	2.62	1.12
14	44.27	NR	4.12	8.12
15	37.9	1.7	-1.27	-0.83
16	40.5	2.5	0.93	0.43
17	41.3	NR	1.61	3.17
18	38.4	1.8	-0.85	-0.53
19	39.2	0.7	-0.17	-0.22
20	39.04	4.68	-0.30	-0.08
21	35	5.3	-3.72	-0.82
22	38.2	2.95	-1.02	-0.40
23	37.46	2.62	-1.64	-0.72
24	37.5	5.5	-1.61	-0.34
25	39.73	2.96	0.28	0.11
26	40	3	0.51	0.20
27	40.88	8.18	1.25	0.18
28	35.52	1.60	-3.28	-2.27
29	39.9	6.0	0.42	0.08
30	39.7	2.4	0.25	0.12

### Statistics

<b>Assigned Value</b>	39.4	0.6
<b>Robust Average</b>	39.2	0.9
<b>Median</b>	39.3	0.8
<b>Mean</b>	39.2	
<b>N</b>	30	
<b>Max</b>	44.27	
<b>Min</b>	35	
<b>Robust SD</b>	2.0	
<b>Robust CV</b>	5.1%	

The assigned value has been calculated as the robust average of the combined results of duplicate pair Samples S1 and S2.

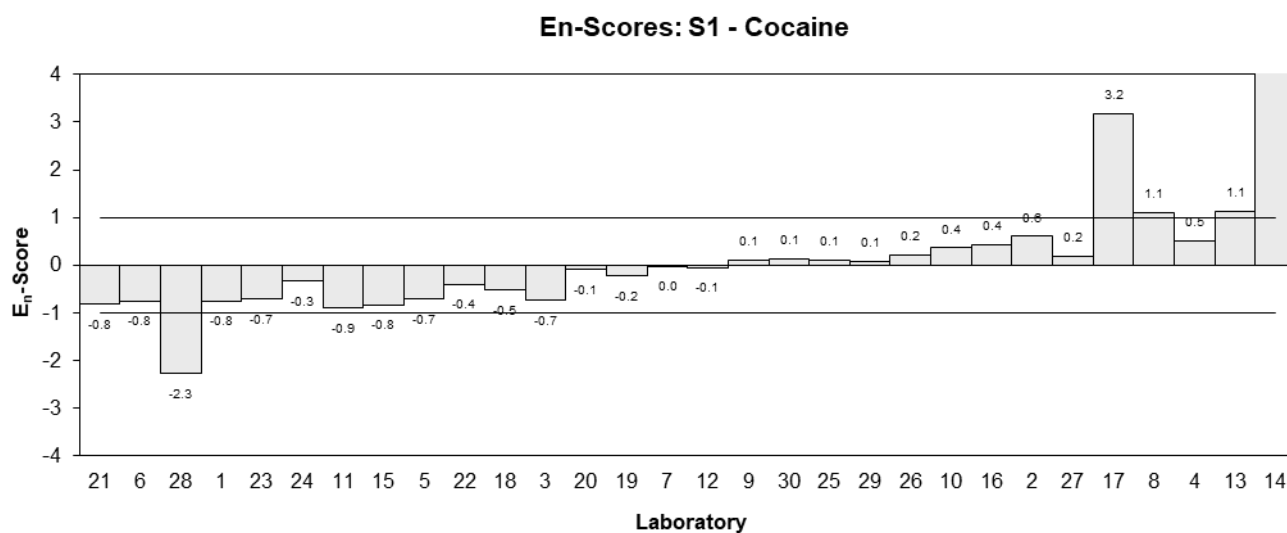
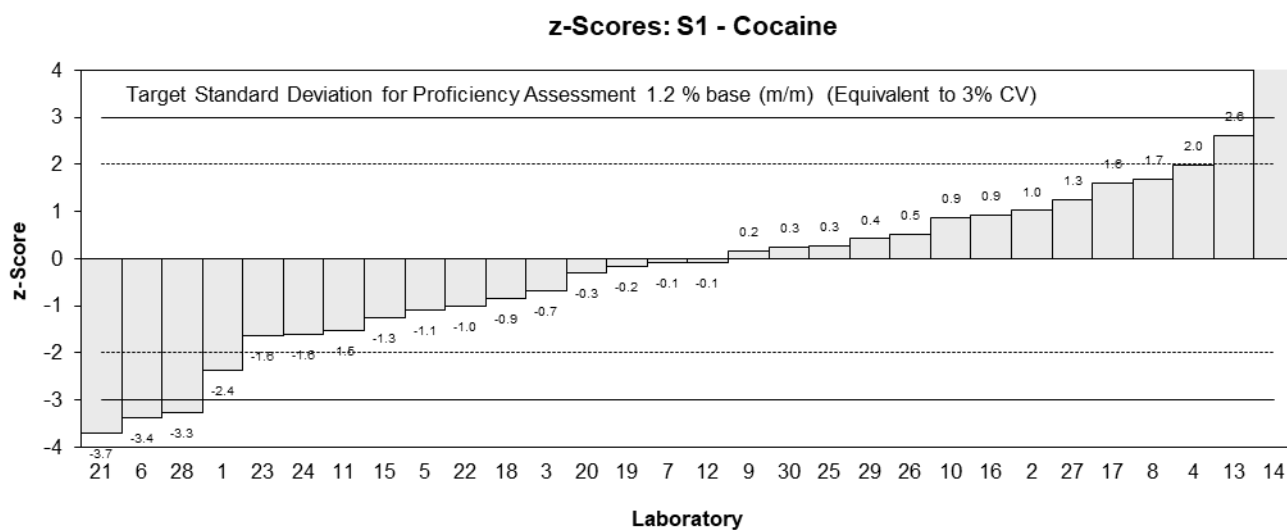
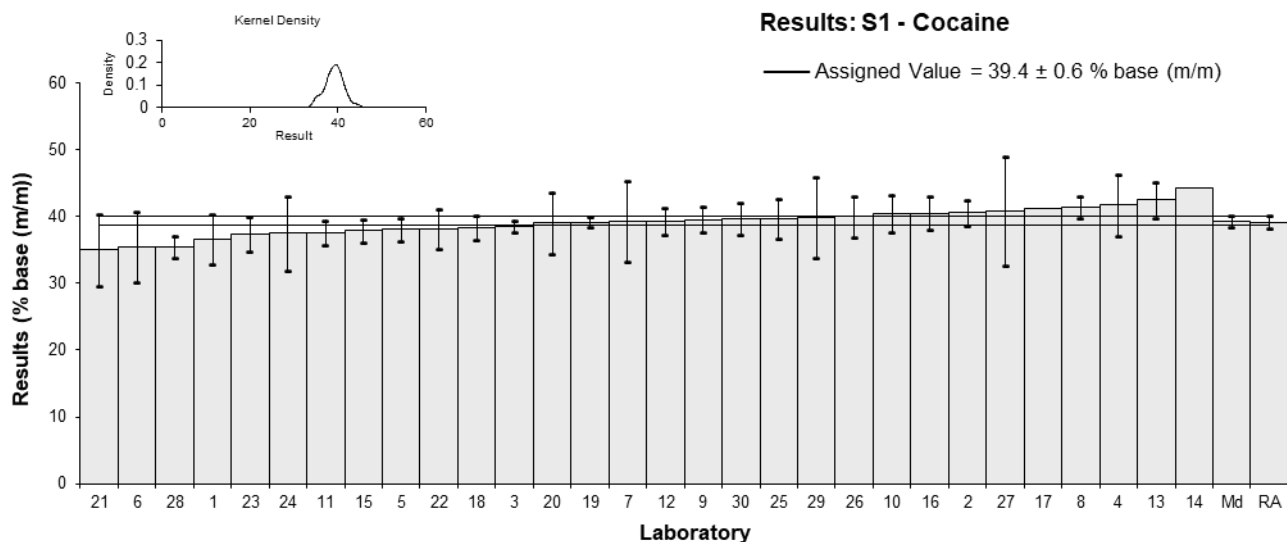


Figure 2

Table 7

**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	38.2	3.8	-1.02	-0.31
2	41.1	1.4	1.44	1.12
3	40.1	0.9	0.59	0.65
4	43.19	4.75	3.21	0.79
5	39.1	1.8	-0.25	-0.16
6	37.2	5.6	-1.86	-0.39
7	40.4	6	0.85	0.17
8	40.3	1.6	0.76	0.53
9	41.6	2	1.86	1.05
10	39.73	2.78	0.28	0.12
11	37.6	1.88	-1.52	-0.91
12	39.2	2.0	-0.17	-0.10
13	43.2	2.8	3.21	1.33
14	40.72	NR	1.12	2.20
15	38.4	1.7	-0.85	-0.55
16	39.9	2.4	0.42	0.20
17	41.8	NR	2.03	4.00
18	38.1	1.7	-1.10	-0.72
19	39.7	0.7	0.25	0.33
20	38.23	4.59	-0.99	-0.25
21	36	5.4	-2.88	-0.63
22	38.97	3	-0.36	-0.14
23	37.86	2.65	-1.30	-0.57
24	37.3	5.4	-1.78	-0.39
25	40.7	3.03	1.10	0.42
26	39.7	3	0.25	0.10
27	42.05	8.41	2.24	0.31
28	36.92	1.60	-2.10	-1.45
29	39.8	6.0	0.34	0.07
30	40.7	2.5	1.10	0.51

**Statistics**

<b>Assigned Value</b>	39.4	0.6
<b>Robust Average</b>	39.6	0.9
<b>Median</b>	39.7	0.9
<b>Mean</b>	39.6	
<b>N</b>	30	
<b>Max</b>	43.2	
<b>Min</b>	36	
<b>Robust SD</b>	1.9	
<b>Robust CV</b>	4.7%	

The assigned value has been calculated as the robust average of the combined results of duplicate pair Samples S1 and S2.

