Australian Government
Department of Industry, Science and Resources

National Measurement Institute

Proficiency Test Final Report
AQA 22-12
Heroin

December 2022

ACKNOWLEDGMENTS

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

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

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

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SUMMARY

AQA 22-12 Heroin commenced in May 2022. Sets of heroin hydrochloride, each containing three test samples, were sent to 29 laboratories, with two laboratories requesting two sample sets to be analysed independently by different analysts. All participants returned results. Samples were prepared at the Sydney NMI laboratory using heroin hydrochloride samples supplied by the Australian Federal Police.

The assigned values in this study 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 participants measuring heroin in samples typical of a routine seizure.*

Participant performance was assessed by z-scores and E_n-scores.

Of 93 z-scores, 84 (90%) returned $|z| \leq 2.0$, indicating a satisfactory performance.

Of 93 E_n-scores, 86 (92%) 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**, **9**, **10**, **11**, **13**, **14**, **15**, **16**, **17**, **19**, **22**, **23**, **25**, **26**, **27**, **29**, **30** and **31** returned satisfactory z-scores and E_n-scores for all results.

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

Of 93 reported results, 90 (97%) were reported with an associated expanded measurement uncertainty. The magnitude of reported uncertainties was within the range 0.5% to 20% relative.

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

Sample S1 was cut with paracetamol and Sample S3 was cut with aspirin. Sample S2 was left uncut.

Thirty participants (97%) reported on the identity of at least one sample's cutting agent. Laboratories **1**, **2**, **3**, **7**, **8**, **9**, **11**, **12**, **13**, **14**, **15**, **16**, **17**, **18**, **20**, **21**, **22**, **24**, **25**, **27**, **30** and **31** correctly reported all cutting agents used.

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

The test samples produced for this study are homogeneous and well characterised. Surplus samples are available for purchase and can be used for quality control and for 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, vegetables and herbs, soil and water;
- petroleum hydrocarbons in soil and water;
- per- and polyfluoroalkyl substances in water, soil, biota and food;
- inorganic analytes in soil, water, filters, food and pharmaceuticals;
- controlled drug assay, drugs in wipes and clandestine laboratory; and
- allergens in food.

1.2 Study Aims

The aims of the study were to:

- assess the proficiency of participants measuring heroin 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 the 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 17043:2010,[1] and The International Harmonized Protocol for the 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 controlled drug study is within the scope of NMI's accreditation.

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitations sent	16/05/2022
Samples sent	18/07/2022
Results due	31/10/2022
Interim report	11/11/2022

The study timeline was extended to accommodate for sample delivery delays to some international participants.

2.2 Participation and Laboratory Code

Twenty-nine laboratories registered to participate, with two laboratories requesting two sets of samples each to be analysed independently by different analysts. All participants were assigned a confidential laboratory code number for this study. All participants submitted results.

2.3 Test Material Specification

Three test samples were prepared in May 2022. The starting material was two batches of heroin hydrochloride, supplied by the Australian Federal Police. The first batch was approximately 75% heroin base (m/m), and was used to prepare Sample S1. The second batch was approximately 78% heroin base (m/m), and was used to prepare Samples S2 and S3.

4-Acetamidophenol (paracetamol) and acetylsalicylic acid (aspirin) purchased from Sigma-Aldrich were used as cutting agents. Sample S1 was cut with paracetamol and Sample S3 was cut with aspirin; no additional cutting agents were added to Sample S2.

The heroin hydrochloride was ground and sieved through a 180 µm sieve. The cutting agents were processed similarly. Test samples were prepared by adding a known mass of sieved cutting agent (if applicable) to a known mass of sieved drug material, and then mixing this in a tumbler overnight. Portions of 150 mg of each of the test samples were weighed into labelled glass vials.

Sample S1 was prepared to contain approximately 22% heroin base (m/m).

Sample S2 was prepared to contain approximately 78% heroin base (m/m).

Sample S3 was prepared to contain approximately 34% heroin 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 (usually < 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, and no additional homogeneity testing was conducted in this proficiency study. Results returned by the participants also gave no reason to question the homogeneity of the test samples.

To assess stability of the samples, results returned by participants were compared to the date of analysis. The results gave no reason to question the stability of the test samples (Section 6.7).

2.5 Sample Dispatch and Receipt

Sets of three test samples, with each sample containing approximately 150 mg of material, were dispatched to participants on 18 July 2022.

The following items were also sent with the samples:

- a covering letter which included a description of the test samples and 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 heroin base by your routine test method.
- Identify and report the diluent(s) and/or adulterant(s) in all samples if this is within your normal scope of analysis.
- For each sample, report % m/m heroin as base. Report this figure as if reporting to a client.
- For each result, report an estimate of your expanded uncertainty as % m/m heroin as base.
- Give brief details of your:
  - basis of uncertainty estimate (e.g. uncertainty budget, repeatability precision)
  - analytical method (e.g. sample treatment, instrument type, calibration method)
  - reference standard (e.g. source, purity)

  as requested by the results sheet.
- Please complete the results sheet by Monday 29 August 2022 and return by email to jenny.xu@measurement.gov.au. Late results may not be included in the study report.

The results due date was extended to 31 October 2022 for all participants due to significant sample delivery delays to several international participants. A further extension was provided to one participant due to additional sample delivery delays affecting this participant only.

2.7 Interim Report

An interim report was emailed to all participants on 11 November 2022.

The interim report release was delayed to accommodate for the sample delivery delays as discussed above.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Methods Reported by Participants

Participants' reported test methods are presented in Table 1. Responses may have been 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	acetonitrile/water (86/14)	none	4	HPLC	DAD	NH2
2	Acetonitrile/Water	None	5	HPLC	UV	Kinetex 5u C18
3	chloroform	C28	4	GC	FID	HP-1
4	Methanol	none	1	HPLC	DAD	Luna 3 µm FPF(2) 100 Å 150x4.6 mm
5	HPLC Methanol	-	1	UPLC	DAD	Thermo Scientific Hypersil-5-ODS
6	HPLC Methanol	-	1	UPLC	DAD	Thermo Scientific Hypersil-5-ODS
7	Deuterium oxide	Maleic Acid		QNMR	Bruker AVIII 600	N/A
8	Ethanol	N/A	4	UHPLC	UV/Vis	Lichrocart 125-4 RP18
9	acetonitrile / water	none	1	HPLC	UV/Vis	Kromasil
10	Chloroform	Octacosane	5	GC	FID	HP5
11	Acetonitrile	Strychnine	6	GC	FID	HP1
12	Methanol	Methadone	4	GC	FID	RXI-5MS
13	acetonitrile/H20 (80/20)	External standard	2	HPLC	DAD	NH2
14	water, acetonitrile and tetrabutylammonium phosphate	none	6	HPLC	UV/Vis	NH2
15	ethanol: dimethylformamide (9:1)	tribenzylamine	6	GC	FID	HP1
16	chloroform	benzopinacolone	4	GC	FID	HP1
17	Methanol	none	5	HPLC	DAD	Kinetex C-18-XB
18	Ethanol	Propyl Paraben	8	UPLC	DAD	BEH Shield RP18
19	water/acetonitrile/ 2.5M sulphuric acid 90:10:1	None	3	HPLC	Diode Array	Shimpack XR-ODS
20	Ethanol	Eicosane	6	GC	FID	HP5
21	Acetonitrile	n/a	7	HPLC	DAD	Luna 3 µm C8(2) 100 x 2 mm

Lab. Code	Extraction Solvent	Internal Standard	Calib. Points	Technique	Detector	Column
22	Acetonitrile/ Methanol (95:5)	Pholcodine 1mg/ml	3	UPLC	PDA	ACQUITY C-18
23	ACN/MeOH/H2O	Analog of heroin	7	UPLC	MSMS	C-18 column
24	Acetonitrile	n/a	7	HPLC	DAD	Luna 3u C8(2) 100x2 mm
25	Acetonitrile:Water (75:25)	Benzocaine	3	UPLC	DAD	Acquity BEH C18
26	Methanol	Mepivacaine	4	UPLC	DAD	Kinetex EVO C18
27	Methanol	NO (External Standard)	7	UPLC	DAD	Poroshell 120 EC-C18 (4.6x150mm, 2.7 microns pore size)
28	Methanol	N/A	3	HPLC	PDA	Silica 15cm
29	Methanol	Diazepam	6	GC	FID	128-5512 DB-5ms
30	Ethanol	Triphenylaceto-phenone (TPAP)	3	GC	FID	HP1-MS
31	Acetonitrile, acetic acid, water	NO ISTD	4	HPLC	UV DAD	Poroshell 120 Ec-18

3.2 Details of Participant Calibration Standards

Participants' responses regarding their calibration standard used are presented in Table 2. Responses may have been modified so that the participant cannot be identified.

Table 2 Participant Calibration Standard

Lab. Code	Reference Standard	Purity (%)
1	Lipomed	99.912
2	British Pharmacopoeia	99.3
3	Siegfried	99.3
4	Lipomed	1 mg/mL
5	Lipomed	99.912 ± 0.018
6	Lipomed	99.912 ± 0.018
7	Sigma Aldrich	99.94
8	Lipomed	99.600 +/- 0.020
9	LIPOMED	99.6
10	NMI	99.3
11	NMI	99.3
12	LGC	1.02mg/ml
13	NMI	99.4
14	Lipomed	99.09
15	LGC	99.4

Lab. Code	Reference Standard	Purity (%)
16	in house synthesis	99.1
17	LGC	99.7
18	NMI	99.4
19	Sigma Aldrich	98.7
20	Alcaliber	99.7
21	NMI	99.4
22	NMI	99.3
23	Lipomed	100
24	NMI	99.4
25	NMI	99.3
26	Lipomed	99.600 +/- 0.020
27	LIPOMED	99.95
28	Johnson Matthey	99.4
29	Lipomed	99.1
30	NMI	99.3 +/- 1.3
31	Lipomed	99.6

3.3 Reported Basis of Participants' Measurement Uncertainty Estimates

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

Table 3 Reported Basis of Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
1	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM	Laboratory bias from PT studies	ISO 11352 and V03-110
2	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Matrix effects Standard purity	Eurachem/CITAC Guide
3	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM Duplicate analysis		Internal document based on Eurachem/CITAC GUIDE, ISO/GUM
4	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Laboratory bias from PT studies	Nordtest Report TR537
5	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
6	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
7	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Control samples - CRM Duplicate analysis	Masses and volumes Standard purity	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
8	Uncertainty Budget Method	Control samples - CRM Duplicate analysis	Instrument calibration Masses and volumes Standard purity	Internal SOP Document: "Uncertainty of Measurement in Drugs Analysis"
9	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - RM		ISO/GUM
10	Top Down - precision and estimates of the method and laboratory bias	Control samples - previously analysed police seizures Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Matrix effects Standard purity	Eurachem/CITAC Guide

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
11	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis	Instrument calibration Masses and volumes Matrix effects Laboratory bias from PT studies Recoveries of SS Standard purity	ISO/GUM
12	Standard deviation of replicate analyses multiplied by 2 or 3	Duplicate analysis	Masses and volumes	ISO/GUM
13		Duplicate analysis	Instrument calibration Standard purity	ISO/GUM
14	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM	Laboratory bias from PT studies	ISO/GUM
15	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM	Standard purity	
16	validation			
17	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - RM Duplicate analysis		Eurachem/CITAC Guide
18				
19	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - CRM Duplicate analysis	Instrument calibration Standard purity	ISO/GUM
20	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis	Matrix effects	ISO/GUM
21	Top Down - precision and estimates of the method and laboratory bias	Control samples - In-house control Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Standard purity	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
22	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
23	Top Down - precision and estimates of the method and laboratory bias			

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
24	Top Down - precision and estimates of the method and laboratory bias	Control samples - In-house control Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Standard purity	NATA GAG Estimating and Reporting Measurement Uncertainty of Chemical Test Results
25	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM Duplicate analysis	Standard purity	Eurachem/CITAC Guide
26	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis	Instrument calibration Homogeneity of sample Matrix effects Laboratory bias from PT studies Recoveries of SS Standard purity	EA-04/16 EA guidelines on the expression of uncertainty in quantitative testing.
27	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis	Instrument calibration Homogeneity of sample Masses and volumes Laboratory bias from PT studies Standard purity	Eurachem/CITAC Guide and Measurement Uncertainty for Weight Determinations in Seized Drug Analysis
28	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Instrument calibration Masses and volumes Recoveries of SS	ISO/GUM
29	Estimating Measurement Uncertainty by black box with pairs of values	Standard deviation from PT studies only		ISO/GUM (ENAC G 09 or ISO 21748)
30	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
31	Accuracy profile based on intermediate precision and repeatability	Control samples - Reference material		ISO 5725-2 and ISO/TS 21748

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

3.4 Participants' Comments

Participants were invited to comment on the samples, their methodology, the PT study in general and suggestions for future PT studies. Such feedback allows for the improvement of future studies. Participants' comments are presented in Table 4, along with the study coordinator's response where appropriate. Some responses may be modified so that the participant cannot be identified.

