CERTIFICATE OF PARTICIPATION

This certificate confirms that:

Public Health Bureau,Yunlin County

took part in:
Food Chemistry Proficiency Test **20185**
Start Date: 15/07/2021
Artificial Colours in Sugar Confectionery (Boiled Sweets)

and were allocated laboratory number **19**.

The performance of the laboratory is shown in the relevant report, which is available from the secure pages at fapas.com

Fapas® – Food Chemistry Proficiency Test Report 20185

Artificial Colours in Sugar Confectionery (Boiled Sweets)

July-September 2021

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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This report is the output of a Management System that is certified by LRQA to ISO 9001:2015 [5, 6].

SUMMARY

1. The test materials for Fapas® – Food Chemistry proficiency test 20185 were dispatched in July 2021. Each participant received a sugar confectionery (boiled sweets) test material to be analysed for a selection of artificial colours.

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.

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

analyte	assigned value, x_a mg/kg	number of scores, $\lvert z \rvert \leq 2$	total number of scores	% $\lvert z \rvert \leq 2$
Allura Red (E129)	21.4	26	36	72
Carmoisine (E122)	11.9	26	34	76
Ponceau 4R (E124)	4.02	27	34	79
Sunset Yellow FCF (E110)	8.85	27	35	77

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 [7, 8].

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 materials were prepared from sugar and water.

Allura Red (E129), Carmoisine (E122), Ponceau 4R (E124) and Sunset Yellow FCF (E110) were spiked into the test material.

Samples were stored frozen 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 [3].

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

2.3. Dispatch

The start date was 15 July 2021. Test materials were sent to 46 participants.

3. RESULTS

The instructions for reporting results were as follows:

Identify and determine the levels of the colours present in mg/kg (corrected for purity of standard). PLEASE NOTE:

- For each colour, select either "Not Detected", "Not Tested" or "Provide Result".
- Enter a default value for "Limit of quantification (LOQ) mg/kg" and "Purity of colour standard (%), & corrected (y/n)?".
- AFTER you have entered your results for each colour you MUST review and if necessary, edit the values for "Limit of quantification (LOQ) mg/kg" and "Purity of colour standard (%), & corrected (y/n)?" that differ from the default value you gave.

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.

This is an identification and quantification proficiency test. Therefore, if you analyse for a residue that is in the test material, and do not identify it, and your limit of quantification is below the level needed for a z-score of -3.0, you will be assessed as if your result was zero.

This Sugar Confectionary (Boiled Sweets) test material may contain any number of the following analytes:

Allura Red (E129)	Brilliant Blue FCF (E133)	Carmoisine (E122)
Erythrosine (E127)	Green S (E142)	Indigo Carmine (E132)
Patent Blue V (E131)	Ponceau 4R (E124)	Quinoline Yellow (E104)
Sunset Yellow FCF (E110)	Tartrazine (E102)	

Results were submitted by 41 participants (89%) before the closing date for this test, 02 September 2021.

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.

There were no results reported for any artificial colours other than Allura Red (E129), Carmoisine (E122), Ponceau 4R (E124) and Sunset Yellow FCF (E110).

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 [9] for each result. The procedure is detailed in the relevant protocols [7, 8].

Further background on the procedure followed can be found in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [10].

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 procedure used to derive this consensus involved:

 i) exclusion, if present, of any non numerical results i.e. qualitative or semi-quantitative results,

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

 iii) exclusion of results that were not corrected for purity of standard,

 iv) exclusion of results where no LoQ was reported or the LoQ was greater than the submitted result.

For all analytes, the mode was chosen as the assigned value because the distribution of results was skewed. For each analyte a kernel density plot showing the distribution of data can be seen in the relevant Figure.

The assigned values for all analytes 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 [11].

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, see Table 4,
and σ_p = the standard deviation for proficiency, see Table 4.

Participants' z-scores for all analytes are given in Table 1 and Table 2 and shown as histograms in Figures 1–4. 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 ≤ z ≤ 2 for all analytes 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 ≤ z ≤ 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 [10].

6. REFERENCES

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10 Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonised
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    Chem.*, **78**, No. 1, 145–196.