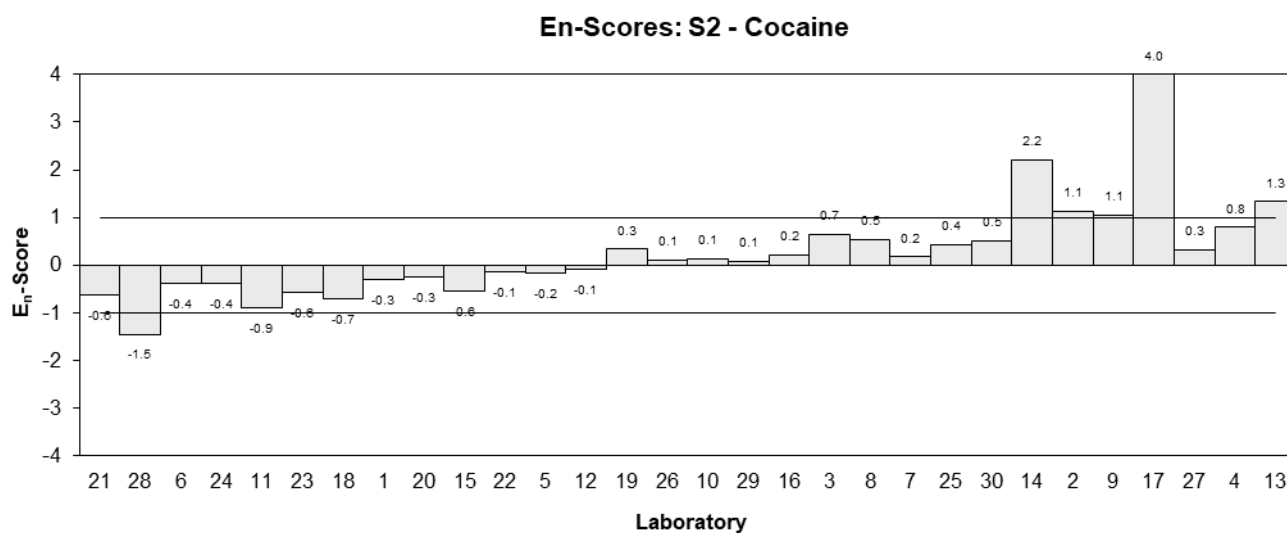
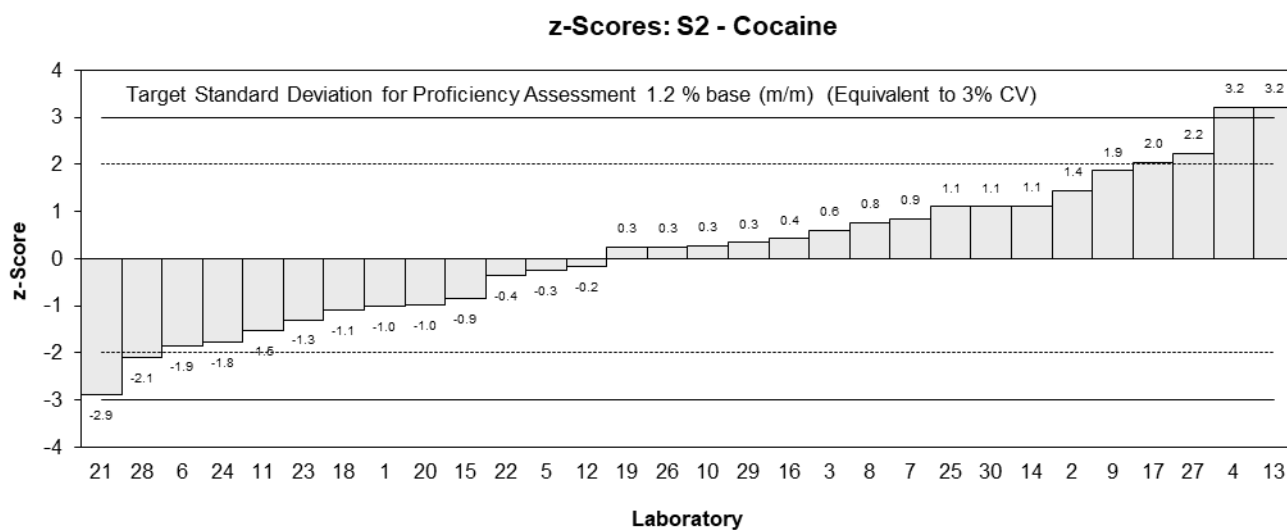
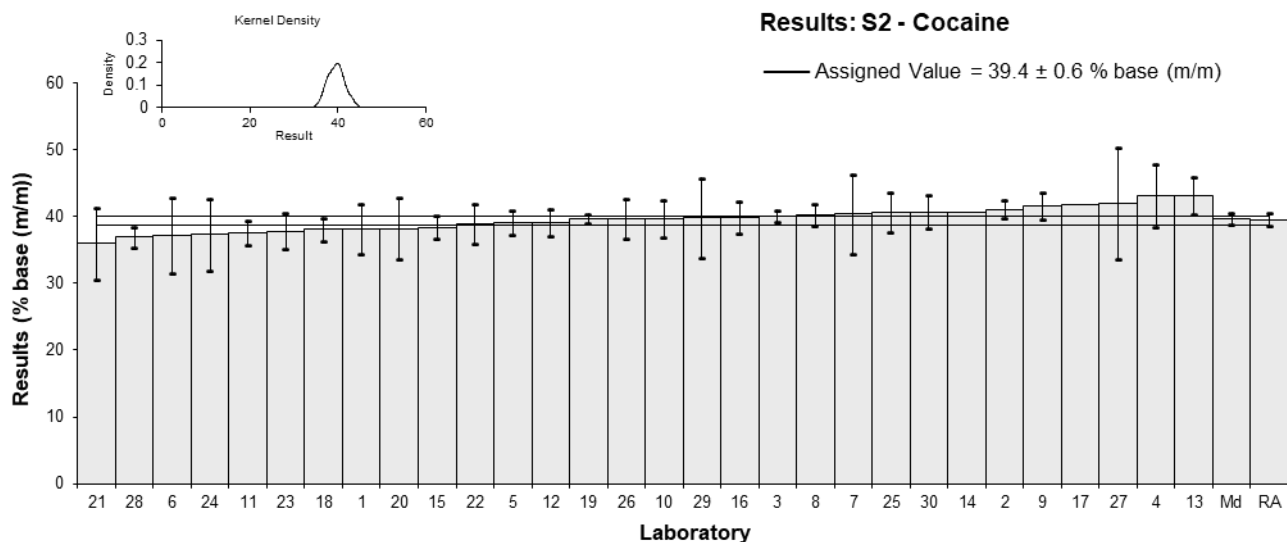


Figure 3

Table 8

**Sample Details**

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

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>Uncertainty</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	71.1	7.1	-1.18	-0.36
2	72.1	2.2	-0.72	-0.63
3	69.8	1.6	-1.76	-1.89
4	76.72	3.07	1.37	0.91
5	71.7	3.4	-0.90	-0.55
6	77.7	11.5	1.81	0.35
7	72.9	10	-0.36	-0.08
8	75.3	3.0	0.72	0.49
9	77	4	1.49	0.78
10	74.22	5.20	0.24	0.10
11	70.8	3.54	-1.31	-0.77
12	75.8	3.8	0.95	0.52
13	76.2	4.9	1.13	0.49
14	75.57	NR	0.85	1.44
15	70.8	3.1	-1.31	-0.86
16	74.0	4.5	0.14	0.06
17	77.2	NR	1.58	2.69
18	71.9	3.3	-0.81	-0.51
19	72.1	1.3	-0.72	-0.87
20	73.98	8.88	0.13	0.03
21	75	11.3	0.59	0.11
22	73.15	5.64	-0.25	-0.10
23	70.28	4.92	-1.55	-0.67
24	68.6	10.0	-2.31	-0.51
25	75.62	5.63	0.87	0.33
26	73.3	3	-0.18	-0.12
27	76.77	15.35	1.39	0.20
28	69.85	2.40	-1.74	-1.41
29	75.6	11.4	0.86	0.17
30	74.1	4.5	0.18	0.09

