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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.

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SUMMARY

AQA 20-19 Cocaine commenced in November 2020. Sample sets, each containing three samples of cocaine hydrochloride, were sent to thirty-two laboratories, with two laboratories 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 an illicit seizure 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 102 z-scores, 89 (87%) returned $|z| \leq 2.0$, indicating a satisfactory performance.

Of 102 E_n -scores, 91 (89%) returned $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratories **1, 5, 6, 7, 10, 11, 13, 14, 15, 16, 17, 18, 21, 22, 23, 26, 27, 28, 30, 31, 32, 33** and **35** returned satisfactory z-scores and E_n -scores for all samples.

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

Of 102 results, 99 (97%) were reported with an associated expanded measurement uncertainty. Laboratory **25** did not report uncertainties for their results; this laboratory was not accredited. The magnitude of reported uncertainties was within 1.5% to 33% 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 niacinamide, Sample S2 was cut with glucodin, and Sample S3 was cut with acetylsalicylic acid.

Twenty-seven participants (74%) reported on the identity of the cutting agents of at least one sample.

Laboratories **1, 14, 22** and **32** correctly identified all cutting agents in the test samples.

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

The test samples of this PT 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 comparison.'¹ NMI PT studies target chemical testing in areas of high public significance such as trade, environment, law enforcement and food safety. NMI offers studies in:

- pesticide residues in fruit and vegetables, soil and water;
- petroleum hydrocarbons in soil and water;
- PFAS in water, soil, biota and food;
- inorganic analytes in soil, water, food and pharmaceuticals;
- controlled drug assay and clandestine laboratory; and
- allergens in food.

1.2 Study Aims

The aims of the study were to:

- assess the proficiency of laboratories measuring cocaine in samples typical of a routine seizure;
- develop a practical application of traceability and measurement uncertainty, and provide participants with information that will assist uncertainty estimates;
- test the ability of participants to identify cutting agents commonly found in controlled drug preparation; and
- produce materials that can be used in method validation and as control samples.

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

1.3 Study Conduct

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

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

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitation issued	2 November 2020
Samples dispatched	27 January 2021
Results due	10 May 2021
Interim report issued	25 May 2021

2.2 Participation and Laboratory Code

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

2.3 Test Material Specification

Three test samples were prepared in November 2020. The starting material was cocaine hydrochloride approximately 84% base (m/m) supplied by the Australian Federal Police.

Niacinamide and acetylsalicylic acid purchased from Sigma Aldrich, and glucodin purchased from a local pharmacy were used as cutting agents. Sample S1 was cut with niacinamide, Sample S2 was cut with glucodin, and Sample S3 was cut with acetylsalicylic acid.

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 54% cocaine base (m/m).

Sample S2 was prepared to contain approximately 24% cocaine base (m/m).

Sample S3 was prepared to contain approximately 72% cocaine base (m/m).

2.4 Test Sample Homogeneity

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.

The procedure for the preparation of the study samples has been validated in previous studies. No homogeneity testing was conducted in this proficiency study. Results returned by the participants gave no reason to question the homogeneity 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 January 2021. 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 e-mailed to participants.

2.6 Instructions to Participants

Participants were asked to analyse the samples using their routine quantitative method and were instructed as follows:

- For each sample report % m/m cocaine as base.
- 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).
- Results are to be returned via email to jenny.xu@measurement.gov.au by 10 March 2021.

Due to the international circumstances occurring over the course of this study, there were delivery delays to some international participants, and so the results due date was extended by 2 months, from 10 March 2021 to 10 May 2021.

2.7 Interim Report

An interim report was emailed to all participants on 25 May 2021.

The interim report release was delayed due to exceptional circumstances requiring further investigation on international sample delivery by the study coordinator.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Methods Reported by Participants

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

Table 1 Summary of Participants' Test Methods

Lab. Code	Extraction Solvent	Internal Standard	Calib. Points	Technique	Detector	Column
1	Methanol	N/A	6	UPLC	DAD	Acquity UPLC BEH C18 1.7 µm, 2.1 x 100 mm
2	water/acetonitrile/ n10 sulphuric acid 90:10:1	None	3	HPLC	Diode Array	Shimpack XR-ODS
3	Methanol	None (external standard)	7	HPLC	DAD	ZORBAX ECLIPSE XDB-C18 (5 micron, 4.6mm X 150 mm)
4	Methanol	Tetracosane	4	GC	FID	SGE 12 x 0.22 mm
5	Acetonitrile/water/ trifluoroacetic acid (25/75/0.1)	N/A	3	HPLC	DAD	ODS2 Interpak column
6	Methanol	None (external calibration)	3	GC	FID	CP sil5CB
7	Acetonitrile	None	5	HPLC	UV	PROTECOL C8 H 5UM 150X4.6MM
8	Methanol	Methadone	4	GC	FID	Hp-5 30mx0.32mm 0.25um
9	Ethanol	Tetracosane	3	GC	FID	RXi-5
10	CDCl ₃	1,4-bis(trimethylsilyl) benzene		QNMR		NA
11	Acetonitrile	Strychnine	6	GC	FID	HP1
12	Methanol	None	6	HPLC	DAD	Phenomenex C18 5um Luna
13	Dichloromethane/ Methanol (50/50)	NO INTERNAL STANDARD	5	HPLC	MS	C18
14	Ethanol	triphenylacetylphenone	3	GC	FID	HP-1MS
15	acetonitrile/water	none	5	HPLC	DAD	Kromasil
16	Ethanol	Tetracosane	6	GC	FID	HP5
17	HPLC Methanol	Vanillin	1	UPLC	DAD	Agilent Lichrospher 60 RP-select B
18	HPLC Methanol	Vanillin	1	UPLC	DAD	Agilent Lichrospher 60 RP-select B
20	Ethanol	Tribenzylamine	4	GC	FID	HP1
21	acetonitrile/water (80/20)	none	3	HPLC	DAD	C8

Lab. Code	Extraction Solvent	Internal Standard	Calib. Points	Technique	Detector	Column
22	deuterium oxide	maleic acid		QNMR	Bruker AVIII 600 with BBFO probe	N/A
23	ACN/MeOH/H2O	Analog of cocaine	7	UPLC	MS/MS	C-18 column
24	Methanol		4	HPLC	DAD	Zorbax Eclipse XDB C18
25	Ethanol	Propylparaben	7	UPLC	DAD	BEH Shield RP18
26	CH3CN/H2O (80/20)	No internal standard	2	HPLC	DAD	C8
27	72% water ultra pure + 28% acetonitrile		5	HPLC	UV/Vis	Kromasil C8
28	Methanol	None	5	HPLC	DAD	Kinetex 2.6 μ XB-C18
29	Methanol	N/A	6	UPLC	DAD	Acquity UPLC BEH C18 1.7 μ m 2.1 x 100 mm
30	Methanol	Diazepam	6	GC	FID	J&W 128-5512
31	Acetonitrile/ Methanol (95:5)	Pholcodine 1mg/ml	3	UPLC	PDA	ACQUITY C-18
32	S1 and S3: mobile phase S2: chloroform	S1 and S3: N/A S2: benzopinacolone (BZP)	S1 and S3: 4 S2: 1	S1 and S3: HPLC S2: GC	S1 and S3: PDA S2: FID	S1 and S3: BondaPak C18 S2: HP1
33	Acetonitrile:water 75:25	Diethylphthalate	3	UPLC	PDA	Acquity UPLC BEH C18 1.7 μ m (2.1 x 100 mm)
34	Ethanol	Tribenzylamine	6	GC	FID	HP5
35	dichloromethane	5 α -cholestane	5	GC	FID	HP5

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

Table 2 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 - In house controls Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Standard purity	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
2	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - CRM Duplicate analysis	Standard purity	ISO/GUM
3	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	Nordtest Report TR537 and Measurement Uncertainty for weight Determination in Seized Drug Analysis

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
4	Top Down - precision and estimates of the method and laboratory bias	Control samples - Authentic powders	Instrument calibration Matrix effects Laboratory bias from PT studies Recoveries of SS Standard purity	
5	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Instrument calibration Masses and volumes Recoveries of SS	ISO/GUM
6	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Instrument calibration Recoveries of SS	ISO/GUM
7	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - RM	Laboratory bias from PT studies	
8	Standard deviation of replicate analyses multiplied by 2 or 3	Duplicate analysis	Masses and volumes	ISO/GUM
9	Uncertainty Budget Method	Control samples - RM Duplicate analysis	Instrument calibration Masses and volumes Standard purity	Internal SOP Document
10	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
11	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM Duplicate analysis	Instrument calibration Masses and volumes Matrix effects Recoveries of SS	ISO/GUM
12	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM	Laboratory bias from PT studies	Eurachem/CITAC Guide
13	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM	Instrument calibration Homogeneity of sample Masses and volumes	
14	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
15	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - RM		ISO/GUM
16	Top Down - precision and estimates of the method and laboratory bias	Control samples – RM (in house) Duplicate analysis	Matrix effects Standard purity	ISO/GUM
17	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Laboratory bias from PT studies Recoveries of SS	Eurachem/CITAC Guide

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
18	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	Laboratory bias from PT studies Recoveries of SS	Eurachem/CITAC Guide
20	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM Duplicate analysis		internal document based on Eurachem/CITAC; ISO/GUM
21	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM	Laboratory bias from PT studies Standard purity	NF V03-110
22	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Control samples - CRM Duplicate analysis	Instrument calibration Masses and volumes Standard purity	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
23	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM		
24	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM Duplicate analysis	Instrument calibration Masses and volumes Recoveries of SS Standard purity	Eurachem/CITAC Guide
25				
26	Global Approach	Control samples		Eurolab Technical Report No1/2007
27	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis	Laboratory bias from PT studies	ISO/GUM
28	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Matrix effects Recoveries of SS Standard purity	Eurachem/CITAC Guide
29	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Standard purity	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
30	Estimating Measurement Uncertainty by black box with pairs of values	Standard deviation from PT studies only		ISO/GUM (ENAC G 09 or ISO 21748)
31	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Standard purity	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
32				
33	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - RM Duplicate analysis	Homogeneity of sample Standard purity	Eurachem/CITAC Guide

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
34	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM	Standard purity	
35	Standard deviation of replicate analysis, uncertainty of reference material and calibration	Control samples - RM Duplicate analysis	Instrument calibration Standard purity	Eurachem/CITAC Guide

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

Table 3 Uncertainty Comments

Lab. Code	Participants' Uncertainty Comments
2	UoM determined from 3 x std deviation of multiple injections expanded by professional judgement. No analysis carried out for inert bulking agents
5	The reported result (in routine case samples) is defined as the average of the individual results multiplied by the uncertainty correction factor and is rounded down to the nearest whole number (unless <1% w/w). The uncertainty correction factor is defined as (mean-2SD)/Mean expressed as a percentage using the relative standard control chart. E.g a result of 53.8% would give a reported result of $53.8 * 0.9709 = 52.23$ therefore rounded down to 52%
35	Based on the repeatability of replicate analysis, the uncertainty of reference material and the uncertainty of the calibration curve.

