Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

# Proficiency Test Report AQA 19-09 Methamphetamine in Wipes

May 2020

## ACKNOWLEDGMENTS

This study was conducted by the National Measurement Institute (NMI). Support funding was provided by the Australian Government Department of Industry, Science, Energy 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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Accredited for compliance with ISO/IEC 17043

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## 1 SUMMARY

This report presents the results of the proficiency test AQA 19-09 Methamphetamine in Wipes. Three test samples of methamphetamine hydrochloride in wipes were sent to eleven laboratories. Ten sets of results were submitted by the due date.

Test samples were prepared at the NMI laboratory in Sydney using methamphetamine hydrochloride synthesised by the NMI.

Two test samples were duplicates (S1/S2). The assigned values for the duplicate pair were calculated as the robust average of the pooled participant results in both samples S1 and S2. The associated uncertainties were estimated from the robust standard deviations of the participants' results for the duplicate pair. The assigned value for sample S3 was the robust average of participants' results. The associated uncertainty for Sample S3 was estimated from the robust standard deviation of the participants' results.

The outcomes of the study were assessed against the aims as follows:

*i. assess the proficiency of laboratories measuring methamphetamine in wipes;*

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

Laboratories **1**, **4**, **5, 7, 10** and **12** returned satisfactory z and $E_n$-scores for all samples.

Of the 30 results for which z-scores were calculated, 29 (97%) returned $|z| \leq 2$ indicating a satisfactory performance.

Of the 30 results for which $|E_n|$-scores were calculated, 23 (77%) returned $|E_n| \leq 1$ indicating agreement of the participants' results with the assigned value within their respective expanded uncertainties.

*ii. evaluate the laboratories methods used in the determination of methamphetamine in wipes;*

Participants used various methods for measurement of methamphetamine wipes and all produced comparable results.

*iii. compare the performance of participant laboratories with their past performance;*

Of the 10 participants who reported results, 7 also reported results for methamphetamine in the previous study AQA 18-08 Methamphetamine and MDMA in Wipes. All but one performed satisfactorily in both studies.

*iv. develop the practical application of traceability and measurement uncertainty and provide participants with information that will be useful in assessing their uncertainty estimates.*

All results were reported with an associated expanded uncertainty.

The magnitude of reported uncertainties was within the range 0.19% to 32% relative.

# 2 INTRODUCTION

## 2.1 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: '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 and food/biota;
- inorganic analytes in soil, water, food and pharmaceuticals;
- allergens in food;
- controlled drug assay.

AQA 19-09 is the second NMI proficiency test of the analysis of methamphetamine in wipes.

## 2.2 Study Background

Illicit laboratory sites (clandestine laboratories, 'clan labs') are places where illegal drugs have been manufactured. During the drug manufacturing process, toxic gases and aerosols are produced. These may be absorbed by flooring, walls, ducting and furnishings. Chemical contamination may remain in the property for many years. Field test kits are used to check the extent of contamination in the premises. Samples may be taken from non-porous surfaces inside a building using wipes.

This scheme was provided to enable laboratories to assess their ability to measure methamphetamine wipes at investigation levels specified in Clandestine Drug Laboratory Remediation Guidelines 2011.

## 2.3 Study Aims

The aims of the study were to:

- assess the proficiency of laboratories measuring methamphetamine in wipes;
- evaluate the laboratories methods used in the determination of methamphetamine in wipes;
- compare the performance of participant laboratories with their past performance; and
- develop the practical application of traceability and measurement uncertainty and provide participants with information that will be useful in assessing their uncertainty estimates.

## 2.4 Study Conduct

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

The conduct of NMI proficiency tests is described in the NMI Chemical Proficiency Testing Study Protocol.[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 17043 and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[4]

## 3 STUDY INFORMATION

### 3.1 Study Timetable

The timetable of the study was:

| | |
|---|---|
| Invitation issued: | 21 October 2019 |
| Samples dispatched: | 10 March 2020 |
| Results due: | 17 April 2020 |
| Interim report issued: | 20 April 2020 |

### 3.2 Participation

Eleven laboratories participated and ten reported results.

### 3.3 Test Material Specification

Three samples were provided for analysis: AQA 19-09 S1, S2 and S3. Each sample consisted of one wipe spiked with methamphetamine

**Sample S1** was prepared to contain 1.20 µg/wipe methamphetamine.

**Sample S2** was identical with Sample S1; and

**Sample S3** was prepared to contain 4.20 µg/wipe methamphetamine.

### 3.4 Laboratory Code

Each participant was randomly assigned a confidential laboratory code.

### 3.5 Sample Preparation, Analysis and Homogeneity Testing

The sample preparation procedure followed was the same procedure as used in previous study.[2] No homogeneity test was conducted for this study as the test samples from previous studies were demonstrated to be sufficiently homogenous for the evaluation of participant's performance.

The preparation procedure is described in Appendix 1.

### 3.6 Stability of Analytes

Results of this study gave no reason to question the stability of the test samples. No correlation between reported results, the received date, the analysis date or the sample condition at arrival was observed (see Appendix 2).

### 3.7 Sample Storage, Dispatch and Receipt

The study samples were stored at 4°C and dispatched by courier on 10 March 2020.

A description of the test sample, instructions to participants, and a form for participants to confirm the receipt of the test sample were sent with the sample.

An Excel spreadsheet for the electronic reporting of results was emailed to participants.

### 3.8 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse each wipe for the amount of methamphetamine using your normal test method.
- If analyses cannot be commenced on the day of receipt, please store the samples refrigerated.
- Report results in µg/wipe drug as base. Report this figure as if reporting to a client.
- For each result report an estimate of your expanded uncertainty as µg/wipe drug as base.

- No limit of reporting has been set for this study. Report results as you would report to a client, applying the limit of reporting of the method used for analysis.
- Give brief details of your:
    - basis of uncertainty estimate (e.g. uncertainty budget, repeatability precision)
    - analytical method (e.g. sample treatment, instrument type and calibration method)
    - reference standard (e.g. source, purity)
- e-mail your results on the results spreadsheet to proficiency@measurement.gov.au.

### 3.9 Interim Report

An interim report was emailed to all participants on 20 April 2020.

## 4 PARTICIPANT LABORATORY INFORMATION

### 4.1 Test Method Summaries

Summaries of test methods used by participants are transcribed in Table 1.

Table 1 Summary of Participants' Test Methods

| Lab. Code | Desorption Solution | Sample Treatment | Filtration | Equipment | Internal Standard | Method Reference |
|---|---|---|---|---|---|---|
| 1 | 0.1 M sulfuric acid | 30 minutes on orbital shaker | Nil | LC-MS/MS | Methamphetamine-$D_{14}$ | NIOSH 9111 |
| 2 | 0.1 M sulfuric acid | 60 minutes on Rotary Mixer | Centrifugation | LC-MS/MS | d,l-Methamphetamine-$D_5$.HCL | Internal Method |
| 3 | 0.1 M sulfuric acid | 1 hr on orbital shaker | 0.2 µm nylon syringe filter | UPLC-MS-MS | Methamphetamine –$D_5$ | Extraction by NIOSH 9111, Analysis by UPLC-MSMS |
| 4 | 0.1 M sulfuric acid | 1 hour on a rotary mixer / Aliquot / Centrifuge | N/A | LC-MS/MS | d,l-Methamphetamine-$D_5$.HCL | Modified NIOSH 9111 |
| 5 | 0.1 M sulfuric acid | 0.5 hour on rotary mixer, pH adjustment | 0.45 µm filter | LC-MS/MS | Methamphetamine –$D_5$ | NIOSH 9111 |
| 6 | 0.1 M sulfuric acid | 1 hour on a rotary mixer | Agilent PES 0.45 µm filter | LC-MS | Methamphetamine-$D_{14}$ | NIOSH 9111 |
| 7 | 0.1 M sulfuric acid | Hour on a rotary mixer, sonication, pH adjustment | 0.2 µm RC Filter | LC-MS/MS | Methamphetamine –$D_5$ | NIOSH 9111 |
| 10 | 0.1 M sulfuric acid | 30 minute tumble end over end | 0.2 µm Nylon filter | UHPLC-MS/MS | Methamphetamine-$D_{14}$ | In house method based on NIOSH 9111 |
| 11 | 0.1 M sulfuric acid | 1 hour on a rotary mixer / Aliquot / Centrifuge | N/A | LC-MS/MS | d,l-Methamphetamine-$D_5$.HCL | Modified NIOSH 9111 |
| 12 | 0.1 M sulfuric acid | 1 hour Linear shaker | N/A | LC-MS/MS | Methamphetamine –$D_9$ | NIOSH 9111 - modified |

### 4.2 Reported Basis of Participants' Measurement Uncertainty Estimates

Participant approaches to measurement uncertainty are listed as received in Table 2.