**Statistics**

<b>Assigned Value</b>	73.7	1.3
<b>Robust Average</b>	73.7	1.3
<b>Median</b>	74.0	1.3
<b>Mean</b>	73.6	
<b>N</b>	30	
<b>Max</b>	77.7	
<b>Min</b>	68.6	
<b>Robust SD</b>	2.8	
<b>Robust CV</b>	3.8%	



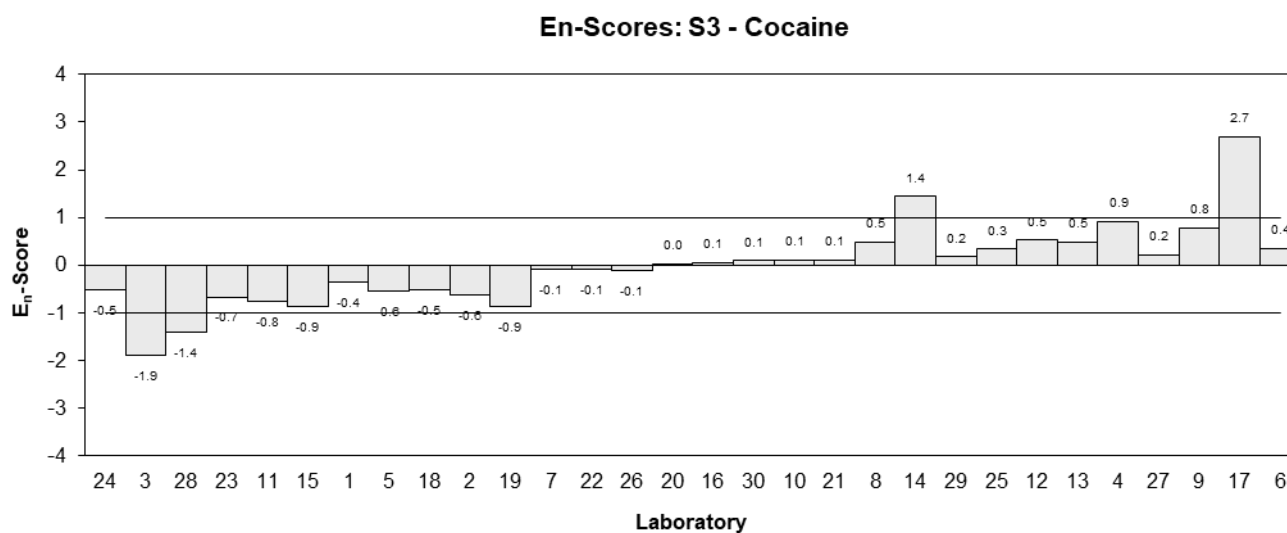
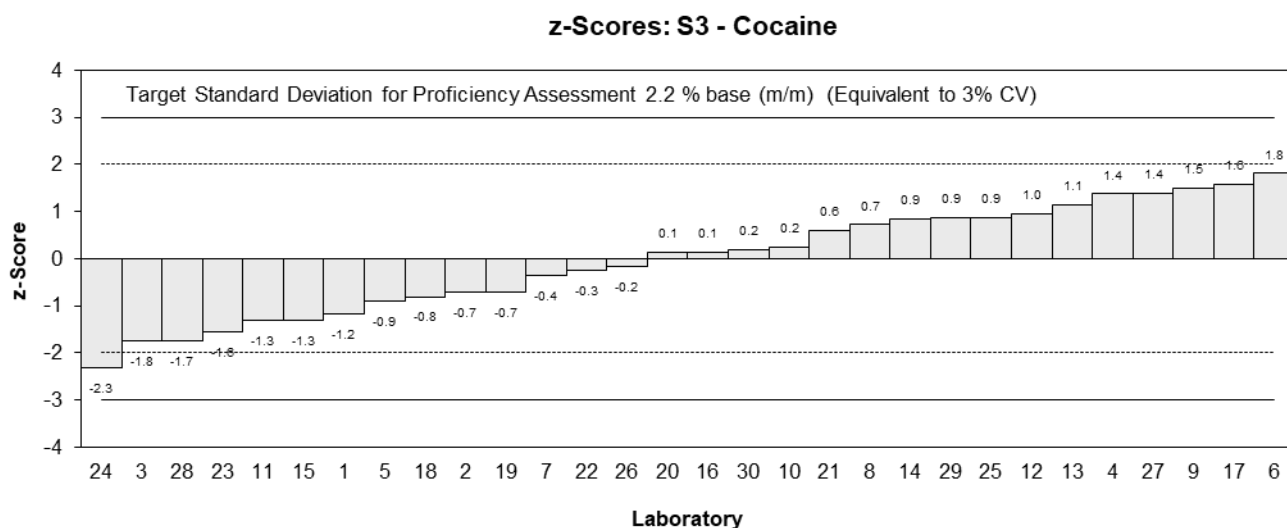
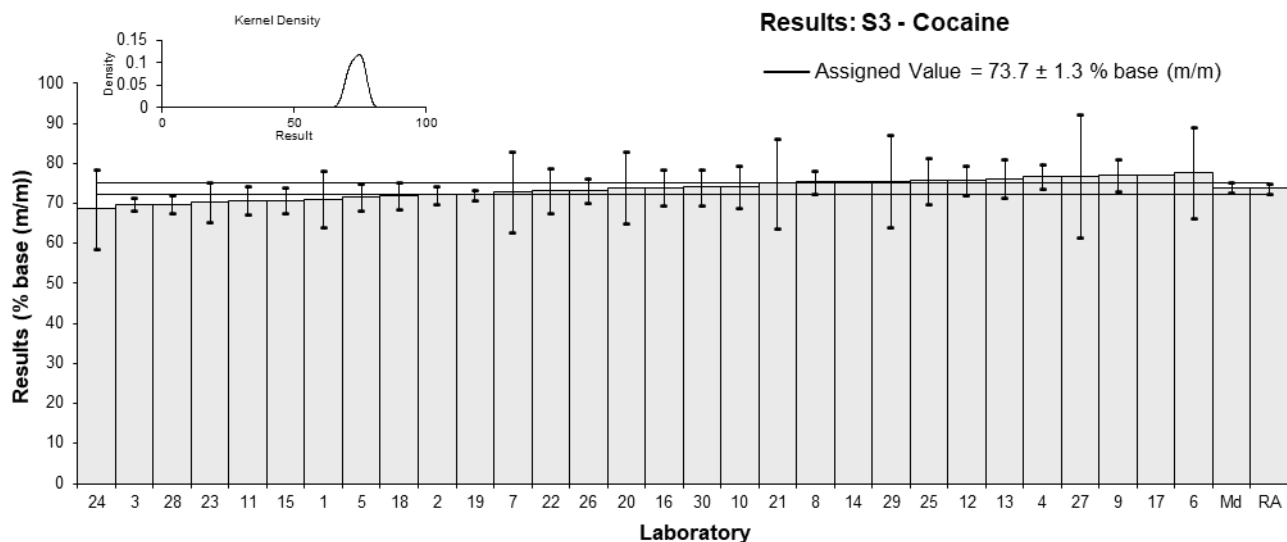


Figure 4

Table 9 Participants' Identification of Cutting Agents\*

Lab. Code	Cutting Agents		
	S1	S2	S3
Preparation	Niacinamide		Sucrose
1	Niacinamide	Niacinamide	Sucrose
2	Niacinamide	Niacinamide	
3	Niacinamide, Saccharin	Niacinamide, Saccharin	Saccharin
4	Niacinamide	Niacinamide	
5	Nicotinamide	Nicotinamide	
6			
7	nicotinamide/niacinamide	nicotinamide/niacinamide	N/A
8	Niacinamide	Niacinamide	
9	Niacinamide	Niacinamide	
10	nicotinamide	nicotinamide	
11	Nicotinamide	Nicotinamide	Sucrose
12			
13	Nicotinamide	Nicotinamide	
14	None detected	None detected	None detected
15	Nicotinamide	Nicotinamide	Saccharose
16	Nicotinamide	Nicotinamide	-
17	Niacinamide	Niacinamide	Sugar
18	niacinamide	niacinamide	
19	nicotinamide, probably inositol	nicotinamide	saccharose
20	Niacinamide	Niacinamide	/
21	none	none	none
22	Niacinamide	Niacinamide	no adulterants detected
23	N/A	N/A	N/A
24	nicotinamide	nicotinamide	
25	Nicotinamide	Nicotinamide	N/A
26	Vitamin B3	Vitamin B3	-
27	nicotinamide	nicotinamide	none
28	Nicotinamide	Nicotinamide	
29	NIACINAMIDE	NIACINAMIDE	/
30	Nicotinamide	Nicotinamide	-

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

## 6 DISCUSSION OF RESULTS

### 6.1 Assigned Value

The assigned values for all scored analytes were the robust averages of participants' results. If there were results less than 50% or greater than 150% of the robust average, these were excluded from the calculation of each assigned value.<sup>3,4</sup> The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528.<sup>5</sup> The calculation of the expanded uncertainty for a robust average is presented in Appendix 1, using Sample S3 as an example.

