Fapas® – Food Chemistry Proficiency Test Report 02357

Phenicols in Bovine Milk

September-October 2018

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.

REPORT INTEGRITY

Fapas® reports are distributed as digitally signed Adobe® PDF files. When these files are opened with Adobe® Reader v9 or later, a blue ribbon and information bar indicates that the certificate has been validated. This confirms that the author of the report is Fapas® and that the document has not been altered since it was signed [1, 2].

The integrity of hard copies of Fapas® reports cannot be assured in this way, i.e. printed copies are not controlled. A watermark stating this appears on every page when a Fapas® report is printed.

End users of Fapas® reports should ensure that either the PDF file they are viewing displays a valid Fapas® digital signature or that the content of their hard copy exactly matches the content of a PDF file that displays a valid Fapas® digital signature.

QUALITY SYSTEMS

Fapas® – Food Chemistry is accredited by UKAS as complying with the requirements of ISO/IEC 17043:2010 [3].

Fera is an ISO 9001 certified organisation.

UKAS PROFICIENCY TESTING
0009

Fera hereby excludes all liability for any claim, loss, demands or damages of any kind whatsoever (whether such claims, loss, demands or damages were foreseeable, known or otherwise) arising out of or in connection with the preparation of any technical or scientific report, including without limitation, indirect or consequential loss or damage; loss of actual or anticipated profits (including loss of profits on contracts); loss of revenue; loss of business; loss of opportunity; loss of anticipated savings; loss of goodwill; loss of reputation; loss or damage to or corruption of data; loss of use of money or otherwise, and whether or not advised of the possibility of such claim, loss demand or damages and whether arising in tort (including negligence), contract or otherwise. This statement does not affect your statutory rights.

Nothing in this disclaimer excludes or limits Fera liability for: (a) death or personal injury caused by Fera negligence (or that of its employees, agents or directors); or (b) the tort of deceit; [or (c) any breach of the obligations implied by Sale of Goods Act 1979 or Supply of Goods and Services Act 1982 (including those relating to the title, fitness for purpose and satisfactory quality of goods);] or (d) any liability which may not be limited or excluded by law (e) fraud or fraudulent misrepresentation.

The parties agree that any matters are governed by English law and irrevocably submit to the non-exclusive jurisdiction of the English courts.

SUMMARY

1. The test materials for Fapas® – Food Chemistry proficiency test 02357 were dispatched in September 2018. Each participant received a bovine milk test material to be analysed for a selection of phenicols.

2. An assigned value (x_a) was determined for each analyte and in conjunction with the standard deviation for proficiency (σ_p) was used to calculate a z-score for each result. However, for Chloramphenicol (using enzyme deconjugation) and Florfenicol-amine we were unable to set an assigned value. For Total Phenicols, the assigned value and z-scores are given *for information only.*

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 (NOT using enzyme deconjugation)	0.800	31	45	69
Florfenicol	5.32	20	26	77
Thiamphenicol	71.7	29	33	88
Total Phenicols	*79.2*	*4*	7	*57*

italics indicate for information only

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 [4, 5].

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 [3].

The test material was prepared from the milk of cows that had been treated with Chloramphenicol. This was blended with drug free bovine milk. Thiamphenicol and Florfenicol were spiked into the test material.

Samples were stored at -20°C until dispatch.

2.2. Homogeneity and Screening

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 [3].

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

Because all possible residues were present, no screening was necessary.

2.3. Dispatch

The start date was 24 September 2018. Test materials were sent to 58 participants.

3. RESULTS

The instructions for reporting results were as follows:

1) Determine the level of residues present in the test material as follows:

analyte	units	comment
Chloramphenicol (using enzyme deconjugation)	µg/kg	corrected for recovery
Chloramphenicol (NOT using enzyme deconjugation)	µg/kg	corrected for recovery
Florfenicol	µg/kg	corrected for recovery
Florfenicol-amine	µg/kg	corrected for recovery
Thiamphenicol	µg/kg	corrected for recovery
Total Phenicols	µg/kg	corrected for recovery

2) Total phenicols is included for the benefit of participants using ELISA test kits or other methods that may not be able to distinguish individual phenicol residues. However, participants using HPLC are welcome to submit results for this analyte as well.

   For total phenicols by HPLC, report results as the sum of chloramphenicol (whether using enzyme deconjugation or not), florfenicol/florfenicol amine and thiamphenicol.

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.

4) 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 [6, 7]. If you do not know the CCβ, you can report your limit of quantification, but please mention this in the comments box.

   Please note: 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.

Results were submitted by 51 participants (88%) before the closing date for this test, 25 October 2018.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1 and Table 2.

If a participant analysed for a residue that was in the test material, but did not identify it, and their limit of quantification was below the level needed for a z-score of -3.0, they were assessed as if their result was zero.

If a participant analysed for a residue that was in the test material, but did not identify it, and their limit of quantification was above the level needed for a z-score of -3.0, the result was recorded as <LoQ.

Participants' comments are given in Table 3.

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 each analyte. The assigned values were then used in combination with the standard deviation for proficiency, σ_p, to calculate a z-score [8] for each result. The procedure is detailed in the relevant protocols [4, 5].

Further background on the procedure followed can be found in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [9].

4.1. Calculation of the Assigned Value, x_a

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

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

i) non-numerical results i.e. qualitative or semi-quantitative results,

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 Total Phenicols, results that were the sum of three recovery-corrected results were deemed to be corrected for recovery.

For Chloramphenicol (using enzyme deconjugation) and Florfenicol-amine there were too few results to set an assigned value. Participants' results are shown in Table 1 and Table 2 respectively.

For Chloramphenicol (NOT using enzyme deconjugation), the major mode was chosen as the assigned value because the distribution of results was skewed. A plot of distribution of results is shown in Figure 1.