3.3 Details of Participant Calibration Standard

Participants were requested to provide information about their calibration standard used. Responses are presented in Table 4. Some responses may be modified so that the participant cannot be identified.

Table 4 Participant Calibration Standard

Lab. Code	Reference Standard	Purity (%)
1	NMI	95.7
2	Sigma Aldrich	99.7
3	LIPOMED	99.9
4	Merck	100
5	Johnson Matthey (MacFarlan Smith)	100.2
6	Duchefa	>99
7	COC-156-FB-100	100
8	LGC	1.01
9	NMI	95.7
10	NMI	100
11	NMI	99.8
12	NMI	96.1
13	sigma	99
14	NMI	95.7
15	Sigma-Aldrich	99.9

Lab. Code	Reference Standard	Purity (%)
16	ALCALIBER	100
17	Lipomed	99.503 ±0.026
18	Lipomed	99.503 ±0.026
20	Fagron	100
21	lipomed	99.503
22	Sigma Aldrich	99.98±0.065
23	Unikem	100
24	LGC Standards	1 ±0.003 mg/mL
25	NMI	96.1
26	NMI	95.7
27	lipomed	99.25
28	Lipomed	>98.5
29	NMI	95.7
30	Lipomed	99.3
31	NMI	95.7
32	Glaxo	99.6
33	NMI	99.8
34	Lipomed	99.503 ± 0.026
35	Chiron	99.6

3.4 Participants' Comments

The study coordinator welcomes comments or suggestions from participants as it can provide information which will improve future studies. Participants' comments are listed in Table 5, along with the study coordinator's response where appropriate. Some responses may be modified so that the participant cannot be identified.

Table 5 Participant Comments

Lab. Code	Participants' Comments	Study Coordinator's Response
3	Qualitative analysis was carried out by GC-MS. No detected diluent have been reported, only the adulterant in sample S3.	
10	Methodology: No reference standard involved	
14	The box containing the AQA samples was not properly sealed, and could possibly been accessed. Methodology: A small amount of dichloromethane was used to dissolve the TPAP.	The cardboard box is taped up but not sealed as it is only for the physical protection of the samples. The outside packaging of the samples themselves does have a seal and the participant reported that this seal was intact and not tampered with. Care will be taken to include additional taping of the box to ensure optimal protection during sample delivery.

Lab. Code	Participants' Comments	Study Coordinator's Response
15	for sample AQA 20-19 S3: unexplained HPLC-DAD coelution on the cocaine peak. Cocaine content subject to change.	
20	No diluent/adulterant detected with available instruments.	
21	Please send 3 samples of different concentrations (e.g. 5%, 40% and 80%) and not 2 samples of the same concentration.	Samples were prepared to be of purities which could cater to the needs of different laboratories. For this study, samples were prepared at three levels at approximately 24%, 54% and 72%.
22	Methodology: Simultaneous observation of analyte and IS peaks in ¹ H NMR spectrum acquired using QNMR conditions	
26	A substance comes out almost at the same retention time of cocaine for the sample S3,	
28	Methodology: Average of two determinations	
32	insufficient sample submitted if repeat analysis is required.	Most participants in this study used 30 mg or less for each analysis. For security and accountability reasons, NMI controlled substance PT studies are conducted using the minimum practical amount.

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.

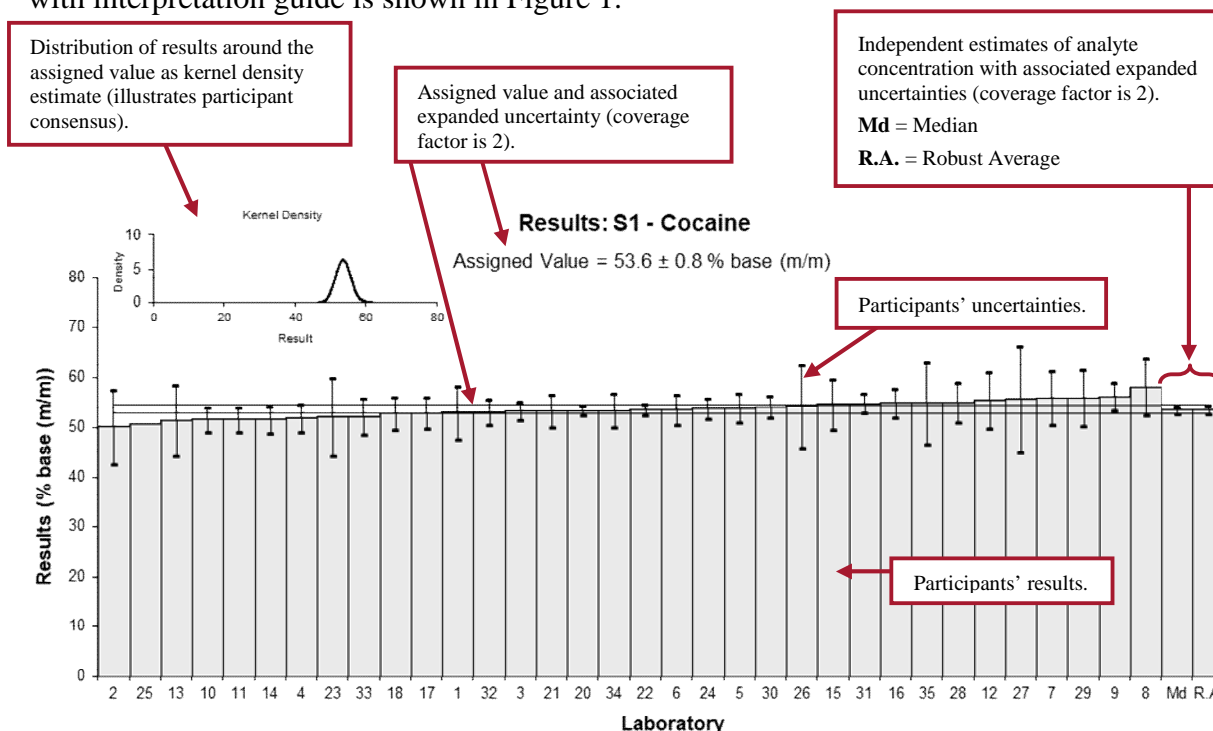


Figure 1 Guide to Presentation of Results

4.2 Assigned Value

The assigned value is defined as the: 'value attributed to a particular property of a proficiency test item'.¹ In this study, the property is the concentration of the analyte in the test samples. 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 13528:2015.⁵

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. It is important to note that the PCV is a value set by the study coordinator; it is not calculated from participants' results. It is based on the levels of analytes in the study and experience from previous studies, and is supported by mathematical models such as the Thompson-Horwitz equation.⁶ By setting a fixed and realistic value for the PCV, a participant's performance does not depend on other participants' performances, and can be compared from study to study.

4.5 Target Standard Deviation

The target standard deviation (σ) is the product of the assigned value (X) and the PCV, as presented in Equation 1. This value is used for calculation of z-scores.

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

4.6 z-Score

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

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

where:

- z is z-score
- χ is a participant's result
- X is the assigned value
- σ is the target standard deviation from Equation 1

For the absolute value of a z-score ($|z|$):

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

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

where:

- E_n is E_n-score
- χ is a participant's result
- X is the assigned value
- U_χ is the expanded uncertainty of the participant's result
- U_X is the expanded uncertainty of the assigned value

For the absolute value of an E_n-score ($|E_n|$):

- $|E_n| \leq 1.0$ is satisfactory;
- $|E_n| > 1.0$ 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.⁷

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

5 TABLES AND FIGURES

Table 6

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z-Score	En-Score
1	53	5.3	-0.37	-0.11
2	50.07	7.5	-2.20	-0.47
3	53.3	1.7	-0.19	-0.16
4	51.9	2.7	-1.06	-0.60
5	53.8	2.91	0.12	0.07
6	53.52	3	-0.05	-0.03
7	55.9	5.40	1.43	0.42
8	58.14	5.67	2.82	0.79
9	56.1	2.7	1.55	0.89
10	51.5	2.3	-1.31	-0.86
11	51.5	2.3	-1.31	-0.86
12	55.5	5.6	1.18	0.34
13	51.4	7	-1.37	-0.31
14	51.5	2.6	-1.31	-0.77
15	54.6	5	0.62	0.20
16	54.9	2.9	0.81	0.43
17	52.9	3.2	-0.44	-0.21
18	52.8	3.2	-0.50	-0.24
20	53.4	1	-0.12	-0.16
21	53.31	3.20	-0.18	-0.09
22	53.5	1.0	-0.06	-0.08
23	52	7.8	-1.00	-0.20
24	53.8	2	0.12	0.09
25	50.7	NR	-1.80	-3.62
26	54.2	8.2	0.37	0.07
27	55.7	10.6	1.31	0.20
28	55	3.9	0.87	0.35
29	56	5.6	1.49	0.42
30	54.1	2.1	0.31	0.22
31	54.76	1.9	0.72	0.56
32	53.1	2.5	-0.31	-0.19
33	52.2	3.7	-0.87	-0.37
34	53.4	3.4	-0.12	-0.06
35	54.9	8.2	0.81	0.16

Statistics

Assigned Value	53.6	0.8
Robust Average	53.6	0.8
Median	53.5	0.7
Mean	53.6	
N	34	
Max.	58.14	
Min.	50.07	
Robust SD	1.8	
Robust CV	3.4%	