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

### 6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an evaluation of the expanded MU associated with their results and the basis of this uncertainty evaluation (Table 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 evaluate the uncertainty of chemical measurements and to report this uncertainty in specific circumstances, including when the client's instruction so requires.<sup>7</sup>

Of 90 numeric results, 84 (93%) were reported with an associated expanded MU. Laboratories **14** and **17** did not report any uncertainties; these participants were not accredited.

The magnitude of reported uncertainties was within the range 1.8% to 20% 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 to be fit for purpose. Of the 84 MUs, 54 (64%) were between 3% and 10% relative to the result, six were less than 3% relative and 24 were greater than 10% relative.

Participants were also requested to report the coverage factor associated with their uncertainties (Table 3). The most common coverage factor was  $k = 2$  (fourteen participants). Two 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  $37.46 \pm 2.62\%$  base (m/m), it is better to report  $37.5 \pm 2.6\%$  base (m/m).<sup>8</sup>

### 6.3 z-Score

A target SD for proficiency assessment 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 for proficiency assessment (as PCV) obtained in this study are presented in Table 9.

Table 10 Comparison of Thompson-Horwitz CVs, Between-Laboratory CVs and Target SDs for Proficiency Assessment

Sample	Analyte	Assigned Value (% base (m/m))	Thompson-Horwitz CV* (%)	Between-Laboratory CV† (%)	Target SD (as PCV) (%)
S1	Cocaine	39.4	1.6	5.1	3
S2	Cocaine			4.7	3
S3	Cocaine	73.7	1.2	3.8	3

\* Calculated from the assigned value.

† Robust between-laboratory CV with outliers removed, if applicable.

Of 90 results for which  $z$ -scores were calculated, 77 (86%) returned a  $z$ -score of  $|z| \leq 2.0$ , indicating an acceptable performance.

Twenty participants received acceptable  $z$ -scores across all three samples: Laboratories **2, 3, 5, 7, 8, 9, 10, 11, 12, 15, 16, 18, 19, 20, 22, 23, 25, 26, 29** and **30**.

Ten participants returned at least one questionable or unacceptable  $z$ -score.

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

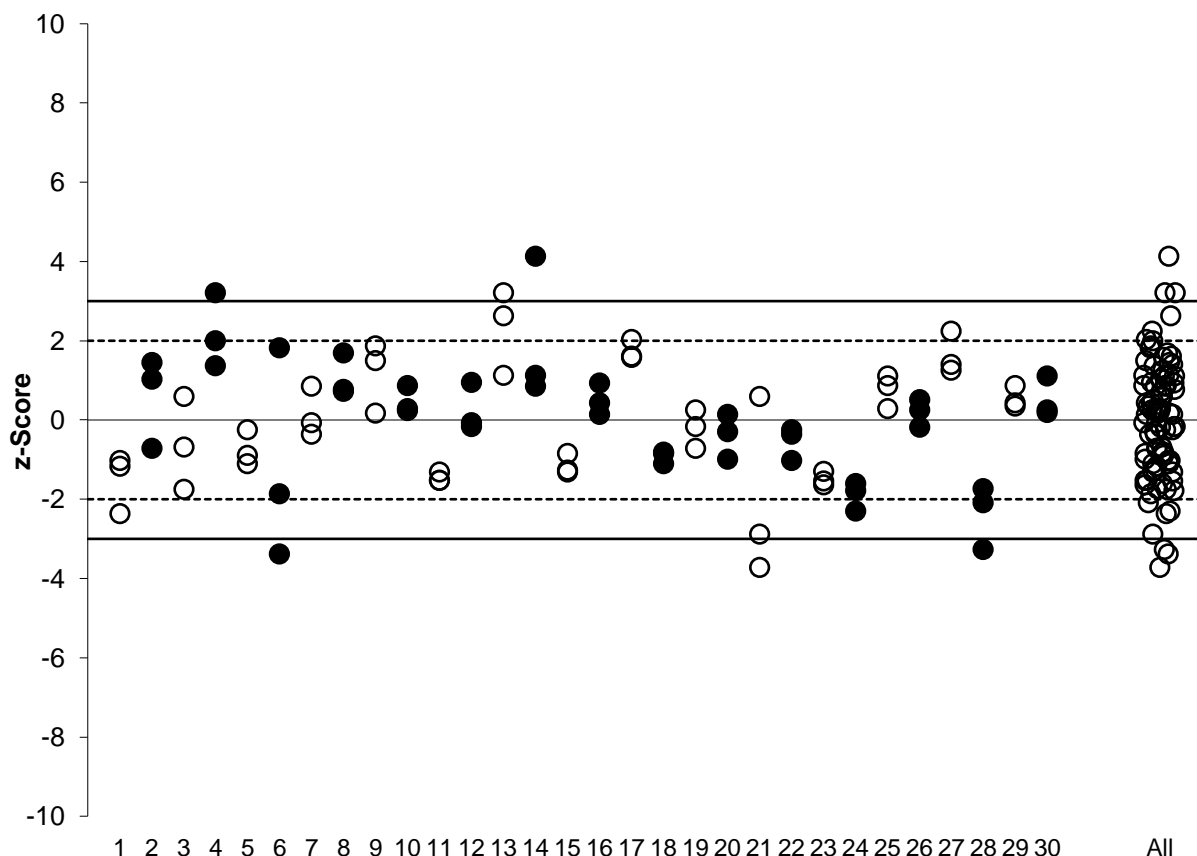


Figure 5  $z$ -Score Dispersal by Laboratory

#### 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 90 results for which  $E_n$ -scores were calculated, 75 (83%) returned an acceptable  $E_n$ -score of  $|E_n| < 1.0$ , indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Twenty-two participants received acceptable  $E_n$ -scores across all three samples: Laboratories **1, 4, 5, 6, 7, 10, 11, 12, 15, 16, 18, 19, 20, 21, 22, 23, 24, 25, 26, 27, 29** and **30**.

Eight participants returned at least one unacceptable  $E_n$ -score. Laboratories **14, 17** and **28** returned unacceptable  $E_n$ -scores for all three samples.