Table 2 Reported Basis of Uncertainty Estimates

| Lab. Code | Approach to Estimating MU | Information Sources for MU Estimation* | | Guide Document for Estimating MU |
|---|---|---|---|---|
| | | Precision | Method Bias | |
| 1 | Standard deviation of replicate analyses multiplied by 2 or 3 | Control Samples – SS<br>Duplicate Analysis<br>Instrument Calibration | CRM<br>Recoveries of SS<br>Instrument Calibration<br>Standard Purity | Eurachem/CITAC Guide |
| 2 | Top Down - precision and estimates of the method and laboratory bias | Duplicate Analysis<br>Instrument Calibration | CRM<br>Standard Purity | NATA 2018-General Accreditation Guidance Estimating and Reporting Measurement Uncertainty of Chemical Test Results |

Table 2 Reported Basis of Uncertainty Estimates (cont.)

| Lab. Code | Approach to Estimating MU | Information Sources for MU Estimation* | | Guide Document for Estimating MU |
|---|---|---|---|---|
| | | Precision | Method Bias | |
| 3 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - SS | Standard Purity<br>Recoveries of SS | NMI Uncertainty Course |
| 4 | Top Down - precision and estimates of the method and laboratory bias | Duplicate Analysis<br>Instrument Calibration | CRM<br>Standard Purity | NATA 2018-General Accreditation Guidance Estimating and Reporting Measurement Uncertainty of Chemical Test Results |
| 5 | Top Down - precision and estimates of the method and laboratory bias | Instrument Calibration | Instrument Calibration | ISO/GUM |
| 6 | NIOSH Method Accuracy Range (A) | Control Samples – SS<br>Duplicate Analysis<br>Instrument Calibration | CRM<br>Instrument Calibration<br>Standard Purity<br>Recoveries of SS | NIOSH Manual of Analytical Methods 3/15/03 Page 208 Part P. Measurement Uncertainty and NIOSH Method Accuracy Range |
| 7 | Top Down - precision and estimates of the method and laboratory bias | Control Samples | Recoveries of SS | NATA 2018-General Accreditation Guidance Estimating and Reporting Measurement Uncertainty of Chemical Test Results |
| 10 | Top Down - precision and estimates of the method and laboratory bias | Control Samples – SS | Recoveries of SS | Eurachem/CITAC Guide |
| 11 | 95% Confidence Level of +/- 0.007 µg/sample | Control Samples<br>Duplicate Analysis<br>Instrument Calibration | CRM<br>Instrument Calibration<br>Laboratory bias from PT studies<br>Standard Purity<br>Recoveries of SS | |
| 12 | | Control Samples – SS<br>Duplicate Analysis<br>Instrument Calibration | Instrument Calibration<br>Laboratory bias from PT studies<br>Standard Purity | Eurachem/CITAC Guide |

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

### 4.3 Details of Participant Calibration Standard

Reference standards used by laboratories are listed as received in Table 3.

Table 3 Participants' Calibration Standard

| Lab. Code | Calibration Standard | Purity (%) |
|---|---|---|
| 1 | Lipomed 1ml d,l-Methamphetamine.HCl solution (1mg/mL) | |
| 2 | Lipomed d,l-Methamphetamine.HCl | 99.6 |
| 3 | CRM – 1000 µg/mL | |
| 4 | Methamphetamine.HCl, LOT: 301.1B6.1L1 Exp: Mar 29 | 99.95 |
| 5 | Lipomed | 99.95 |
| 6 | CRM Ceriliant product M-009, 1 mg/mL | |

Table 3 Participants' Calibration Standard (cont.)

| Lab. Code | Calibration Standard | Purity (%) |
|---|---|---|
| 7 | Chiron 1000 ppm mixed std | |
| 10 | Cerilliant ampule 1000 µg/mL | |
| 11 | Methamphetamine.HCl, LOT: 301.1B6.1L1 Exp: Mar 29 | 99.95 |
| 12 | | 99.8 |

### 4.4 Participants' Comments

The study manager welcomes comments or suggestions from participants as it provides information which will improve future studies. All returns are listed as received in Table 4 along with the study manager's response, where appropriate.

Table 4 Participants' Comments

| Lab. Code | Participant Comments | Study Manager's Comments |
|---|---|---|
| 1 | S1, S2, S3 NOTE: Multiple sets of the ILCP were purchased and an averaged result is provided above. | |
| 4 | Laboratory estimated measurement uncertainty by using parameters from bottom-up and top-down approaches. | |

## 5 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

### 5.1 Results Summary

Participant results are listed in Tables 5 to 7 with resultant summary statistics: mean, median, maximum, minimum, robust average, 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.

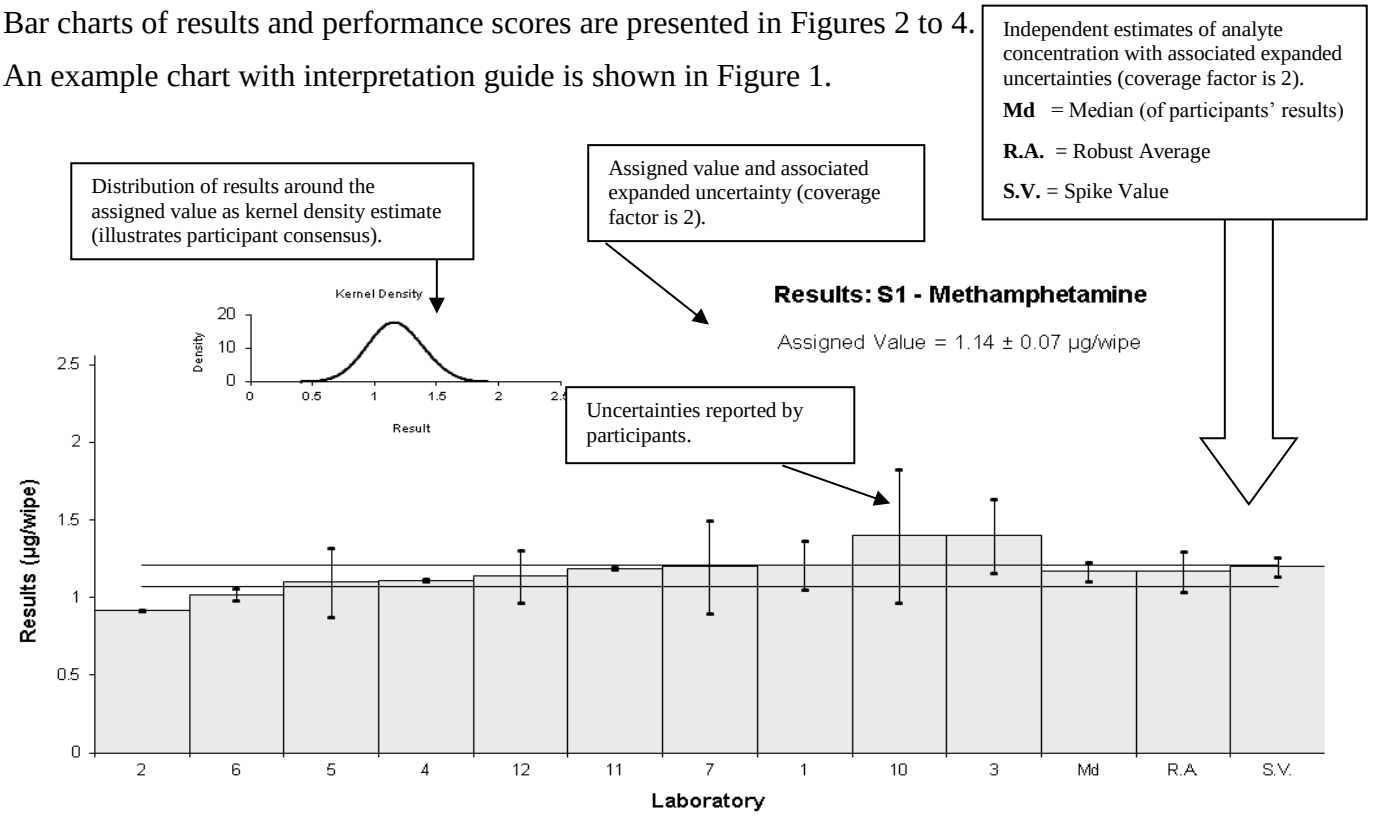



Figure 1 Guide to Presentation of Results

### 5.2 Assigned Value

Assigned value is defined as: 'the value attributed to a particular quantity and accepted, sometimes by convention, as having an uncertainty appropriate for a given purpose'.[1]

For a proficiency test, the assigned value is the best available measurement of the true concentration of an analyte in the test sample.

### 5.3 Robust Average

The robust averages and associated expanded measurement uncertainties were calculated using the procedure described in the 'Statistical methods for use in proficiency testing by inter-laboratory comparisons, ISO 13528:2015(E)'.[5]

### 5.4 Robust Between-Laboratory Coefficient of Variation

The robust between-laboratory coefficient of variation (robust CV) is a measure of the variability of participants' results and was calculated using the procedure described in ISO 13528:2015(E).[5]

### 5.5 Target Standard Deviation

The target standard deviation (σ) is the product of the assigned value (*X)* and the performance coefficient of variation (PCV) as presented in Equation 1. This value is used for calculation of participant z-score.

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

It is important to note that the PCV is a fixed value established by the study coordinator and is not the standard deviation of participants' results. By setting a fixed value for the PCV, the participants' performance can be compared from study to study.

### 5.6 z-Score

For each participants' result a z-score is calculated according to Equation 2 below:

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

where:

$z$ is z-score

$\chi$ is participants' result $X$ is the study assigned value

$\sigma$ is the target standard deviation

A z-score with absolute value (|z|):

- $|z| \leq 2$ is satisfactory;
- $2 < |z| < 3$ is questionable;
- $|z| \geq 3$ is unsatisfactory.

### 5.7 $E_n$-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 below:

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

where:

$E_n$ is $E_n$-score

$\chi$ is participants' result $X$ is the study assigned value

$U_\chi$ is the expanded uncertainty of the participant's result

$U_X$ is the expanded uncertainty of the assigned value

An $E_n$-score with absolute value ($|E_n|$):

- $|E_n| \leq 1$ is satisfactory;
- $|E_n| > 1$ is unsatisfactory.