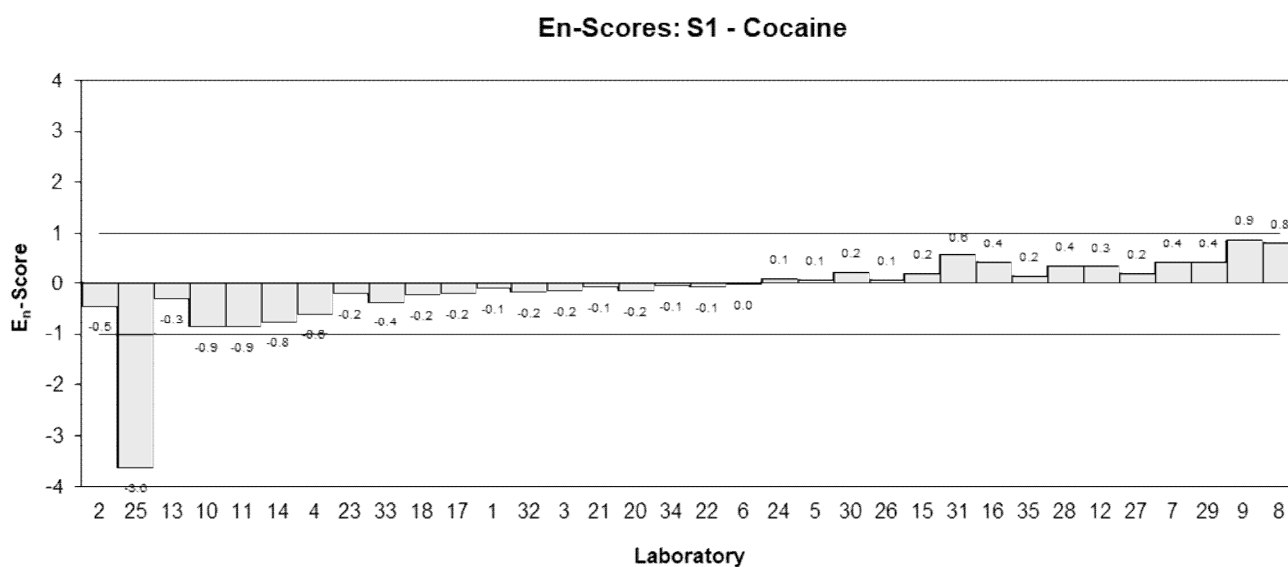
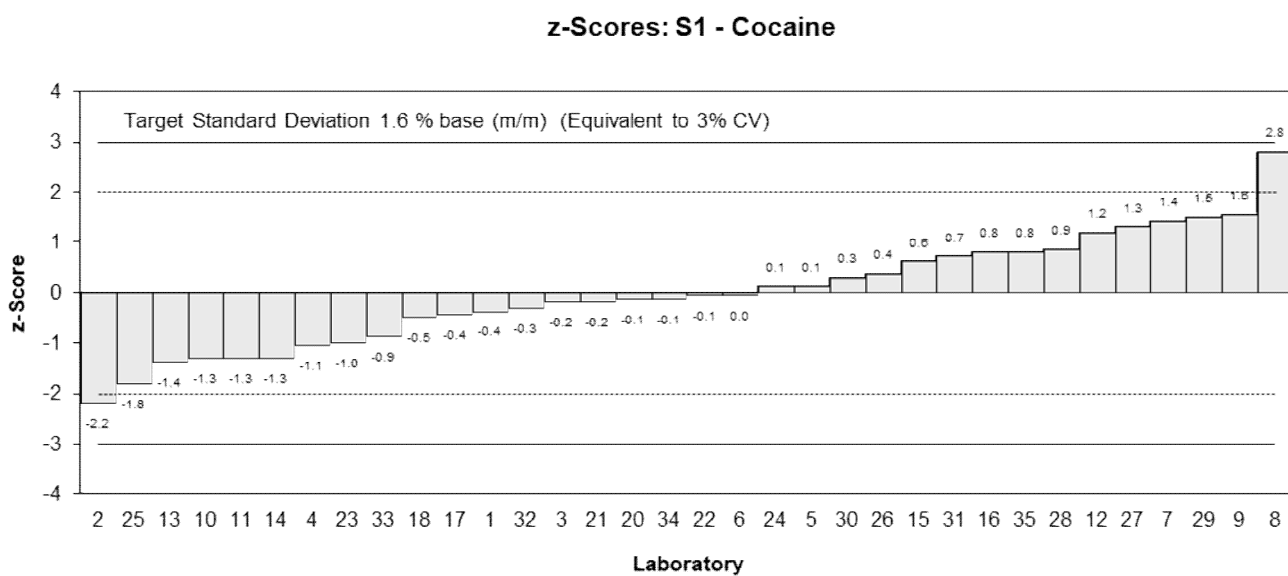
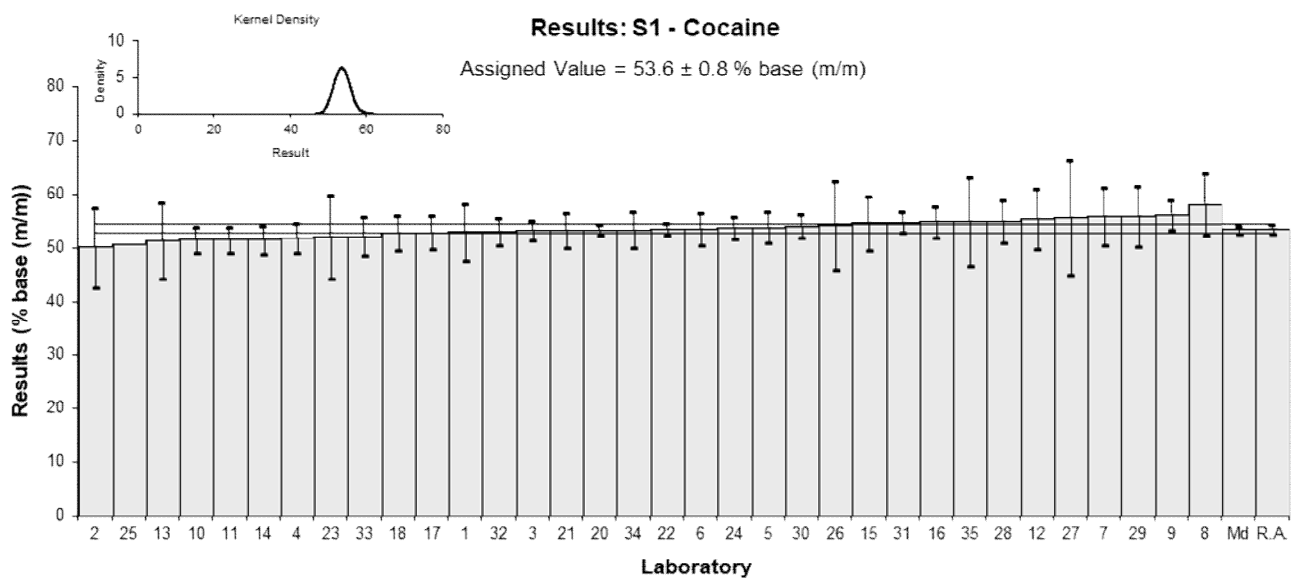


Figure 2

Table 7

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z-Score	E _n -Score
1	23	2.3	1.52	0.43
2	21.3	3.2	-1.06	-0.22
3	19.9	2.1	-3.18	-0.98
4	21.5	1.1	-0.76	-0.43
5	21.3	2.91	-1.06	-0.24
6	22.67	3	1.02	0.22
7	22.2	5.40	0.30	0.04
8	24.41	2.38	3.65	1.00
9	21.1	2.7	-1.36	-0.33
10	22.2	1	0.30	0.19
11	21.3	0.9	-1.06	-0.71
12	24.6	2.5	3.94	1.03
13	21.3	7	-1.06	-0.10
14	21.7	1.1	-0.45	-0.26
15	22.7	2	1.06	0.34
16	21.8	1.1	-0.30	-0.17
17	22.6	1.4	0.91	0.41
18	22.4	1.4	0.61	0.27
20*	71.3	1.3	74.70	36.25
21	21.25	1.28	-1.14	-0.56
22	22.4	0.9	0.61	0.41
23	22	3.3	0.00	0.00
24	23.7	1	2.58	1.58
25	21.2	NR	-1.21	-2.00
26	22	3.3	0.00	0.00
27	22.2	4.2	0.30	0.05
28	22	1.5	0.00	0.00
29	25	2.5	4.55	1.18
30	21.8	1.7	-0.30	-0.11
31	23.11	1.3	1.68	0.82
32	22.4	3.3	0.61	0.12
33	21.8	1.5	-0.30	-0.13
34	20.4	1.3	-2.42	-1.18
35	21.4	3.2	-0.91	-0.19

Statistics*

Assigned Value	22.0	0.4
Robust Average	22.0	0.4
Median	22.0	0.4
Mean	22.1	
N	33	
Max.	25	
Min.	19.9	
Robust SD	0.90	
Robust CV	4.1%	

* Result from Laboratory 20 was excluded from all statistical calculations (gross error).