Table 4 Participant Comments

Lab. Code	Participants' Comments	Study Coordinator's Response
1	we would like to receive 3 samples of very different concentration for example 3%, 30% and 80%	A range of drug purities are selected to cater for the needs of different laboratories. In this study, the samples were prepared to contain approximately 22%, 34% and 78% heroin base (m/m).
3	Uncertainty: Expanded measurement uncertainty= 95% confidence level	
4	Uncertainty: Method validation is on-going. Expanded uncertainly is calculated with 10 % which is the requirement in the validation plan.	
7	Methodology: Simultaneous observation of analyte and IS peaks in 1H NMR spectrum acquired using QNMR conditions	
19	Analysis for Inert cutting agents not undertaken as part of standard analytical procedure for S1 and S2 samples Uncertainty: MuM determined from multiple injections of reference material. 3x(Std Dev/mean)x100. no analysis undertaken for inert bulking agents for S1 and S2 samples	
21	Acetylcodeine and monoacetylmorphine likely present due to the manufacture process for heroin	
27	Qualitative analysis was carried out by GC-MS	
28	Routine case samples would always be round down i.e. -3.92% for example: 21.7*0.9608 = 20.8 Uncertainty: The reported result (in routine case samples) is defined as the mean of the individual results multiplied by the uncertainty correction factor and is rounded down to the nearest whole number (unless <1%w/w). e.g. a mean result of 21.7% with an uncertainty correction value of 96.08% would give a reported result of 21.7*0.9608 = 20.8 therefore rounded down to 20%.	
30	Methodology: Dichloromethane (30mL/L of ethanol) was used to dissolve the TPAP	
31	Methodology: 0 ; 5 ; 20 ; 100 mg/l	

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 5 to 7 with summary statistics: robust average, median, mean, number of numerical 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.

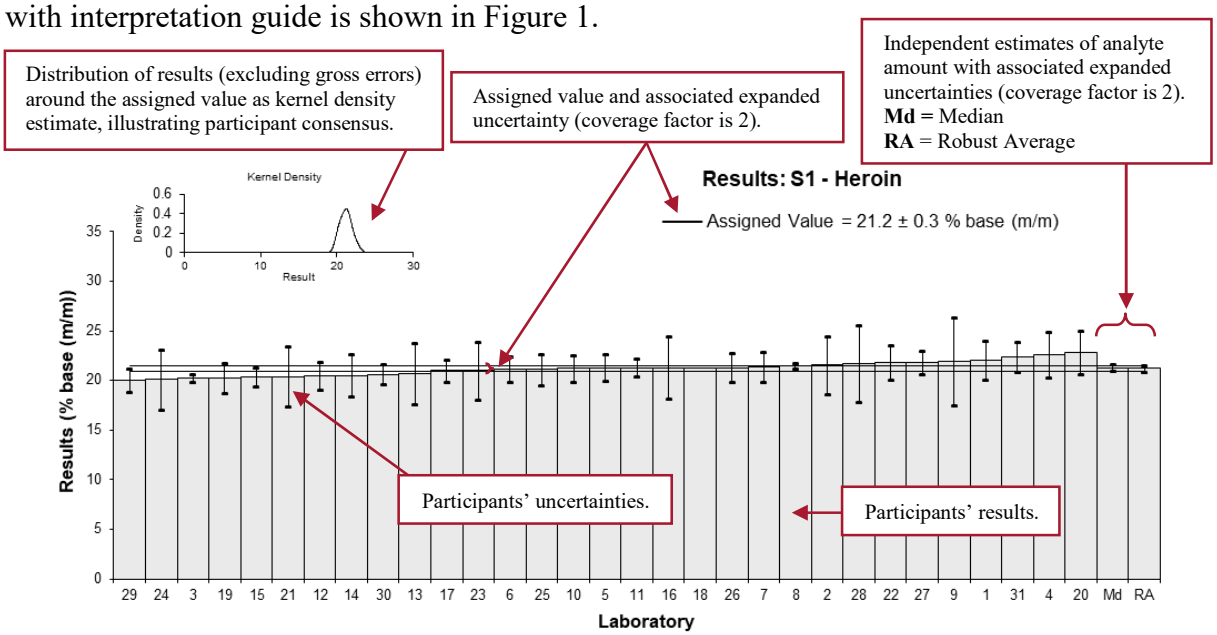


Figure 1 Guide to Presentation of Results

4.2 Outliers and Gross Errors

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

4.3 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 % heroin base (m/m) 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).

4.4 Robust Average and Robust Standard Deviation

The robust averages and associated expanded MUs, and robust SDs (a measure of the variability of participants' results), were calculated using the procedure described in ISO 13528:2022.[5]

4.5 Performance Coefficient of Variation (PCV)

The performance coefficient of variation (PCV) is a fixed 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, and is supported by mathematical models such as the Thompson-Horwitz equation.[6] It is important to note that this is a performance measure set by the study coordinator and it is not the robust CV of participants' results. 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.

4.6 Target Standard Deviation for Proficiency Assessment

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

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

4.7 *z*-Score

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

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

where:

z is z-score

χ is a participant's result

X is the assigned value

σ is the target standard deviation for proficiency assessment from Equation 1

For the absolute value of a z-score:

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

4.8 E_n-Score

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

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

where:

E_n is E_n-score

χ is a participant's result

X is the assigned value

U_χ is the expanded uncertainty of the participant's result

U_X is the expanded uncertainty of the assigned value

For the absolute value of an E_n-score:

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

4.9 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC 17025 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.[7]

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

5 TABLES AND FIGURES

Table 5

Sample Details

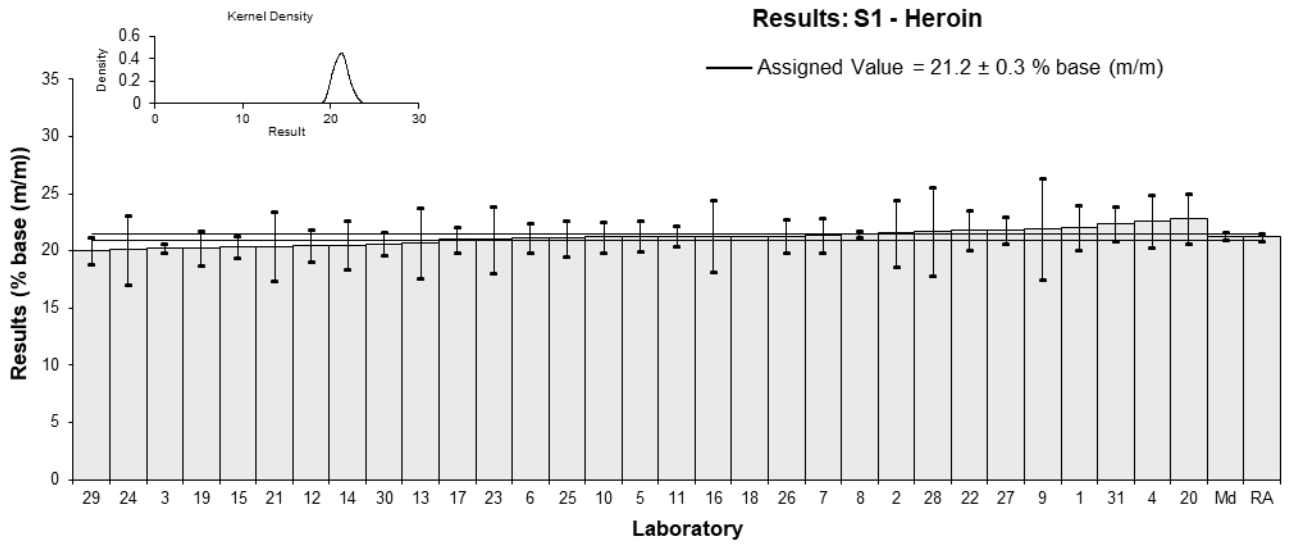
Sample No.	S1
Matrix	Powder
Analyte	Heroin
Unit	% base (m/m)

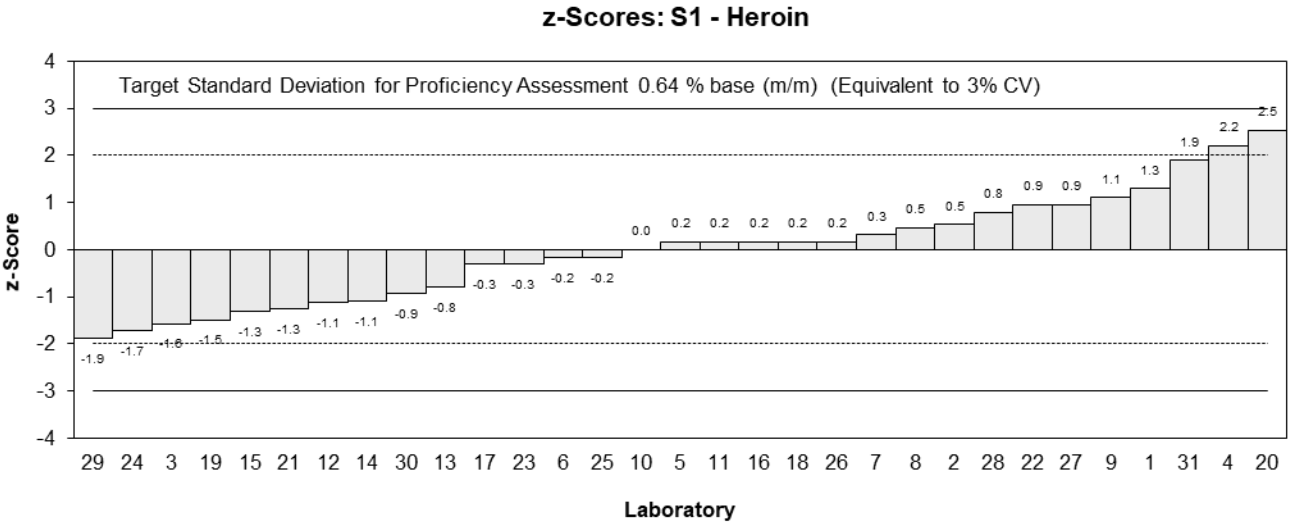
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	22.02	1.98	1.29	0.41
2	21.54	2.93	0.53	0.12
3	20.2	0.4	-1.57	-2.00
4	22.6	2.3	2.20	0.60
5	21.3	1.3	0.16	0.07
6	21.1	1.3	-0.16	-0.07
7	21.4	1.5	0.31	0.13
8	21.5	0.3	0.47	0.71
9	21.9	4.4	1.10	0.16
10	21.2	1.3	0.00	0.00
11	21.3	0.9	0.16	0.11
12	20.48	1.36	-1.13	-0.52
13	20.7	3.1	-0.79	-0.16
14	20.5	2.1	-1.10	-0.33
15	20.36	1	-1.32	-0.80
16	21.3	3.1	0.16	0.03
17	21	1.1	-0.31	-0.18
18	21.3	NR	0.16	0.33
19	20.25	1.54	-1.49	-0.61
20	22.8	2.2	2.52	0.72
21	20.4	3	-1.26	-0.27
22	21.8	1.70	0.94	0.35
23	21	2.9	-0.31	-0.07
24	20.1	3	-1.73	-0.36
25	21.1	1.6	-0.16	-0.06
26	21.3	1.5	0.16	0.07
27	21.8	1.2	0.94	0.49
28	21.7	3.92	0.79	0.13
29	20.0	1.2	-1.89	-0.97
30	20.6	1.0	-0.94	-0.57
31	22.4	1.49	1.89	0.79