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

For Total Phenicols, the median was chosen as the assigned value because of the low number of valid results. The assigned value was checked against the sum of the assigned values of the individual residues. Due to the low number of valid results, the assigned value and z-scores are given *for information only*.

The assigned values for all analytes, except for Chloramphenicol (using enzyme deconjugation) and Florfenicol-amine are shown in Table 4.

4.2. Standard Deviation for Proficiency, σ_p

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

For all analytes, σ_p was derived from the appropriate form of the Horwitz equation [10].

The values for σ_p used to calculate z-scores from the reported results of this test are given in Table 4.

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 σ_p = the standard deviation for proficiency.

Participants' z-scores for all analytes, except for Chloramphenicol (using enzyme deconjugation) and Florfenicol-amine are given in Table 1 and Table 2 and shown as histograms in Figures 1–4. Those for Total Phenicols are given *for information only*. 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 4.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for all analytes, except for Chloramphenicol (using enzyme deconjugation) and Florfenicol-amine, are given in Table 5.

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 [9].

6. REFERENCES

1 Adobe Approved Trust List, https://helpx.adobe.com/acrobat/kb/approved-trust-list2.html#Whatisit accessed 13/11/2018.

2 GlobalSign PDF Signing Tool, https://www.globalsign.com/en/digital-signatures/ accessed 13/11/2018.

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

4 Fapas®, 2017, Protocol for Proficiency Testing Schemes, Version 6, April 2017, Part 1 – Common Principles.

5 Fapas®, 2017, Protocol for Proficiency Testing Schemes, Version 5, April 2017, Part 2 – Fapas® Food Chemistry scheme (FAPAS).

6 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.

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

8 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.

9 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.

10 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 for Chloramphenicol (using enzyme deconjugation), Chloramphenicol (NOT using enzyme deconjugation), Florfenicol and Florfenicol-amine

laboratory number	analyte													
	Chloramphenicol (using enzyme deconjugation) assigned value: not issued			Chloramphenicol (NOT using enzyme deconjugation) assigned value: 0.800 µg/kg				Florfenicol assigned value: 5.32 µg/kg				Florfenicol-amine assigned value: not issued		
	result	IS/% rec	CCβ	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ
001	#			0.941			0.8	5.811			0.4	#		
002	#			0.78	M	0.2	-0.1	#				#		
003	#			0.76	M		-0.2	6.18	M		0.7	#		
004	0			19	100	0	**103.3**	0			**-4.5**	0		
005	#			#				#				#		
006	#			0.6475	Y	0.012	-0.9	#				#		
007	#			3.9	Yes		**17.6**	6.5	Yes		1.0	#		
008	#			0.992	M	0.053	1.1	#				#		
009	0.81	Y, 98	0.3	#				5.7	Y, 82	1.0	0.3	#		
010	#			0.66	Y/ 98	0.05	-0.8	#				#		
011	#			0.90	82%	0.1	0.6	#				5.17	71%	10
012	#			0.66	Y,M,S	0.1	-0.8	6.77	Y,M,S	0.2	1.2	0		
013	#			0.98	Y	0.12	1.0	#				#		
014	#			0.685		0.1	-0.7	4.485		0.3	-0.7	#		
015	#			0.75	Y/106.33/M	0.2	-0.3	2.92	Y/97.89/M	0.5	**-2.1**	#		

= not analysed 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 for Chloramphenicol (using enzyme deconjugation), Chloramphenicol (NOT using enzyme deconjugation), Florfenicol and Florfenicol-amine

laboratory number	analyte													
	Chloramphenicol (using enzyme deconjugation) assigned value: not issued			Chloramphenicol (NOT using enzyme deconjugation) assigned value: 0.800 µg/kg				Florfenicol assigned value: 5.32 µg/kg				Florfenicol-amine assigned value: not issued		
	result	IS/% rec	CCβ	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ
016	#			0.782	Y	0.3	-0.1	#				#		
017	#			0.916	M; Y	0.09	0.7	#				#		
018	#			0.903	Y, M	0.09	0.6	#				#		
019	0			#				#				#		
020	#			1.71	M	0.3	**5.2**	#				#		
021	3.90	S	0.15	#				3.80	S	355	-1.3	0		
022	#			0.75	Y	0.25	-0.3	4.40	Y	2.00	-0.8	#		
023	#			0.349	Y	0.1	**-2.6**	4.68	95.6%	0.1	-0.6	#		
024	#			0.77	80%	0.3	-0.2	#				#		
025	#			0.94	Y	0.1	0.8	5.32	Y	2	0.0	<10	Y	10
026	#			0			**-4.5**	5.1			-0.2	#		
027	#			0.691	Y, 100%		-0.6	#				#		
028	#			0.32	Yes	0.015	**-2.7**	#				#		
029	#			1.04	Y M	0.25	1.4	#				#		
030	#			1.07	Y/M		1.5	5.22	Y/M		-0.1	0		