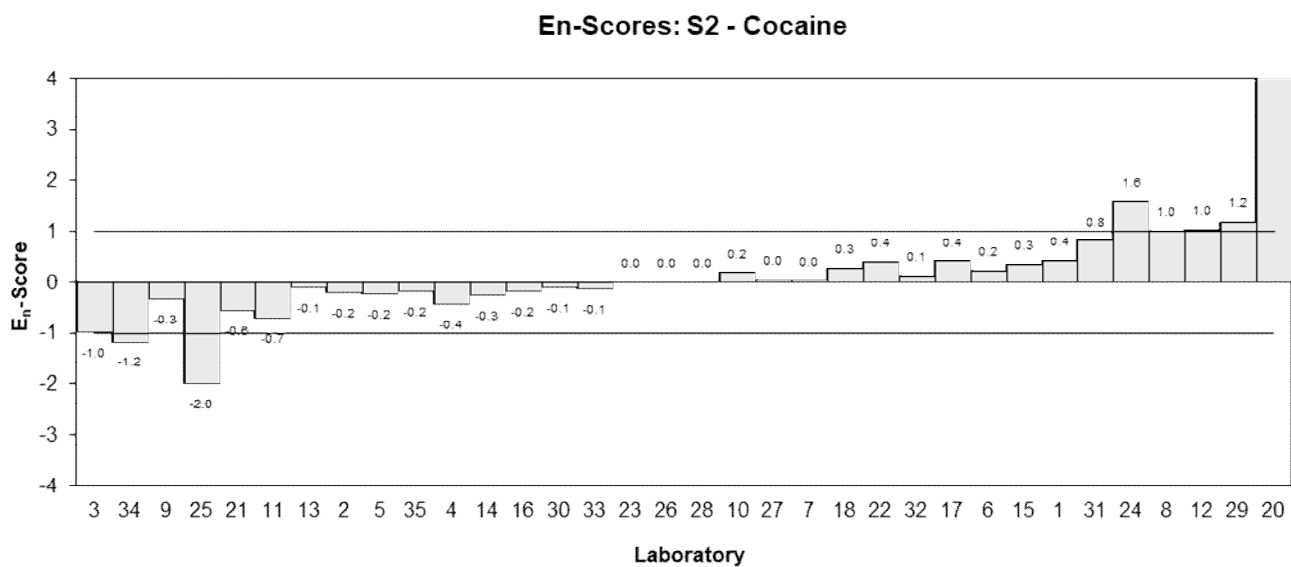
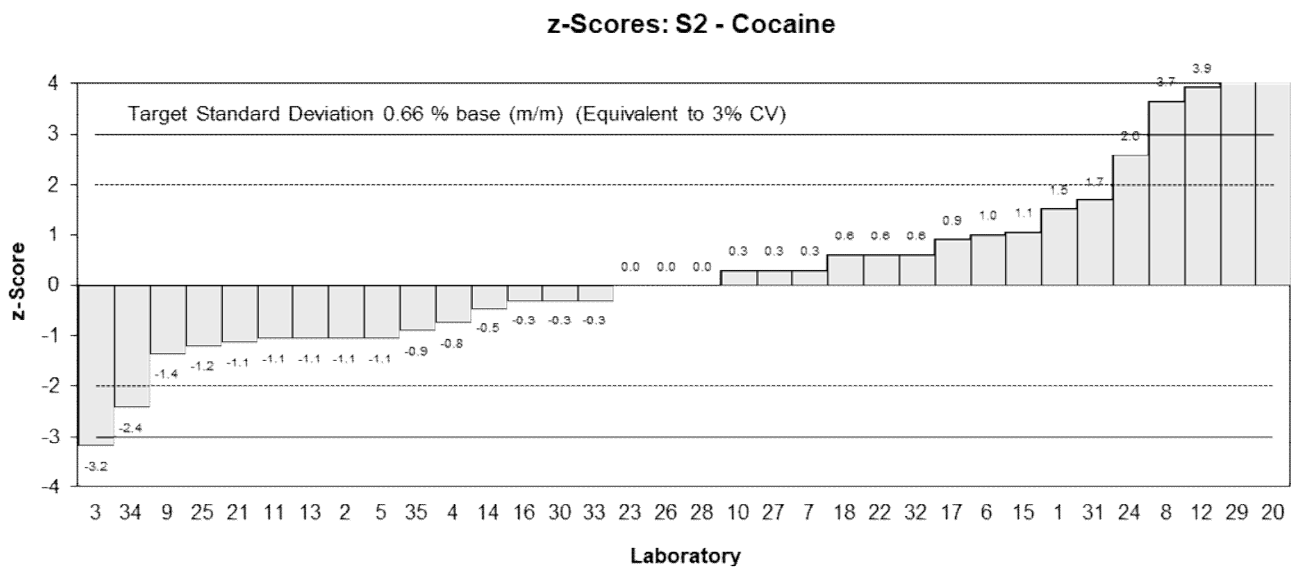
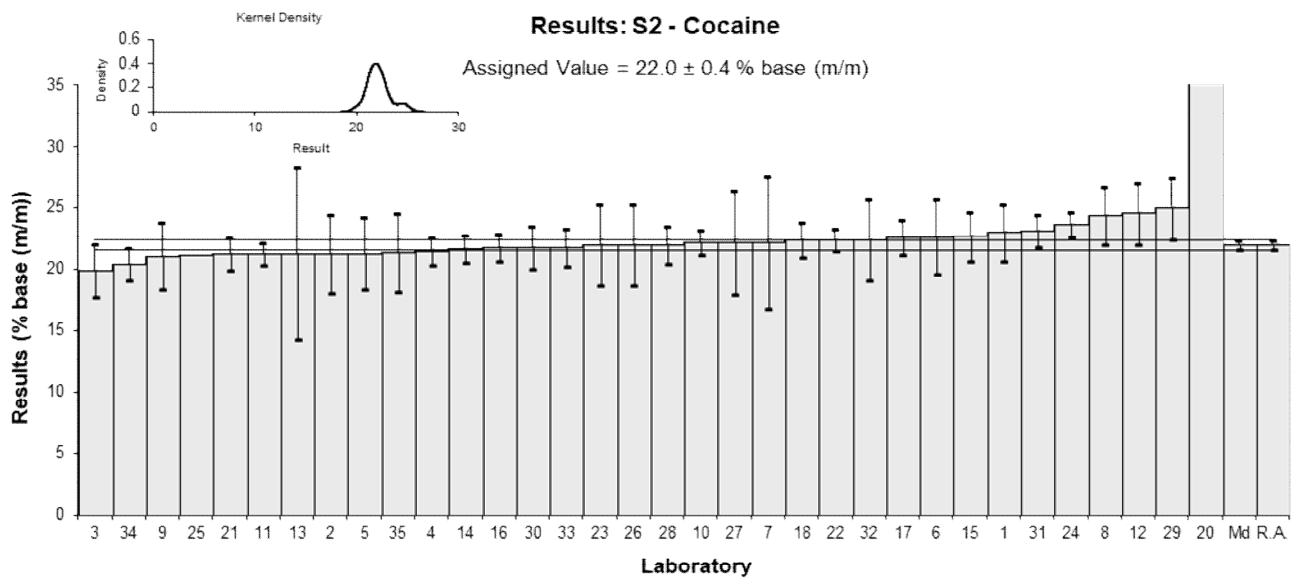


Figure 3

Table 8

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z-Score	E _n -Score
1	68	6.8	-1.68	-0.53
2	72.86	10.93	0.59	0.12
3	73.8	2.1	1.02	0.99
4	65.7	3.4	-2.75	-1.70
5	70.7	2.91	-0.42	-0.30
6	71.53	3	-0.03	-0.02
7	73	5.40	0.65	0.26
8	76.75	7.49	2.40	0.68
9	75.8	3.2	1.96	1.28
10	70.4	3.2	-0.56	-0.37
11	69.9	3.1	-0.79	-0.53
12	73.3	7.4	0.79	0.23
13	72.5	7	0.42	0.13
14	68.5	3.5	-1.44	-0.87
15	72.3	7	0.33	0.10
16	71.8	3.7	0.09	0.05
17	71.1	4.3	-0.23	-0.11
18	70.9	4.3	-0.33	-0.16
20*	21.6	0.4	-23.28	-62.02
21	70.62	4.24	-0.46	-0.23
22	71.0	1.1	-0.28	-0.46
23	71	10.7	-0.28	-0.06
24	72.4	4	0.37	0.20
25	65.6	NR	-2.79	-8.57
26	71	10.7	-0.28	-0.06
27	71.2	13.5	-0.19	-0.03
28	71	5.0	-0.28	-0.12
29	71	7.1	-0.28	-0.08
30	72.4	2.9	0.37	0.27
31	73.18	2.4	0.74	0.63
32	71.5	3.4	-0.05	-0.03
33	70.8	5.1	-0.37	-0.16
34	71.8	4.6	0.09	0.04
35	75.5	11.3	1.82	0.34

Statistics*

Assigned Value	71.6	0.7
Robust Average	71.6	0.7
Median	71.2	0.6
Mean	71.5	
N	33	
Max.	76.75	
Min.	65.6	
Robust SD	1.7	
Robust CV	2.4%	

* Result from Laboratory 20 was excluded from all statistical calculations (gross error).

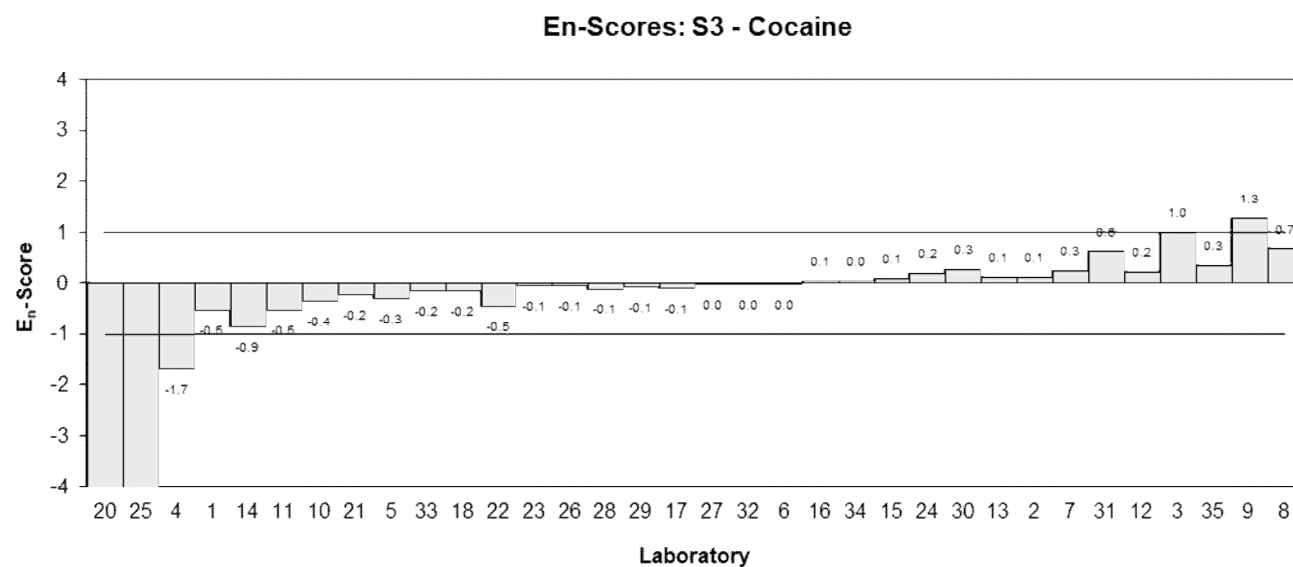
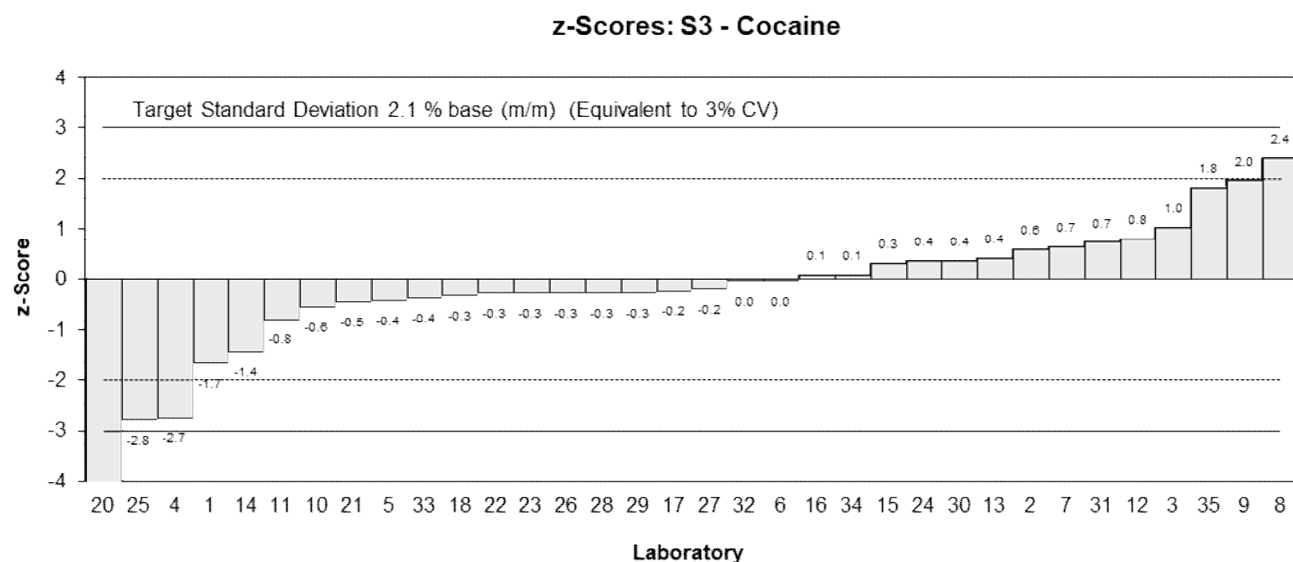
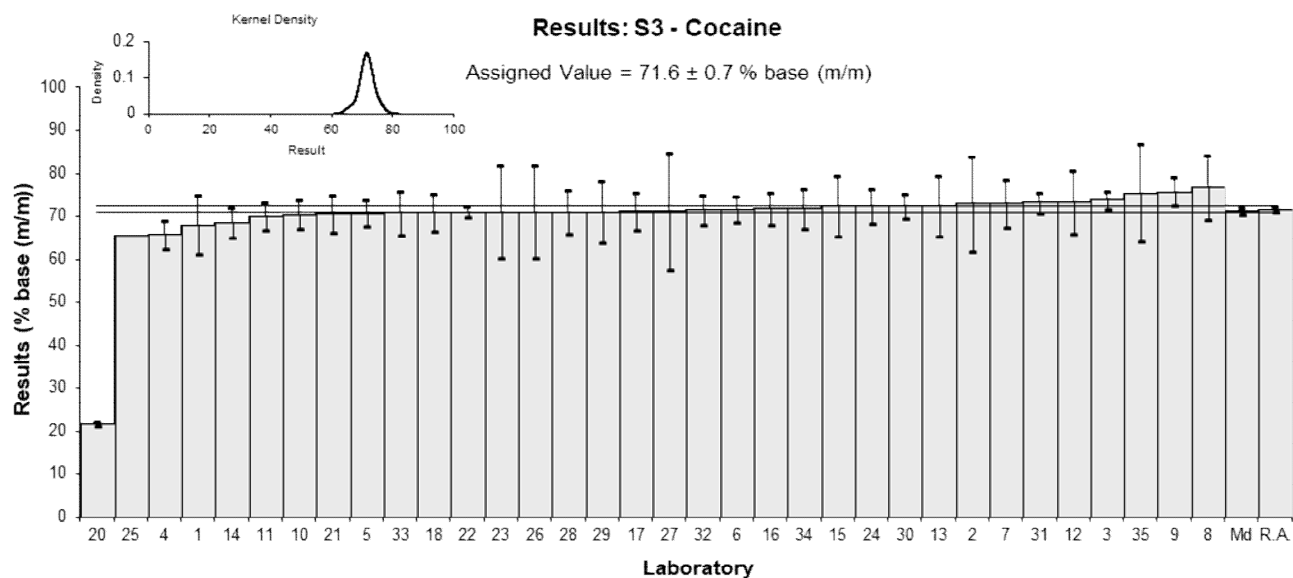