### 5.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2018[6] must establish and demonstrate the traceability and measurement uncertainty associated with their test results. Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide. [7]

## 6 TABLES AND FIGURES

Table 5

**Sample Details**

| | |
|---|---|
| **Sample No.** | S1 |
| **Matrix** | Wipe |
| **Analyte** | Methamphetamine |
| **Units** | µg/wipe |

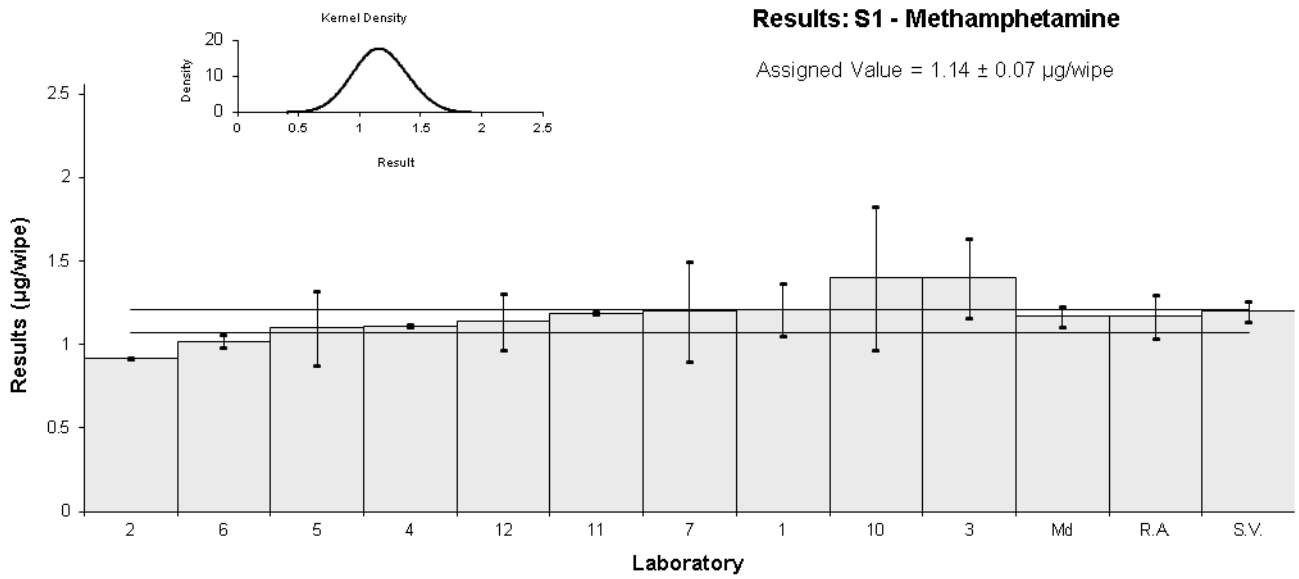
**Participant Results**

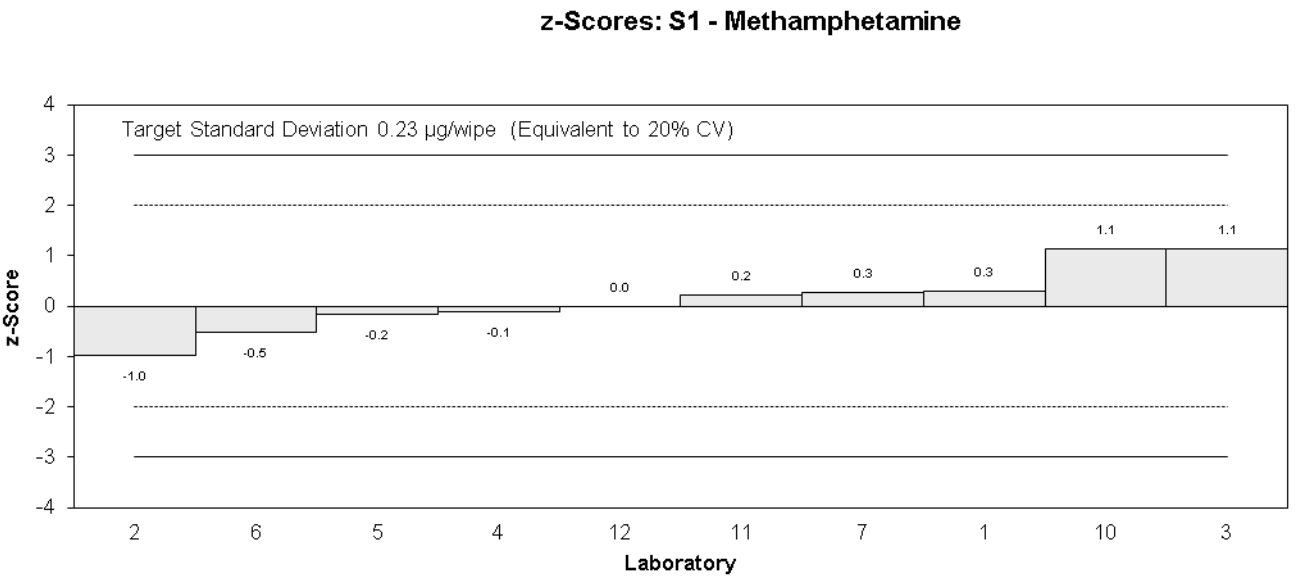
| Lab Code | Result | Uncertainty | Recovery | z-Score | $E_n$-Score |
|---|---|---|---|---|---|
| **1** | 1.21 | 0.16 | 106 | 0.31 | 0.40 |
| **2** | 0.918 | 0.007 | NR | -0.97 | -3.16 |
| **3** | 1.4 | 0.24 | NR | 1.14 | 1.04 |
| **4** | 1.113 | 0.007 | NR | -0.12 | -0.38 |
| **5** | 1.1 | 0.22 | 98 | -0.18 | -0.17 |
| **6** | 1.02 | 0.04 | 98 | -0.53 | -1.49 |
| **7** | 1.2 | 0.3 | 101 | 0.26 | 0.19 |
| **10** | 1.4 | 0.43 | 107.5 | 1.14 | 0.60 |
| **11** | 1.19 | 0.007 | 95 | 0.22 | 0.71 |
| **12** | 1.14 | 0.17 | NR | 0.00 | 0.00 |

**Statistics**

| | | |
|---|---|---|
| **Assigned Value*** | 1.14 | 0.07 |
| **Spike** | 1.20 | 0.06 |
| **Robust Average** | 1.17 | 0.13 |
| **Median** | 1.17 | 0.06 |
| **Mean** | 1.17 | |
| **N** | 10 | |
| **Max.** | 1.4 | |
| **Min.** | 0.918 | |
| **Robust SD** | 0.17 | |
| **Robust CV** | 15% | |

*The assigned value was calculated as the robust average of the combined results of duplicate pair samples S1 and S2


Kernel Density
Density
Result
Results: S1 - Methamphetamine
Assigned Value = 1.14 ± 0.07 µg/wipe
Results (µg/wipe)
Laboratory
2
6
5
4
12
11
7
1
10
3
Md
R.A
S.V.


z-Scores: S1 - Methamphetamine
Target Standard Deviation 0.23 µg/wipe (Equivalent to 20% CV)
z-Score
Laboratory
-1.0
-0.5
-0.2
-0.1
0.0
0.2
0.3
0.3
1.1
1.1

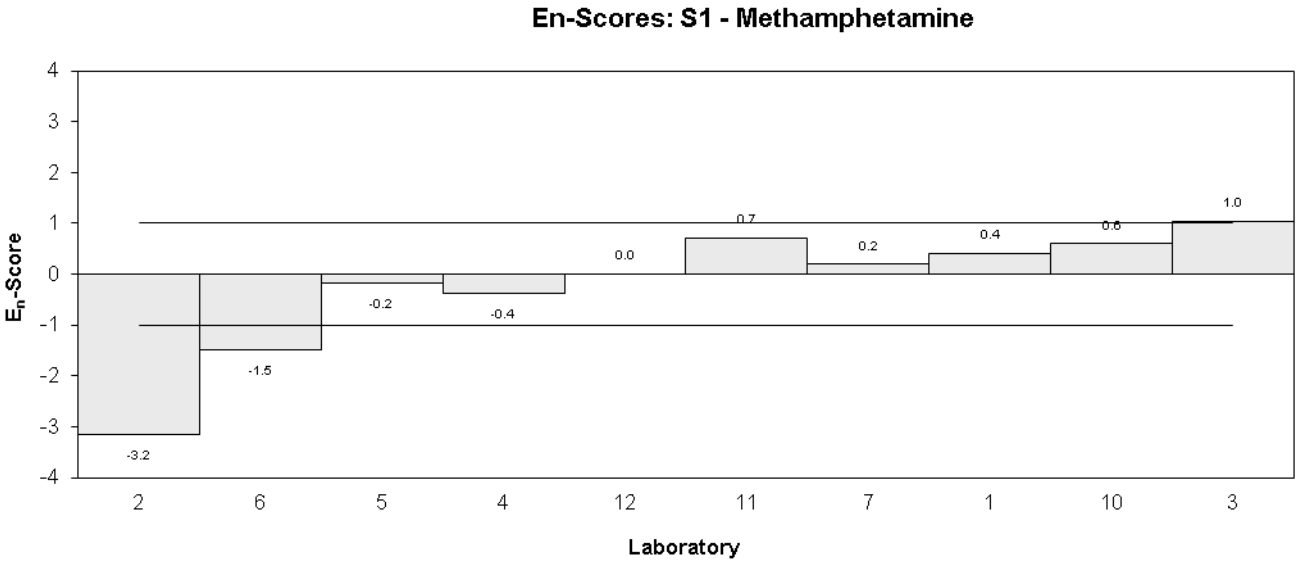

En-Scores: S1 - Methamphetamine
En-Score
Laboratory
-3.2
-1.5
-0.2
-0.4
0.0
0.7
0.2
0.4
0.6
1.0

Figure 2

Table 6

**Sample Details**

| | |
|---|---|
| **Sample No.** | S2 |
| **Matrix** | Wipe |
| **Analyte** | Methamphetamine |
| **Units** | µg/wipe |