= not analysed 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 for Chloramphenicol (using enzyme deconjugation), Chloramphenicol (NOT using enzyme deconjugation), Florfenicol and Florfenicol-amine

laboratory number	analyte														
	Chloramphenicol (using enzyme deconjugation) assigned value: not issued			Chloramphenicol (NOT using enzyme deconjugation) assigned value: 0.800 µg/kg				Florfenicol assigned value: 5.32 µg/kg				Florfenicol-amine assigned value: not issued		
	result	IS/% rec	CCβ	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ
031	#			1.14	Y	0.0560	1.9	2.12	Y	0.0114	**-2.7**	#		
032	#			0.83	S	0.3	0.2	6.55	S	0.3	1.0	#		
033	#			0.68		0.15	-0.7	#				#		
034	#			1.2			**2.3**	#				#		
035	#			0.61	Y/95%	0.1	-1.1	5.15	Y/104%	0.1	-0.1	#		
036	1.67	Y	0.10	#				0			**-4.5**	3.8	M	0.57
037	#			0.385	Y	0.02	**-2.4**	#				#		
038	#			1.18			**2.2**	#				#		
039	#			1.38	Y M 100.48%	0.30	**3.3**	5.55	Y,M 103.70%	5.00	0.2	0		
040	#			0.79	Y, M	0.08	-0.1	#				#		
041	#			1.1	Y		1.7	5.7	Y		0.3	0		
042	#			0.78	Yes	0.15	-0.1	6.2	Yes	1	0.7	6.2	Yes	1
043	0			0.280	Y	0.01	**-3.0**	0			**-4.5**	0		
044	0			0.778	101%		-0.1	0			**-4.5**	0		
045	#			1.50	Y % M S	0.05	**4.0**	5.19	Y % M S	0.05	-0.1	#		

= not analysed 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 for Chloramphenicol (using enzyme deconjugation), Chloramphenicol (NOT using enzyme deconjugation), Florfenicol and Florfenicol-amine

laboratory number	analyte													
	Chloramphenicol (using enzyme deconjugation) assigned value: not issued			Chloramphenicol (NOT using enzyme deconjugation) assigned value: 0.800 µg/kg				Florfenicol assigned value: 5.32 µg/kg				Florfenicol-amine assigned value: not issued		
	result	IS/% rec	CCβ	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ
046	#			0.47		0.17	-1.9	#				#		
047	#			0.741		0.1	-0.3	#				#		
048	#			0.79	Y		-0.1	7.30			1.7	#		
049	#			<1				#				#		
050	#			1.48	R	0.56	**3.9**	#				#		
051	#			1.25	97%		**2.6**	5.58	98%		0.2	#		

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

Table 2: Results and z-Scores for Thiamphenicol and Total Phenicols

laboratory number	analyte							
	Thiamphenicol assigned value: 71.7 µg/kg				Total Phenicols assigned value: *79.2* µg/kg			
	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ	z-score
001	73.398			0.1	#			
002	#				#			
003	75.52	M		0.2	#			
004	0			**-4.5**	0			*-4.5*
005	75.13			0.2	#			
006	#				#			
007	74.5	Yes		0.2	#			
008	#				#			
009	83	Y, 117	1.0	0.7	#			
010	65.1	Y/ 95	0.05	-0.4	#			
011	79.37	90%	0.4	0.5	#			
012	64.1	Y,M,S	0.4	-0.5	#			
013	#				#			
014	75.1		0.3	0.2	80.27		0.3	*0.1*
015	37.09	Y/98.86/M	0.5	**-2.2**	#			

= not analysed Y = internal standard used M = matrix-extracted calibration S = standard addition
z-scores outside |z| >2 are shown in **bold,** see Section 5 *italics indicate for information only*

Table 2 (continued): Results and z-Scores for Thiamphenicol and Total Phenicols

laboratory number	analyte							
	Thiamphenicol assigned value: 71.7 µg/kg				Total Phenicols assigned value: *79.2* µg/kg			
	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ	z-score
016	#				#			
017	80.1	M;Y	0.25	0.5	#			
018	#				#			
019	75	Y	1	0.2	#			
020	99.37	M	50	1.8	#			
021	69.1	S	75.0	-0.2	76.8			*-0.1*
022	71.50	Y	64.00	0.0	#			
023	68.4	77.4%	0.1	-0.2	#			
024	#				#			
025	72.93	Y	2	0.1	79.19	Y		*0.0*
026	69			-0.2	#			
027	70.3	Y, 100%		-0.1	#			
028	#				#			
029	#				#			
030	54.26	Y/M		-1.1	#			

= not analysed Y = internal standard used M = matrix-extracted calibration S = standard addition
z-scores outside |z| >2 are shown in **bold,** see Section 5 *italics indicate for information only*

Table 2 (continued): Results and z-Scores for Thiamphenicol and Total Phenicols

laboratory number	analyte							
	Thiamphenicol assigned value: 71.7 µg/kg				Total Phenicols assigned value: *79.2* µg/kg			
	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ	z-score
031	42.2	Y	0.0115	-1.9	#			
032	47.69	S	0.3	-1.5	#			
033	85.00		63	0.8	#			
034	#				#			
035	#				#			
036	100	Y	2.5	1.8	#			
037	#				#			
038	#				#			
039	72.43	Y,M 95.06%	5.00	0.0	#			
040	#				#			
041	69.6	Y		-0.1	#			
042	72.3	Yes	25	0.0	#			
043	0			**-4.5**	0			*-4.5*
044	0			**-4.5**	0			*-4.5*
045	82.34	Y % M S	0.05	0.7	89.03	Y % M S	0.15	*0.6*

= not analysed Y = internal standard used M = matrix-extracted calibration S = standard addition
z-scores outside |z| >2 are shown in **bold,** see Section 5 *italics indicate for information only*

Table 2 (continued): Results and z-Scores for Thiamphenicol and Total Phenicols

laboratory number	analyte							
	Thiamphenicol assigned value: 71.7 µg/kg				Total Phenicols assigned value: *79.2* µg/kg			
	result	IS/% rec	CCβ	z-score	result	IS/% rec	CCβ	z-score
046	#				#			
047	#				#			
048	80.0			0.5	#			
049	#				#			
050	#				#			
051	79.39	101%		0.5	#			