Figure 4

Table 9 Participants' Identification of Cutting Agents*

Lab. Code	Cutting Agents		
	S1	S2	S3
Preparation	Niacinamide	Glucodin	Acetylsalicylic acid
1	Nicotinamide	Dextrose	Acetylsalicylic acid
2			Benzocaine
3			acetyl salicylic acid
4	Nicotinamide		Salicylic acid
5	Niacinamide	None	None
6	Vitamin B3 (Nicotinamide)		
7			
8	Nicotinamide	Glucose	
9	Nicotinamide	Glucose (indicated)	
10	-	-	-
11	Nicotinamide		
12			
13	nothing	nothing	nothing
14	Nicotinamide	Glucose	Aspirin
15	nicotinamide	glucose	
16	Nicotinamide		Ethyl salicylate
17	Nicotinamide	-	-
18	Nicotinamide	-	-
20	Nicotinamide	Glucose	No diluent/adulterant detected with available instruments.
21		glucose	
22	Nicotinamide 36.2%	Glucose 67.5%	Acetylsalicylic acid
23	none	none	none
24	Nicotinamide		Salicylic acid+ Acetylsalicylic acid
25	Niacinamide		Acetylsalicylic acid
26	Nicotinamide	Sugars	Methyl Salicylate
27	nicotinamide	insoluble not identified	no
28	Nicotinamide	N/A	Salicylic acid
29	Niacinamide (aka nicotinamide)	Dextrose	None identified
30	Niacinamide		
31	nicotinamide		aspirin
32	niacinamide	glucose	aspirin
33			
34	Nicotinamide (not quantified)	Sugars (not quantified)	Acetylsalicylic acid (not quantified)
35	not analysed	not analysed	not analysed

* 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 13528:2015.⁵ Results less than 50% and greater than 150% of the robust average were removed before calculation of the assigned value, if applicable.^{3,4} The calculation of the expanded uncertainty for robust averages is presented in Appendix 1, with Sample S1 as an example.

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

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded MU associated with their results and the basis of this uncertainty estimate (Section 3.2).

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

Of 102 results, 99 (97%) were reported with an associated expanded MU. Laboratory **25** did not report uncertainties for their results; this laboratory reported that they were not accredited.

The magnitude of reported uncertainties was within the range 1.5% to 33% relative.

Of 99 expanded measurement uncertainties, 67 (68%) 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.

Laboratories with results having a satisfactory z-score but an unsatisfactory E_n -score are likely to have underestimated the expanded uncertainty associated with that result.

In some cases the results were reported with an inappropriate number of significant figures. 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 $53.31 \pm 3.20\%$, it is better to report this as $53.3 \pm 3.2\%$.⁸

6.3 z-Score

A target SD equivalent to 3% PCV was used to calculate z-scores. The CVs predicted by the Thompson-Horwitz equation,⁶ target SDs, and between laboratories CVs obtained in this study are presented in Table 10.

Table 10 Comparison of Target SDs, Thompson-Horwitz CVs and Between Laboratories CVs

Sample	Analyte	Assigned value (% base (m/m))	Thompson-Horwitz CV (%)	Target SD (as PCV) (%)	Between Laboratories CV (%)
S1	Cocaine	53.6	1.4	3	3.4
S2	Cocaine	22.0	2.1	3	4.1
S3	Cocaine	71.6	1.2	3	2.4

Of 102 results for which z-scores were calculated, 89 (87%) returned a z-score of $|z| \leq 2.0$, indicating a satisfactory performance.

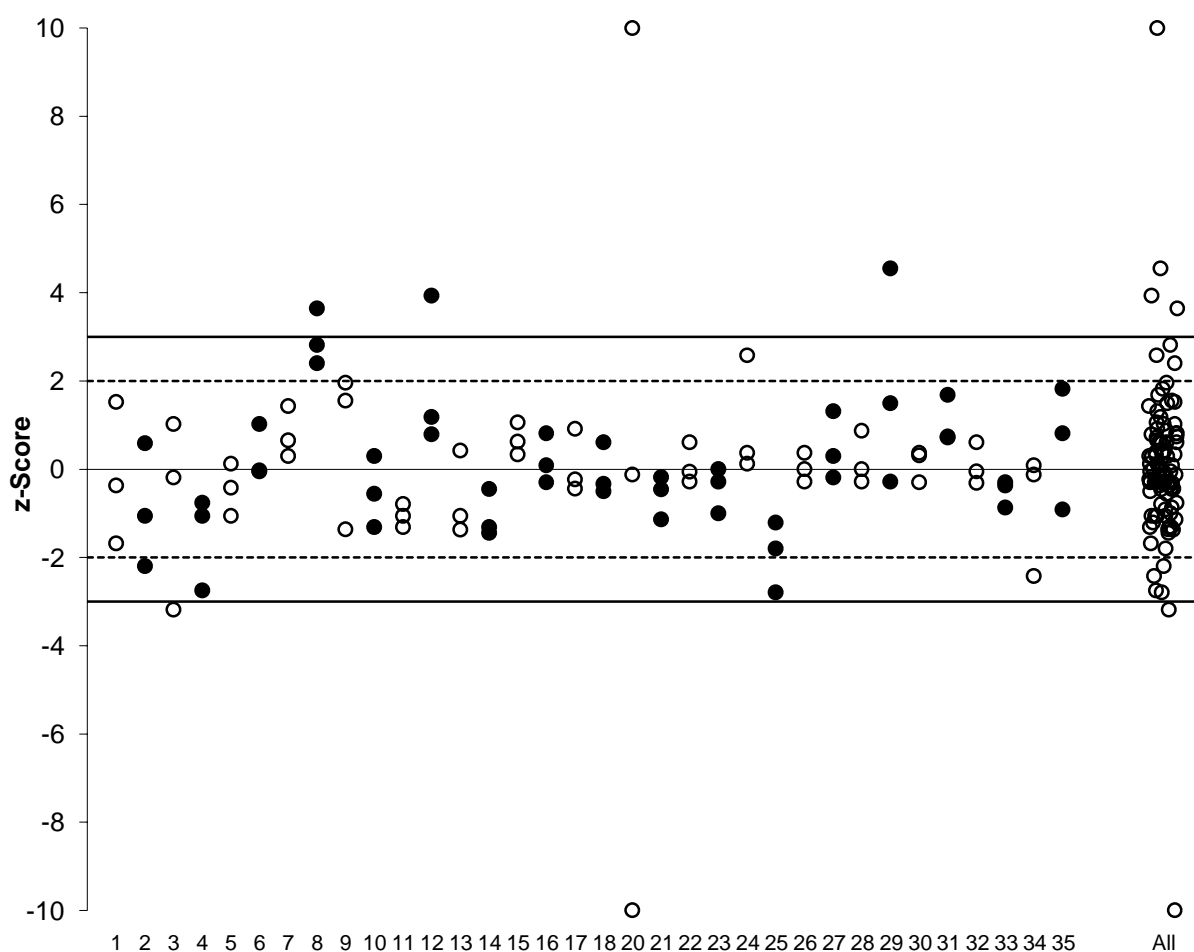
Twenty-four participants: **1, 5, 6, 7, 9, 10, 11, 13, 14, 15, 16, 17, 18, 21, 22, 23, 26, 27, 28, 30, 31, 32, 33** and **35** returned satisfactory z-scores for all three samples.

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

Laboratory **8** returned questionable or unsatisfactory z-scores for all three samples, with all reported results higher than the assigned value (positive bias). This participant may need to check if they have reported results as % salt (m/m) instead of % base (m/m), or investigate their source of bias.

Laboratory **20** has likely transposed their Sample S2 and S3 results; their results have been excluded from all statistical calculations for these two samples.

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



Scores greater than 10 or less than -10 have been plotted at 10 and -10 respectively.

