# Fapas® – Food Chemistry Proficiency Test Report 02308

## Chloramphenicol in Prawns

## November-December 2016

## PARTICIPANT LABORATORY NUMBER

Participants can log in to Fapas® SecureWeb at any time to obtain their laboratory number for this proficiency test.

Laboratory numbers are displayed in SecureWeb next to the download link for this report.

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## SUMMARY

1. The test material for Fapas® – Food Chemistry proficiency test 02308 was dispatched in November 2016. Each participant received a prawns test material to be analysed for chloramphenicol.

2. An assigned value ($x_a$) was determined for chloramphenicol and in conjunction with the standard deviation for proficiency ($\sigma_p$) was used to calculate a z-score for each result.

3. Results for this proficiency test are summarised as follows:

| analyte | assigned value, $x_a$ µg/kg | number of scores, \|z\| ≤2 | total number of scores | % \|z\|≤2 |
|---|---|---|---|---|
| Chloramphenicol | 0.857 | 93 | 110 | 85 |

## CONTENTS

# 1. INTRODUCTION

## 1.1. Proficiency Testing

Proficiency testing aims to provide an independent assessment of the competence of participating laboratories. Together with the use of validated methods, proficiency testing is an essential element of laboratory quality assurance.

Further details of the Fapas® – Food Chemistry proficiency testing scheme are available in our protocols [3, 4].

# 2. TEST MATERIAL

## 2.1. Preparation

Preparation of the samples for this proficiency test was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation [2].

The test material was prepared from Whiteleg shrimp (*Litopenaeus vannamei*). Chloramphenicol was spiked into the test material.

Samples were stored at -80°C until dispatch.

## 2.2. Homogeneity

To test for homogeneity, randomly selected test materials were analysed in duplicate. Testing was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation [2].

These data showed sufficient homogeneity and were not included in the subsequent calculation of the assigned value.

## 2.3. Dispatch

The start date was 22 November 2016. Test materials were sent to 130 participants.

# 3. RESULTS

The instructions for reporting results were as follows:

1) Determine the level of chloramphenicol present in the test material, in µg/kg, as received, corrected for recovery. PLEASE NOTE:
2) Chloramphenicol is to be measured as the parent compound.
3) In the Internal Standard/Recovery Correction column on the results form:
   - If you added an internal standard at the start, enter 'Y',
   - If you have measured your % recovery, enter your % recovery,
   - If you used a matrix-extracted calibration curve, enter 'M',
   - If you used standard addition, enter 'S'.

   It is important that you report the results in this way so that we can include as many results as possible in the statistical analysis.

Please report CCβ (CCbeta) instead of limit of quantification (LoQ). CCβ, the 'detection capability' is defined as: the smallest content of the analyte that may be detected in a sample with a chance of 5% of a false negative decision [5, 6]. If you do not know the CCβ, you can report your limit of quantification, but please mention this in the comments box.

Results were submitted by 114 participants (88%) before the closing date for this test, 22 December 2016.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1.

Participants' comments are given in Table 2.

The analytical methods used by each participant are summarised in APPENDIX I.

## 4. STATISTICAL EVALUATION OF RESULTS

The results submitted by participants were statistically analysed in order to provide an assigned value for chloramphenicol. The assigned values were then used in combination with the standard deviation for proficiency, $\sigma_p$, to calculate a z-score [7] for each result. The procedure is detailed in the relevant protocols [3, 4].

Further background on the procedure followed can be found in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [8].

### 4.1. Calculation of the Assigned Value, $x_a$

The assigned value, $x_a$, for chloramphenicol was derived from the consensus of the results submitted by participants.

The following results were excluded from the calculation of the assigned value:

i) qualitative or semi-quantitative results, e.g. >0.2,

ii) results reported as approximately 10, 100 or 1000 × greater or smaller than the majority of submitted results (as these were considered to be reporting errors),

iii) results where use of neither a matrix-extracted calibration curve, nor a recovery %, nor standard addition was reported (as these results were considered to be uncorrected for recovery).

For chloramphenicol, this procedure was straightforward and the robust mean was chosen as the assigned value.

The assigned value for chloramphenicol is shown in Table 3.

### 4.2. Standard Deviation for Proficiency, $\sigma_p$

The standard deviation for proficiency, $\sigma_p$, was set at a value that reflects best practice for the analyses in question.

For chloramphenicol, $\sigma_p$ was derived from the appropriate form of the Horwitz equation [9].

The values for $\sigma_p$ used to calculate z-scores from the reported results of this test are given in Table 3.

### 4.3. Individual z-Scores

Participants' z-scores were calculated as:

$$z = \frac{(x - x_a)}{\sigma_p}$$

where $x$ = the participant's reported result,
$x_a$ = the assigned value
and $\sigma_p$ = the standard deviation for proficiency.

Participants' z-scores for chloramphenicol are given in Table 1 and shown as a histogram in Figure 1. It is possible for the z-scores published in this report to differ slightly from the z-score that can be calculated using the formula given above. These differences arise from the necessary rounding of the actual assigned values and standard deviations for proficiency prior to their publication in Table 3.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for chloramphenicol are given in Table 4.

## 5. INTERPRETATION OF SCORES

In normal circumstances, over time, about 95% of z-scores will lie in the range $-2 \leq z \leq 2$. Occasional scores in the range $2 < |z| < 3$ are to be expected, at a rate of 1 in 20. Whether or not such scores are of importance can only be decided by considering them in the context of the other scores obtained by that laboratory.