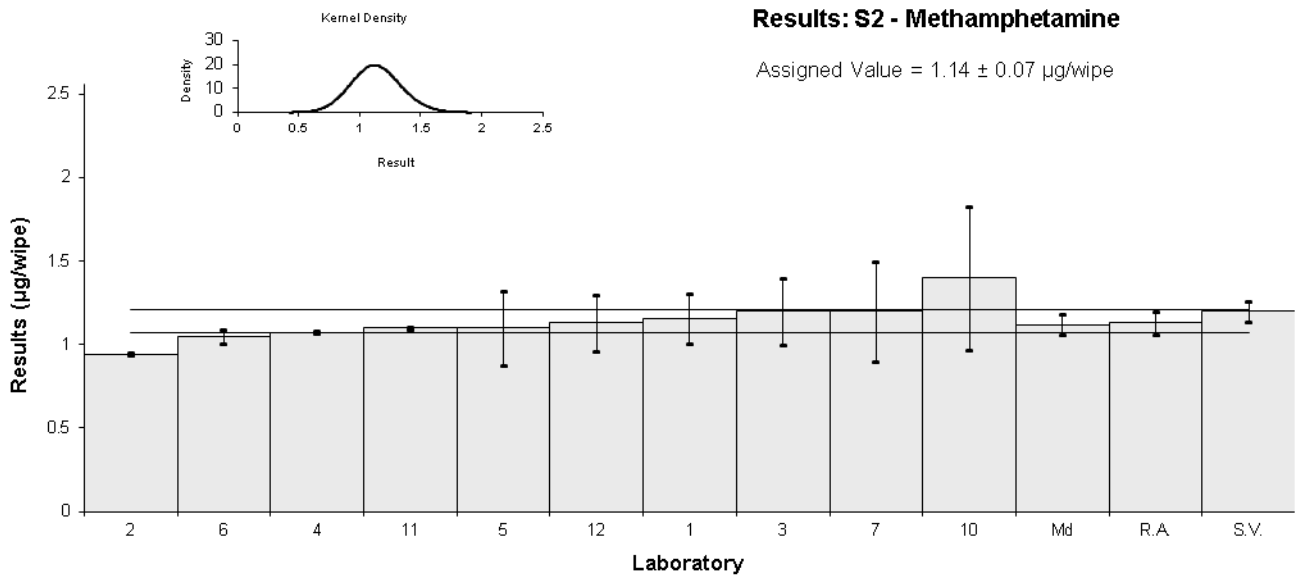
**Participant Results**

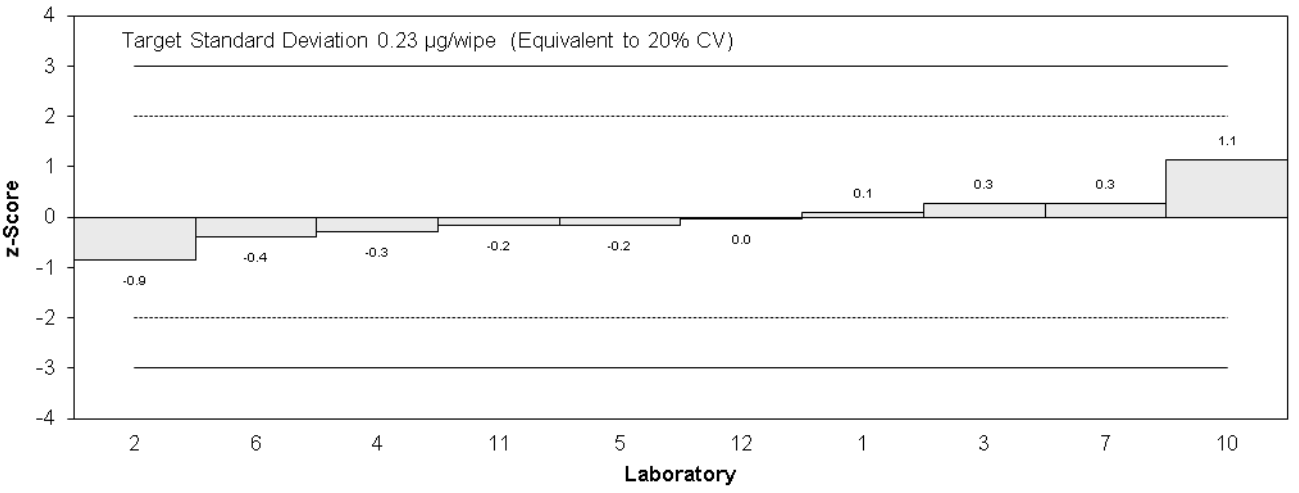
| Lab Code | Result | Uncertainty | Recovery | z-Score | $E_n$-Score |
|---|---|---|---|---|---|
| **1** | 1.16 | 0.15 | 106 | 0.09 | 0.12 |
| **2** | 0.944 | 0.007 | NR | -0.86 | -2.79 |
| **3** | 1.2 | 0.20 | NR | 0.26 | 0.28 |
| **4** | 1.073 | 0.007 | NR | -0.29 | -0.95 |
| **5** | 1.1 | 0.22 | 104 | -0.18 | -0.17 |
| **6** | 1.05 | 0.04 | 98 | -0.39 | -1.12 |
| **7** | 1.2 | 0.3 | 103 | 0.26 | 0.19 |
| **10** | 1.4 | 0.43 | 107.5 | 1.14 | 0.60 |
| **11** | 1.10 | 0.007 | 95 | -0.18 | -0.57 |
| **12** | 1.13 | 0.17 | NR | -0.04 | -0.05 |

**Statistics**

| | | |
|---|---|---|
| **Assigned Value*** | 1.14 | 0.07 |
| **Spike** | 1.20 | 0.06 |
| **Robust Average** | 1.13 | 0.07 |
| **Median** | 1.12 | 0.06 |
| **Mean** | 1.14 | |
| **N** | 10 | |
| **Max.** | 1.4 | |
| **Min.** | 0.944 | |
| **Robust SD** | 0.09 | |
| **Robust CV** | 8% | |

*The assigned value was calculated as the robust average of the combined results of duplicate pair samples S1 and S2


Kernel Density
Density
Result
Results: S2 - Methamphetamine
Assigned Value = 1.14 ± 0.07 µg/wipe
Results (µg/wipe)
Laboratory
2
6
4
11
5
12
1
3
7
10
Md
R.A
S.V.


z-Scores: S2 - Methamphetamine
Target Standard Deviation 0.23 µg/wipe (Equivalent to 20% CV)
z-Score
Laboratory
-0.9
-0.4
-0.3
-0.2
-0.2
0.0
0.1
0.3
0.3
1.1

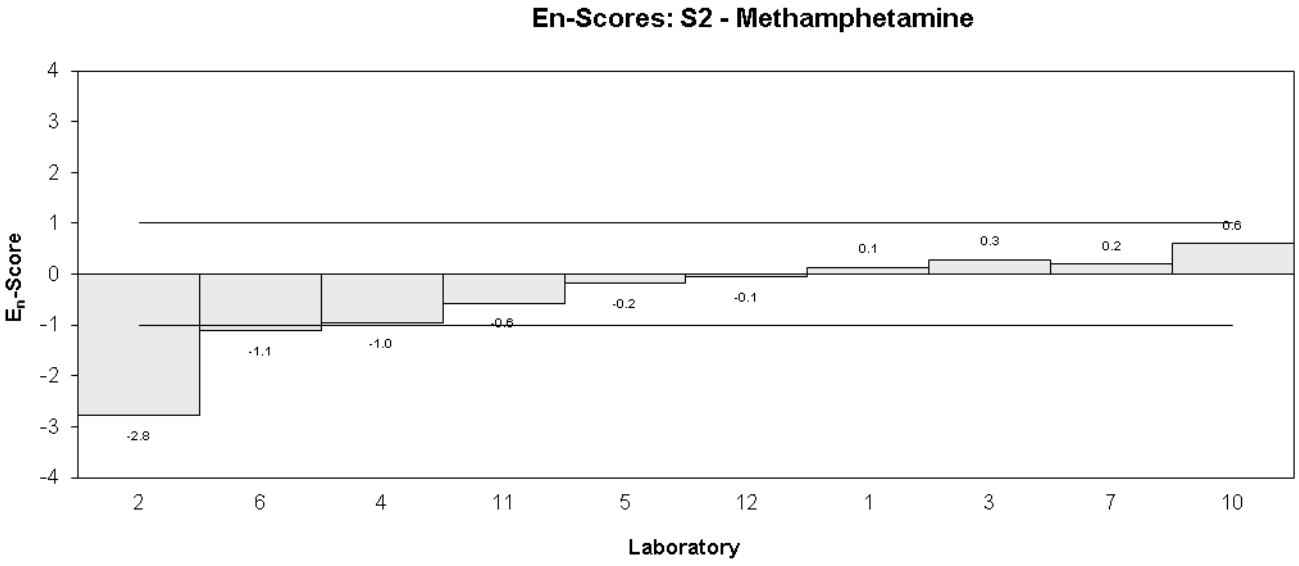

En-Scores: S2 - Methamphetamine
En-Score
Laboratory
-2.8
-1.1
-1.0
-0.6
-0.2
-0.1
0.1
0.3
0.2
0.6

Figure 3

Table 7

**Sample Details**

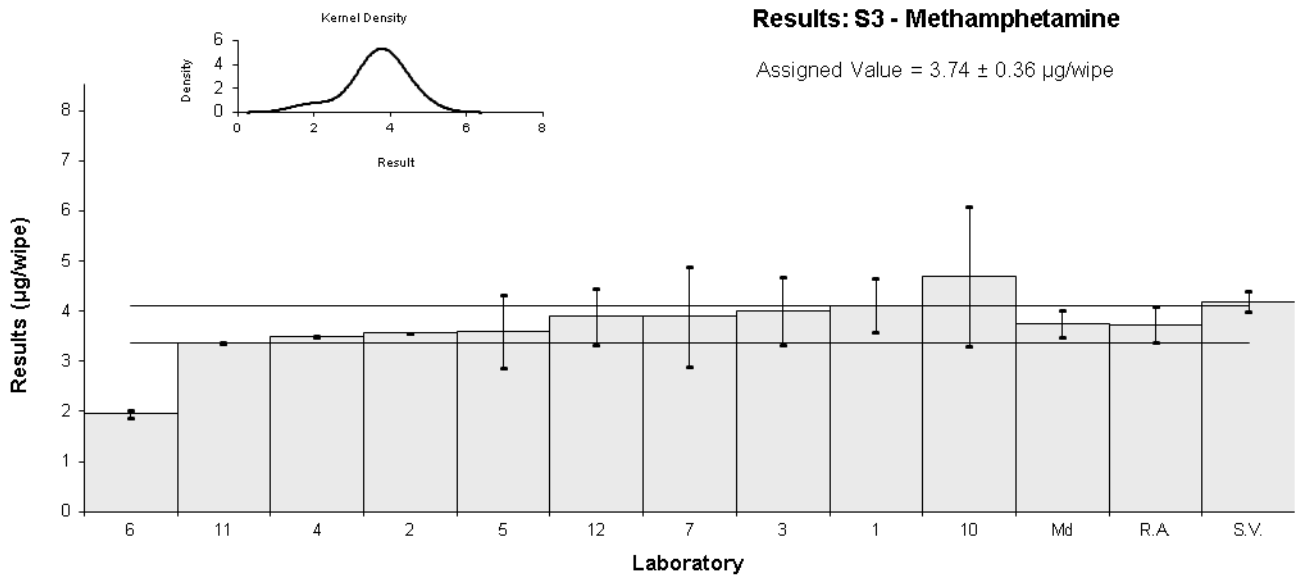
| | |
|---|---|
| **Sample No.** | S3 |
| **Matrix** | Wipe |
| **Analyte** | Methamphetamine |
| **Units** | µg/wipe |