Statistics

Assigned Value	21.2	0.3
Robust Average	21.2	0.3
Median	21.3	0.3
Mean	21.2	
N	31	
Max	22.8	
Min	20	
Robust SD	0.77	
Robust CV	3.6%	


Results: S1 - Heroin
Assigned Value = 21.2 ± 0.3 % base (m/m)
Kernel Density
Density
Result
Results (% base (m/m))
Laboratory
29 24 3 19 15 21 12 14 30 13 17 23 6 25 10 5 11 16 18 26 7 8 2 28 22 27 9 1 31 4 20 Md RA


z-Scores: S1 - Heroin
Target Standard Deviation for Proficiency Assessment 0.64 % base (m/m) (Equivalent to 3% CV)
z-Score
Laboratory
29 24 3 19 15 21 12 14 30 13 17 23 6 25 10 5 11 16 18 26 7 8 2 28 22 27 9 1 31 4 20

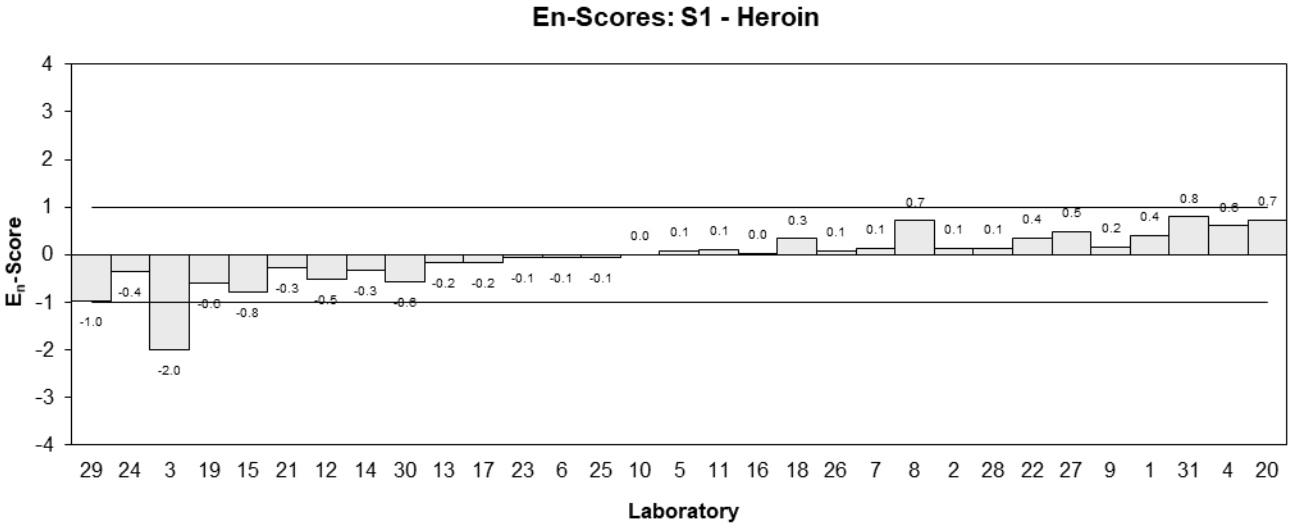

En-Scores: S1 - Heroin
En-Score
Laboratory
29 24 3 19 15 21 12 14 30 13 17 23 6 25 10 5 11 16 18 26 7 8 2 28 22 27 9 1 31 4 20

Figure 2

Table 6

Sample Details

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

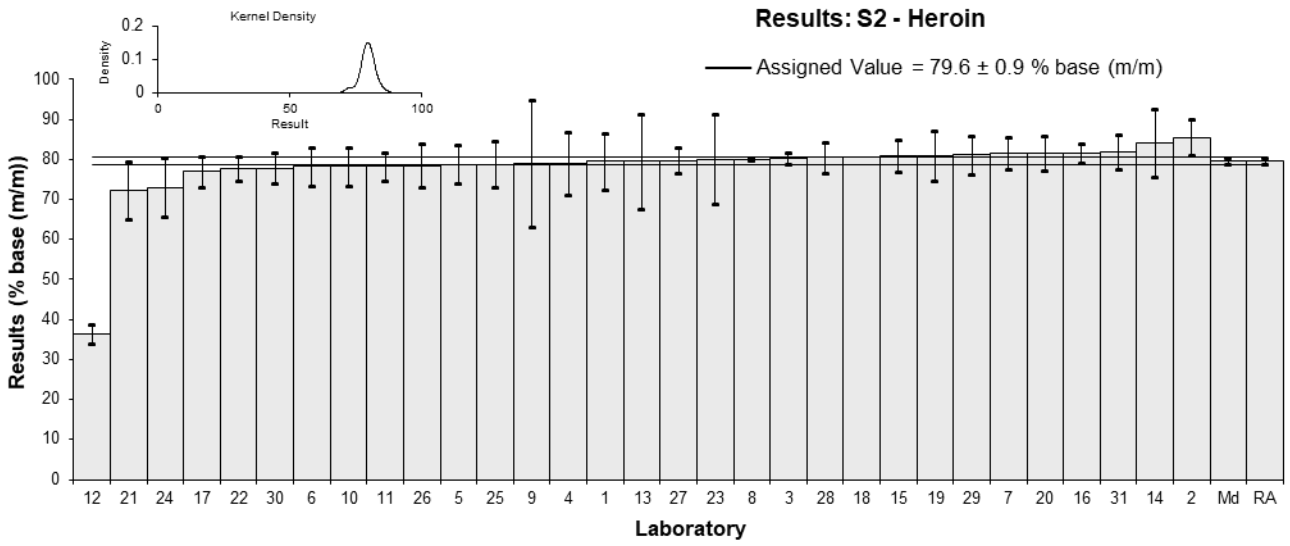
Participant Results

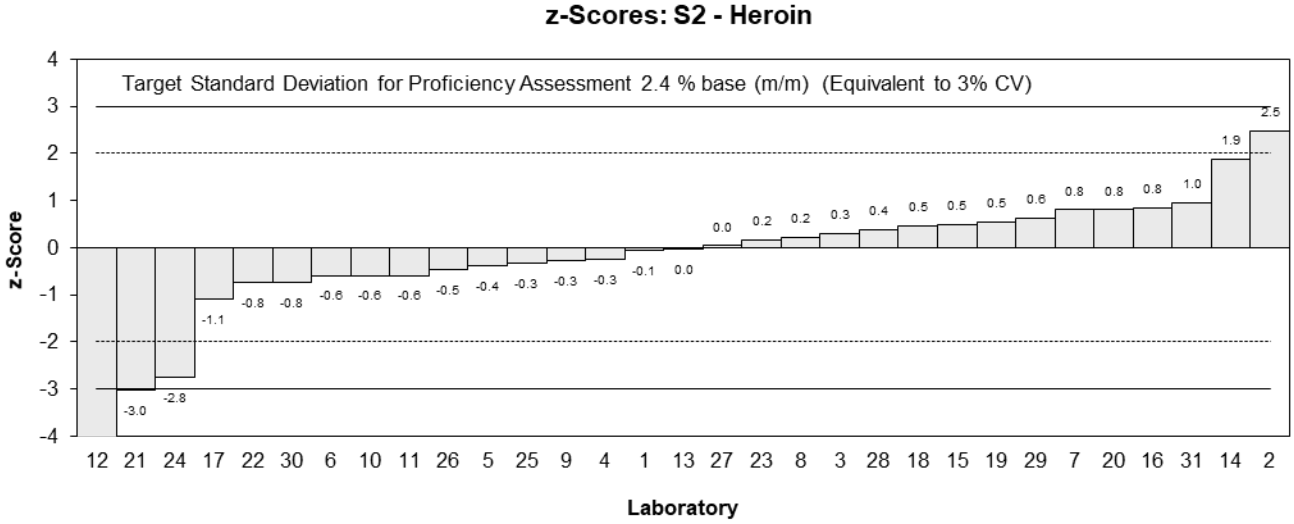
Lab. Code	Result	Uncertainty	z	E_n
1	79.48	7.15	-0.05	-0.02
2	85.52	4.36	2.48	1.33
3	80.3	1.5	0.29	0.40
4	79.0	7.9	-0.25	-0.08
5	78.7	4.8	-0.38	-0.18
6	78.2	4.7	-0.59	-0.29
7	81.5	4	0.80	0.46
8	80.1	0.4	0.21	0.51
9	78.9	15.8	-0.29	-0.04
10	78.2	4.8	-0.59	-0.29
11	78.2	3.4	-0.59	-0.40
12**	36.32	2.41	-18.12	-16.82
13	79.5	11.9	-0.04	-0.01
14	84.1	8.4	1.88	0.53
15	80.77	4	0.49	0.29
16	81.6	2.4	0.84	0.78
17	77	3.9	-1.09	-0.65
18	80.7	NR	0.46	1.22
19	80.88	6.15	0.54	0.21
20	81.5	4.4	0.80	0.42
21	72.4	7.2	-3.02	-0.99
22	77.8	3.10	-0.75	-0.56
23	80	11.2	0.17	0.04
24	73	7.3	-2.76	-0.90
25	78.8	5.7	-0.34	-0.14
26	78.5	5.5	-0.46	-0.20
27	79.7	3.2	0.04	0.03
28	80.5	3.92	0.38	0.22
29	81.1	4.9	0.63	0.30
30	77.8	3.8	-0.75	-0.46
31	81.9	4.3	0.96	0.52

** Gross Error, see Section 4.2

Statistics

Assigned Value	79.6	0.9
Robust Average	79.6	0.9
Median	79.6	0.9
Mean	79.5	
N	30	
Max	85.52	
Min	72.4	
Robust SD	1.9	
Robust CV	2.4%	


Results: S2 - Heroin
Assigned Value = 79.6 ± 0.9 % base (m/m)
Kernel Density
Density
Result
Results (% base (m/m))
Laboratory
12 21 24 17 22 30 6 10 11 26 5 25 9 4 1 13 27 23 8 3 28 18 15 19 29 7 20 16 31 14 2 Md RA


z-Scores: S2 - Heroin
Target Standard Deviation for Proficiency Assessment 2.4 % base (m/m) (Equivalent to 3% CV)
z-Score
Laboratory
12 21 24 17 22 30 6 10 11 26 5 25 9 4 1 13 27 23 8 3 28 18 15 19 29 7 20 16 31 14 2