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

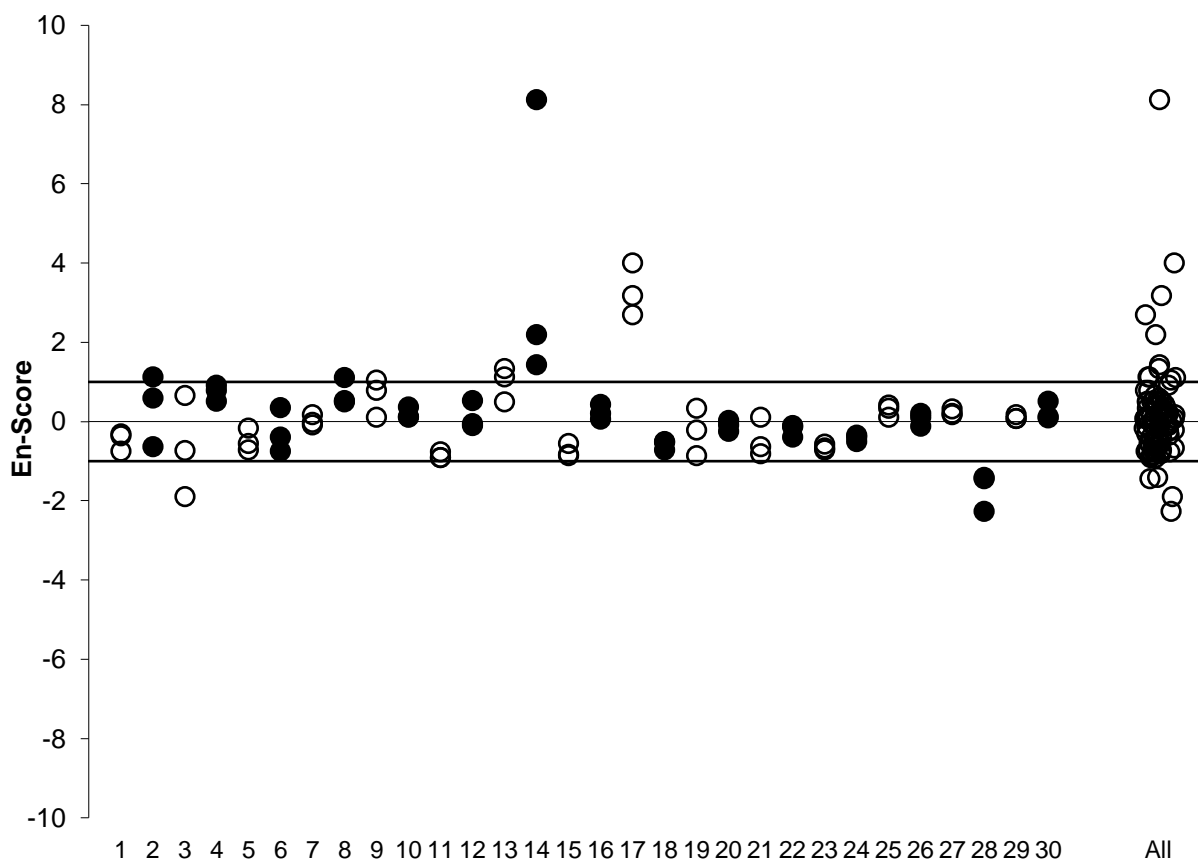


Figure 6  $E_n$ -Score Dispersal by Laboratory

### 6.5 Identification of Cutting Agent

The test samples were prepared using seizures of cocaine hydrochloride supplied by the Australian Federal Police. Niacinamide was added to blind duplicate Samples S1 and S2, and sucrose was added to Sample S3.

Twenty-five participants (83%) reported on the identity of the cutting agent(s) in at least one sample (Table 8).

Laboratories **1, 11** and **15** correctly identified all cutting agents in the samples.

For Samples S1 and S2, most participants reporting on cutting agents correctly identified the presence of niacinamide in these samples. Laboratory **3** incorrectly reported saccharin additionally in these samples. Laboratory **19** incorrectly reported inositol additionally for Sample S1 only. Laboratory **26** reported Vitamin B3 in these samples, which may refer to niacinamide, but may also refer to related compounds nicotinic acid and nicotinamide riboside.

A significantly smaller proportion of participants was able to identify sucrose as the cutting agent in Sample S3 (4 participants). Laboratory **17** reported sugar more generally. Laboratory **3** incorrectly reported saccharin, an artificial sweetener not structurally related to sucrose.

## 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 11 Summary of Participants' Analyses

		Lab. Code
Accreditation	Yes to ISO/IEC 17025	1, 2, 3, 4, 5, 6, 7, 8, 11, 13, 15, 16, 18, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 30
	Not Accredited / Not Reported	9, 10, 12, 14, 17, 29
Average Sample Mass Used per Analysis (mg)	< 20	3, 4, 5, 8, 9, 10, 11, 12, 20, 24, 27, 29
	20 – 30	1, 6, 13, 14, 16, 19, 22, 23, 25, 28, 30
	31 – 50	7, 17, 18, 21, 26
	51 – 100	2, 15
Instrument Used for Quantification*	HPLC-DAD	1 (214 nm), 2, 9 (230 nm), 10 (230 nm), 22 (230 nm), 23, 27, 29
	HPLC-UV/Vis	4 (233 nm), 6, 14, 20
	UPLC-DAD	5 (275 nm), 12, 16 (230 nm) 17, 28, 30 (230 nm)
	UPLC-MS/MS	21
	GC-FID	7, 8, 11, 13, 15, 18, 19, 24, 25, 26
	HPLC (detector not reported)	3
Solvent	Acetonitrile	6, 15
	Acetonitrile/Water(/Acid)	4, 5, 10, 12, 20, 22, 27, 29
	Acetonitrile/Methanol(/Water)	21, 28
	Ethanol	11, 13, 17, 19
	Methanol	1, 2, 7, 8, 9, 14, 16, 23, 25, 26, 30
	Other	18, 24
	Not Reported	3
Sources of Calibration Standard	NMI Australia	1, 5, 11, 15, 17, 18, 28
	Lipomed	2, 6, 8, 10, 12, 13, 16, 20, 23, 27, 29, 30
	Other	4, 14, 19, 21, 22, 24, 25, 26
	Not Reported	3, 7, 9

\* If the participant reported the wavelength used, this has been reported in parentheses.

Plots of the  $z$ -score versus various methodology parameters are presented in Figures 7 to 10. Where charts refer to  $n = x$ , this corresponds to  $x$  number of participants using that methodology. No significant trend was observed.