Scores where $|z| > 3$ are to be expected at a rate of about 1 in 300. Given this rarity, such z-scores very strongly indicate that the result is not fit-for-purpose and almost certainly requires investigation.

The consideration of a set or sequence of z-scores over time provides more useful information than a single z-score. Examples of suitable methods of comparison are provided in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [8].

## 6. REFERENCES


1 Adobe Certified Document Services, http://www.adobe.com/misc/pki/cds_cp.html, accessed 12/05/2016.

2 ISO/IEC 17043:2010, Conformity assessment – General requirements for proficiency testing.

3 Fapas®, 2016, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 5, Issued September 2016.

4 Fapas®, 2016, Protocol for Proficiency Testing Schemes, Part 2 – Fapas® – Food Chemistry Version 4, Issued September 2016.

5 Commission Decision 2002/657/EC of 12 August 2002 implementing Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results.

6 ISO 11843-1:1997, Capability of detection -- Part 1: Terms and definitions.

7 AMC Tech Brief No. 74, z-Scores and other scores in chemical proficiency testing – their meanings, and some common misconceptions, Anal. Methods, 2016, 8, 5553.

8 Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem.*, **78**, No. 1, 145–196.

9 Thompson, M., 2000, Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing, *Analyst*, **125**, 385-386.

## Table 1: Results and z-Scores

| laboratory number | analyte | | | |
|---|---|---|---|---|
| | Chloramphenicol assigned value: 0.857 µg/kg | | | |
| | result | IS %rec. | CCβ | z-score |
| 001 | 0.92 | Y | 0.4 | 0.3 |
| 002 | 0.98 | Y M | 0.1 | 0.7 |
| 003 | 0.91 | Y | 0.1 | 0.3 |
| 004 | > 0.2 | | 0.2 | |
| 005 | 1 | 1 | 0.25 | 0.8 |
| 006 | 0.38 | | | **-2.5** |
| 007 | 1.03 | | | 0.9 |
| 008 | 1.102 | | | 1.3 |
| 009 | 0.93 | | 0.3 | 0.4 |
| 010 | 1.01 | | 0.2 | 0.8 |
| 011 | 0.89 | Y | | 0.2 |
| 012 | 0.48 | Y | 0.2 | -2.0 |
| 013 | 0.65 | | | -1.1 |
| 014 | 1.01 | Y | 0.5 | 0.8 |
| 015 | 0.82 | Y | 0.15 | -0.2 |
| 016 | 0.90 | Y | 0.1 | 0.2 |
| 017 | 0.95 | Y % S | 0.05 | 0.5 |
| 018 | 0.982 | Y, 113 | 0.3 | 0.7 |
| 019 | 0.70 | M | 0.09 | -0.8 |
| 020 | 0.91 | | | 0.3 |
| 021 | 0.83 | | | -0.1 |
| 022 | 0.838 | Y 93%, M | | -0.1 |
| 023 | 0.792 | Y 106% M | 0.069 | -0.3 |
| 024 | 13.976 | M | ≤6.5 | **69.5** |
| 025 | 0.88 | Y M | 0.13 | 0.1 |

Y = internal standard used M = matrix-extracted calibration S = standard addition
z-scores outside |z| >2 are shown in **bold,** see Section 5

## Table 1 (continued): Results and z-Scores

| laboratory number | analyte | | | |
|---|---|---|---|---|
| | Chloramphenicol assigned value: 0.857 µg/kg | | | |
| | result | IS %rec. | CCβ | z-score |
| 026 | 1.13 | | 0.025 | 1.4 |
| 027 | 0.348 | 80% | 0.0125 | **-2.7** |
| 028 | 0.95 | Y | 50 | 0.5 |
| 029 | 0.76 | Y M | 0.02 | -0.5 |
| 030 | 1.01 | Y M | 0.38 | 0.8 |
| 031 | 0.99 | 81.5 % | | 0.7 |
| 032 | 0.93 | Y 98.2% | 0.1 | 0.4 |
| 033 | 0.868 | Y | 0.1 | 0.1 |
| 034 | 0.94 | Y 65% M | 0.058 | 0.4 |
| 035 | 0.966 | Y | 0.1 | 0.6 |
| 036 | 1.0 | Y | 0.07 | 0.8 |
| 037 | 0.659 | 118.45% | 0.2 | -1.1 |
| 038 | <0.2 | Y | | |
| 039 | 0.9 | Y | 0.5 | 0.2 |
| 040 | 0.63 | Y | 0.5 | -1.2 |
| 041 | 0.99 | Y | 0.20 | 0.7 |
| 042 | 0.67 | Y 89.10% | | -1.0 |
| 043 | 0.65 | Y | 0.35 | -1.1 |
| 044 | 1.0 | Y M | 0.30 | 0.8 |
| 045 | 0.86 | Y M | 0.1 | 0.0 |
| 046 | 0.113 | Y | 0.12 | **-3.9** |
| 047 | 0.94 | Y 77% | 0.1 | 0.4 |
| 048 | 1.2472 | | | **2.1** |
| 049 | 1.15 | Y | 0.14 | 1.6 |
| 050 | 0.396 | M | 0.3 | **-2.4** |

Y = internal standard used M = matrix-extracted calibration
z-scores outside |z| >2 are shown in **bold,** see Section 5