= not analysed *italics indicate for information only*

Table 3: Participants' Comments

laboratory number	comments
002	0.2 limit of quantification
009	CCbeta is our LoQ. Sample was held in customs for extended period of time, and slight curderling was noticed at the top of the sample.
011	LOQ instead of CCbeta
014	Total Phenicols = sum of chloramphenicol + florfenicol + thiamphenicol
015	Values added in CCbeta column are LoQ
016	Reporting limit: 0.3 µg/kg
017	CCÃ? = LOQ
020	LOQ values are given as CCbeta
023	Not counting the total results
025	We use LOQ as ccbeta.
029	0.25 ug/kg is CCbeta, CC alpha submitted in results form
033	Analytical method used was Quenchers, for Chloramphenicol Internal standard was used,
036	The analysis involved two independent methods for CAP/THIA and FFA.
045	CCbeta is our LoQ.
047	0.741 µg/kg Ã?Â±0.053 µg/kg. The FAPAS TEST 02357 was also analyzed by ELISA Ridascreen with a result greater than 9.7 ug / kg of Chloramphenicol (Kit specificity: 100% Chloramphenicol and approx. 51-68% Chloramphenicol Glucuronide).

comments are as submitted by participants; but some may have been edited to maintain participant anonymity

Table 4: Assigned Values and Standard Deviations for Proficiency

analyte	data points, *n*	assigned value, x_a µg/kg	uncertainty, *u*	standard deviation for proficiency, σ_p	
Chloramphenicol (NOT using enzyme deconjugation)	36	0.800	0.030	Horwitz [10]	0.176
Florfenicol	18	5.32	0.26	Horwitz [10]	1.17
Thiamphenicol	24	71.7	2.4	Horwitz [10]	15.8
Total Phenicols	3	*79.2*	*2.0*	Horwitz [10]	*17.4*

italics indicate for information only

Table 5: Number and Percentage of z-Scores where |z| ≤2

analyte	number of scores where |z| ≤2	total number of scores	% |z|≤2
Chloramphenicol (NOT using enzyme deconjugation)	31	45	69
Florfenicol	20	26	77
Thiamphenicol	29	33	88
Total Phenicols	*4*	7	*57*

italics indicate for information only

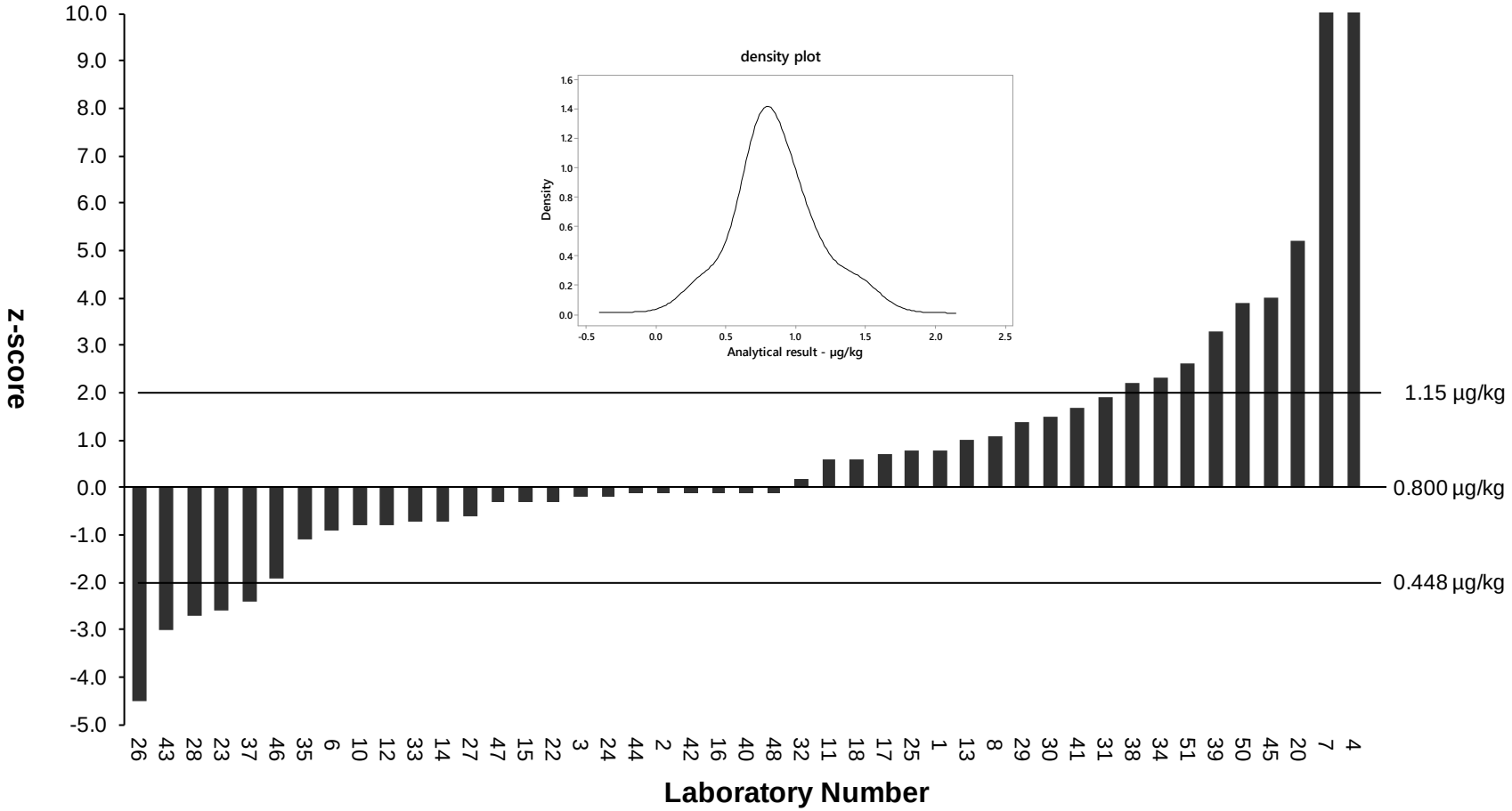

Figure 1: z-Scores for Chloramphenicol (NOT using enzyme deconjugation)