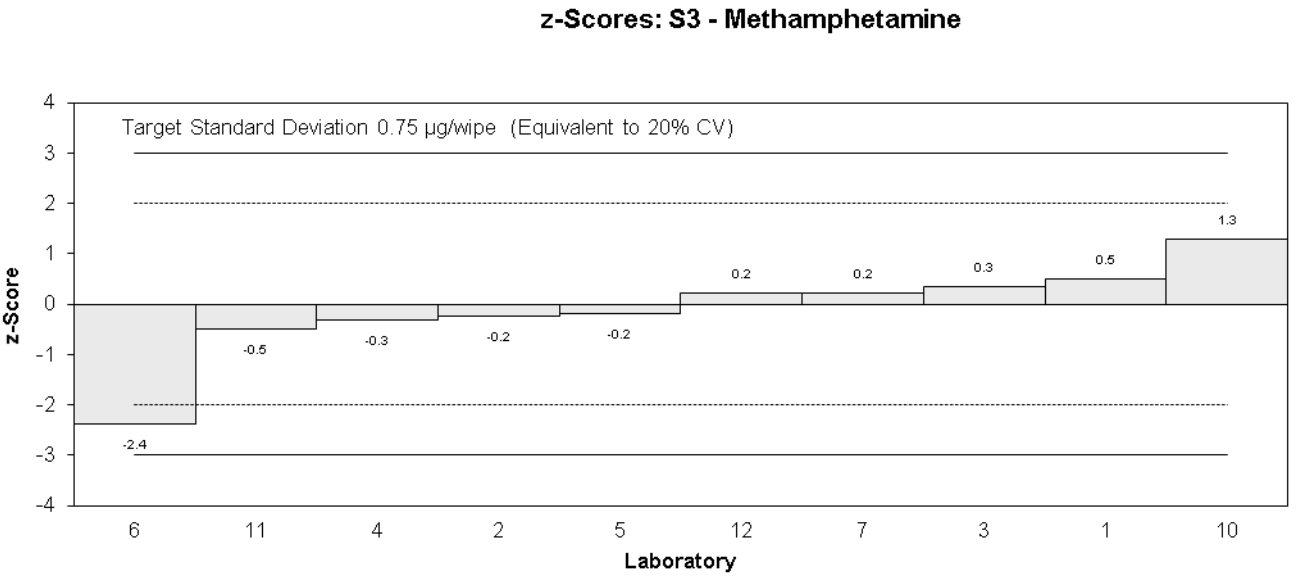
**Participant Results**

| Lab Code | Result | Uncertainty | Recovery | z-Score | $E_n$-Score |
|---|---|---|---|---|---|
| **1** | 4.12 | 0.54 | 106 | 0.51 | 0.59 |
| **2** | 3.566 | 0.007 | NR | -0.23 | -0.48 |
| **3** | 4.0 | 0.68 | NR | 0.35 | 0.34 |
| **4** | 3.497 | 0.007 | NR | -0.32 | -0.67 |
| **5** | 3.6 | 0.72 | 107 | -0.19 | -0.17 |
| **6** | 1.95 | 0.07 | 98 | -2.39 | -4.88 |
| **7** | 3.9 | 1 | 102 | 0.21 | 0.15 |
| **10** | 4.7 | 1.4 | 107.5 | 1.28 | 0.66 |
| **11** | 3.37 | 0.007 | 95 | -0.49 | -1.03 |
| **12** | 3.9 | 0.57 | NR | 0.21 | 0.24 |

**Statistics**

| | | |
|---|---|---|
| **Assigned Value** | 3.74 | 0.36 |
| **Spike** | 4.20 | 0.21 |
| **Robust Average** | 3.74 | 0.36 |
| **Median** | 3.75 | 0.27 |
| **Mean** | 3.66 | |
| **N** | 10 | |
| **Max.** | 4.7 | |
| **Min.** | 1.95 | |
| **Robust SD** | 0.45 | |
| **Robust CV** | 12% | |


Kernel Density
Density
Result
Results: S3 - Methamphetamine
Assigned Value = 3.74 ± 0.36 µg/wipe
Results (µg/wipe)
Laboratory
6
11
4
2
5
12
7
3
1
10
Md
R.A.
S.V.


z-Scores: S3 - Methamphetamine
Target Standard Deviation 0.75 µg/wipe (Equivalent to 20% CV)
z-Score
Laboratory
-2.4
-0.5
-0.3
-0.2
-0.2
0.2
0.2
0.3
0.5
1.3

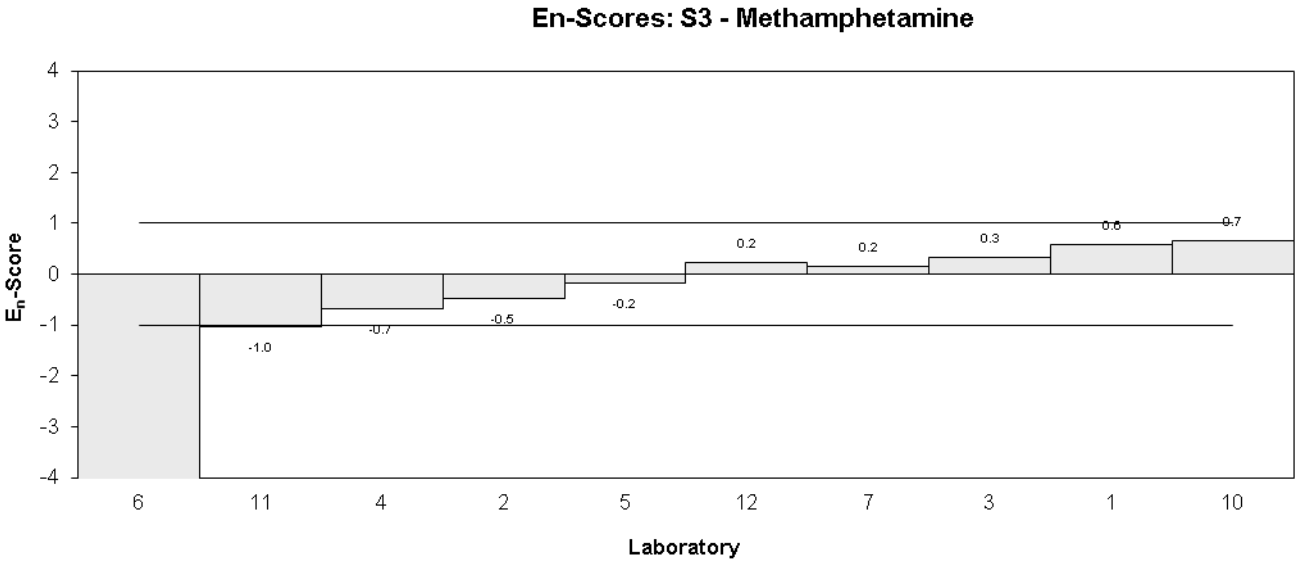

En-Scores: S3 - Methamphetamine
En-Score
Laboratory
-1.0
-0.7
-0.5
-0.2
0.2
0.2
0.3
0.6
0.7