## Table 1 (continued): Results and z-Scores

| laboratory number | analyte | | | |
|---|---|---|---|---|
| | Chloramphenicol assigned value: 0.857 µg/kg | | | |
| | result | IS %rec. | CCβ | z-score |
| 051 | 0.141 | 100.91% | 38.24% | **-3.8** |
| 052 | 0.81 | 104 | 0.5 | -0.3 |
| 053 | 0.85 | Y | | 0.0 |
| 054 | 0.88 | Y | 1.0 | 0.1 |
| 055 | 0.22 | Y 97.0% & 97.5% M | 0.3 | **-3.4** |
| 056 | 1.08 | M | 0.5 | 1.2 |
| 057 | 0.75 | Y 103% S | 0.1 | -0.6 |
| 058 | 1.16 | Y 106.5% S | 0.2 | 1.6 |
| 059 | 0.800 | 112% | 0.07 | -0.3 |
| 060 | 0.51 | Y 108% | 0.1 | -1.8 |
| 061 | 1.15 | Y | 0.1 | 1.6 |
| 062 | 0.956 | Y | 0.1 | 0.5 |
| 063 | 0.97 | Y | | 0.6 |
| 064 | 0.74 | 90.18% | 0.04 | -0.6 |
| 065 | 1.07 | 90% | 0.1 | 1.1 |
| 066 | 0.6741 | Y | 0.3 | -1.0 |
| 067 | 1.25 | Y | | **2.1** |
| 068 | 0.88 | 109 | 0.3 | 0.1 |
| 069 | 0.68 | 90.5% | 0.2 | -0.9 |
| 070 | 0.59 | 82% | 0.3 | -1.4 |
| 071 | 0.94 | Y | 0.12 | 0.4 |
| 072 | >0.3 | NA | | |
| 073 | 0.061 | 100.20% | | **-4.2** |
| 074 | 0.82 | Y 99% M | 0.3 | -0.2 |
| 075 | 0.71 | Y 93.60% | | -0.8 |

Y = internal standard used
M = matrix-extracted calibration
S = standard addition
z-scores outside |z| >2 are shown in **bold,** see Section 5

## Table 1 (continued): Results and z-Scores

| laboratory number | analyte | | | |
|---|---|---|---|---|
| | Chloramphenicol assigned value: 0.857 µg/kg | | | |
| | result | IS %rec. | CCβ | z-score |
| 076 | 0.11 | Y 97.7% M S | 0.05 | **-4.0** |
| 077 | 0.90 | Y M | 0.04 | 0.2 |
| 078 | 0.29 | 84% | | **-3.0** |
| 079 | 0.80 | Y S | 0.1 | -0.3 |
| 080 | 0.76 | | 0.24 | -0.5 |
| 081 | 1.12 | 95% | 0.1 | 1.4 |
| 082 | 0.86 | Y 103% | 0.1 | 0.0 |
| 083 | 0.81 | Y 106.6 M | 0.1 | -0.3 |
| 084 | 0.82 | Y | 0.15 | -0.2 |
| 085 | 0.82 | Y | 0.1 | -0.2 |
| 086 | 0.91 | Y 90% | 0.05 | 0.3 |
| 087 | 0.74 | Y M | | -0.6 |
| 088 | 0.820 | Y | 0.2 | -0.2 |
| 089 | 0.141 | | | **-3.8** |
| 090 | 1.09 | / | 0.1 | 1.2 |
| 091 | 0.990 | 112.8% | 0.0 | 0.7 |
| 092 | 0.89 | Y M | 0.07 | 0.2 |
| 093 | 0.91 | Y 95% M | 0.22 | 0.3 |
| 094 | 0.74 | Y | | -0.6 |
| 095 | 0.92 | Y M | 0.13 | 0.3 |
| 096 | 0.749 | 81.6 | 0.08 | -0.6 |
| 097 | 1.00 | Y | 0.3 | 0.8 |
| 098 | 0.92 | Y | | 0.3 |
| 099 | 1.1 | n/a | n/a | 1.3 |
| 100 | 0.843 | Y 82.9% | 0.2 | -0.1 |

Y = internal standard used M = matrix-extracted calibration S = standard addition
z-scores outside |z| >2 are shown in **bold,** see Section 5

## Table 1 (continued): Results and z-Scores

| laboratory number | analyte | | | |
|---|---|---|---|---|
| | Chloramphenicol assigned value: 0.857 µg/kg | | | |
| | result | IS %rec. | CCβ | z-score |
| 101 | 0.55 | Y | | -1.6 |
| 102 | 0.810 | N | 0.1 | -0.3 |
| 103 | 0.54 | | | -1.7 |
| 104 | 1.46 | 100% | | **3.2** |
| 105 | 0.69 | | | -0.9 |
| 106 | 1.24 | Y | 0.2 | 2.0 |
| 107 | 0.216 | | | **-3.4** |
| 108 | 0.80 | | | -0.3 |
| 109 | 3.0 | 100 | 0.5 | **11.4** |
| 110 | 0.435 | | | **-2.2** |
| 111 | ND (Not Detected) | Y | 0.100 | |
| 112 | 1.17 | Y | | 1.7 |
| 113 | 0.82 | 100 % | | -0.2 |
| 114 | 0.55 | | | -1.6 |

Y = internal standard used N = internal standard not used
z-scores outside |z| >2 are shown in **bold,** see Section 5