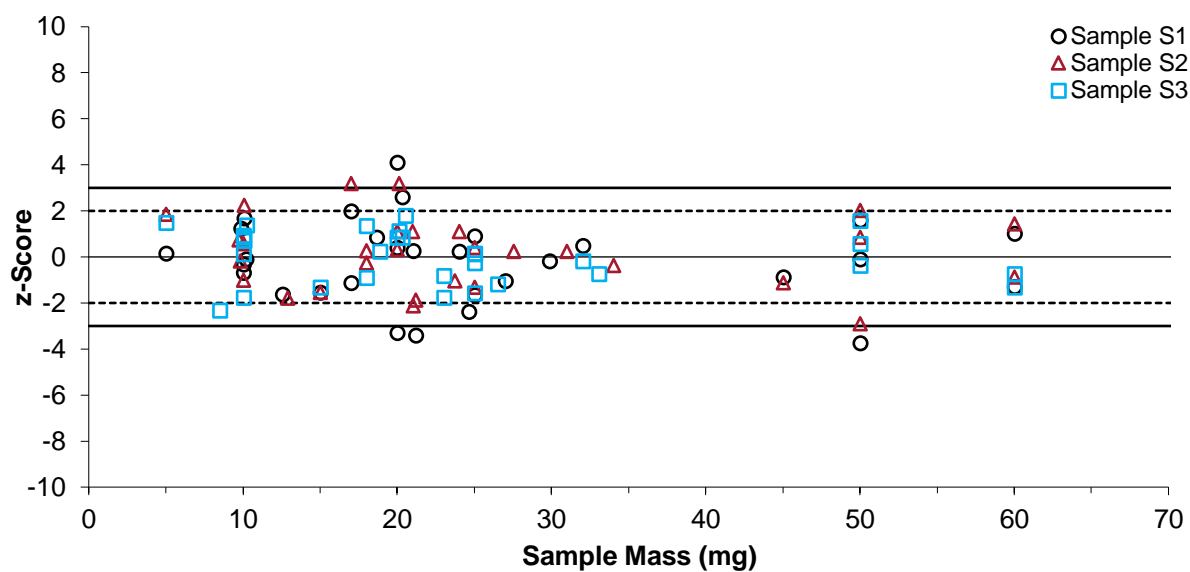


Figure 7 z-Score vs Sample Mass Used for Analysis

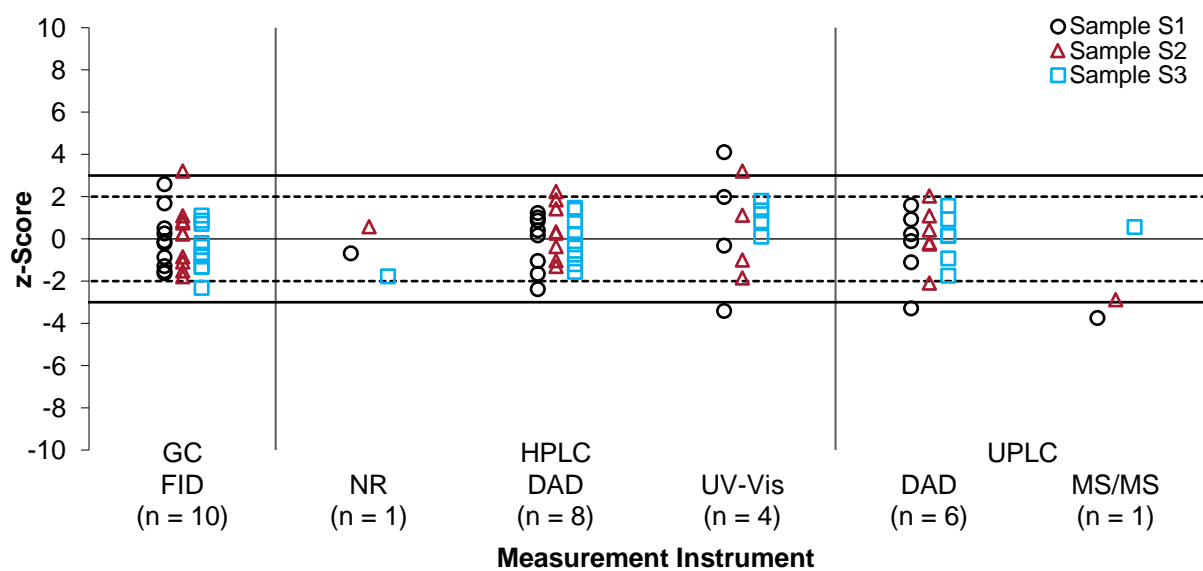


Figure 8 z-Score vs Measurement Instrument

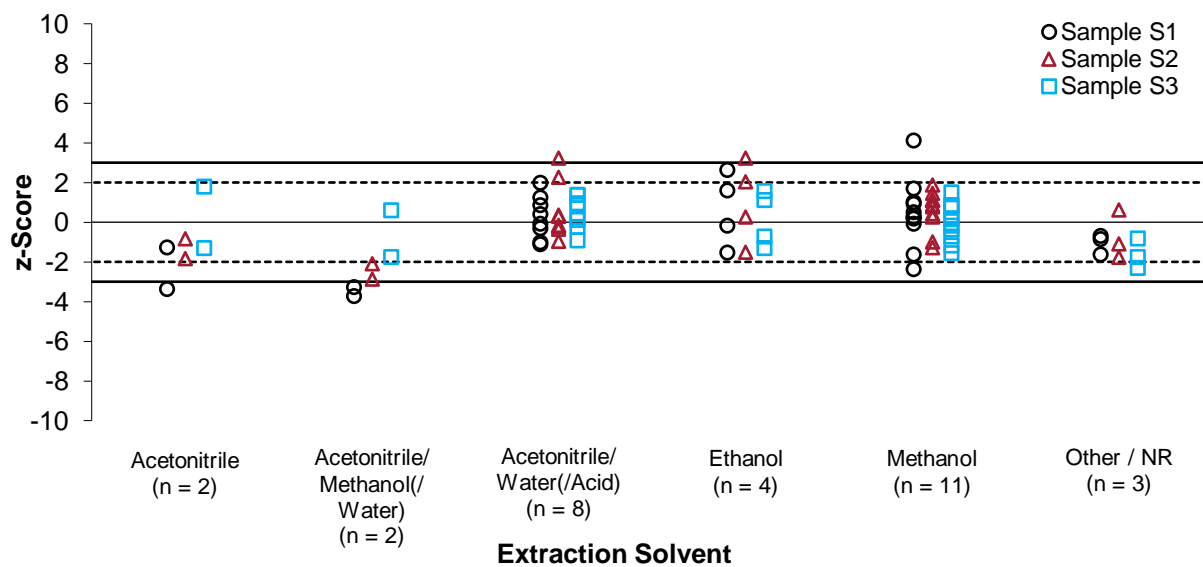


Figure 9 z-Score vs Extraction Solvent

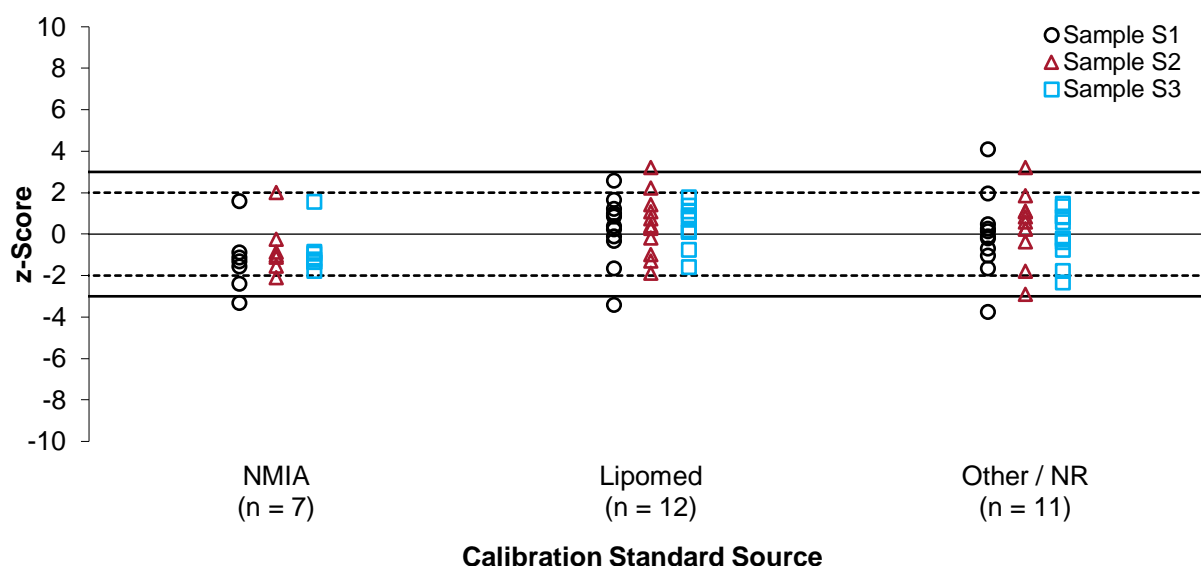


Figure 10  $z$ -Score vs Source of Calibration Standard

## 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 six months. No trend was found between when the samples were analysed and the results obtained (Figure 11).