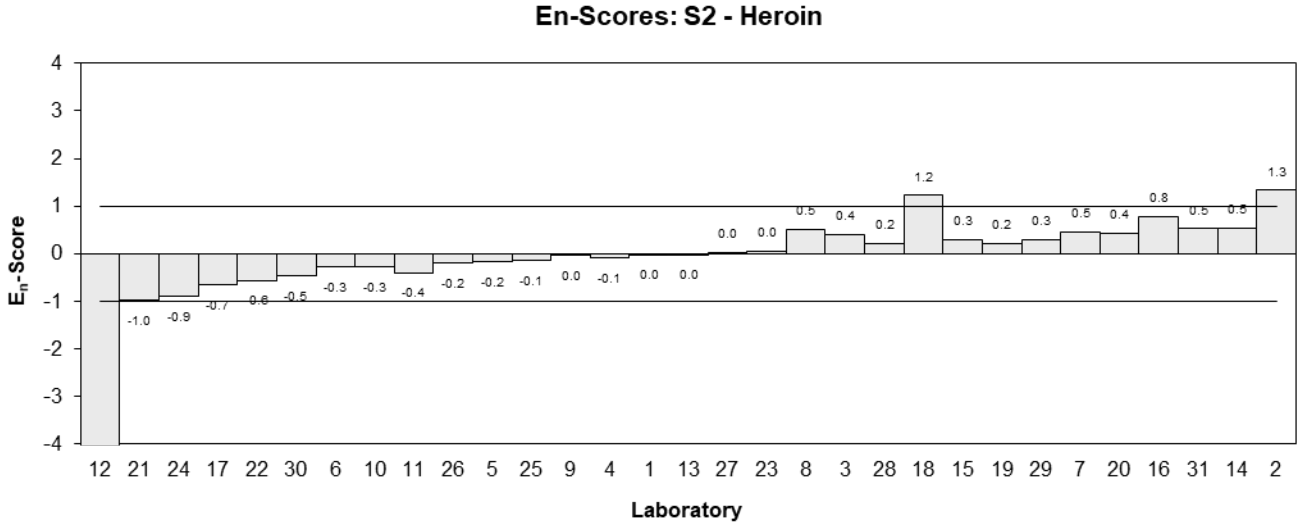

En-Scores: S2 - Heroin
En-Score
Laboratory
12 21 24 17 22 30 6 10 11 26 5 25 9 4 1 13 27 23 8 3 28 18 15 19 29 7 20 16 31 14 2

Figure 3

Table 7

Sample Details

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

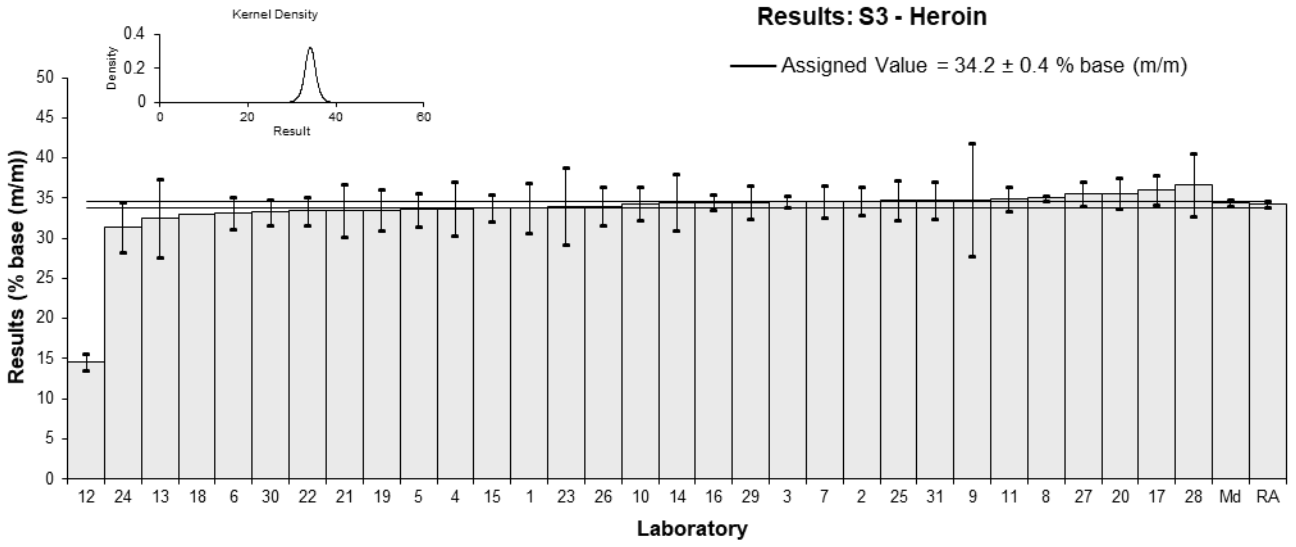
Participant Results

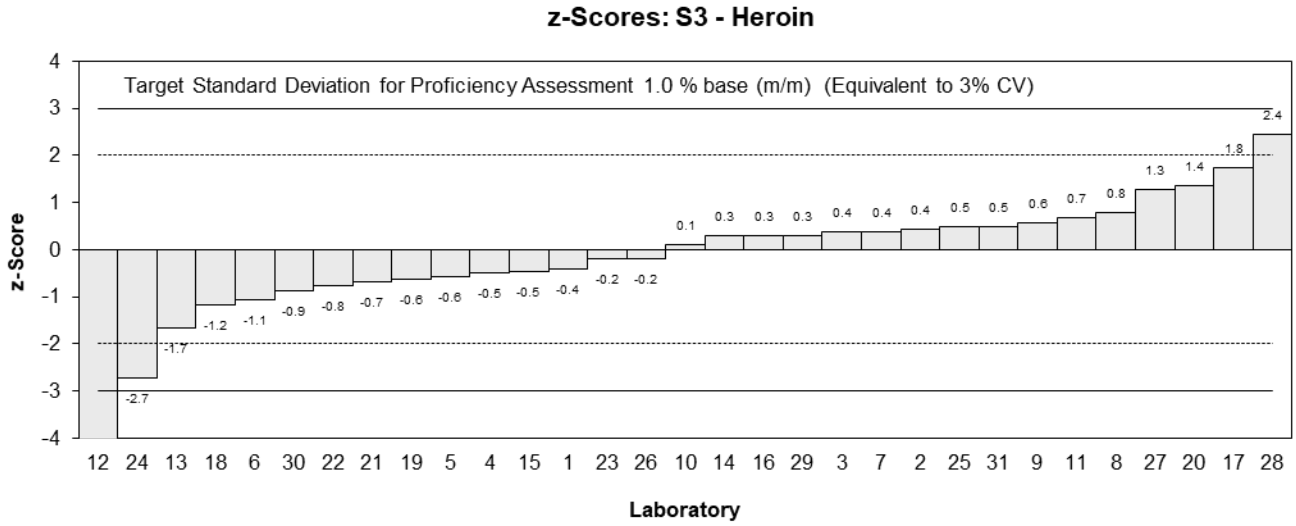
Lab. Code	Result	Uncertainty	z	E_n
1	33.78	3.04	-0.41	-0.14
2	34.65	1.77	0.44	0.25
3	34.6	0.7	0.39	0.50
4	33.7	3.4	-0.49	-0.15
5	33.6	2.1	-0.58	-0.28
6	33.1	2	-1.07	-0.54
7	34.6	2	0.39	0.20
8	35.0	0.3	0.78	1.60
9	34.8	7	0.58	0.09
10	34.3	2.1	0.10	0.05
11	34.9	1.5	0.68	0.45
12**	14.6	0.97	-19.10	-18.68
13	32.5	4.9	-1.66	-0.35
14	34.5	3.5	0.29	0.09
15	33.73	1.7	-0.46	-0.27
16	34.5	1.0	0.29	0.28
17	36	1.8	1.75	0.98
18	33.0	NR	-1.17	-3.00
19	33.54	2.55	-0.64	-0.26
20	35.6	1.9	1.36	0.72
21	33.5	3.3	-0.68	-0.21
22	33.4	1.80	-0.78	-0.43
23	34	4.8	-0.19	-0.04
24	31.4	3.1	-2.73	-0.90
25	34.7	2.5	0.49	0.20
26	34	2.4	-0.19	-0.08
27	35.5	1.5	1.27	0.84
28	36.7	3.92	2.44	0.63
29	34.5	2.1	0.29	0.14
30	33.3	1.6	-0.88	-0.55
31	34.7	2.3	0.49	0.21

** Gross Error, see Section 4.2

Statistics

Assigned Value	34.2	0.4
Robust Average	34.2	0.4
Median	34.4	0.4
Mean	34.2	
N	30	
Max	36.7	
Min	31.4	
Robust SD	0.96	
Robust CV	2.8%	


Results: S3 - Heroin
Kernel Density
Density
Result
Assigned Value = 34.2 ± 0.4 % base (m/m)
Results (% base (m/m))
12 24 13 18 6 30 22 21 19 5 4 15 1 23 26 10 14 16 29 3 7 2 25 31 9 11 8 27 20 17 28 Md RA
Laboratory


z-Scores: S3 - Heroin
Target Standard Deviation for Proficiency Assessment 1.0 % base (m/m) (Equivalent to 3% CV)
z-Score
12 24 13 18 6 30 22 21 19 5 4 15 1 23 26 10 14 16 29 3 7 2 25 31 9 11 8 27 20 17 28
Laboratory

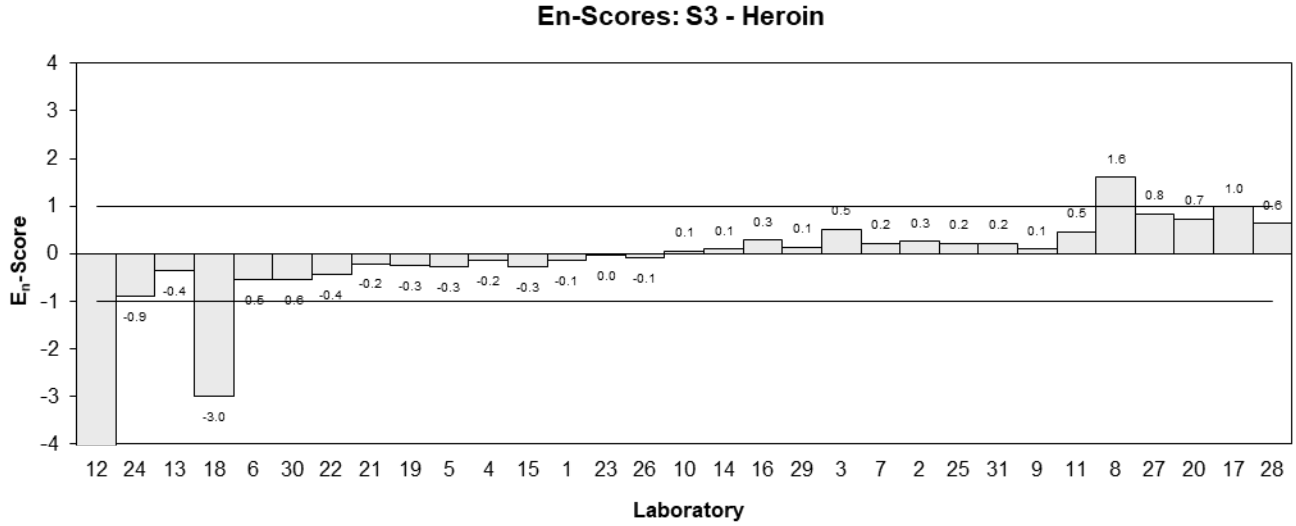

En-Scores: S3 - Heroin
En-Score
12 24 13 18 6 30 22 21 19 5 4 15 1 23 26 10 14 16 29 3 7 2 25 31 9 11 8 27 20 17 28
Laboratory

Figure 4

Table 8 Reported Cutting Agents*

Lab. Code	Cutting Agents		
	S1	S2	S3
Preparation	4-Acetamidophenol (Paracetamol)	N/A	Acetylsalicylic Acid (Aspirin)
1	paracetamol, acetyl codeine, monoacetyl morphine	acetyl codeine, monoacetyl morphine	aspirin, acetyl codeine, monoacetyl morphine
2	Paracetamol		Aspirin
3	acetaminophen	-	aspirin (acetylsalicylic acid)
4			
5	Paracetamol	-	-
6	Paracetamol	-	-
7	Paracetamol	uncut	Acetylsalicylic acid
8	Paracetamol	Not detected	Aspirin
9	Acetaminophen		Acetylsalicylic acid
10	paracetamol		
11	Acetaminophen		Acetylsalicylic acid
12	Paracetamol	None	Aspirin
13	acetaminophen	/	aspirin
14	Paracetamol	none	Aspirin
15	paracetamol (72.31%)		acetylsalicylic acid (not quantified)
16	paracetamol	-	aspirin
17	paracetamol, acetylcodeine	acetylcodeine, MAM	acetylcodeine, MAM, acetylsalicylic acid
18	Acetaminophen : 73.3 %		Aspirin
19	Acetylcodeine, Paracetamol	Acetylcodeine	Acetylcodeine, Salicylic acid (aspirin)
20	Paracetamol		Acetylsalicylic acid
21	Acetylcodeine, Paracetamol	Acetylcodeine, Monoacetylmorphine	Acetylcodeine, Aspirin
22	Paracetamol		Aspirin
23	paracetamol	none	salicylic acid
24	Acetylcodeine, Paracetamol	Acetylcodeine	Acetylcodeine, Aspirin
25	Acetylcodeine, Acetylmorphine & Paracetamol	Acetylcodeine & Acetylmorphine	Acetylcodeine, Acetylmorphine & Aspirin
26	paracetamol		salicylic acid or acetylsalicylic acid
27	Paracetamol	NA	Acetylsalicylic acid
28	Paracetamol	None	Salicylic Acid
29	Acetylcodeine, 6-Monoacetylmorphine and acetaminophen	Acetylcodeine and 6-Monoacetylmorphine	Acetylcodeine and 6-Monoacetylmorphine
30	Paracetamol	None identified	Acetylsalicylic acid
31	Acetaminophen	-	Acetyl salicylic acid

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

6 DISCUSSION OF RESULTS

6.1 Assigned Value

The robust averages of participants' results were used as the assigned values in this study. The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528:2022.[5] Results less than 50% and greater than 150% of the robust average were removed before the calculation of the assigned value, if applicable.[3,4] The calculation procedure for the expanded uncertainty of the robust average, using Sample S1 as an example, is presented in Appendix 1.