## Table 2: Participants' Comments

| laboratory number | Comments |
|---|---|
| 002 | The sample was first screened positive with ELISA (Europroxima) |
| 004 | Screening Elisa |
| 009 | Confirmed chloramphenical at 0.95 ug/kg with LC-MS/MS using FDA-LIB 4473. Internal Standard added at the start; Chloramphenicol D5 with 100 % recovery. |
| 011 | Chloramphenicol LOQ: 0.1µg/kg |
| 016 | The given value for the ccbeta ist the LOQ. |
| 017 | CCbeta is our LoQ |
| 018 | LOQ used in place of CCbeta |
| 022 | ELISA method, LoQ 0.1µg/kg. Sample box was not seal upon arrival, with temp. of 25.7C, the meat had decomposed smell, slight pink color. The bottle was not hard or soft swollen, kept at -20C immediately until analysis |
| 027 | 0.0125µg/kg is the limit of quantification |
| 028 | Instead of CCbeta LOQ is given |
| 029 | LoQ instead of CCbeta |
| 031 | LOD = 0.002 µg/kg and LOQ = 0.008 µg/kg |
| 032 | LOQ=0.1µg/kg |
| 037 | LoQ |
| 038 | LOQ = 0.2ug/kg |
| 039 | LoQ is reported in CCbeta column |
| 041 | CCbeta entered is the LOQ of the method we used. |
| 042 | LoQ 0.1 µg/kg |
| 044 | LoD reported |
| 045 | LOQ=0.1 (µg/kg) |
| 047 | Results are corrected with recovery and % Recovery is 77% |
| 049 | Chloramphenicol + Chloramphenicol-Glucuronide: 1.55 µg/kg |
| 050 | use limit of quantification(LOQ) |
| 051 | % recovery correction of 0.3 ppb |
| 053 | LOQ is 0.05 µg/kg |
| 055 | LOQ: 0.3µg/kg |
| 056 | 0.5 is limit of quantification |
| 057 | CCbeta is replaced by limit of quantification(LOQ) |
| 061 | limit of quantification |
| 064 | Our laboratory method limit of quantification = 0.04 ug/kg |
| 068 | LOQ instead of CC-Beta |
| 070 | limit of quantification is filled in CCbeta-Box |

## Table 2 (continued): Participants' Comments

| laboratory number | Comments |
|---|---|
| 071 | CCbeta info is LOQ |
| 072 | Reported as per Lab Instructions |
| 073 | Limit of Quantification is 0.050 ug/kg |
| 087 | LoQ |
| 090 | 0.1ug/kg (LOQ) |
| 091 | *limit of quantification |
| 092 | Matrix matched calibration used |
| 095 | results are based on matrix based calibration curve |
| 099 | Determination for Chloramphenicol for UPLCMSMS |
| 100 | Internal standard of injection and not of preparation- LOQ and not CCbeta. |
| 102 | Analyte detected in blank below LOQ. Reported result not corrected. |
| 106 | cc beta value is LOQ |
| 111 | Sample received in a bad condition, packaging was bulging and bad smell |

comments are as submitted by participants

## Table 3: Assigned Values and Standard Deviations for Proficiency

| analyte | data points, *n* | assigned value, $x_a$ µg/kg | uncertainty, *u* | standard deviation for proficiency, $\sigma_p$ | |
|---|---|---|---|---|---|
| Chloramphenicol | 86 | 0.857 | 0.0203 | Horwitz [9] | 0.189 |

## Table 4: Number and Percentage of z-Scores where |z| ≤2

| analyte | number of scores where \|z\| ≤2 | total number of scores | % \|z\|≤2 |
|---|---|---|---|
| Chloramphenicol | 93 | 110 | 85 |