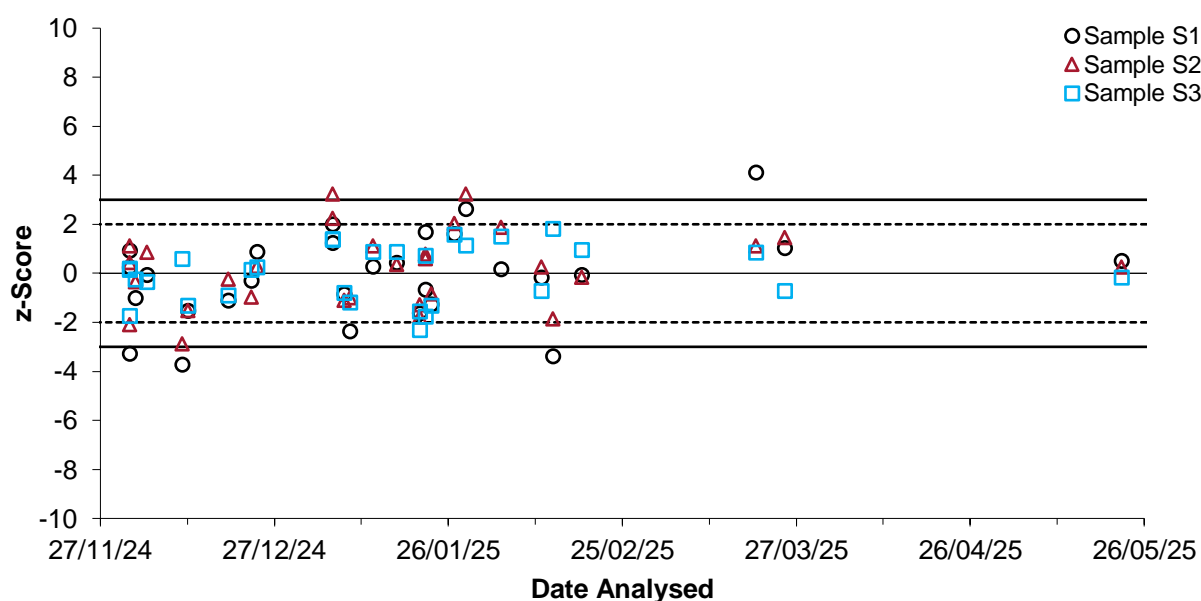


Figure 11  $z$ -Score vs Sample Analysis Date

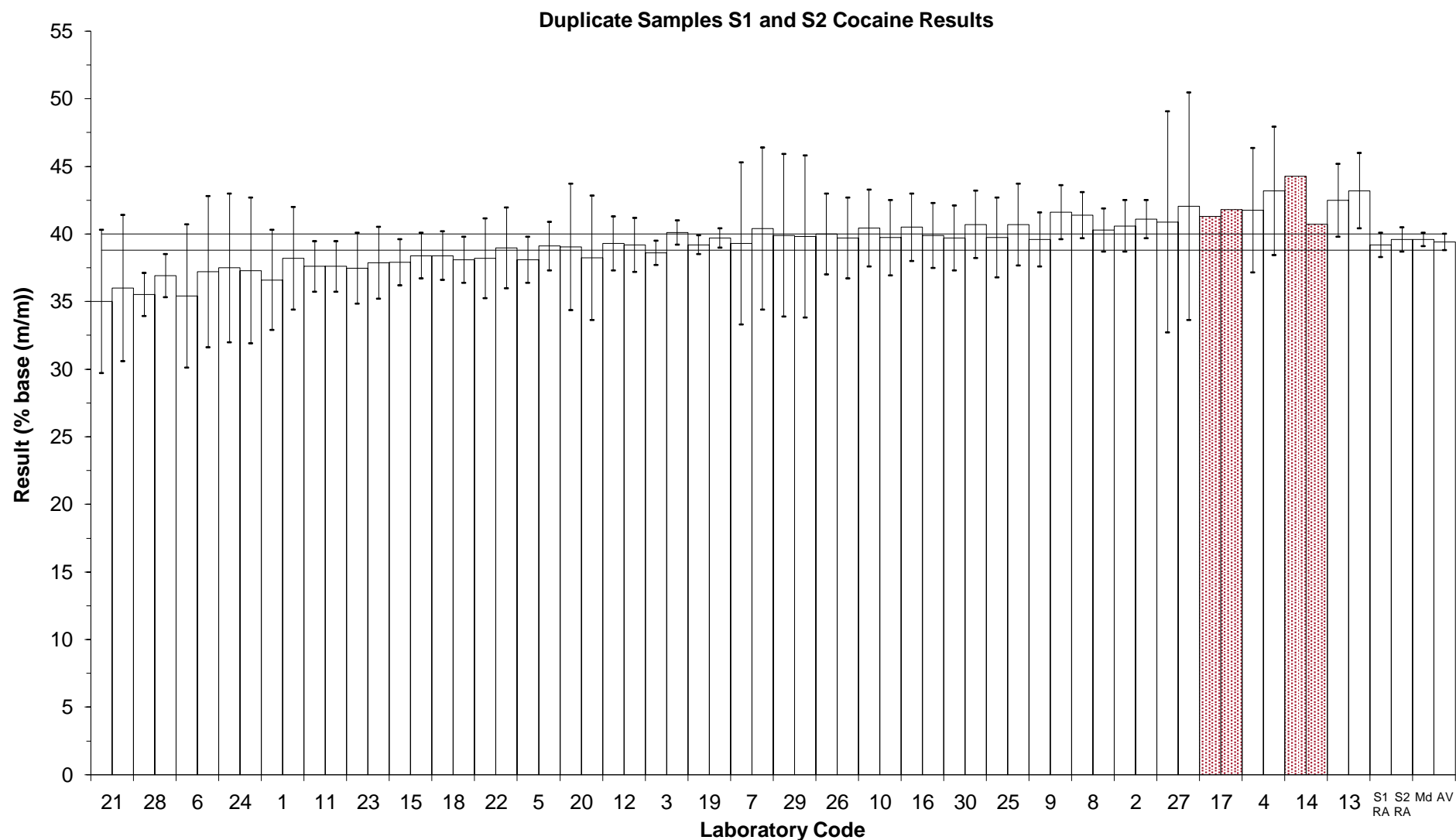
## 6.8 Duplicate Samples S1 and S2

Samples S1 and S2 were blind duplicate samples. Participants' results for these samples, along with the robust averages, median and assigned value, are presented in Figure 12.

The majority of participants' results for these samples were in agreement with each other within their reported expanded uncertainties, reflecting suitable within laboratory repeatability.

As Laboratories 14 and 17 did not report identical results, and also did not report uncertainties, their duplicate results were not in agreement. For both participants, their results may have been in agreement with each other if they had reported reasonable expanded uncertainties.





Horizontal lines are the assigned value  $\pm$  U. Participants' results which are not in agreement with each other within reported uncertainties are shaded. RA = Robust Average, Md = Median, AV = Assigned Value.

Figure 12 Results for Blind Duplicate Samples S1 and S2

## 6.9 Comparison with Previous Cocaine PT Studies

To enable direct comparison with previous Cocaine PT studies, the target SD for proficiency assessment 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 2015 to 2024 (last 10 studies) are presented in Figure 13. The average proportion of acceptable  $z$ -scores and  $E_n$ -scores over this period is 84% and 85% respectively.

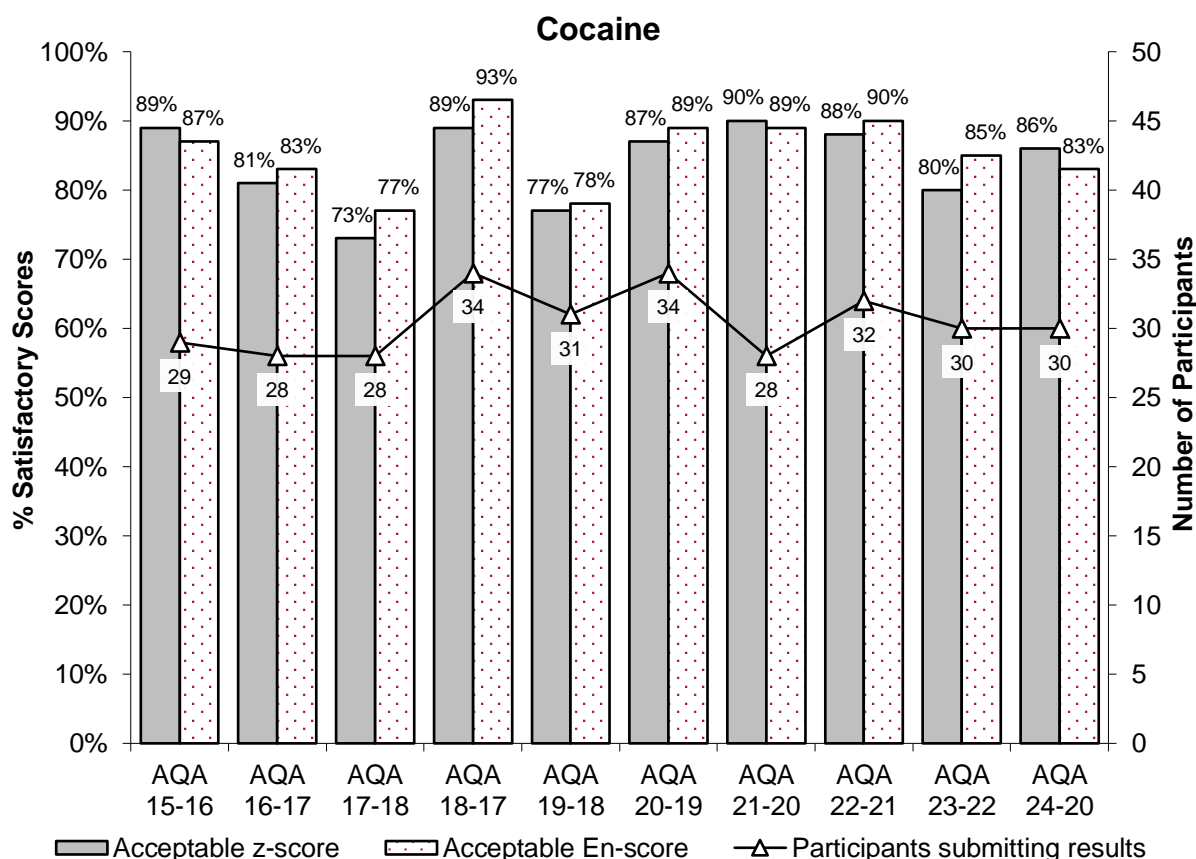


Figure 13 Summary of Participants' Performance in Cocaine PT Studies

Several participants have consistently participated in NMIA 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.

For those laboratories consistently participating in NMIA Cocaine PT studies, a summary of individual laboratory's performances over the last ten studies is presented in Figures 14 and 15 for Australian and international laboratories respectively. One Australian and three international laboratories have achieved acceptable  $z$ -scores across all samples in all NMIA Cocaine PT studies participated in over this period.