Figure 5 z-Score Dispersal by Laboratory

6.4 E_n-Score

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

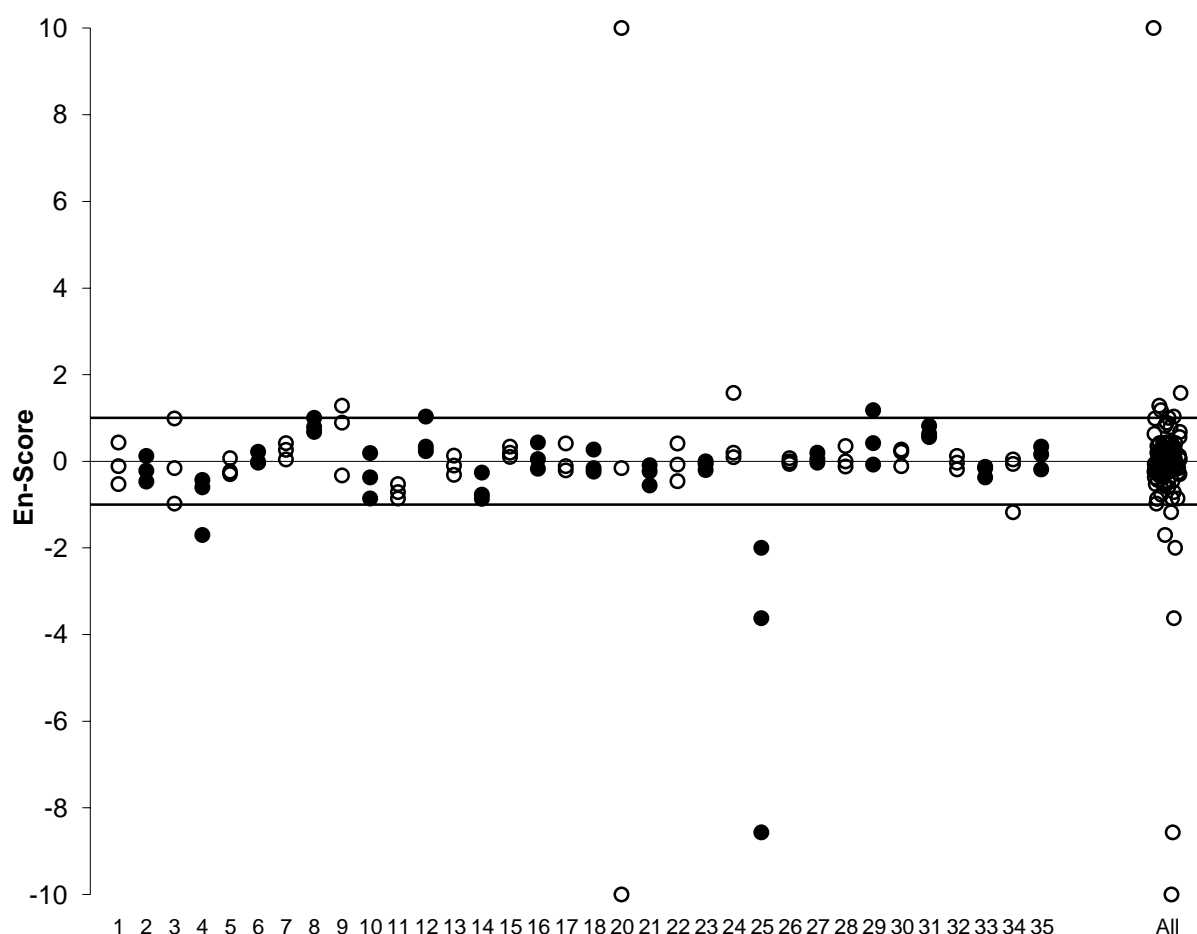
Of 102 results for which E_n-scores were calculated, 91 (89%) returned a satisfactory E_n-score of $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Twenty-six participants: **1, 2, 3, 5, 6, 7, 8, 10, 11, 13, 14, 15, 16, 17, 18, 21, 22, 23, 26, 27, 28, 30, 31, 32, 33** and **35** returned satisfactory E_n-scores for all three samples.

Eight participants returned at least one unsatisfactory E_n -score.

Laboratory **25** returned unsatisfactory E_n -scores for all three samples.

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



Scores greater than 10 or less than -10 have been plotted at 10 and -10 respectively.

Figure 6 E_n -score Dispersal by Laboratory

6.5 Identification of Cutting Agent

Samples were prepared using a seizure of cocaine hydrochloride, approximately 84% base (m/m) supplied by the Australian Federal Police. The study coordinator added niacinamide to Sample S1, glucodin to Sample S2, and acetylsalicylic acid to Sample S3.

Twenty-seven participants (74%) reported on the identity of the cutting agents in at least one sample (Table 9).

Laboratories **1**, **14**, **22** and **32** correctly identified all cutting agents in the test samples.

For Sample S1, 24 participants correctly identified that niacinamide was used as the cutting agent.

For Sample S2, 10 participants correctly identified that glucodin was used as the cutting agent, with a further 2 participants reporting “sugars”. One participant reported the cutting agent as an insoluble substance.

For Sample S3, 8 participants correctly identified that acetylsalicylic acid was used as the cutting agent. A number of participants reported other structurally similar compounds, such as salicylic acid, ethyl salicylate and methyl salicylate.

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 is presented in Table 11.

Table 11 Summary of Participants' Analyses

		Lab. Code
Accreditation	Yes to ISO/IEC 17025	1, 2, 3, 4, 5, 6, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 20, 23, 27, 28, 29, 32, 33, 34, 35
	Not Accredited / Not Reported	7, 21, 22, 24, 25, 26, 30, 31
Average Sample Mass Used per Analysis (mg)	5 – 10	7, 12, 15, 16, 24, 30
	11 – 30	1, 2, 5, 6, 8, 9, 10, 11, 14, 17, 18, 20, 21, 22, 26, 27, 28, 29, 31, 33, 34, 35
	31 – 50	4, 23, 25, 32
	51 – 100	3
	Not Reported	13
Conversion to Base?	Yes	3, 4, 5, 6, 9, 10, 15, 16, 21, 27, 31, 32, 33, 34
	No	1, 8, 11, 12, 13, 14, 17, 18, 20, 22, 24, 25, 26, 28, 29, 30, 35
	Not Reported	2, 7, 23
Instrument Used for Quantification	HPLC-DAD	2, 3, 5, 12, 15, 21, 24, 26, 28, 32
	HPLC-UV/Vis	7, 27
	HPLC-MS	13
	UPLC-DAD	1, 17, 18, 25, 29, 31, 33
	UPLC-MS/MS	23
	GC-FID	4, 6, 8, 9, 11, 14, 16, 20, 30, 34, 35
	QNMR	10, 22
Solvent	Acetonitrile/Water(/Other)	2, 5, 15, 21, 23, 26, 27, 33
	Methanol	1, 3, 4, 6, 8, 12, 17, 18, 24, 28, 29, 30
	Ethanol	9, 14, 16, 20, 25, 34
	Other / Not reported	7, 10, 11, 13, 22, 31, 32, 35
Sources of Calibration Standard	NMI Australia	1, 9, 10, 11, 12, 14, 25, 26, 29, 31, 33
	Lipomed	3, 7, 17, 18, 21, 27, 28, 30, 34
	LGC	8, 24
	Merck / Sigma Aldrich	2, 4, 13, 15, 22
	Johnson Matthey / MacFarlan Smith	5
	Other	6, 16, 20, 23, 32, 35

Plots of the z-score versus various methodology parameters are presented in Figures 7 to 11. Results excluded from all statistical calculations (gross errors) have not been plotted.