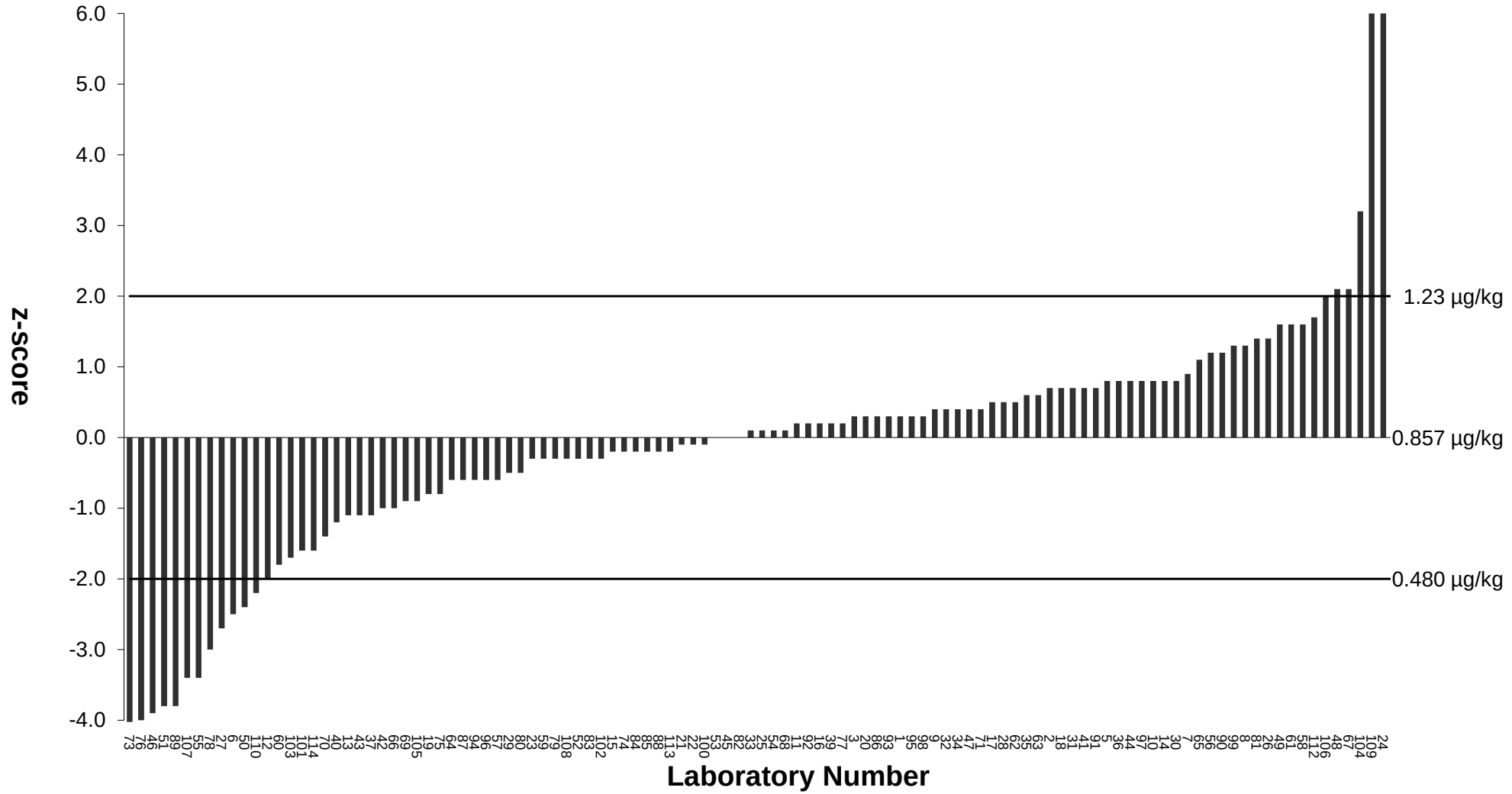


**Figure 1: z-Scores for Chloramphenicol**

## APPENDIX I: Analytical Methods Used by Participants

Methods are tabulated according to the information supplied by participants, but some responses may have been combined or edited for clarity.

| Accredited Method Used | laboratory number |
|---|---|
| yes | 001 005 010 011 013 014 015 016 017 018 024 025 029 030 033 035 036 038 040 041 042 044 046 047 049 052 053 054 055 056 057 060 061 062 063 064 065 066 069 071 074 075 078 079 080 081 082 083 084 085 087 090 092 093 094 095 099 100 102 103 105 106 107 111 |
| no | 002 003 006 007 008 009 012 020 023 026 032 037 039 073 076 077 088 089 098 109 114 |

| Method Based On | laboratory number |
|---|---|
| International Standard | 016 024 046 053 054 063 065 080 083 084 094 102 105 |
| National Standard | 007 011 012 018 029 032 035 039 040 041 042 052 056 057 060 061 062 074 075 078 079 081 082 085 087 088 090 103 |
| Paper Published In An International Journal | 037 043 047 071 092 099 |
| Manufacturer/Kit Instructions/Technical Note | 006 008 009 013 020 026 069 073 089 107 114 |
| In house method | 001 002 003 005 010 014 015 017 023 025 030 036 038 044 049 055 064 066 076 077 093 095 098 100 106 109 111 |

| Sample Weight (g) | laboratory number |
|---|---|
| ≥1 - <2 | 003 006 012 035 037 047 054 061 062 063 076 081 082 087 093 098 105 |
| ≥2 - <5 | 001 008 009 010 011 013 015 016 017 018 020 023 025 029 036 038 040 042 043 046 049 052 066 069 073 077 078 079 083 085 089 090 092 094 095 102 103 107 109 111 114 |
| ≥5 - <10 | 002 005 007 024 030 032 039 041 044 055 056 057 060 064 065 074 075 080 084 088 099 100 106 |
| ≥10 - <25 | 014 053 071 |

| Extraction Procedure | laboratory number |
|---|---|
| centrifuge | 009 |
| cold solvent extraction at atmospheric pressure | 005 012 018 030 036 037 041 060 065 066 074 080 087 098 107 114 |
| homogenize | 039 |
| maceration/homogenisation | 007 015 043 052 056 061 069 071 075 088 |
| QUECHERS | 109 |
| shaking | 002 003 010 017 023 024 025 029 035 038 042 044 053 055 062 063 077 079 081 082 084 085 100 105 106 111 |
| sonicate/ultrasonic bath | 001 032 077 |
| vortex mix | 006 008 011 013 014 016 020 046 047 049 054 057 064 076 077 078 083 089 090 092 093 094 095 099 102 103 |