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

6.2 Measurement Uncertainty Reported by Participants

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

It is a requirement of ISO/IEC 17025 that laboratories have procedures to estimate the uncertainty of chemical measurements and to report this uncertainty in specific circumstances, including when the client's instruction so requires.[7] From 1 July 2012, this is also a requirement of ANAB-ASCLD/LAB accreditation program.[10]

Of 93 reported results, 90 (97%) were reported with an associated expanded MU. Laboratory **18** did not report any uncertainties; this participant reported that they were not accredited.

The magnitude of reported uncertainties was within the range 0.5% 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 and not fit for purpose. Of the 90 expanded MUs, 63 (70%) were between 3% and 10% relative to the result, eight were less than 3% and 19 were greater than 10%.

Uncertainties associated with results returning a satisfactory z-score but an unsatisfactory E_n-score may have been underestimated.

In some cases, 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 reporting 33.78 ± 3.04%, the recommended format is 33.8 ± 3.0%.[8]

6.3 *z*-Score

Target SDs equivalent to 3% PCV was used to calculate z-scores. CVs predicted by the Thompson-Horwitz equation,[6] target SDs (as PCVs) and between-laboratory CVs obtained in this study are presented for comparison in Table 9.

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

Sample	Analyte	Assigned Value (% base (m/m))	Thompson-Horwitz CV (%)	Target SD (as PCV) (%)	Between-Laboratory CV (%)
S1	Heroin	21.2	2.2	3	3.6
S2	Heroin	79.6	1.1	3	2.4
S3	Heroin	34.2	1.7	3	2.8

Of 93 results for which *z*-scores were calculated, 84 (90%) returned a *z*-score with $|z| \leq 2.0$, indicating a satisfactory performance.

Twenty-four participants: **1**, **3**, **5**, **6**, **7**, **8**, **9**, **10**, **11**, **13**, **14**, **15**, **16**, **17**, **18**, **19**, **22**, **23**, **25**, **26**, **27**, **29**, **30** and **31** returned satisfactory *z*-scores for all three samples. Seven participants returned at least one questionable or unsatisfactory *z*-score.

Laboratory **12**'s results for Samples S2 and S3 were approximately 50% of the assigned value. This participant should ensure that they have not made a dilution calculation or related error in reporting their result.

The dispersal of participants' *z*-scores is presented graphically in Figure 5. *z*-Scores less than -10.0 have been plotted at -10.0.

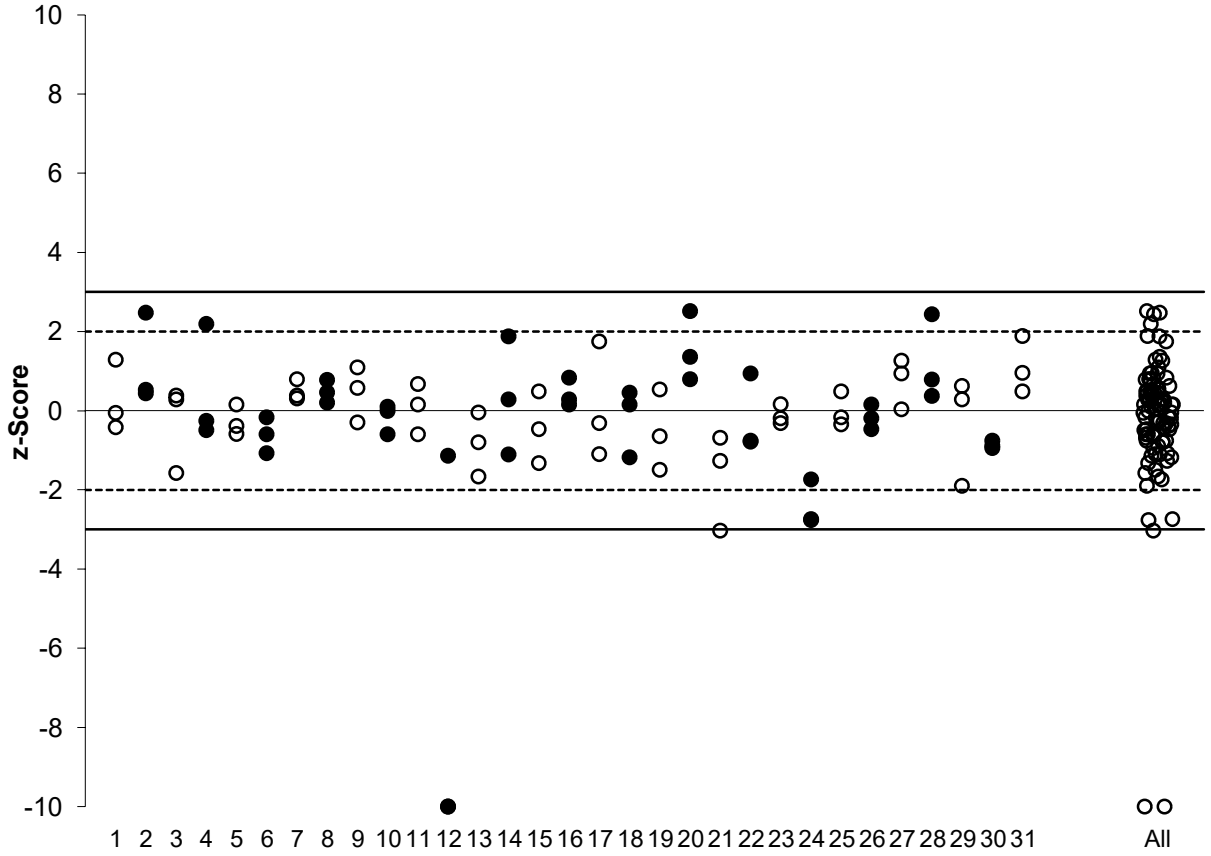


Figure 5 *z*-Score Dispersal by Laboratory

6.4 E_n-Score

E_n-scores can be interpreted in conjunction with *z*-scores, as an unsatisfactory E_n-score can be caused by an inappropriate measurement or uncertainty estimation, 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 93 results for which E_n-scores were calculated, 86 (92%) 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**, **4**, **5**, **6**, **7**, **9**, **10**, **11**, **13**, **14**, **15**, **16**, **17**, **19**, **20**, **21**, **22**, **23**, **24**, **25**, **26**, **27**, **28**, **29**, **30** and **31** returned satisfactory E_n-scores for all three samples. Five participants returned at least one unsatisfactory E_n-score.

The dispersal of participants' E_n-scores is presented graphically in Figure 6. E_n-Scores less than -10.0 have been plotted at -10.0.

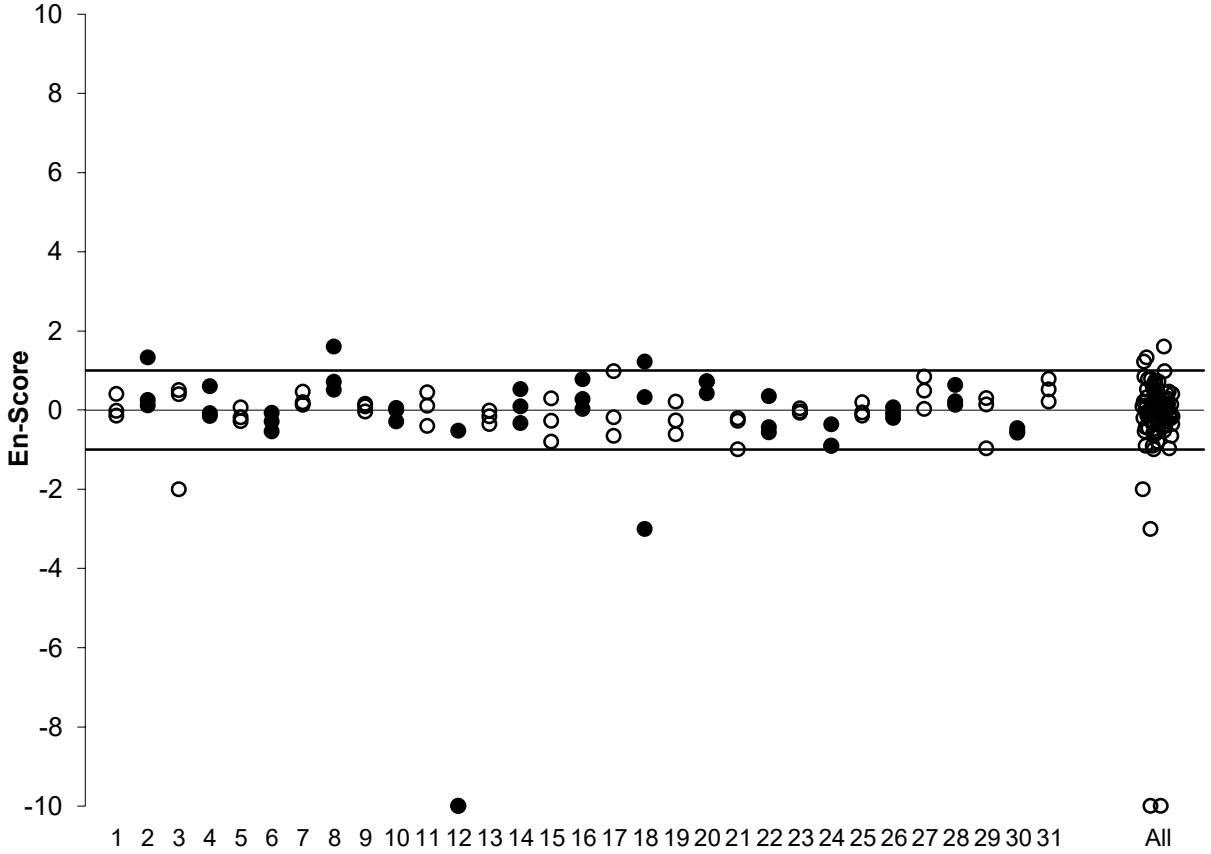


Figure 6 E_n-Score Dispersal by Laboratory

6.5 Identification of Cutting Agents

Cutting agents were added to Samples S1 (paracetamol) and S3 (aspirin). Sample S2 was left uncut.

Thirty participants (97%) reported on the identity of at least one sample's cutting agent. Results reported by participants are presented in Table 8.

Laboratories **1**, **2**, **3**, **7**, **8**, **9**, **11**, **12**, **13**, **14**, **15**, **16**, **17**, **18**, **20**, **21**, **22**, **24**, **25**, **27**, **30** and **31** correctly reported all cutting agents used.