Figure 4

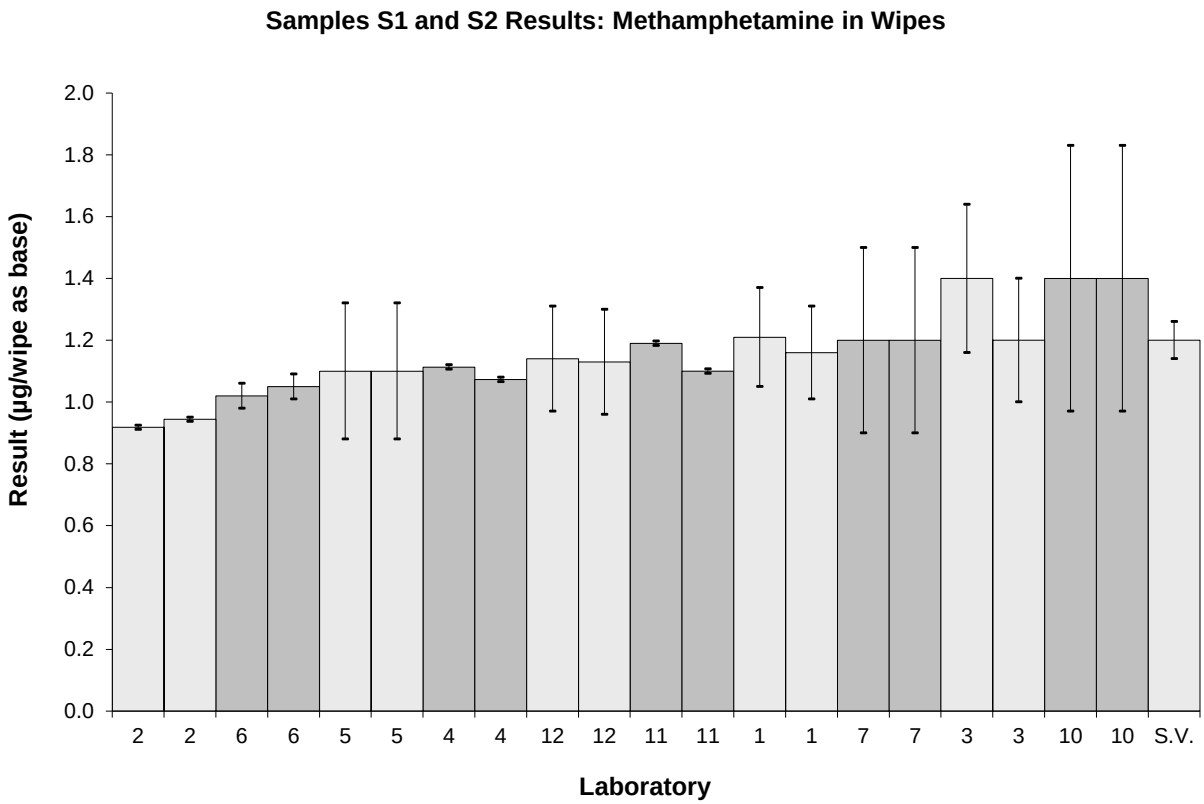



Figure 5 Results for Methamphetamine in Duplicate Pair Samples S1 and S2

## 7 DISCUSSION OF RESULTS

### 7.1 Assigned Value

Assigned values for methamphetamine in the duplicate pair of Samples S1 and S2 were calculated as the robust average of the combined results of both samples. The assigned value for sample S3 was the robust average of participants' results. All assigned values were in good agreement with the spike values.

**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.

### 7.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded measurement uncertainty associated with their results. All results were reported with an expanded measurement uncertainty, indicating that laboratories have addressed this requirement of ISO 17025.[6] The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Table 2.

The magnitude of reported uncertainties was within the range of 0.19% to 32% relative.

Laboratories with a satisfactory z-score and an unsatisfactory $E_n$-score are likely to have underestimated the expanded uncertainty associated with the result.

Laboratories 2, 4 and 11 reported results for the duplicate pair of Samples S1 and S2 that do not agree within their reported uncertainties (see Figure 5).

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 1.655 ± 0.331 µg/wipe drug as base, the recommended format is 1.66 ± 0.33 µg/wipe).[7]

### 7.3 z-Score

A target standard deviation equivalent to 20% PCV was used to calculate z-scores. Target SDs, the between-laboratory coefficient of variation predicted by the Thomson-Horwitz equation[8] and participants' coefficient of variation obtained in this study are presented in Table 8.

A summary of z-scores by laboratory is presented in Figure 6.

Of 30 numeric results, 29 (97%) returned a satisfactory z-score with $|z| \leq 2$.

- Nine participants (**1, 2, 3, 4, 5, 7, 10, 11** and **12)** returned satisfactory z-scores for all samples;
- One participant returned one questionable z-score;
- There were no reported results that returned an unsatisfactory z-score of greater than or equal to 3.

Table 8 Target SD (as PCV), Thompson Horwitz CV and Participants CV

| Sample | Analyte | Assigned Value (µg/wipe as base) | Target SD (as PCV) | Thompson Horwitz CV | Participants CV |
|---|---|---|---|---|---|
| S1 | Methamphetamine | 1.14 | 20% | 22% | 15% |
| S2 | Methamphetamine | 1.14 | 20% | 22% | 8% |
| S3 | Methamphetamine | 3.74 | 20% | 22% | 12% |

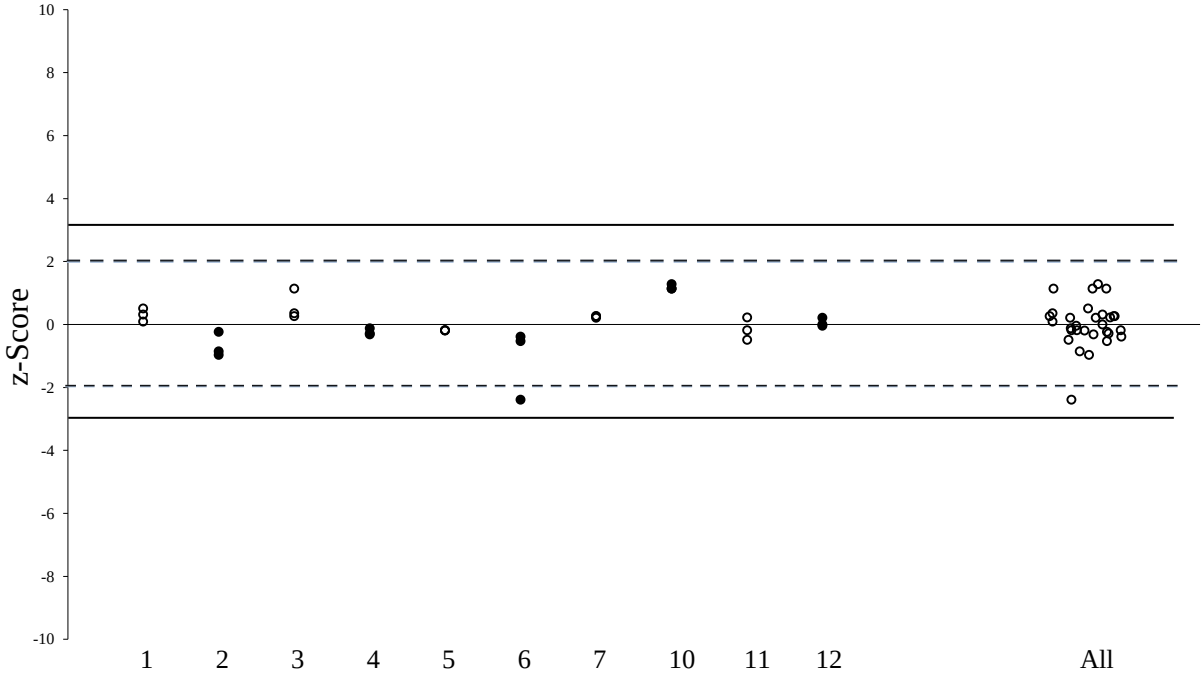



Figure 6 Summary of Participants' z-Scores

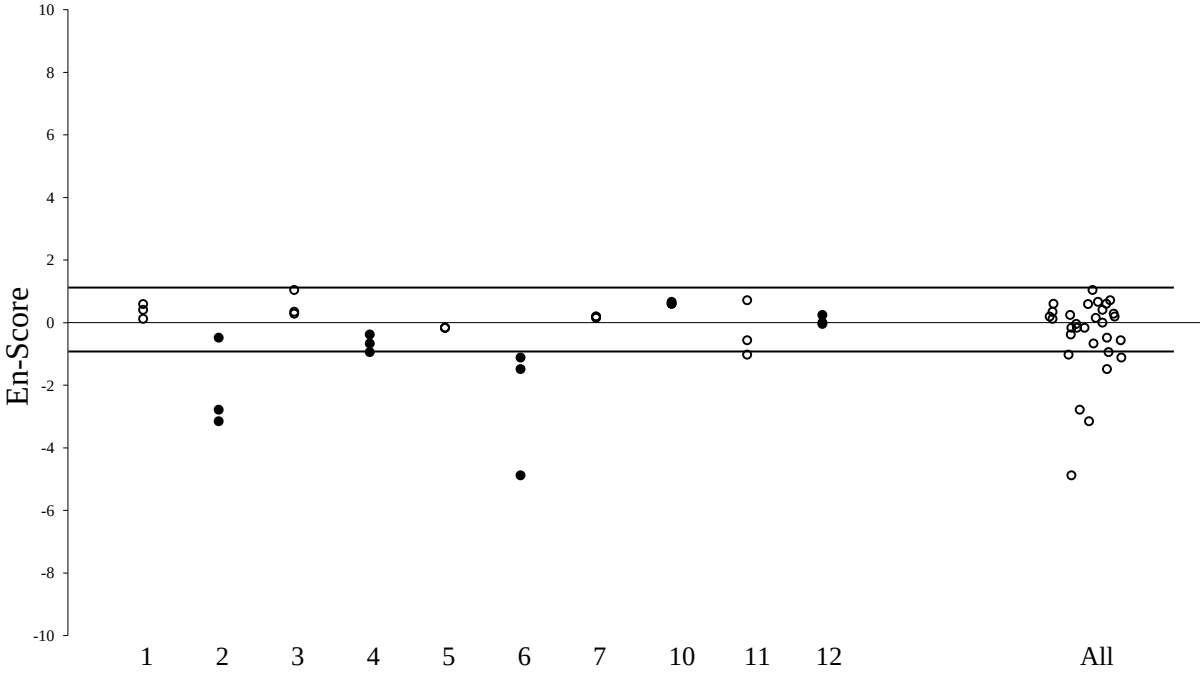



Figure 7 Summary of Participants' $E_n$-Scores

### 7.4 $E_n$-Score

The dispersal of participants' $E_n$-scores is graphically presented in Figure 7. Where a laboratory did not report an expanded uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the $E_n$-score.

Of 30 numeric results, 23 (77%) returned a satisfactory $E_n$-score with $|E_n| \leq 1$.

- Six participants (**1, 4, 5, 7, 10** and **12)** returned satisfactory $E_n$-scores for all samples.
- Four laboratories returned at least one questionable $E_n$-score; and
- Laboratory **6** returned an unsatisfactory $E_n$-score for all samples.

### 7.5 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 participants' accreditation status, methods and reference standards is presented in Table 9 and Figures 8 and 9. No trend in sample preparation method or instrumental technique was evident.