| Extraction Solvent Components | laboratory number |
|---|---|
| acetate buffer | 030 |
| acetone | 071 |
| acetonitrile | 002 009 012 015 016 017 036 039 041 042 043 044 049 055 057 063 065 077 079 082 085 087 105 107 109 |
| ammonium hydroxide | 018 |
| diethyl ether | 060 |
| ethyl acetate | 001 003 006 008 009 010 011 013 014 018 020 023 024 025 029 032 035 036 037 038 046 047 053 054 061 062 064 066 069 073 074 075 076 077 078 079 080 081 083 084 089 090 092 093 094 095 099 100 102 111 114 |
| formic acid | 044 |
| hexane | 009 020 036 053 075 076 077 080 111 |
| McIlvane Buffer | 103 |
| metaphosphoric acid | 007 052 056 088 |
| methanol | 002 007 052 056 075 076 077 080 088 111 |
| NaCl | 077 |
| phosphate buffer | 005 077 106 |
| water | 007 009 041 052 063 075 077 098 111 |

| Extraction Time (mins) | laboratory number |
|---|---|
| ≥0.1 - <1 | 015 036 066 |
| ≥1 - <2 | 040 065 073 094 |
| ≥2 - <5 | 006 007 030 041 049 052 053 054 056 064 081 093 100 109 111 |
| ≥5 - <10 | 011 016 025 039 043 062 063 083 092 095 105 107 |
| ≥10 - <30 | 003 008 010 014 017 020 023 024 029 032 035 038 042 046 057 060 061 069 074 078 080 082 085 087 088 089 090 098 102 103 114 |
| ≥30 - <60 | 002 005 009 012 013 037 047 071 077 079 106 |
| ?60 | 001 018 044 055 075 076 084 099 |

| Enzyme Deconjugation Used | laboratory number |
|---|---|
| yes | 009 020 030 036 046 049 078 |
| no | 002 003 005 006 007 008 010 011 012 013 014 015 016 017 018 023 024 025 026 029 032 035 037 038 039 041 042 043 044 047 052 053 054 055 056 057 060 061 062 063 064 065 066 069 071 074 075 076 077 079 080 081 083 084 085 087 088 089 090 092 093 095 098 099 102 103 105 107 109 114 |

| Enzyme Used | laboratory number |
|---|---|
| beta-glucuronidase | 030 036 049 |
| chloramphenicol enzyme conjugate | 020 |
| enzyme conjugate | 089 |
| no | 095 |
| not specified by extraction kit. | 009 |

| Sample Work Up | laboratory number |
|---|---|
| acid hydrolysis with HCl | 043 |
| back-extraction | 015 |
| centrifuge | 003 012 016 020 023 025 029 030 039 041 042 046 071 074 077 082 085 088 099 103 105 106 111 |
| defatted with hexane | 006 009 013 018 024 037 038 039 049 053 060 066 069 071 075 077 079 083 084 089 114 |

| Sample Work Up (continued) | laboratory number |
|---|---|
| defatted with iso-octane | 092 |
| dilute | 064 |
| dry over Na2SO4 | 002 011 014 054 061 090 |
| evaporate | 001 036 039 055 057 065 077 080 095 099 107 |
| filter | 007 062 |
| shaking | 077 |
| none | 032 099 109 |

| Sample Clean-up Technique | laboratory number |
|---|---|
| centifuge | 035 |
| extraction on shaker | 002 |
| filter | 024 077 080 |
| hexane | 073 |
| liquid extraction | 089 |
| liquid/liquid extraction | 001 011 013 016 020 023 029 036 037 039 046 047 049 053 054 060 066 071 076 077 079 081 084 087 090 092 093 095 099 102 105 106 111 |
| solid phase extraction (SPE) (column/cartridge) | 003 007 010 012 015 018 030 032 038 041 042 043 052 055 056 057 061 062 064 065 074 075 077 088 098 103 106 107 |
| solid phase extraction (SPE) (dispersive) | 025 063 |
| solvent exchange | 009 064 083 092 095 |
| none | 005 085 109 |

| SPE Sorbent Type | laboratory number |
|---|---|
| Bond Elut AccuCAT | 015 |
| C18 | 014 018 041 043 055 062 064 065 077 083 103 106 107 |
| MCX | 074 075 094 |
| Oasis HLB | 007 010 012 032 038 052 056 061 088 |
| silica | 042 057 |
| Chem Elut | 030 |
| MIP | 003 |
| PSA and C18 | 025 063 |
| Strata-X | 098 |

| Calibrations | laboratory number |
|---|---|
| solvent | 007 011 014 018 036 037 042 049 053 057 063 079 082 085 102 106 |
| matrix-matched | 001 002 003 010 012 015 023 025 029 030 035 038 044 052 055 056 074 075 076 077 080 081 083 084 087 092 093 094 095 098 099 103 |
| single-level | 043 111 |
| multi-level | 002 003 009 016 017 018 024 030 032 036 039 041 046 047 049 053 054 060 061 062 063 064 065 066 069 071 076 077 079 087 088 090 092 106 109 114 |
| standard addition | 005 008 013 020 041 073 076 079 089 105 107 |

| Type of Internal Standard Added | laboratory number |
|---|---|
| stable isotope labelled analogue | 001 002 003 010 011 012 014 015 016 017 018 023 025 029 030 035 036 038 039 041 042 043 044 047 049 053 055 057 060 061 062 063 065 066 071 074 075 076 077 079 081 083 084 087 088 090 092 093 095 098 105 106 107 111 |
| structural analogue | 005 032 082 |
| chloramphenicol | 085 |
| none | 007 009 013 020 024 037 052 069 073 080 089 099 102 103 109 |