All participants reporting on cutting agents correctly identified paracetamol in Sample S1.

No cutting agents were added to Sample S2, and no participants reported cutting agents in this sample.

For Sample S3, 22 participants correctly identified aspirin as the cutting agent. Laboratory **26** reported that the cutting agent was aspirin or the closely related compound salicylic acid. Laboratory **19** reported salicylic acid, though they identified this as aspirin. Laboratories **23** and **28** reported salicylic acid.

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 methodologies provided by participants are presented in Table 1.

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

Table 10 Summary of Participants' Analytical Methods

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

Plots of *z*-scores against various parameters are presented in Figures 7 to 11 (gross errors have not been plotted). No trend was observed in this study.

The majority of participants used around 20 – 30 mg for sample analysis. The most common methodology used was extraction with acetonitrile/water(/other) and analysis on HPLC-DAD, or extraction with methanol and analysis on UPLC-DAD.

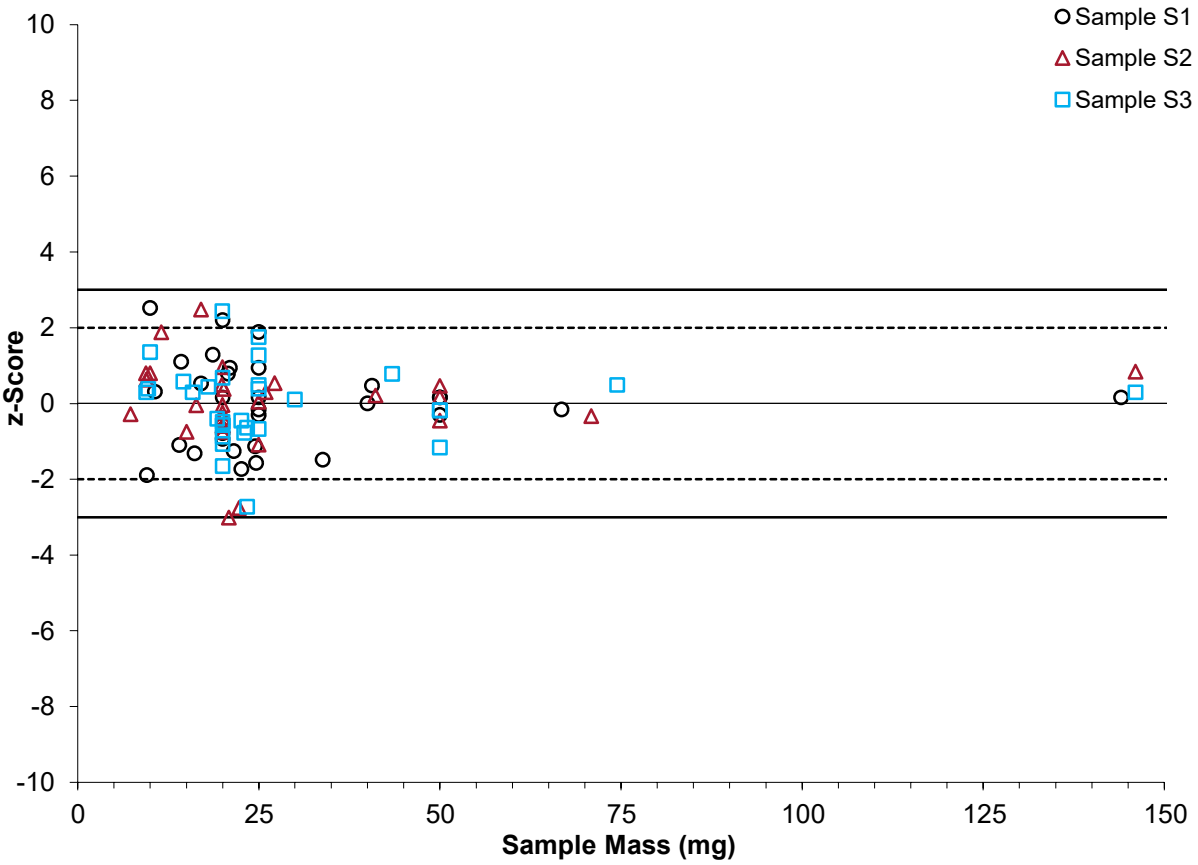


Figure 7 *z*-Score vs Sample Mass Used per Analysis

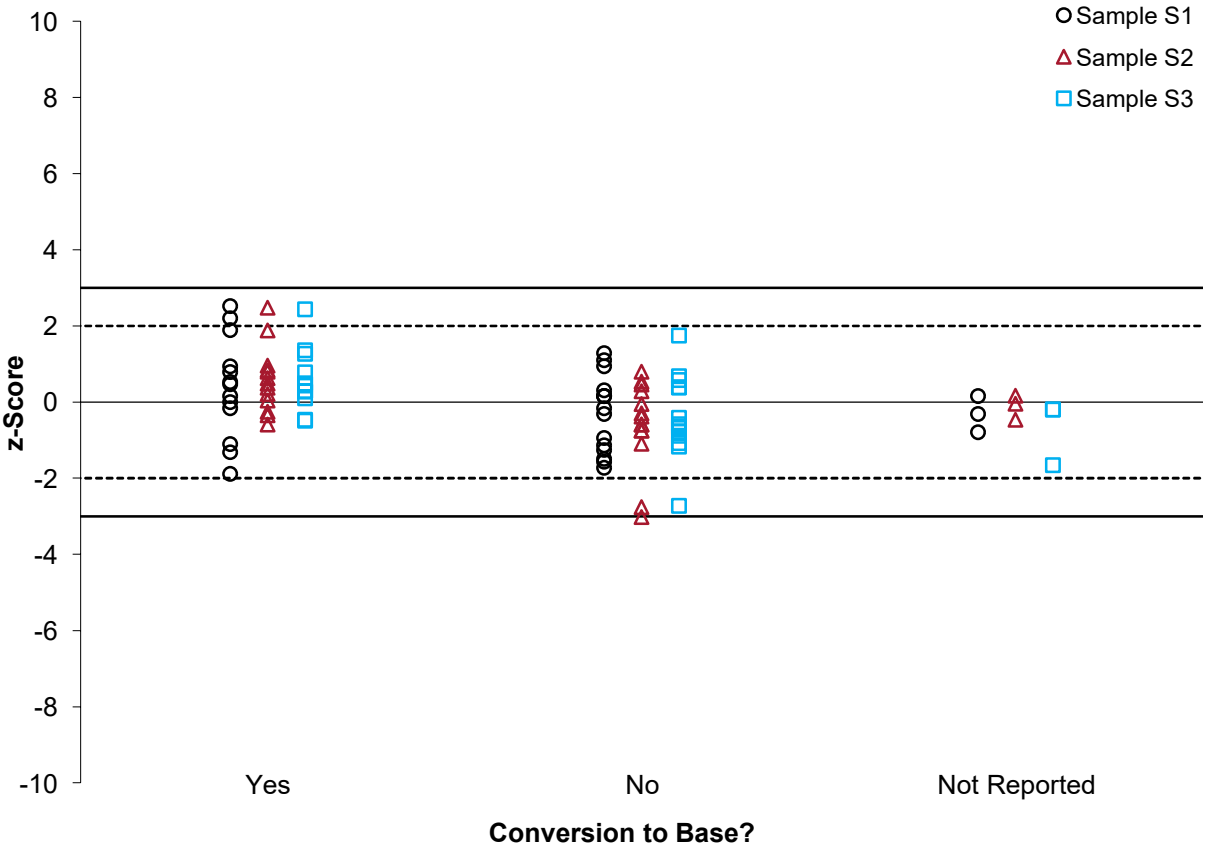


Figure 8 *z*-Score vs Sample Processing

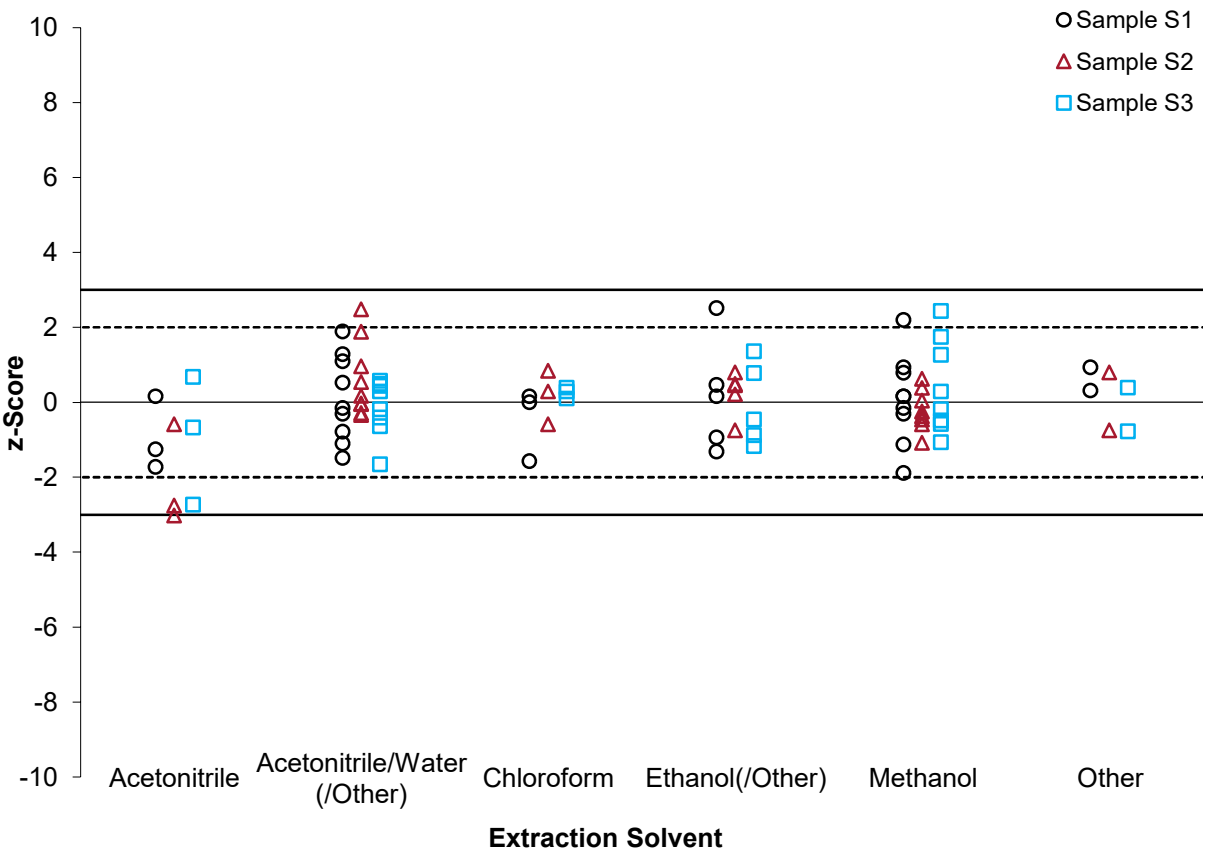


Figure 9 *z*-Score vs Extraction Solvent

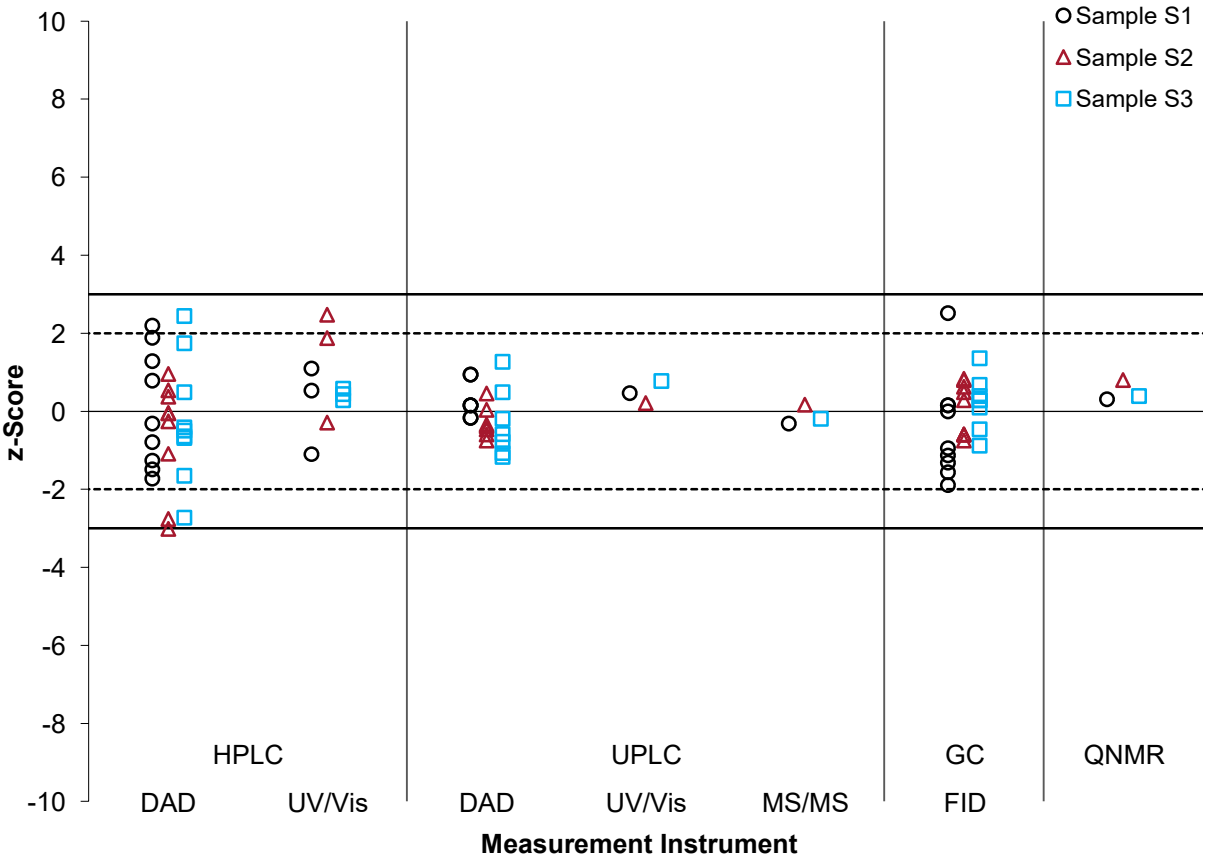


Figure 10 *z*-Score vs Measurement Instrument

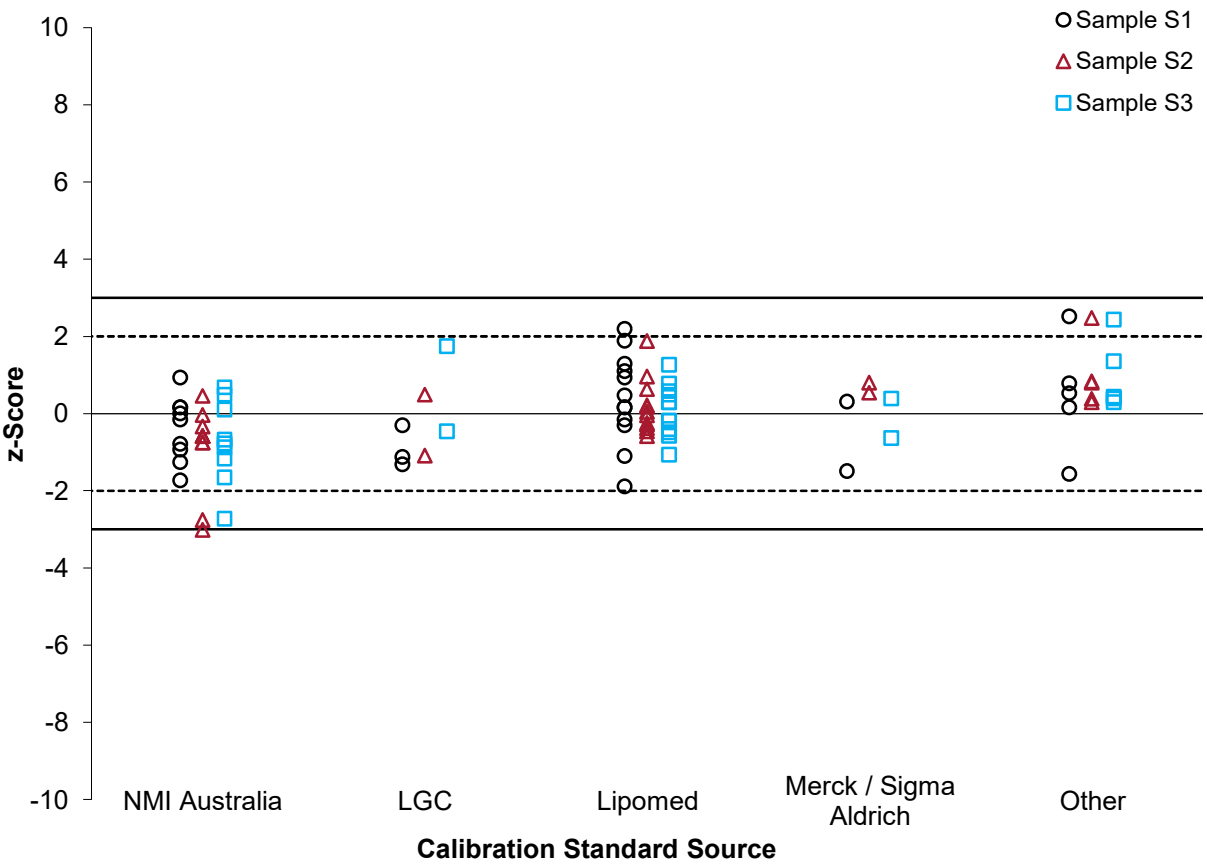


Figure 11 *z*-Score vs Calibration Standard Source

6.7 Comparison of Results and Date of Analysis