Table 9 Summary of Participants' Accreditation Status, Methods and Reference Standards

| **Accreditation status** | **Laboratory Code** |
|---|---|
| Yes to ISO 17025 | 1, 2, 3, 4, 5, 6, 7, 10, 11, 12 |

| **Sample Treatment** | **Laboratory Code** |
|---|---|
| Rotary mixer/shaking/tumbling | 1, 2, 3, 4, 5, 6, 7, 10, 11, 12 |
| Centrifuge | 2, 4, 11 |
| Sonication | 7 |
| pH adjustment | 5, 7 |

| **Desorption Solution** | **Laboratory Code** |
|---|---|
| 0.1 M Sulfuric acid | 1, 2, 3, 4, 5, 6, 7, 10, 11, 12 |

| **Instrumental technique** | **Laboratory Code** |
|---|---|
| LCMS/LCMSMS | 1, 2, 4, 5, 6, 7, 11, 12 |
| UPLC-MSMS | 3, 10 |

| **Sources of Calibration Standard** | **Laboratory Code** |
|---|---|
| Lipomed | 1, 2, 5 |
| Cerilliant | 6, 10 |
| Chiron | 7 |
| Other/none/unspecified | 3, 4, 11, 12 |

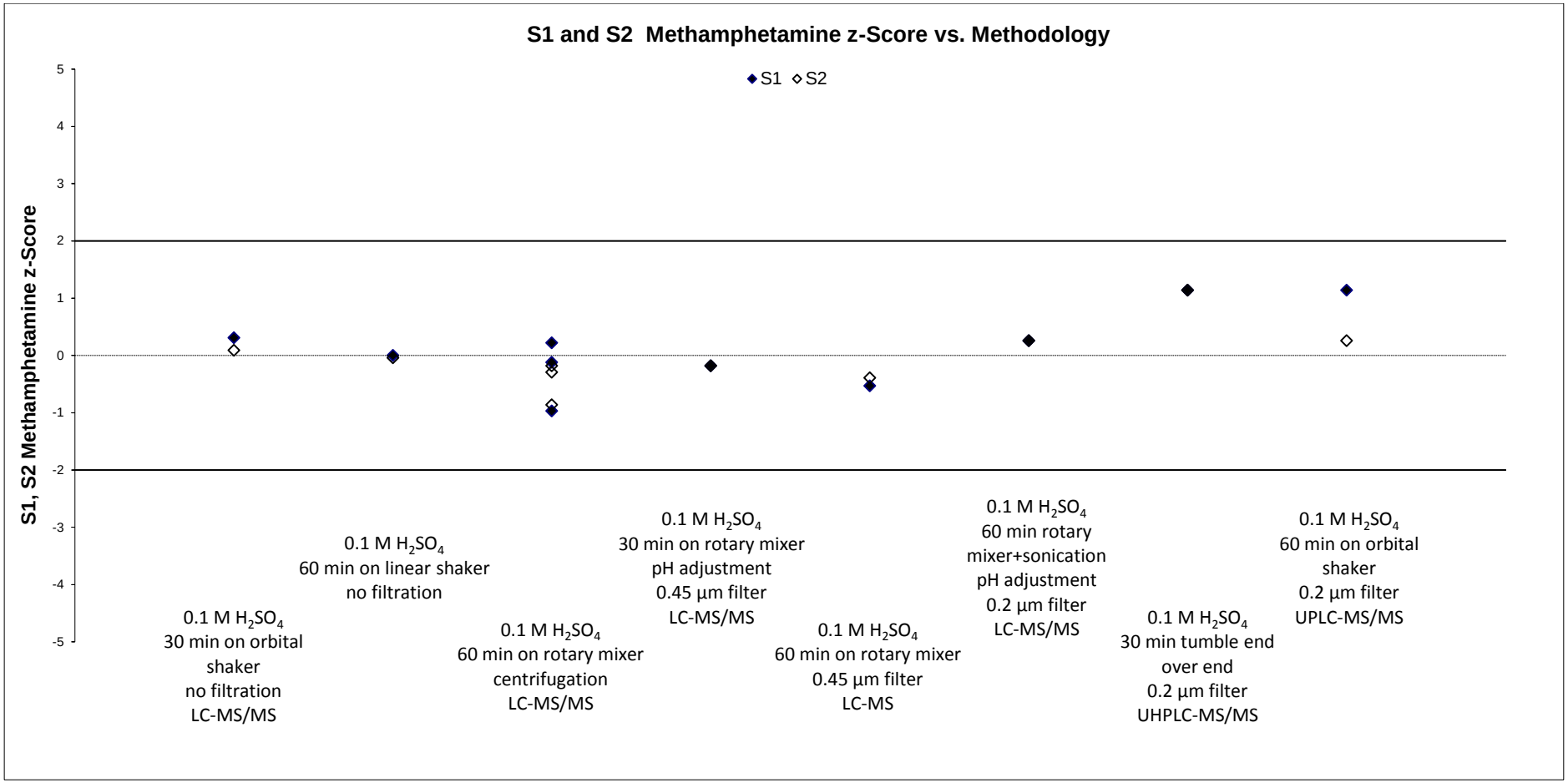



Figure 8 Participants' Performance for Methamphetamine in S1 and S2 versus Methodology

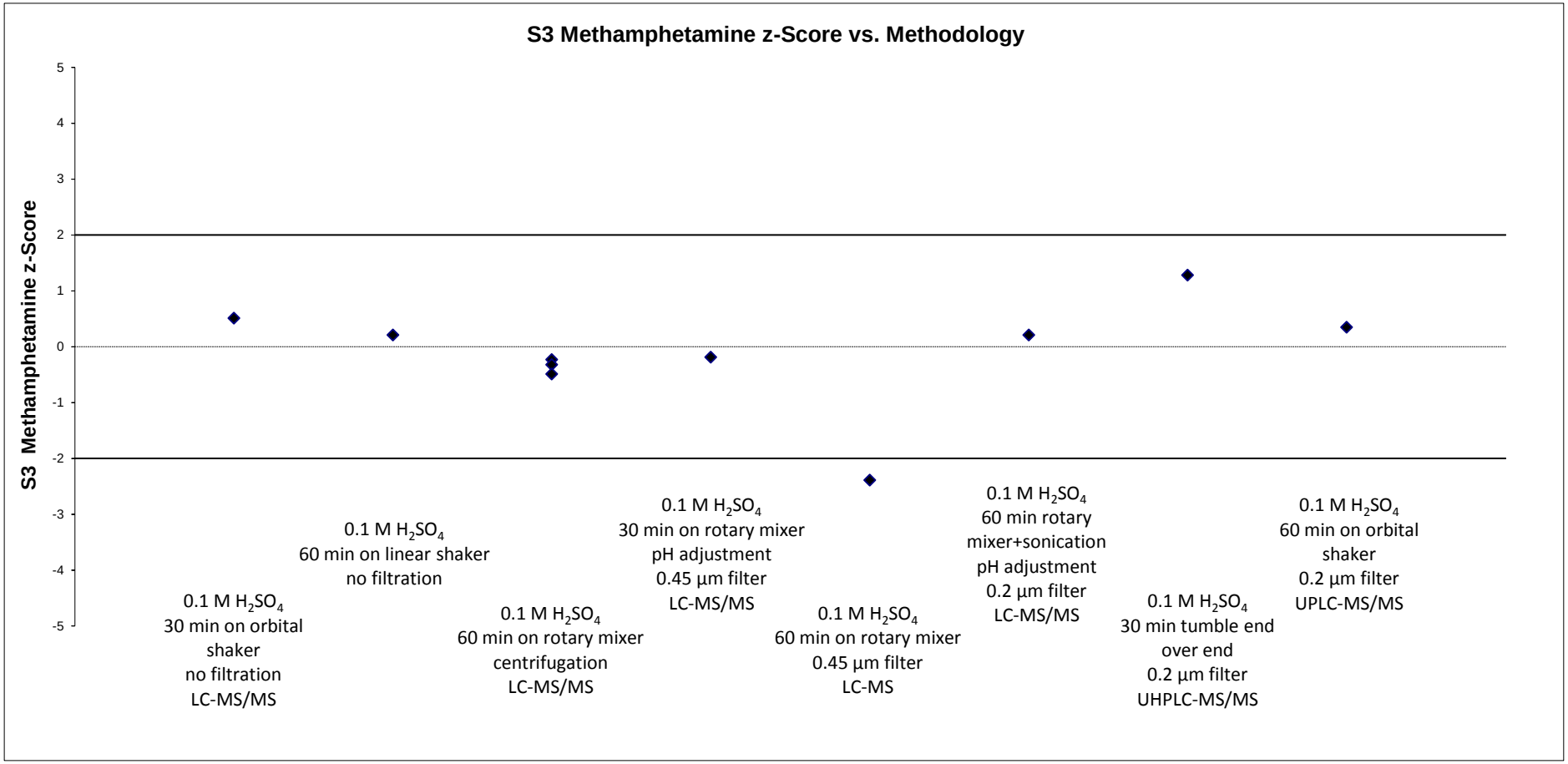



Figure 9 Participants' Performance for Methamphetamine in S3 versus Methodology

### 7.6 Participants' Within – Laboratory Repeatability

The study included one pair of duplicate samples (Samples S1 and S2). The same target standard deviation was used to calculate z-scores for methamphetamine in both samples. This allowed for the evaluation of the within-laboratory repeatability of laboratories.

Scatter plots of z-scores for S1 and S2 are presented in Figure 10. Most laboratories are plotted in the upper-right or lower-left quadrants. This is consistent with systematic bias being the major contributor to the observed variation in results.