insert shows a plot of the distribution of results

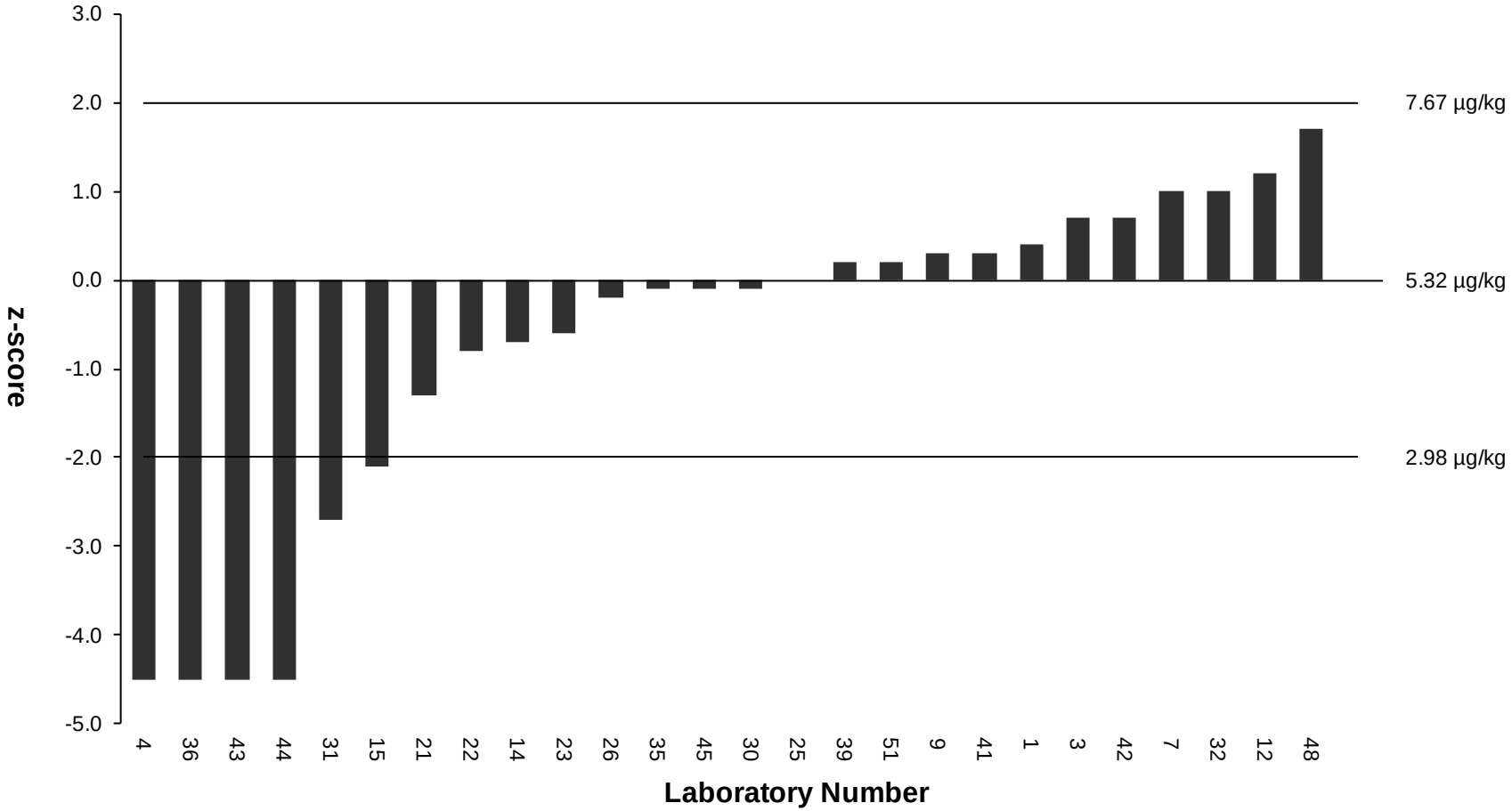

Figure 2: z-Scores for Florfenicol

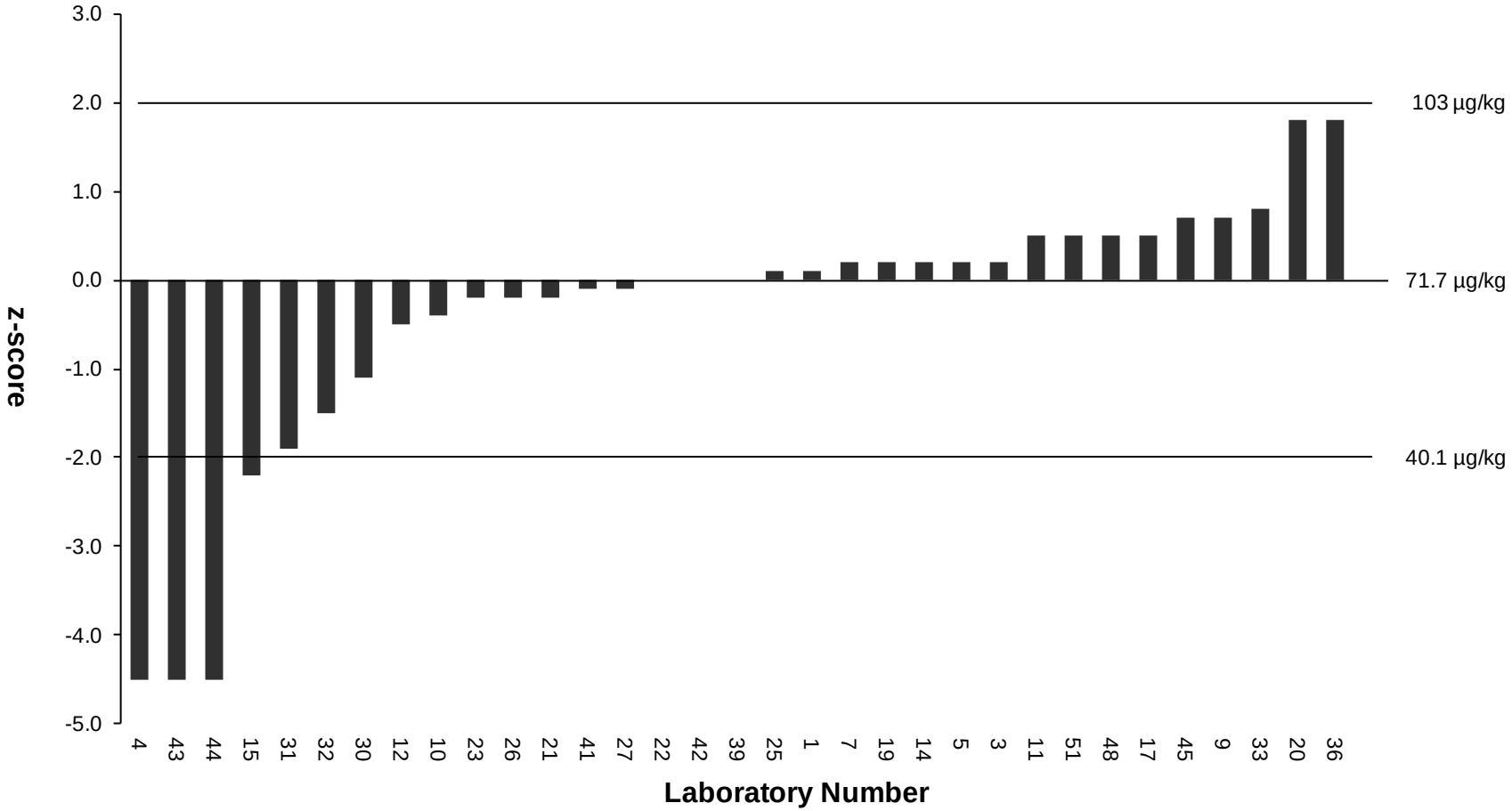

Figure 3: z-Scores for Thiamphenicol