As there were delays with sample delivery to some participants, the test samples were analysed over the course of approximately three months. There was no evidence of sample degradation over this period, and there was no trend between when the samples were analysed and the results obtained by participants (Figure 12).

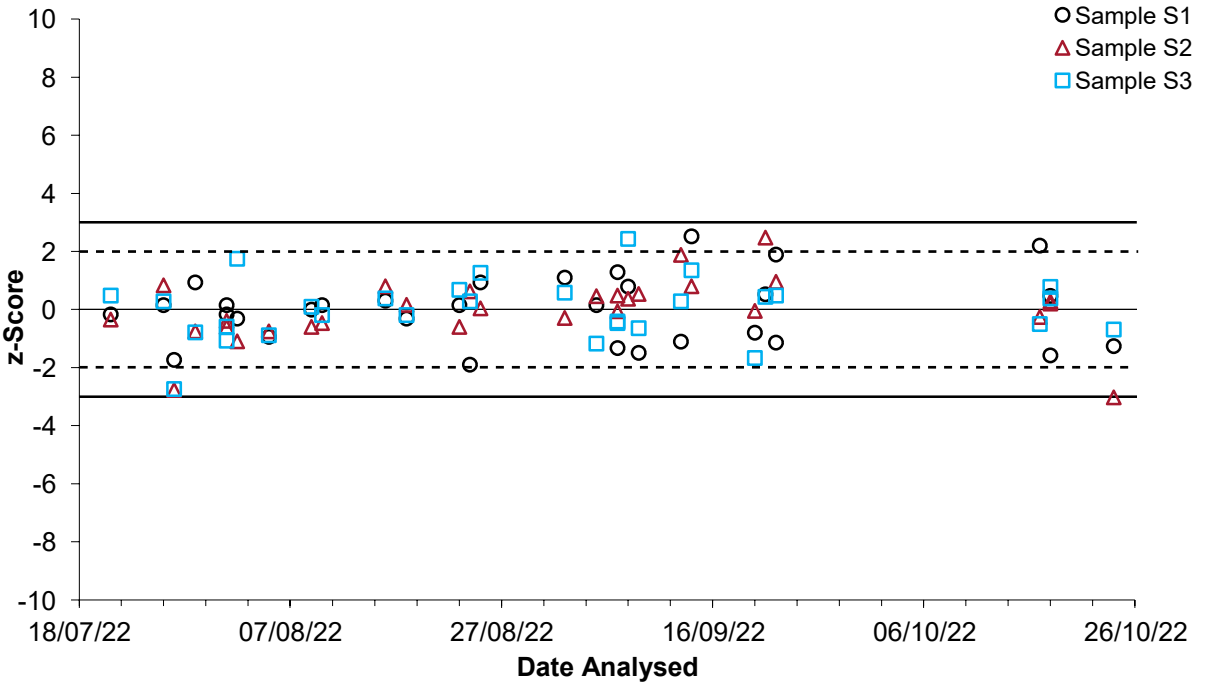


Figure 12 *z*-Score vs Sample Analysis Date

6.8 Comparison with Previous Heroin PT Studies

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

A summary of the satisfactory performance, presented as a percentage of the total number of scores, obtained by participants from 2013 to 2022 (last ten studies) is presented in Figure 13. The proportion of satisfactory *z*-scores and E_n-scores over this period on average is 82% for both. While each PT study has a different group of participants, taken as a group, the performance over this period has improved.

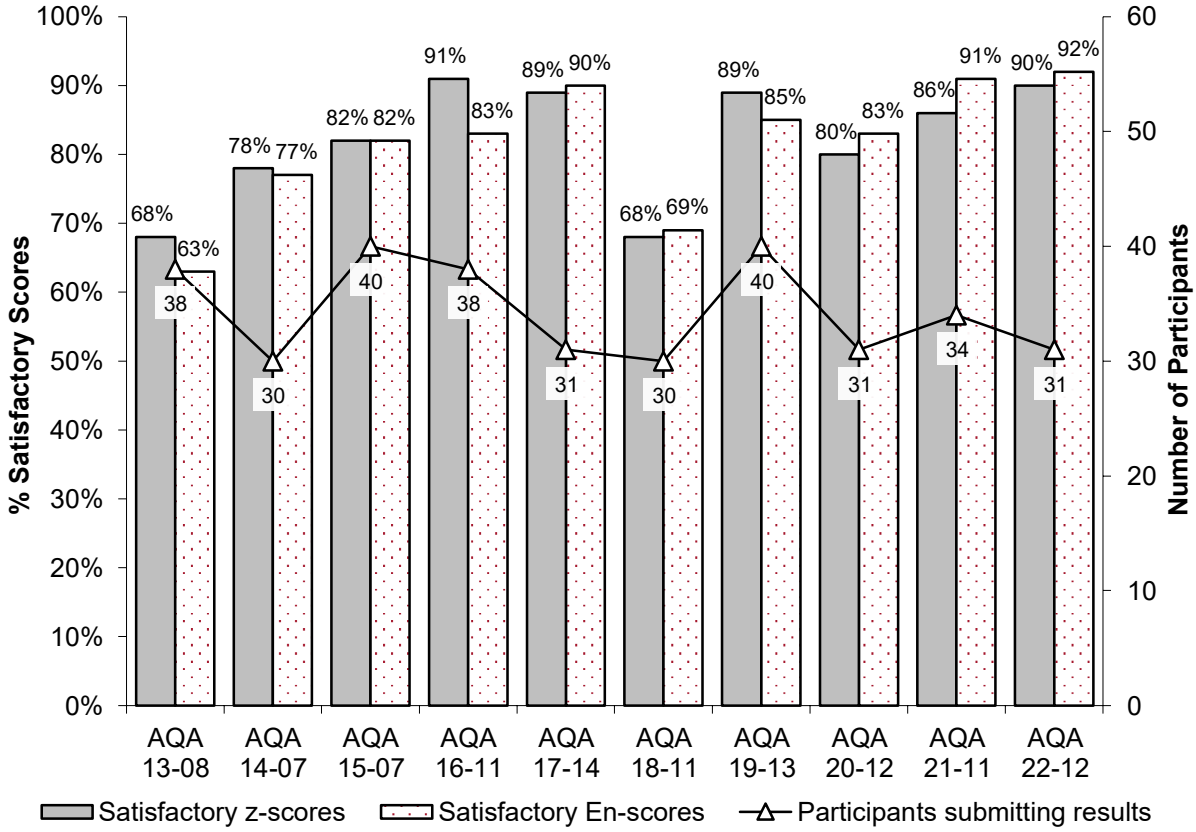


Figure 13 Summary of Participants' Performance in NMI Heroin PT Studies

A number of participants have consistently participated in NMI heroin PT studies, and individual performance history reports are emailed to each participant at the end of each study. The consideration of *z*-scores for an analyte over time provides much more useful information than a single *z*-score. Over time, laboratories should expect at least 95% of their scores to lie within the range $|z| \leq 2.0$. Scores in the range $2.0 < |z| < 3.0$ can occasionally occur, however, these should be interpreted in conjunction with the other scores obtained by that laboratory. For example, a trend of *z*-scores on one side of the zero line is an indication of method or laboratory bias.