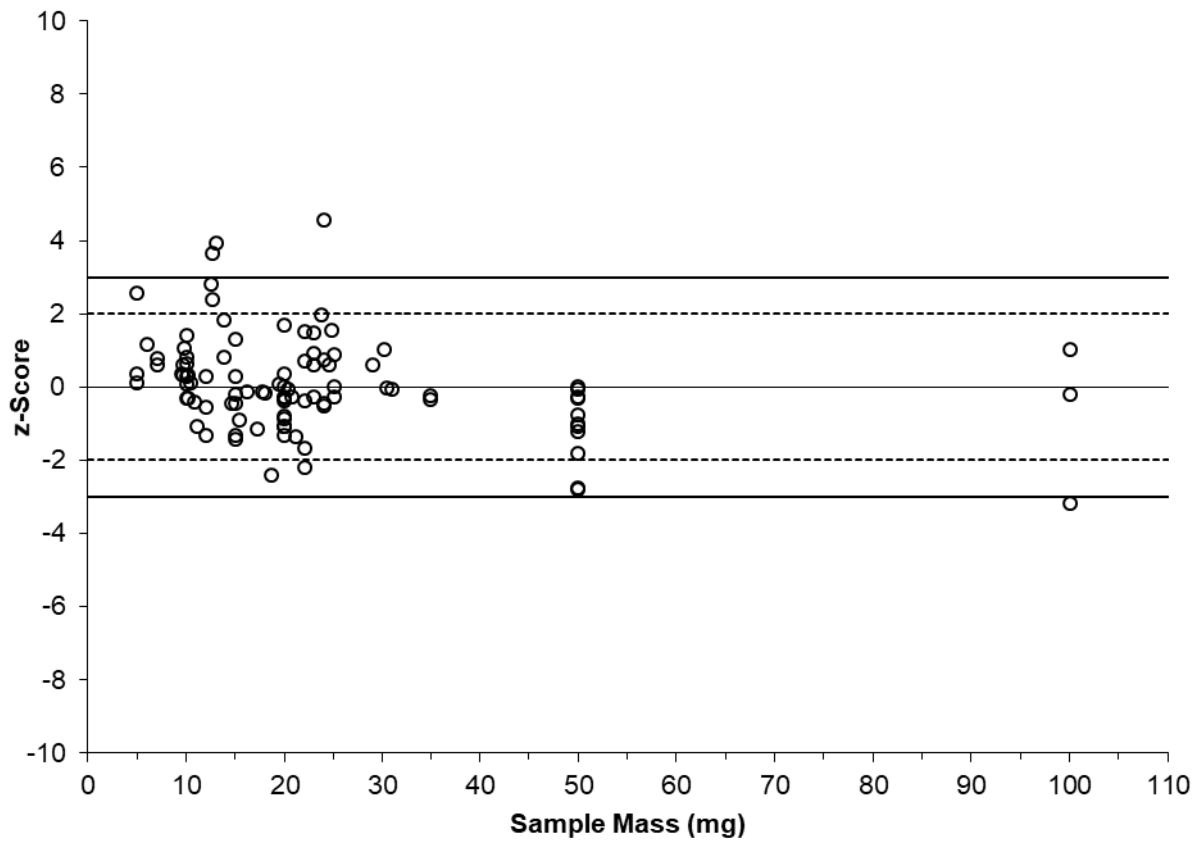


Figure 7 z-Score vs Sample Mass Used For Analysis

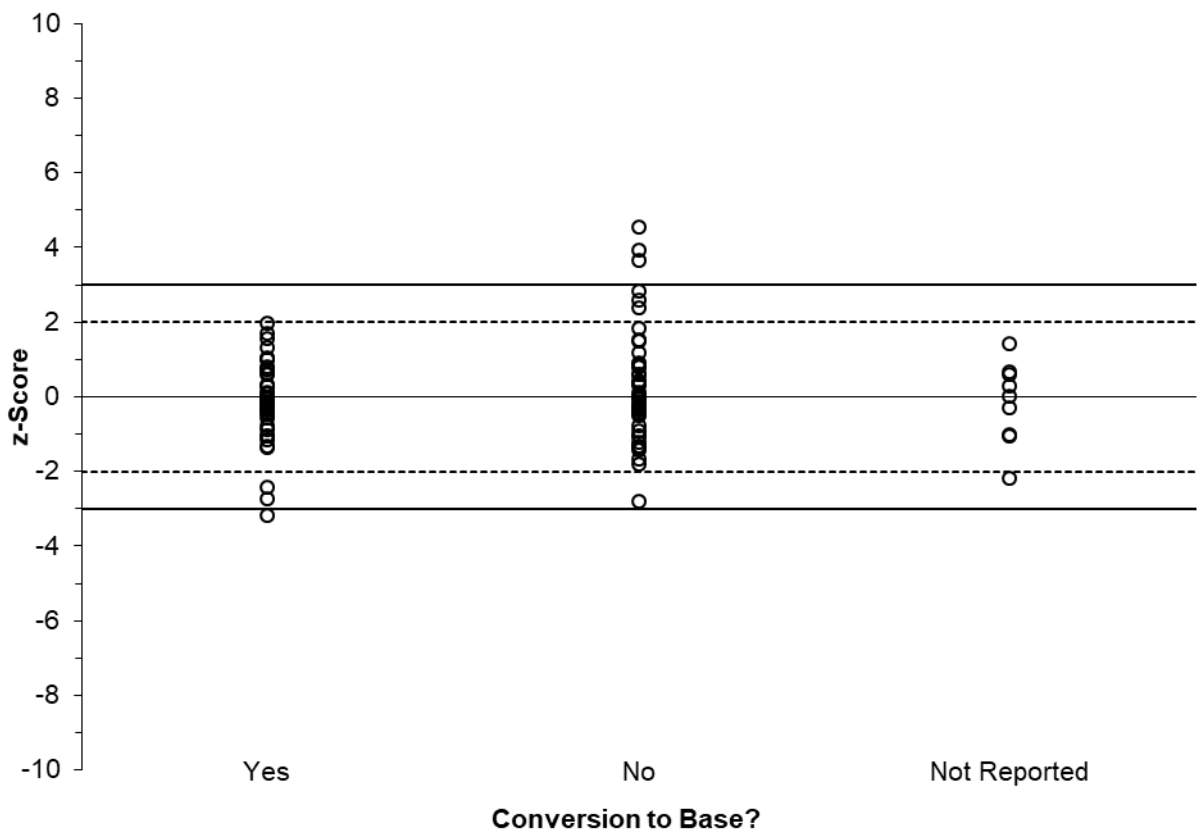


Figure 8 z-Score vs Sample Processing

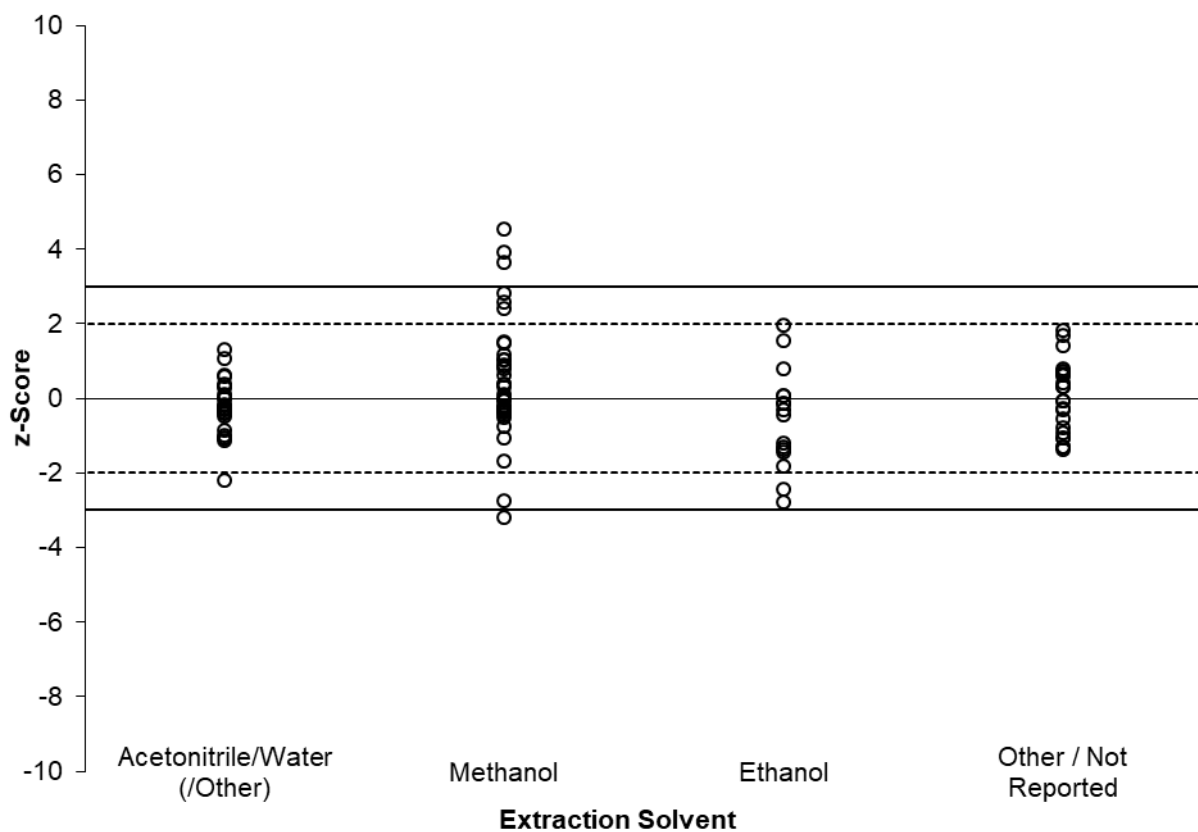


Figure 9 z-Score vs Extraction Solvent

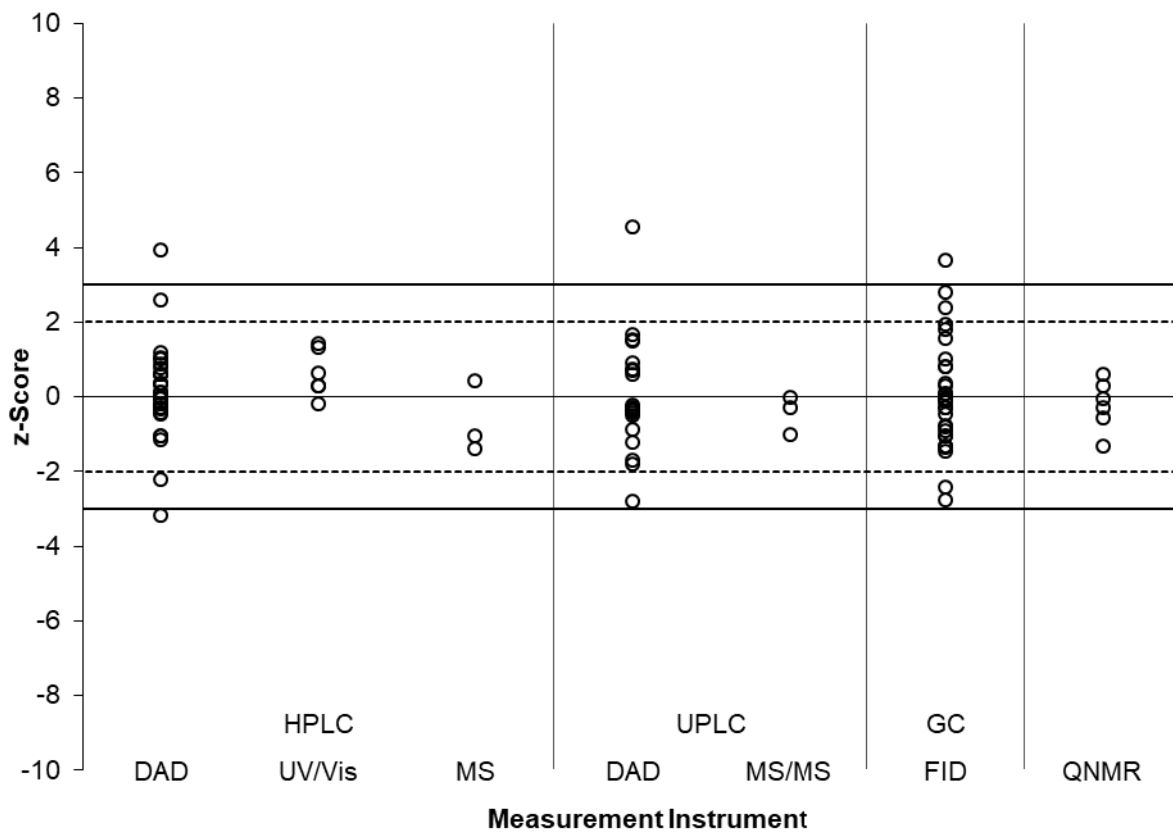


Figure 10 z-Score vs Measurement Instrument

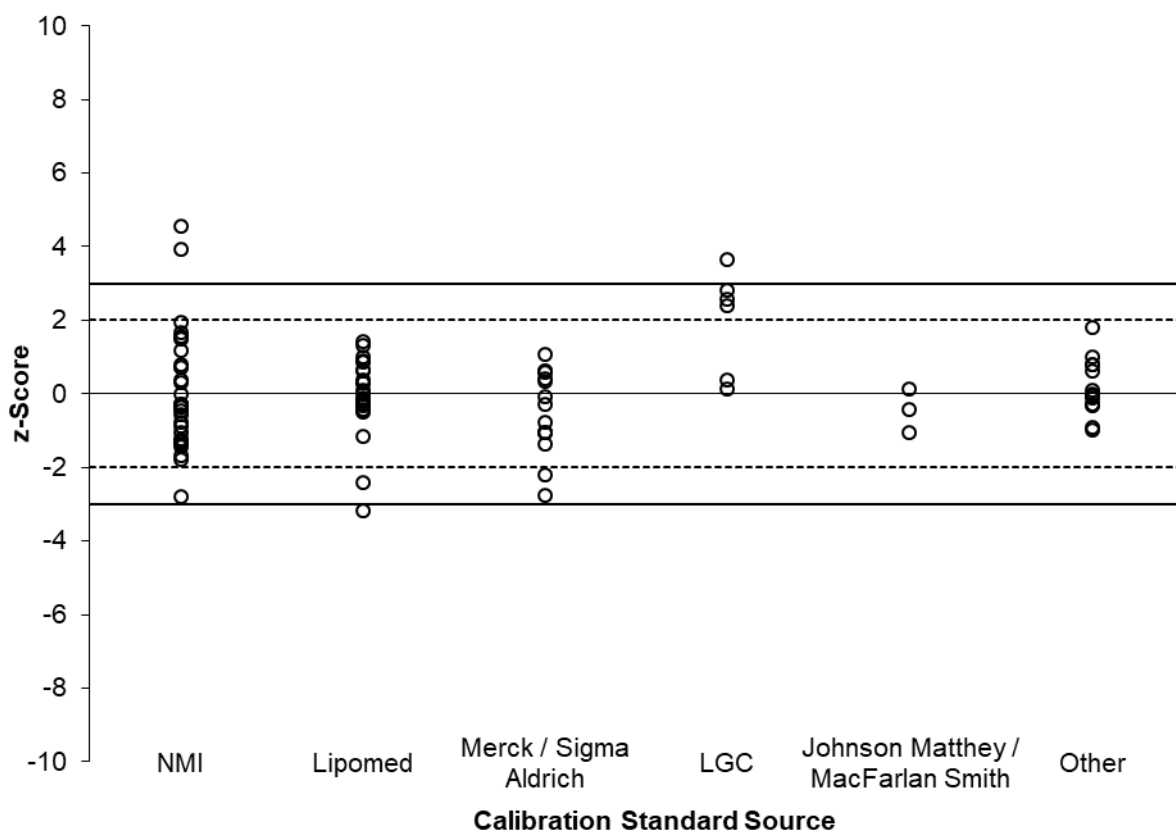


Figure 11 z-Score vs Source of Calibration Standard

As there were delays with sample delivery to some participants due to the ongoing international circumstances, the samples were analysed by participants over the course of approximately 3 months. No trend was found between when the samples were analysed and the results obtained (Figure 12).

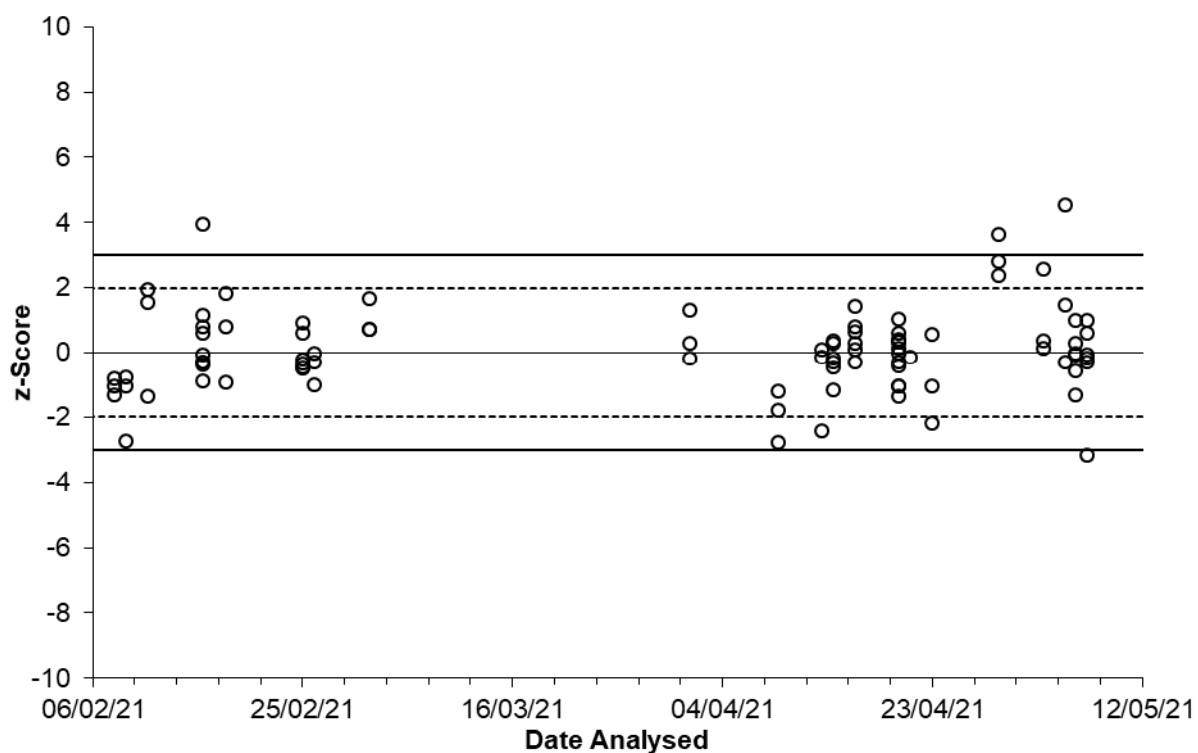


Figure 12 z-Score vs Sample Analysis Date

6.7 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 2011 to 2020 (last 10 studies) are presented in Figure 13. The average proportion of satisfactory z-scores and E_n -scores over this period is 80% and 83% respectively. While each PT study has a different group of participants, taken as a group, the performance over this period has improved.

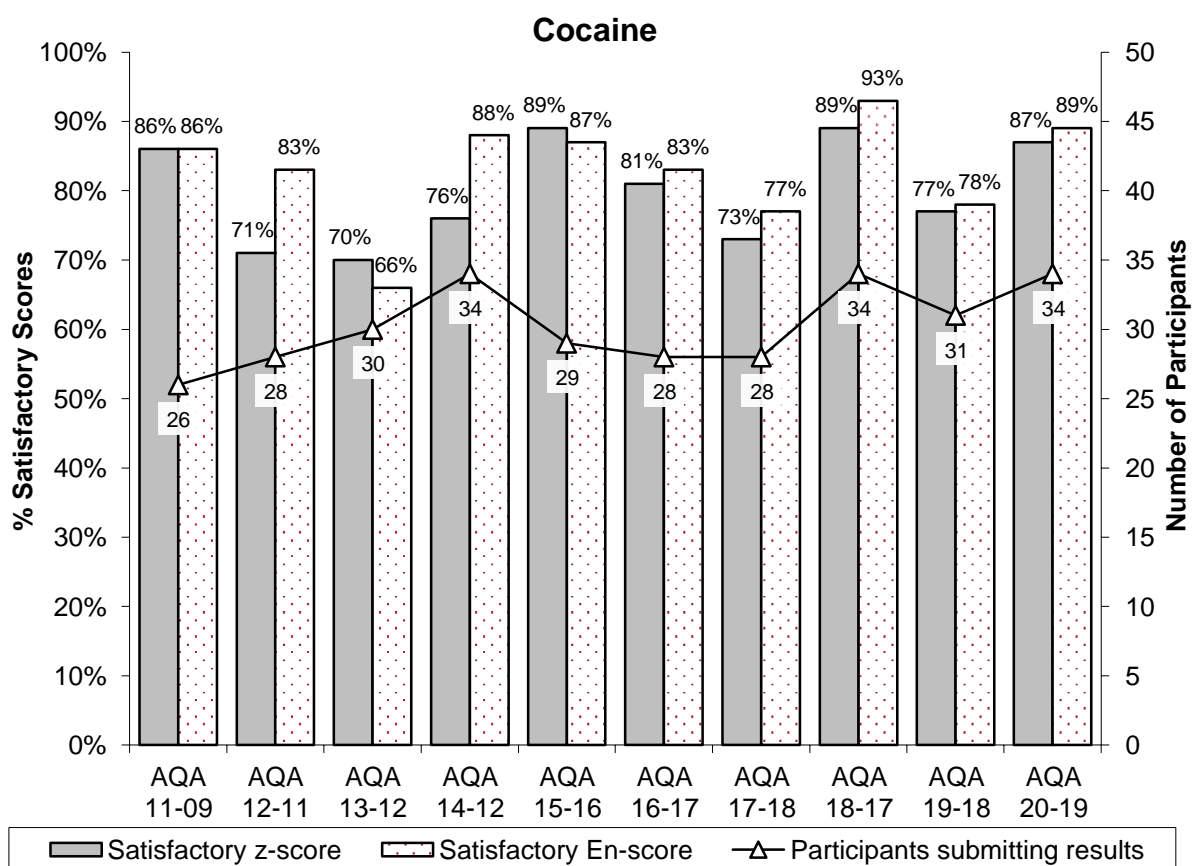


Figure 13 Summary of Participants' Performance in Cocaine PT Studies

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.

7 REFERENCES

- [1] ISO/IEC 17043:2010, *Conformity assessment – General requirements for proficiency testing*.
- [2] NMI, 2020, *Study Protocol for Proficiency Testing*, viewed May 2021, <https://www.industry.gov.au/sites/default/files/2020-10/cpt_study_protocol.pdf>
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- [7] ISO/IEC 17025:2017, *General requirements for the competence of testing and calibration laboratories*.
- [8] Eurachem/CITAC Guide CG 4, 2012, *Quantifying Uncertainty in Analytical Measurement*, 3rd Edition, viewed May 2021, <http://eurachem.org/images/stories/guides/pdf/quam2012_P1.pdf>