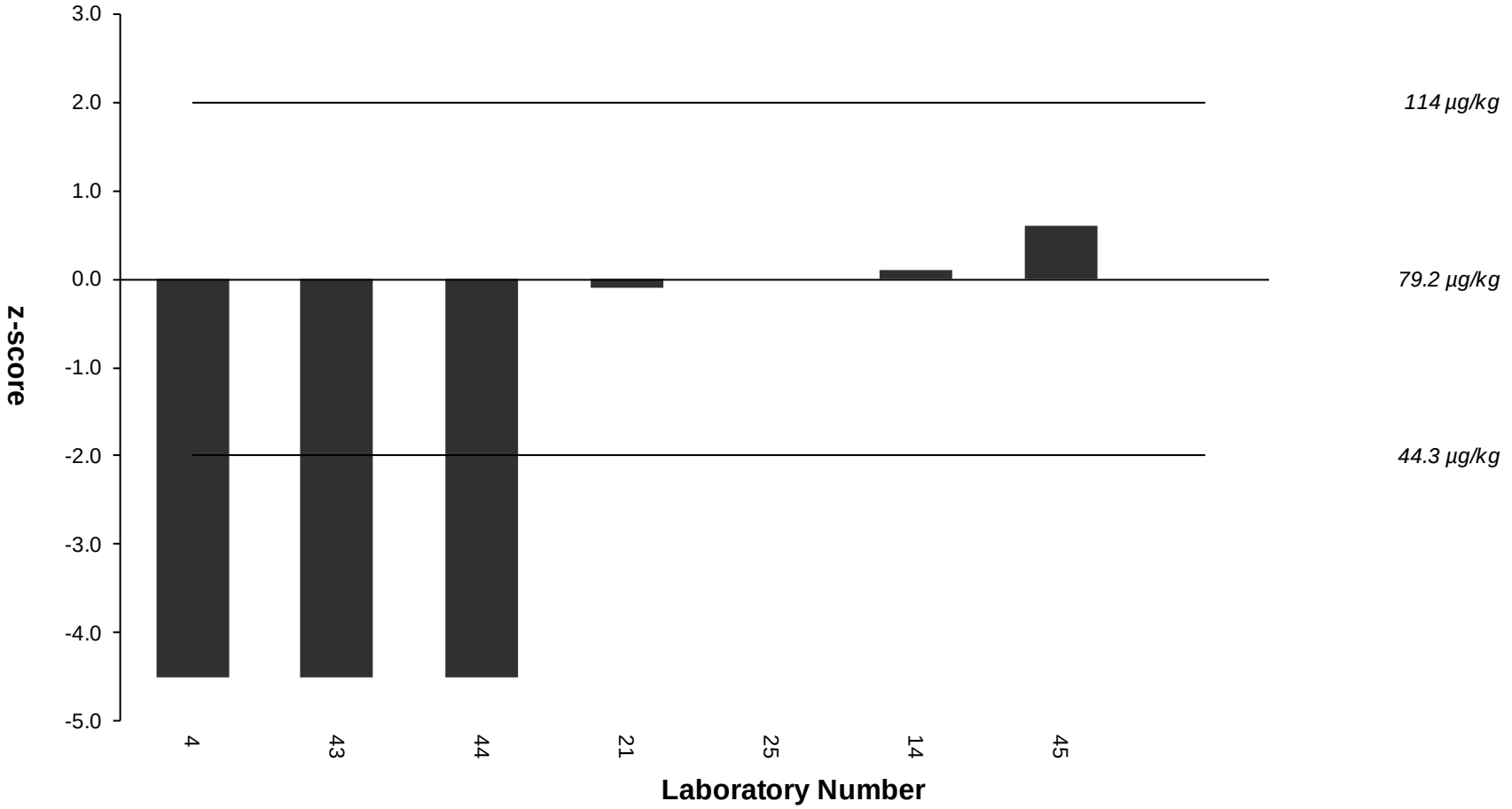

Figure 4: z-Scores for Total Phenicols

this histogram is given for information only

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.