| Method of Separation | laboratory number |
|---|---|
| ELISA | 005 006 008 009 013 020 026 046 069 073 089 102 114 |
| HPLC | 001 003 007 010 012 014 015 016 017 018 023 024 025 029 030 032 035 037 038 039 041 042 043 044 049 052 053 054 056 057 060 061 062 063 064 065 066 071 074 076 077 079 080 081 082 083 084 085 087 088 092 094 095 100 103 105 106 107 109 111 |
| HPLC-MS/MS | 011 047 090 093 |
| UPLC | 002 098 |
| UPLC-MS/MS | 055 099 |

| HPLC Column Packing | laboratory number |
|---|---|
| C18 | 001 002 007 010 011 014 015 016 018 023 024 029 030 032 035 036 037 038 039 041 042 043 047 052 053 054 055 056 057 060 061 062 063 064 065 066 071 075 076 077 079 080 081 082 083 084 085 087 088 090 092 093 094 095 098 099 100 106 107 109 111 |
| C8 | 103 105 |
| biphenyl | 025 049 |
| F5 | 003 |
| PFP | 074 |
| phenyl | 017 044 |

| Mobile Phase Components | laboratory number |
|---|---|
| ammonium acetate | 012 035 041 043 044 052 060 065 092 107 |
| ammonium formate | 014 015 047 093 109 |
| ethanoic acid (acetic acid) | 002 049 071 |
| acetonitrile | 002 007 010 016 023 024 030 032 035 037 039 042 049 053 056 057 071 077 079 081 082 085 087 088 092 094 107 111 |
| formic acid | 003 007 016 039 044 052 053 056 074 106 |
| ion pair agent | 111 |
| methanol | 002 003 011 017 018 025 029 036 038 041 044 047 052 054 055 061 062 063 064 066 074 075 076 080 082 083 084 087 090 095 098 099 100 105 106 |
| water | 001 002 003 007 011 016 017 018 024 025 029 030 032 035 036 037 038 042 044 047 052 053 061 062 063 064 066 071 074 075 076 077 079 082 083 084 087 090 092 095 098 099 103 105 106 107 111 |

| HPLC Post Column Derivatisation | laboratory number |
|---|---|
| none | 007 010 011 015 017 018 023 024 030 037 038 042 043 049 052 054 055 057 060 061 062 066 071 075 077 080 081 082 083 085 087 088 092 095 099 103 105 107 109 |

| HPLC Detector Type | laboratory number |
|---|---|
| MS-MS | 001 002 007 010 011 014 015 016 017 018 023 024 025 029 030 032 035 037 038 039 041 042 047 049 052 053 054 055 056 057 060 061 062 063 064 066 071 074 075 076 077 079 080 081 082 083 084 085 087 088 090 092 093 094 095 099 100 103 105 106 107 109 111 |
| HR-MS | 043 |

| ELISA Test Kit Name | laboratory number |
|---|---|
| AgraQuant Chloramphenicol, AgraQuant Chloramphenicol Plus | 006 |
| Chloramphenicol EIA | 114 |
| Chloramphenicol ELISA Diagnostic Kit | 013 |
| EuroProxima | 008 |
| Ridascreen | 078 102 |
| RIDASCREEN (R) Chloramphenicol | 020 026 |
| Ridascreen Art. No. R1505 | 009 |
| TABP (Taiwan Advence Bio-Pharm) ELISA | 046 |
| TABP Chloramphenicol | 089 |

| ELISA Kit Manufacturer | laboratory number |
|---|---|
| Neogen | 102 |
| R-Biopharm | 009 020 026 069 073 078 |
| EuroProxima B.V | 008 |
| Romer Labs | 006 |
| Taiwan Advance Bio- Pharmaceutical, Inc. | 013 046 |
| XEMA Ltd. | 114 |

| ELISA Antibody Description | laboratory number |
|---|---|
| monoclonal | 005 |
| polyclonal | 013 089 102 114 |
| no info | 008 009 020 026 069 073 078 095 |

| ELISA Standard Material | laboratory number |
|---|---|
| provided by test manufacturer | 006 008 009 013 020 026 046 069 078 089 102 114 |

| ELISA Number of Standards | laboratory number |
|---|---|
| 5 | 006 046 |
| 6 | 005 008 009 013 020 026 069 073 078 089 102 114 |

| ELISA Time Requirement for Testing (min) [not sample preparation] | laboratory number |
|---|---|
| ≥30 - <60 | 005 006 013 026 102 |
| ≥60 - <90 | 008 009 020 046 069 073 089 114 |
| ≥120 | 078 |

| ELISA Calculation of Results | laboratory number |
|---|---|
| 4 parameter | 069 |
| cubic spline | 026 073 078 |
| logit/log | 006 008 009 013 089 102 114 |
| point to point | 005 |

| ELISA Extraction Solvent | laboratory number |
|---|---|
| 50% methanol | 114 |
| ethyl acetate | 006 009 013 026 |
| extraction buffer provided | 089 |
| none | 005 |

| ELISA Sample Extraction (weight/volume, g/ml) | laboratory number |
|---|---|
| <0.5 | 020 |
| ≥0.5 - <1 | 006 013 089 |
| ≥2 - <5 | 005 008 009 026 069 073 078 102 114 |
| ≥5 - <10 | 046 |

| ELISA Dilution Factor of Sample Preparation | laboratory number |
|---|---|
| <1:1 | 026 069 |
| 1:1 | 009 013 046 073 078 089 114 |
| 1:2 | 006 008 102 |
| 1:5 - 1:9 | 005 |
| 1:26 - 1:50 | 020 |