APPENDIX 1 – ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, Z-SCORE AND E_N-SCORE CALCULATIONS

A1.1 Robust Average and Associated Uncertainty

When the assigned value is calculated as the robust average using the procedure described in ISO 13528:2015,⁵ the uncertainty is estimated as:

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

where:

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

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

p is the number of results

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

A worked example for Sample S1 is set out below in Table 12.

Table 12 Uncertainty of Assigned Value for Sample S1 as % base (m/m)

No. results (p)	34
Robust average	53.6
$S_{\text{rob average}}$	1.8
$u_{\text{rob average}}$	0.4
k	2
$U_{\text{rob average}}$	0.8

Therefore, the assigned value for Sample S1 is $53.6 \pm 0.8\%$ 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.

A worked example is set out below in Table 13.

Table 13 z-Score and E_n-Score Calculation for Sample S1 Result Reported by Laboratory 1

Participant Results (% base (m/m))	Assigned Value (% base (m/m))	Target SD	z-Score	E _n -Score
53 ± 5.3	53.6 ± 0.8	3% as PCV, or: 0.03 × 53.6 = 1.608% base (m/m)	$z\text{-Score} = \frac{53-53.6}{1.608}$ = -0.37	$E_n\text{-Score} = \frac{53-53.6}{\sqrt{5.3^2+0.8^2}}$ = -0.11

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 Detector
FID	Flame Ionization Detector
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
Max.	Maximum value in a set of results
Md	Median
Min.	Minimum value in a set of results
MS	Mass Spectrometry
MS/MS	Tandem Mass Spectrometry
MU	Measurement Uncertainty
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 Test
QNMR	Quantitative Nuclear Magnetic Resonance
R.A.	Robust Average
RM	Reference Material
SD	Standard Deviation
SI	International System of Units
SS	Spiked Samples
UPLC	Ultra Performance Liquid Chromatography
UV	Ultraviolet
Vis	Visible

END OF REPORT