Is the Method Used Accredited?	laboratory number
no	001 010 015 019 034 040 044 047
yes	002 007 008 011 012 013 016 017 018 020 022 023 025 026 029 030 031 032 033 035 036 041 043 048 051

What is Your Method Based On?	laboratory number
International Standard	051
National Standard	008 023 030 035 041
Manufacturer/Kit Instructions/Technical Note	034 044
In house method	001 002 007 010 011 012 013 015 016 017 018 019 020 022 025 026 029 031 032 033 036 040 047 048

Sample Weight (g)	laboratory number
<1	012 044 048
≥1 - <2	010 011 023 025 029 031 033 034 035 051
≥2 - <5	001 002 007 008 013 016 018 019 020 022 032 036 040 047
≥5 - <10	015 017 030 041
≥10 - <25	043

Extraction Procedure	laboratory number
cold solvent extraction at atmospheric pressure	007 012 020 030 033 036 040 051
maceration/homogenisation	022 032
shaking	008 015 016 023 031 035 047 048
sonicate/ultrasonic bath	002 013
vortex mix	001 010 019 025 029 034 041 044
1st step) SPE with Water and Acetonitrile. 2nd step) dryness with N2	018
SPE	017

Extraction Solvent Components	laboratory number
acetonitrile	001 007 008 010 015 016 018 019 022 023 025 029 032 033 035 048 051
ethyl acetate	012 013 017 018 020 031 036 047
hexane	031
methanol	031
petroleum ether/spirit	012
phosphate buffer	002 013 040
trichloroacetic acid	030
water	018 030 034

Extraction Time (mins)	laboratory number
≥0.1 - <1	016 034
≥1 - <2	010 012 033 036 048
≥2 - <5	022 031
≥5 - <10	001 002 007 017 025 044 051
≥10 - <30	008 013 018 019 023 029 030 032 035 040
≥30 - <60	020 047
≥60	015

Enzyme Deconjugation Used?	laboratory number
no	001 002 007 008 010 011 012 013 015 016 017 018 019 020 022 023 029 030 031 032 033 034 035 040 043 044 047 048 051
yes	036

Enzyme Used	laboratory number
beta-glucuronidase	036
Detection without enzymatic solution	023
no	010

Sample Work Up	laboratory number
Acid Hydrolysis with HCl	008 017
Carrez I & II	020
centrifuge	001 012 016 023 025 029 030 031 032 033 035 051
defatted with hexane	047
dilute	019 034
evaporate	013
pH adjustment	040
centrifuge after precipitating with ACN	010
FFA Acid Hydrolysis followed by Ethyl Acetate extraction. CAP/THIA defatted using Hexane.	036
none	044

Sample Clean-up Technique	laboratory number
filter	010 016
liquid/liquid extraction	001 007 012 016 020 022 025 031 033 035 041 047
solid phase extraction (SPE) (column/cartridge)	002 013 017 018 022 023 030 032 035 036 040
solid phase extraction (SPE) (dispersive)	015 019 051
solvent exchange	036
Enfriar y centrifugar	048
none	029 034 044

SPE Sorbent Type	laboratory number
C18	015 036 043 051
MCX	030 041
NH2	042
Oasis HLB	002 013 018 032 040
silica	023 035
Chem Elut	017
EMR-L	019

Calibrations	laboratory number
solvent	016 023 048 051
matrix-matched	001 002 012 015 017 019 020 022 029 030 036 040 041 042 047
single-level	010 011
multi-level	007 012 017 020 025 031 033 036 044 048
standard addition	012 013 032 034 035

Type of Internal Standard Added	laboratory number
none	001 011 034
stable isotope labelled analogue	002 007 010 012 013 016 017 018 022 025 029 030 031 032 033 035 036 040 042 048 051
structural analogue	015 019 023

Method of Separation	laboratory number
ELISA	034 044
HPLC	002 007 010 011 012 013 015 016 017 018 019 020 022 023 025 029 030 031 032 035 036 040 041 042 043 047 048 051

HPLC Column Packing	laboratory number
C18	001 002 007 010 013 015 016 017 018 019 020 023 029 030 031 033 035 036 040 041 042 043 047 048 051
BEH Phenyl	012
phenyl	032

Mobile Phase Components	laboratory number
ammonium acetate	010 016 017 032 047
ammonium formate	020 042 051
acetonitrile	001 007 013 017 018 022 023 029 031 033 035 040
formic acid (methanoic acid)	015 020 022 030 031 032
methanol	010 012 016 020 030 033 047 048
water	012 013 016 017 018 020 022 025 030 031 033 035 047 048
0.1 % formic acid in water and acetonitrile	019
ACN/water	002
CAP/THIA -Methanol & Water. FFA - Acetonitrile & Ammonia/Water	036

HPLC Post Column Derivatisation	laboratory number
none	001 007 010 012 013 015 016 017 018 019 023 030 031 032 033 040 044 048 051

HPLC Detector Type	laboratory number
Diode Array Detector	043
MS-MS	001 002 007 010 011 012 013 015 016 017 018 019 020 022 023 025 029 030 031 032 033 035 036 040 041 042 047 048 051