A summary of individual laboratories' performances over the last ten studies is presented in Figures 14 and 15 for Australian and international laboratories respectively. *z*-Scores greater than 10.0 or less than -10.0 have been plotted at 10.0 and -10.0 respectively. Two Australian and three international laboratories have achieved satisfactory *z*-scores across all samples in all heroin PT studies participated in over this period.

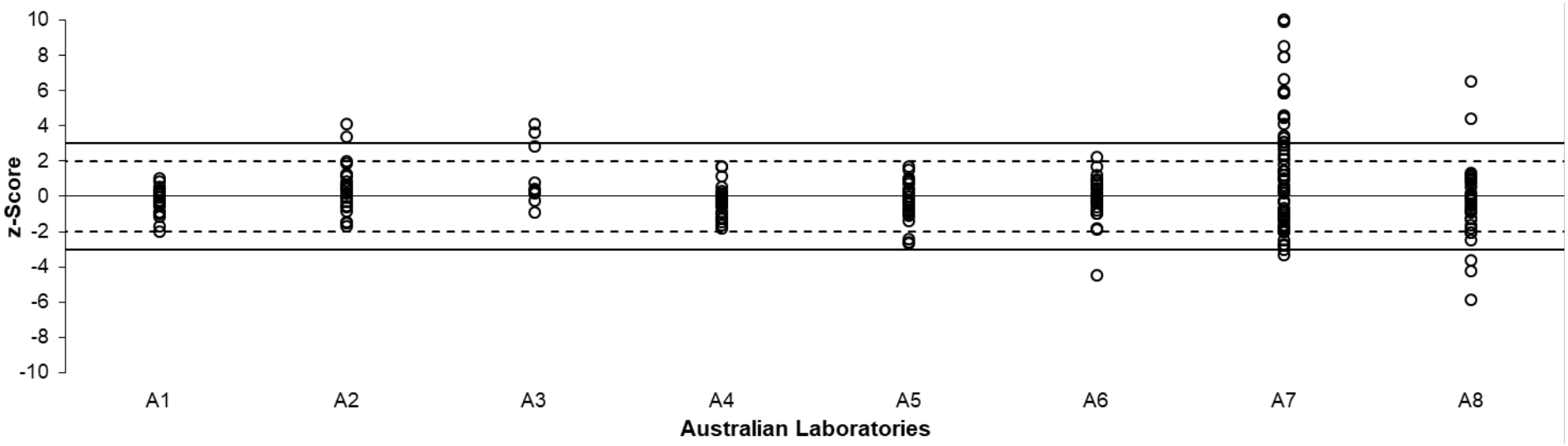


Figure 14 Summary of Australian Participants' *z*-Scores in NMI Heroin PT Studies

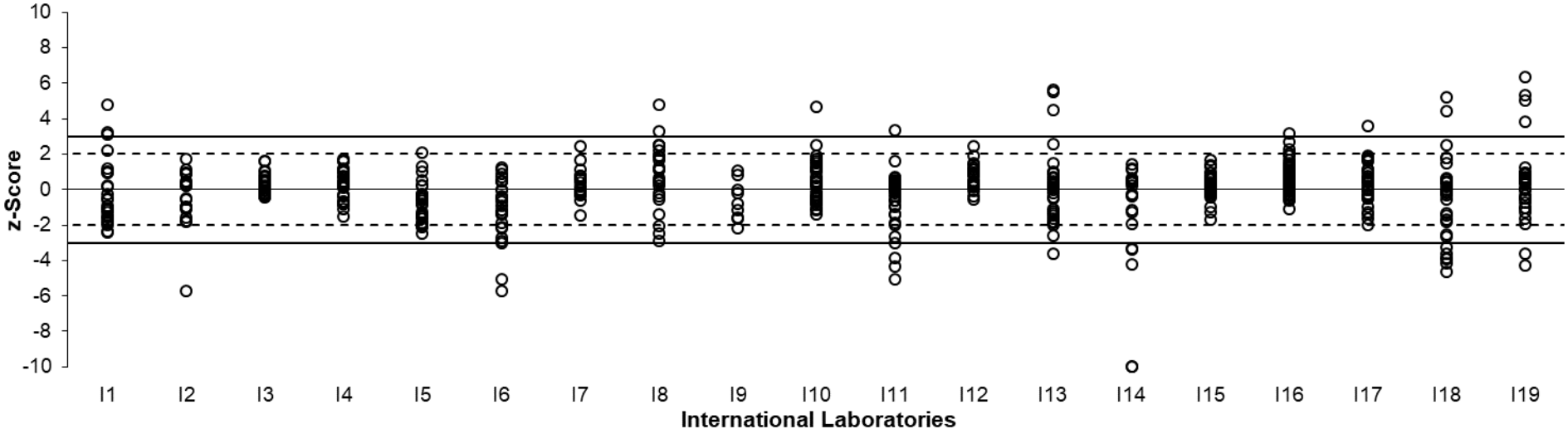


Figure 15 Summary of International Participants' *z*-Scores in NMI Heroin PT Studies

A comparison of all results from Australian and international laboratories in NMI heroin PT studies over the last ten studies is presented in Figure 16. Overall, both groups have performed very similarly, with Australian and international laboratories achieving 81% and 83% satisfactory *z*-scores respectively over this period.

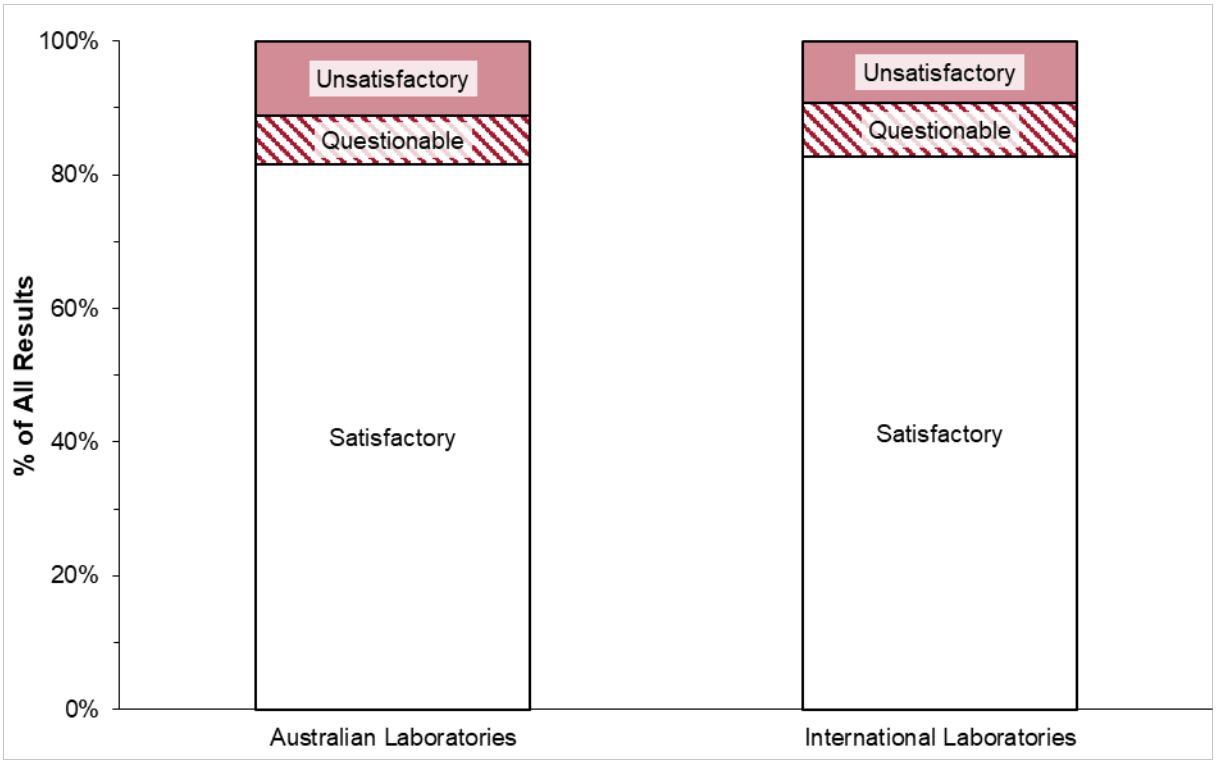


Figure 16 Comparison of Australian and International Laboratories in NMI Heroin PT Studies

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

A1.1 Robust Average and Associated Uncertainty

Robsut averages were calculated using the procedure described in ISO 13528:2022.[5] The associated uncertainties were estimated as according to Equation 4.

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

where:

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

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

p is the number of results

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

A worked example is set out below in Table 11.

Table 11 Uncertainty Estimate for Robust Average of Sample S1

Number of Results (p)	31
Robust Average	21.17% base (m/m)
$S_{rob\ average}$	0.77% base (m/m)
$u_{rob\ average}$	0.17% base (m/m)
k	2
$U_{rob\ average}$	0.34% base (m/m)

Therefore, the robust average for Sample S1 is 21.2 ± 0.3% base (m/m).

A1.2 z-Score and E_n-Score Calculations

For each participant's result, a z-score and E_n-score are calculated according to Equations 2 and 3 respectively (Section 4).

A worked example is set out below in Table 12.

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

Participant Result (% base (m/m))	Assigned Value (% base (m/m))	Target Standard Deviation	z-Score	E_n-Score
22.02 ± 1.98	21.2 ± 0.3	3% as PCV, or: 0.03 × 21.2 = 0.636% base (m/m)	z-Score = $\frac{22.02-21.2}{0.636}$ = 1.29	E_n-Score = $\frac{22.02-21.2}{\sqrt{1.98^2+0.3^2}}$ = 0.41

APPENDIX 2 ACRONYMS AND ABBREVIATIONS

ANAB	ANSI (American National Standards Institute) National Accreditation Board
ASCLD/LAB	American Society of Crime Laboratory Directors – Laboratory Accreditation Board
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
DAD	Diode Array Detection
EA	European Accreditation
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
Max	Maximum
Md	Median
Min	Minimum
MS	Mass Spectrometry
MS/MS	Tandem Mass Spectrometry
MU	Measurement Uncertainty
N	Number of numeric results
NATA	National Association of Testing Authorities, Australia
NMI	National Measurement Institute, Australia
NR	Not Reported
PCV	Performance Coefficient of Variation
PDA	Photodiode Array Dectection
PT	Proficiency Testing
QNMR	Quantitative Nuclear Magnetic Resonance Spectroscopy
RA	Robust Average
RM	Reference Material
SD	Standard Deviation
SI	International System of Units
SS	Spiked Samples
TS	Technical Specification (ISO)
U(H)PLC	Ultra (High) Performance Liquid Chromatography
UV/Vis	Ultraviolet/Visible Detection

END OF REPORT