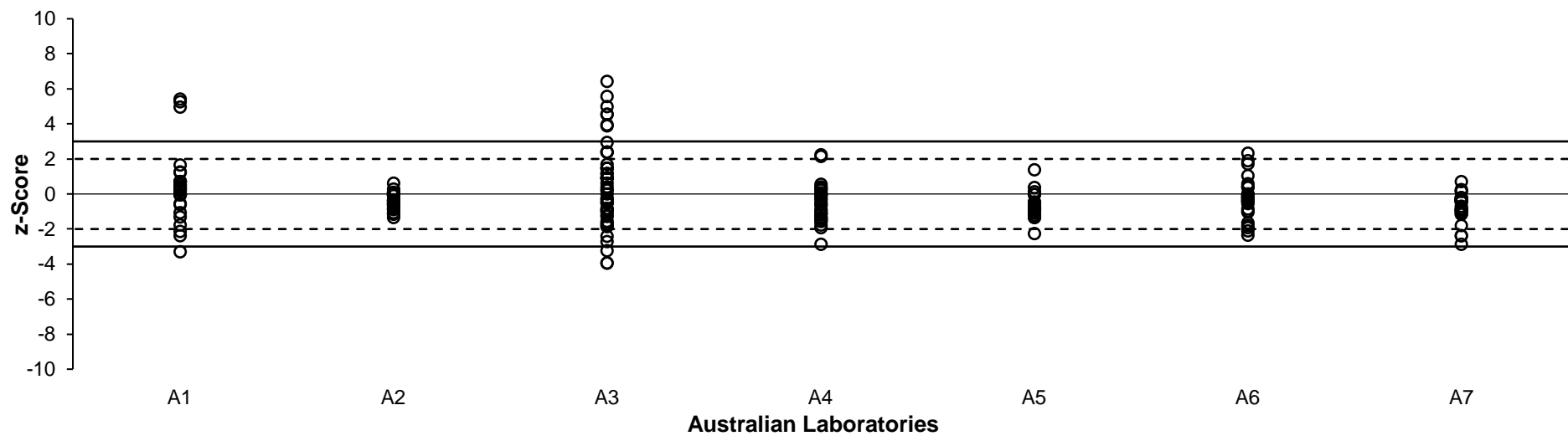


Figure 14 Summary of Australian Participants'  $z$ -Scores in NMIA Cocaine PT Studies

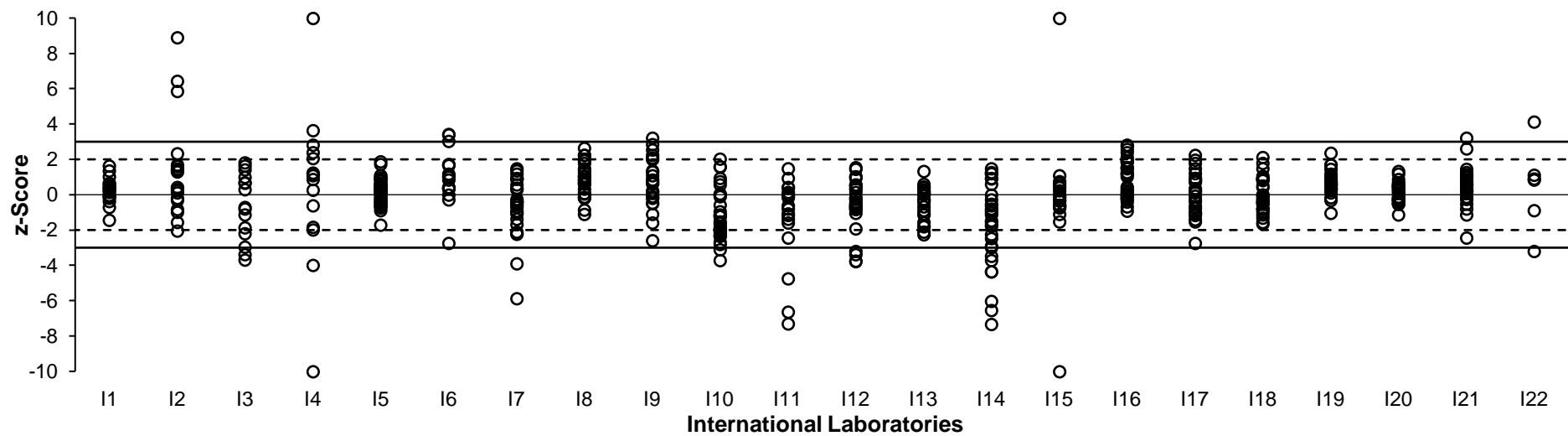
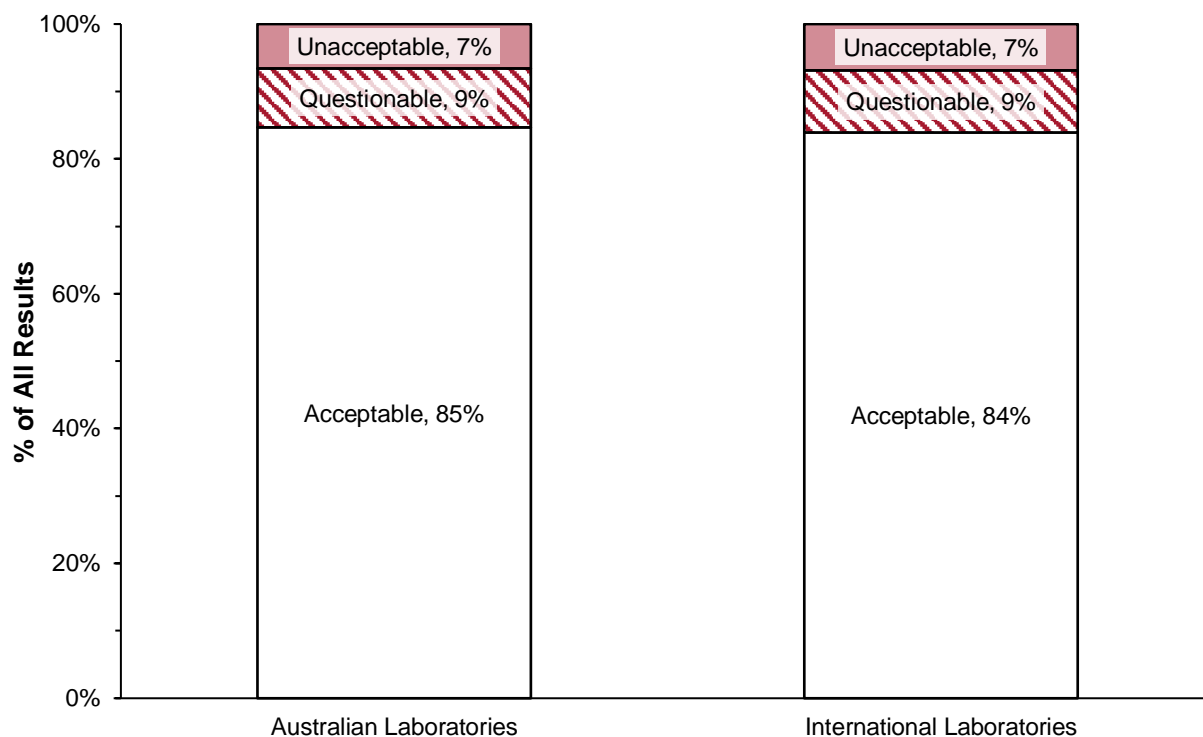


Figure 15 Summary of International Participants'  $z$ -Scores in NMIA Cocaine PT Studies

A comparison of all results from Australian and international laboratories in NMIA Cocaine PT studies over the last ten years is presented in Figure 16. Overall, both groups have performed similarly, achieving 85% and 84% acceptable z-scores over this period for Australian and international laboratories respectively.



Values are rounded and therefore may not add up to 100%.

Figure 16 Comparison of Australian and International Laboratories in NMIA 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, *Conformity assessment — General requirements for the competence of proficiency testing providers*.
- [2] Commonwealth of Australia, Department of Industry, Science and Resources, NMIA, 2024, *Study Protocol for Proficiency Testing*, viewed May 2025, <[https://www.industry.gov.au/sites/default/files/2020-10/cpt\\_study\\_protocol.pdf](https://www.industry.gov.au/sites/default/files/2020-10/cpt_study_protocol.pdf)>
- [3] Commonwealth of Australia, Department of Industry, Science and Resources, NMIA, 2024, *Chemical Proficiency Testing Statistical Manual*, viewed May 2025, <[https://www.industry.gov.au/sites/default/files/2019-07/cpt\\_statistical\\_manual.pdf](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*.
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## APPENDIX 1 ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE AND E<sub>n</sub>-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 evaluated 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 12 Uncertainty of Sample S3 Robust Average\*

No. results ( $p$ )	30
Robust Average	73.67% base (m/m)
$S_{rob\ av}$	2.83% base (m/m)
$u_{rob\ av}$	0.65% base (m/m)
$K$	2
$U_{rob\ av}$	1.29% base (m/m)

\* Values presented in this table have been rounded, however calculations were performed on exact values.

Therefore, the robust average of Sample S3 is  $73.7 \pm 1.3\%$  base (m/m).

### A1.2 z-Score and E<sub>n</sub>-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 13 z-Score and  $E_n$ -Score Calculation for Sample S3 Result Reported by Laboratory 1

Participant Result (% base (m/m))	Assigned Value (% base (m/m))	Target SD	z-Score	$E_n$ -Score
71.1 ± 7.1	73.7 ± 1.3	3% as PCV, or: $0.03 \times 73.7 =$ 2.211% base (m/m)	$z = \frac{71.1 - 73.7}{2.211}$ $= -1.18$	$E_n = \frac{71.1 - 73.7}{\sqrt{7.1^2 + 1.3^2}}$ $= -0.36$

## **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
NMIA	National Measurement Institute Australia
PCV	Performance Coefficient of Variation
PDA	Photodiode Array
PT	Proficiency Testing
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**