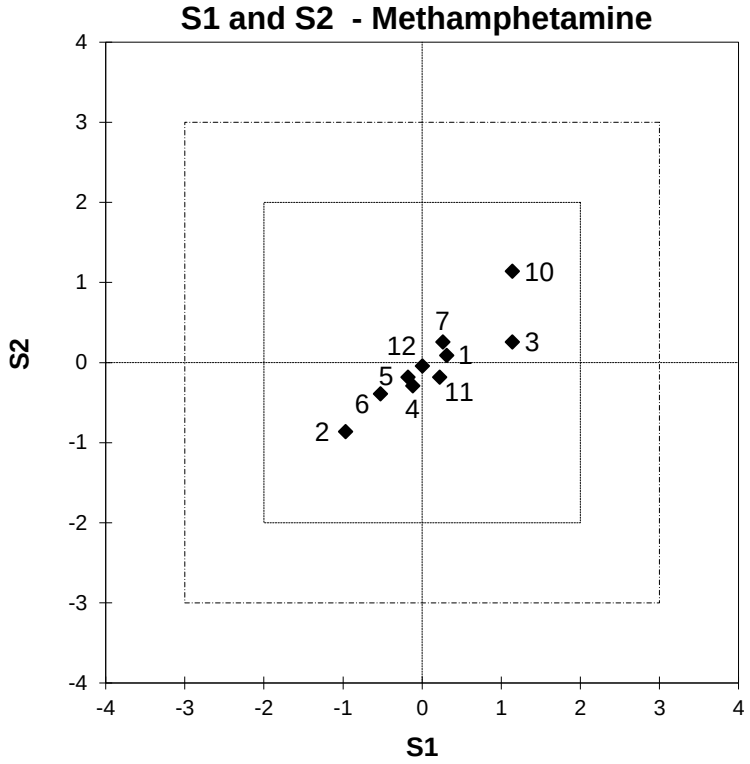



Figure 10 z-Score Scatter Plots for S1 and S2

### 7.7 Comparison with Previous NMI Proficiency Tests of Metals in Soil

AQA 19-09 is the second NMI proficiency test of illicit drugs in wipes.

Of the 84 results for which z-scores were calculated in the previous study, 81 (96%) returned satisfactory z-scores whilst in the present study of the 30 reported results, 29 (97%) were satisfactory.

Of the 10 participants who reported results, 7 also reported results for methamphetamine in the previous study AQA 18-08 Methamphetamine and MDMA in wipes. All performed satisfactorily in both studies except for one.

## 8 REFERENCES


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

[2] NMI 2016, *Chemical Proficiency Testing Study Protocol*, viewed 29 April 2020, <http://www.industry.gov.au>.

[3] NMI 2016, *Chemical Proficiency Testing Statistical Manual*, viewed 29 April 2020, <http://www.industry.gov.au>.

[4] Thompson, M., Ellison, SLR. & Wood, R. 2006. 'The international harmonized protocol for proficiency testing of (chemical) analytical laboratories', *Pure Appl. Chem*, vol 78, pp 145-196.

[5] ISO/IEC 13528 2015(E), *Statistical methods for use in proficiency testing by interlaboratory comparisons*.

[6] ISO/IEC 17025 2018, *General requirements for the competence of testing and calibration laboratories.*

[7] Eurachem 2012, *Quantifying Uncertainty in Analytical Measurement*, 3rd edition, viewed 29 April 2020, <http://www.eurachem.org/images/stories/Guides/pdf/QUAM2012_P1.pdf>.

[8] Thompson M. and Lowthian, P.J. 1995. 'A Horwitz-like function describes precision in a proficiency test', *Analyst*, vol 120, pp 271-272.

## APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

### A1.1 Sample Preparation

Samples used were large Liv-Wipe alcohol wipes bought from a local supplier. The wipes were removed from the individual packaging using tweezers and long-nosed pliers and unfolded. The analytes were spiked onto the wipes using calibrated positive displacement pipettes. After spiking, the methanol solvent was allowed to evaporate and the wipes were placed in amber glass jars, labelled and placed in a refrigerator.

Methamphetamine in S1 and S2 was at the same level and approximately half the concentration of S3.

### A1.2 Homogeneity Testing

No homogeneity test was conducted for this study. The same sample preparation procedure was followed as in the previous study. The test samples from previous studies were demonstrated to be sufficiently homogenous for the evaluation of participants' performance. The results reported gave no reason to question the samples' homogeneity.

## APPENDIX 2 – STABILITY STUDY

Participants were advised to store the samples refrigerated if analyses cannot be commenced on the day of receipt.

Sample condition on receipt and the date when the samples were received and analysed by the participants are presented in Table 10.

Table 10 Condition on Receipt and the Date When the Samples Were Received and Analysed

| Lab Code | Received Date | Arrival Condition | Analysis Date |
|---|---|---|---|
| 1 | 09/03/2020 | Acceptable | 16, 17, 18/03/2020 |
| 2 | 10/03/2020 | Good | 11/03/2020 |
| 3 | 10/03/2020 | Good | 10/03/2020 |
| 4 | 12/03/2020 | Excellent | 16/03/2020 |
| 5 | 10/03/2020 | good | 03/04/2020 |
| 6 | 10/03/2020 | good condition | 17/03/2020 |
| 7 | 10/03/2020 | good | 06/04/2020 |
| 10 | 10/03/2020 | Good | 11/03/2020 |
| 11 | 12/03/2020 | Good | 16/03/2020 |
| 12 | 10/04/2020 | | 17/04/2020 |

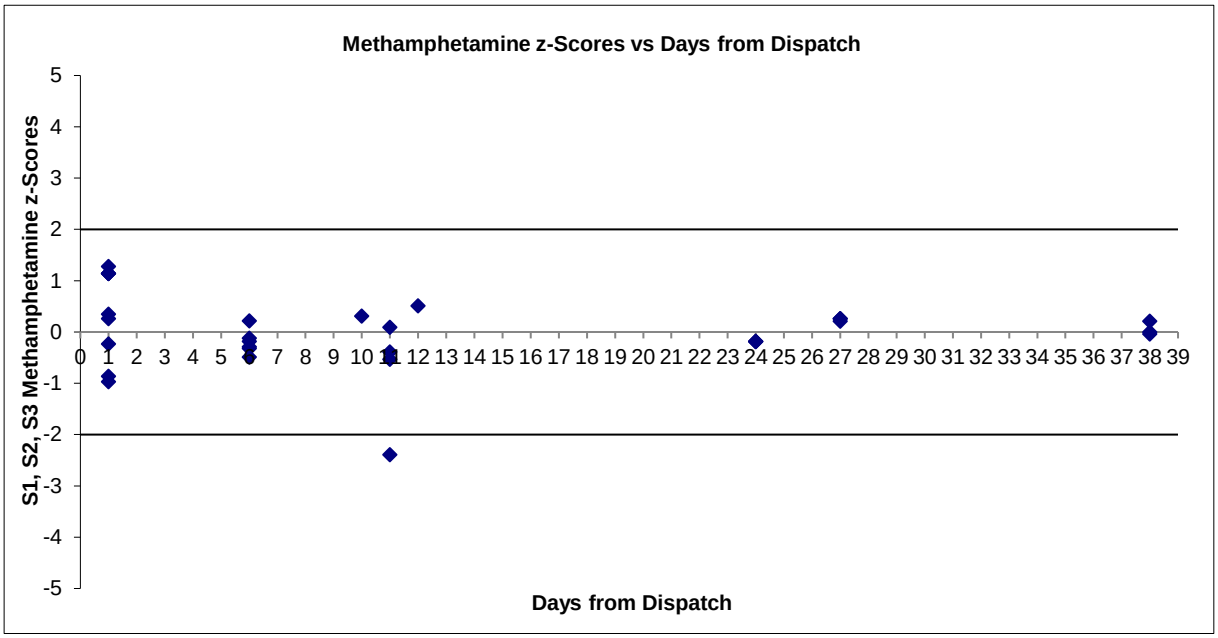



Figure 91 Methamphetamine z-Scores in S1, S2 and S3 vs. Analysis Date

No correlation between reported results, the received date, the analysis date or the sample condition at arrival was observed (Table 10 and Figure 11).

## APPENDIX 3 - MEASUREMENT UNCERTAINTY OF THE ROBUST AVERAGE

When the robust average is calculated using the procedure described in 'ISO13528:2015(E), Statistical methods for use in proficiency testing by interlaboratory comparisons – Annex C'[5], the uncertainty is estimated as:

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

where:

| | |
|---|---|
| $u_{rob\ average}$ | robust average standard uncertainty |
| $S_{rob\ average}$ | robust average standard deviation |
| $p$ | number of results |

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

A worked example is set out below in Table 11.

Table 11 Uncertainty of Assigned Value for Methamphetamine in Sample S3 as ug/wipe.

| | |
|---|---|
| No. results ($p$) | 10 |
| Robust average | 3.74 |
| $S_{rob\ average}$ | 0.45 |
| $u_{rob\ average}$ | 0.18 |
| $k$ | 2 |
| $U_{rob\ average}$ | 0.36 |

The robust average for **methamphetamine** in Sample S3 is **3.74 ± 0.36** µg/wipe as base.

## APPENDIX 4 - ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| CITAC | Cooperation on International Traceability in Analytical Chemistry |
| CRM | Certified Reference Material |
| CV | Coefficient of Variation |
| $\|E_n\|$ | Absolute value of an $E_n$-score |
| GUM | Guide to the expression of uncertainty in measurement |
| HPLC | High Performance Liquid Chromatography |
| ISO | International Standards Organisation |
| LC | Liquid Chromatography |
| 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 |
| NATA | National Association of Testing Authorities |
| NIOSH | National Institute for Occupational Safety and Health |
| NMI | National Measurement Institute Australia |
| NR | Not Reported |
| NT | Not Tested |
| PFAS | Per- and poly fluorinated alkyl substances |
| PT | Proficiency Test |
| PCV | Performance Coefficient of Variation |
| Robust CV | Robust between-laboratory Coefficient of Variation |
| Robust SD | Robust Standard Deviation |
| SI | International System of Units |
| Target SD ($\sigma$) | Target standard deviation |
| U(H)PLC | Ultra (High) Performance Liquid Chromatography |
| $\|z\|$ | Absolute value of a z-score |

**END OF REPORT**