ELISA Test Kit Name	laboratory number
RIDASCREEN Chloramphenicol Art.No.R1511	044
TABP Chloramphenicol ELISA Diagnostic Kit	034

ELISA Kit Manufacturer	laboratory number
R-Biopharm	044
Taiwan Advance Bio- Pharmaceutical, Inc.	034

ELISA Antibody Description	laboratory number
monoclonal	044
polyclonal	034
no info	001 033

ELISA Standard Material	laboratory number
other (please specify)	001 033
provided by test manufacturer	034 044

ELISA Number of Standards	laboratory number
6	034 044

ELISA Time Requirement for Testing (min) [not sample preparation]	laboratory number
≥30 - <60	034 044

ELISA Calculation of Results	laboratory number
logit / log	044
semi-logarithmic system	034

ELISA Sample Extraction (weight/volume, g/ml)	laboratory number
<0.5	034
≥1 - <2	044

ELISA Dilution Factor of Sample Preparation	laboratory number
undiluted	044
1:3	034

Chloramphenicol (using enzyme deconjugation)

CC alpha (decision limit) (µg/kg)	laboratory number
≥0.01 - <0.1	036

CC beta (detection capability) (µg/kg)	laboratory number
≥0.1 - <1	036

MS-MS Transitions Monitored	laboratory number
320.8 > 151.9; 320.8 > 121.1	036

Chloramphenicol (NOT using enzyme deconjugation)

Limit of Detection (µg/kg)	laboratory number
<0.01	007
≥0.01 - <0.1	010 011 017 023 031 033 037 042 044 047 051
≥0.1 - <1	002 012 016 018 020 025 029 030 032 034 035 048

CC alpha (decision limit) (µg/kg)	laboratory number
<0.01	007
≥0.01 - <0.1	008 012 017 018 031 033 037 040 042
≥0.1 - <1	002 013 025 029 032 035 051

CC beta (detection capability) (µg/kg)	laboratory number
<0.01	007
≥0.01 - <0.1	008 018 031 037 040 042 051
≥0.1 - <1	002 012 013 017 025 032 033 035

MS-MS Transitions Monitored	laboratory number
320.9>151.9	023
320.9>152	042
320.9>152; 320.9>256.9	031
320.9>257, 320.9>176, 320.9>152	048
320.9-151.7	051
321 > 152	010
321 > 152 321 > 257	030
321,0 > 152,1	011
321.0>152	032
321.0>152.0 321.0>194.0 321.0>257.0 323.0>152.0	025
321.2>152 , 321.2>257	033
321.73>151.83 ; 321.73>256.93	018
321>152	002
321>152*, 321>257	016
321>152, 257	029
321>152, 321>257	013 035 040
321>257.1, 152	047
321>257; 321>194; 321>152; 323>259; 323>152	017
321>257>152, 354>290>240>227, 356>336>185	015
321-152, 321-257, 326-157	037

For MS only, Single Ions Monitored, m/z	laboratory number
152, 257, 157	037

For HR -MS only, Ions Monitored, m/z	laboratory number
321,321, 326	037

Wavelength (absorbance)(nm)	laboratory number
450	034 044

Florfenicol

Limit of Detection (µg/kg)	laboratory number
≥0.01 - <0.1	023 031 051
≥0.1 - <1	012 032 035
≥1 - <10	007 025 030 042 048

CC alpha (decision limit) (µg/kg)	laboratory number
≥0.01 - <0.1	007 012 031
≥0.1 - <1	032 035 051
≥1 - <10	042

CC beta (detection capability) (µg/kg)	laboratory number
≥0.01 - <0.1	012 051
≥0.1 - <1	007 035
≥1 - <10	032 042

MS-MS Transitions Monitored	laboratory number
355.8>185; 355.8>336	031
355.8>336, 355.8>185	048
355.9-335.8	051
356 > 336 356 > 185	015 030 035
356>185	023 032 042

Florfenicol-amine

Limit of Detection (µg/kg)	laboratory number
≥0.1 - <1	042
≥1 - <10	011 025

CC alpha (decision limit) (µg/kg)	laboratory number
≥0.1 - <1	036 042

CC beta (detection capability) (µg/kg)	laboratory number
≥0.1 - <1	036 042

MS-MS Transitions Monitored	laboratory number
248 > 230.1 > 130.1; 248 > 230.1 > 131.1	036
248,10 > 130,0	011
248.1>130.1	042

Thiamphenicol

Limit of Detection (µg/kg)	laboratory number
≥0.01 - <0.1	010 012 017 019 023 031 051
≥0.1 - <1	011 032
≥1 - <10	007 025 030 033 036 048
≥10 - <100	020 042

CC alpha (decision limit) (µg/kg)	laboratory number
≥0.01 - <0.1	012 017 031
≥0.1 - <1	019 032 051
≥10 - <100	007 033 042

CC beta (detection capability) (µg/kg)	laboratory number
≥0.01 - <0.1	012 031 051
≥0.1 - <1	017 032
≥10 - <100	007 033 042

MS-MS Transitions Monitored	laboratory number
326 > 157	010
354>290	023
353.8>185; 353.8>240	031
353.9 > 289.9; 353.9 > 184.9	036
353.9>185	042
353.9>289.9, 353.9>239.9	033
354 > 185 354 > 290	030 048
354,0 > 290	011
354.0-184.9	051
354>185	032
354>290 354>227	015 019
354>290; 354>185; 356>185	017

Total Phenicols

Limit of Detection (µg/kg)	laboratory number
≥1 - <10	025

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 [4, 5] 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:

Fapas®

Fera Science Ltd (Fera)
National Agri-Food Innovation Campus
Sand Hutton
York
YO41 1LZ
UK

Tel: +44 (0)1904 462100
Fax: +44 (0)1904 500440

info@fapas.com

www.fapas.com