11 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 Allura Red (E129) and Carmoisine (E122)

laboratory number	analyte							
	Allura Red (E129) assigned value: 21.4 mg/kg				Carmoisine (E122) assigned value: 11.9 mg/kg			
	result	purity of colour standard (%), & corrected (y/n)?	loq	z-score	result	purity of colour standard (%), & corrected (y/n)?	loq	z-score
001	21.3	y	1.5	-0.1	11.6	y	1.5	-0.2
002	20.8			-0.3	11.2			-0.5
003	20.7	78% corrected (y)	1	-0.3	13.2	90% corrected (y)	2	1.0
004	21.9	86.13%, Y	0.6	0.2	11.8	86.24%, Y	0.6	0.0
005	16.07	99%	0.5	**-2.5**	9.25	99%	0.5	-2.0
006	0			**-9.9**	0			**-9.1**
007	detected	na	>1.5		detected	na	>5.0	
008	24.2	98.3, y	0.45	1.3	13.8	93.9, y	0.46	1.5
009	7.36	85% & n	1	**-6.5**	6.86	85% & n	1	**-3.8**
010	detected		2.5		detected		2.5	
011	/	/	/		0	/	/	**-9.1**
012	19.66	86.3	1.4	-0.8	10.99	78	1.5	-0.7
013	18.32	80, y	0.5	-1.4	11.85	97, y	0.5	0.0
014	26.4	85%/Y	1.00	**2.3**	15.9	85%/Y	1.00	**3.1**
015	22	91%,y	<2,0mg/kg	0.3	12	90%, y	<2,0mg/kg	0.1
016	41.29	85%, y	5	**9.2**	23.13	87% y	5	**8.6**
017	22.3	84.6 %, & (y)	2.0	0.4	18.0	99,4 %, & (y)	2.0	**4.7**
018	22.6	100%ï¼? Y	2.5	0.5	#			
019	20.51	80%, y	1	-0.4	13.10	100%, y	1	1.0
020	17.92		1	-1.6	12.49		1	0.5
021	#				#			

z-scores outside $|z| > 2$ are shown in **bold**, see Section 5
\# = not analysed   loq = limit of quantification

Table 1 (continued): Results and z-Scores for Allura Red (E129) and Carmoisine (E122)

laboratory number	analyte							
	Allura Red (E129) assigned value: 21.4 mg/kg				Carmoisine (E122) assigned value: 11.9 mg/kg			
	result	purity of colour standard (%), & corrected (y/n)?	loq	z-score	result	purity of colour standard (%), & corrected (y/n)?	loq	z-score
022	13.58	57, y	12.9	**-3.6**	Detected	87.4, y		
023	15.66	98	0.61	**-2.7**	10.42	85	0.34	-1.1
024	#				10.098	100	10	-1.3
025	19.1	96.93, y	0.20	-1.1	10.5	97.14, y	0.20	-1.0
026	15.13	86 %, y	3 mg/kg	**-2.9**	12.6	87 %, y	3 mg/kg	0.6
027	17	85, y	2	-2.0	11	87.5, y	3	-0.7
028	20.2	84.6, y	2	-0.6	10.6	67.3, y	2	-1.0
029	21.5216	84.6%, Y	30	0.0	11.3502	87.4%, Y	30	-0.4
030	19.6	n	10	-0.8	4<x<10	n	10	
031	19.48	80 % Y	0.1	-0.9	11.28	80 % Y	0.1	-0.4
032	23.0	93.7 % (Y)	2	0.7	11.9	78.8 % (Y)	2	0.0
033	20.95	> 80 not corrected	0.1	-0.2	11.61	> 80 not corrected	0.1	-0.2
034	15.7	100%,y	1.00	**-2.6**	7.4	100%,y	1.00	**-3.4**
035	21.0	98 - n	2.0	-0.2	14.5	85 - y	2.0	2.0
036	21.93	Concentration confirmed by absorbance.	2.5	0.2	#			
037	22.18	91.11	2	0.4	12.30	73.14	2	0.3
038	30	results are not corrected	5	**4.0**	15	results are not corrected	5	**2.4**
039	20.44	98 %, y	1 mg/kg	-0.5	14.09	98 %, y	1 mg/kg	1.7
040	22.7	85.2 & Y	5.0mg/Kg	0.6	9.8	85.3 & Y	5.0mg/Kg	-1.6
041	17		5	-2.0	10		5	-1.4

z-scores outside |z| >2 are shown in **bold,** see Section 5
\# = not analysed   loq = limit of quantification

Table 2: Results and z-Scores for Ponceau 4R (E124) and Sunset Yellow FCF (E110)