| Limit of Detection (µg/kg) | laboratory number |
|---|---|
| <0.01 | 010 020 046 077 |
| ≥0.01 - <0.1 | 002 006 012 013 014 016 017 024 026 029 033 044 049 053 063 064 071 076 083 084 089 095 099 102 109 114 |
| ≥0.1 - <1 | 007 009 011 015 018 025 030 032 035 037 038 039 040 041 042 043 047 052 054 055 056 057 060 061 062 065 074 075 079 080 081 082 085 087 088 090 093 100 106 107 111 |

| CC alpha (decision limit) µg/kg | laboratory number |
|---|---|
| <0.01 | 079 |
| ≥0.01 - <0.1 | 002 003 013 014 016 023 033 035 036 037 044 046 047 049 077 083 084 092 095 109 111 |
| ≥0.1 - <1 | 001 007 012 015 018 025 030 043 052 060 065 074 080 082 085 087 088 093 107 |
| ≥1 - <10 | 024 054 |
| ≥100 | 010 |

| CC beta (detection capability) µg/kg | laboratory number |
|---|---|
| <0.01 | 010 |
| ≥0.01 - <0.1 | 002 013 014 016 023 026 033 036 044 046 053 060 076 077 083 089 092 111 |
| ≥0.1 - <1 | 001 007 012 015 018 025 030 035 037 038 039 043 047 049 052 054 065 069 074 079 080 082 084 085 087 088 093 095 102 107 109 |
| ≥1 - <10 | 024 |

| MS-MS Transitions Monitored | laboratory number |
|---|---|
| 256.8>175 | 083 |
| 320.6> 256.9 | 017 |
| 320.7> 256.7, 194.0; 151.8 | 064 |
| 320.7>151.7 | 056 |
| 320.8>256.8 151.8 | 092 |
| 320.827>152.000 | 039 |
| 320.84>151.83 | 082 |
| 320.842>256.928, 151.819 | 023 |
| 320.85>256.9, 193.91, 151.85 | 002 |
| 320.853>257.00, 151.888 | 095 |
| 320.9>151.993 | 018 |
| 320.9>152.0 | 010 |
| 320.9>152.1 | 035 |
| 320.9>194, 176 | 111 |
| 320.9>256.7, 193.9, 151.9;<br>322.9>258.5, 193.5, 151.1 | 107 |
| 320.9>256.8, 151.8 | 060 |
| 320.9>256.9, 151.9 | 036 077 |
| 320.9>257.0, 152.0 | 090 |
| 321>152 | 007 013 015 029 052 062 |
| 321>157, 152 | 038 |
| 321>194, 152 | 100 |
| 321>194, 152, 121 | 025 |
| 321>194, 176, 152 | 033 |
| 321>256, 152 | 080 |
| 321>257, 152 | 001 003 011 012 014 024 037 049 055 057<br>074 076 084 087 109 |
| 321>257, 152.0 | 098 |
| 321>257, 152; 323>152 | 106 |
| 321>257, 194, 152 | 063 |
| 321>257, 194, 152;<br>323>259; 152 | 030 |
| 321>257, 321.0>152 | 047 |
| 321>257.03, 152.04 | 071 |
| 321>257; 194, 152 | 041 |
| 321.0>256.0, 152.0 | 054 |
| 321.0>256.9, 152.1 | 079 |
| 321.0>257.0, 152.0 | 061 |

| MS-MS Transitions Monitored (continued) | laboratory number |
|---|---|
| 321.0>257.0, 194.0, 152.0 | 044 |
| 321.0>257.0, 194.0, 152.0; 323.0>152.0 | 016 |
| 321.05>256.7, 151.7 | 053 |
| 321.1>152.0 | 032 |
| 321.1>256.6 | 085 |
| 321.11>151.85 | 099 |
| 321.155>151.978 | 088 |
| 321.2>257.2, 152.2 | 093 |
| 326>157 | 042 |

| For MS only, Single Ions Monitored, m/z | laboratory number |
|---|---|
| 321.0 | 054 |
| 321.35 | 071 |
| 466 | 041 |

| For HR -MS only, Ions Monitored, m/z | laboratory number |
|---|---|
| 152.0 | 054 |
| 321.00505 | 043 |

| Wavelength (absorbance)(nm) | laboratory number |
|---|---|
| 285 | 024 |
| 450 | 013 020 026 069 078 |

# APPENDIX II: Fapas® SecureWeb, Protocol and Contact Details

## 1. Fapas® SECUREWEB

Access to the secure area of our website is only available to participants in our proficiency tests. Please contact us if you require a UserID and Password. Fapas® SecureWeb allows participants to:

- Obtain their laboratory numbers for the proficiency tests in which they have participated.
- View the results they submitted in past and current proficiency tests.
- Submit their results and methods for current tests.
- Review future tests they have ordered.
- Order proficiency tests, reference materials and quality control materials.
- Freely download copies of reports (PDF file), of proficiency tests in which they have participated.
- View charts of their z-scores obtained in previous Fapas® – Food Chemistry proficiency tests.

## 2. PROTOCOL

The Protocols [3, 4] set out how Fapas® – Food Chemistry is organised. Copies can be downloaded from our website.

## 3. CONTACT DETAILS

This report was prepared and authorised on behalf of Fapas® by Michael Knaggs (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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