laboratory number	analyte							
	Ponceau 4R (E124) assigned value: 4.02 mg/kg				Sunset Yellow FCF (E110) assigned value: 8.85 mg/kg			
	result	purity of colour standard (%), & corrected (y/n)?	loq	z-score	result	purity of colour standard (%), & corrected (y/n)?	loq	z-score
001	4.12	y	1.5	0.2	8.80	y	1.5	-0.1
002	4.3			0.5	8.6			-0.2
003	4.8	87% corrected (y)	2	1.5	9.9	91% corrected (y)	1	1.0
004	4.14	80.56%, Y	0.6	0.2	9.23	88.42%, Y	0.4	0.4
005	3.67	99%	0.5	-0.7	7.30	99%	0.5	-1.5
006	0			**-7.7**	0			**-8.7**
007	detected	na	>1.5		detected	na	>1.5	
008	4.7	96.9, y	0.49	1.3	9.2	94.5, y	0.23	0.3
009	3.00	70% & n	1	-2.0	5.14	90% & n	1	**-3.6**
010	detected		2.5		detected		12.5	
011	/	/	/		/	/	/	
012	3.96	89.8	0.8	-0.1	8.43	90	0.7	-0.4
013	4.57	91, y	0.5	1.1	9.16	99, y	0.5	0.3
014	6.74	80%/Y	1.00	**5.2**	11.6	85%/Y	1.00	**2.7**
015	4.1	82%, y	<2,0mg/kg	0.2	8.3	79%, y	<2,0mg/kg	-0.5
016	9.55	87%, y	5	**10.6**	16.71	76%, y	5	**7.7**
017	5.0	99.0 %, & (y)	2.0	1.9	10.4	99.0 %, & (y)	2.0	1.5
018	3.61	100%ï¼? Y	0.5	-0.8	7.43	100%ï¼? Y	0.5	-1.4
019	3.34	75%, y	1	-1.3	8.98	90%, y.	1	0.1
020	3.82		1	-0.4	7.5		1	-1.3
021	#				#			

z-scores outside |z| >2 are shown in **bold,** see Section 5
= not analysed   loq = limit of quantification

Table 2 (continued): Results and z-Scores for Ponceau 4R (E124) and Sunset Yellow FCF (E110)

laboratory number	analyte							
	Ponceau 4R (E124) assigned value: 4.02 mg/kg				Sunset Yellow FCF (E110) assigned value: 8.85 mg/kg			
	result	purity of colour standard (%), & corrected (y/n)?	loq	z-score	result	purity of colour standard (%), & corrected (y/n)?	loq	z-score
022	Detected	99, y	12.6		Detected	77.4, y	16.1	
023	2.65	99	0.68	**-2.6**	4.92	95	0.69	**-3.9**
024	4.018	90.6	10	0.0	8.458	99.8	10	-0.4
025	3.7	83.25, y	0.20	-0.6	8.1	93.11, y	0.20	-0.7
026	3.63	81 %, y	3 mg/kg	-0.7	9.24	78 %, y	3 mg/kg	0.4
027	4	80.7, y	3	0.0	9	81.8, y	3	0.1
028	3.6	80.2, y	2	-0.8	7.8	87.2, y	2	-1.0
029	4.2968	87%, Y	30	0.5	8.90	77.4%, Y	30	0.0
030	<LOQ	n	10		4<x<10	n	10	
031	3.41	75 % Y	0.2	-1.2	8.45	90% Y	0.1	-0.4
032	4.37	81.9 % (Y)	2	0.7	8.94	85.8 % (Y)	2	0.1
033	4.12	> 80 not corrected	0.1	0.2	9.27	> 80 not corrected	0.1	0.4
034	2.06	100%,y	1.00	**-3.8**	7.3	100%,y	1.00	-1.5
035	0			**-7.7**	12.3	85 - y	2.0	**3.4**
036	4.03	Concentration confirmed by absorbance.	2.5	0.0	8.35	Concentration confirmed by absorbance.	2.5	-0.5
037	4.08	88.60	2	0.1	8.51	90.63	2	-0.3
038	10	results are not corrected	5	**11.5**	15	results are not corrected	5	**6.0**
039	4.20	99 %, y	1 mg/kg	0.3	8.97	95 %, y	1 mg/kg	0.1
040	<5.0	82.6 & Y	5.0mg/Kg		8.9	79.3 & Y	5.0mg/Kg	0.0
041	3		5	-2.0	6		5	**-2.8**

z-scores outside |z| >2 are shown in **bold,** see Section 5
\# = not analysed   loq = limit of quantification

Table 3:   Participants' Comments

laboratory number	comments
004	The material was not received frozen.
005	Carmoisine (122) is not accrediated in our method but still we have in our scope.
019	TFDAA0057.02 Method of Test for Colors in Foods (2)
032	Qualitative detection with accredited method YK010 and quantitive analysis with non-accredited method TLAB-YK011
038	QMAA-IA-30:2020-12 (HPLC-DAD)
039	Eignungspruefung im Rahmen der Verifizierung der Methode
040	Ponceau 4R was detected at less than limit of quantification <5.0mg/Kg (1.6mg/Kg)

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 mg/kg	uncertainty, u	standard deviation for proficiency, σ_p	
Allura Red (E129)	22	21.4	0.5	Horwitz [11]	2.16
Carmoisine (E122)	20	11.9	0.3	Horwitz [11]	1.31
Ponceau 4R (E124)	20	4.02	0.16	Horwitz [11]	0.521
Sunset Yellow FCF (E110)	22	8.85	0.19	Horwitz [11]	1.02

Table 5:   Number and Percentage of z-Scores where $|z| \leq 2$

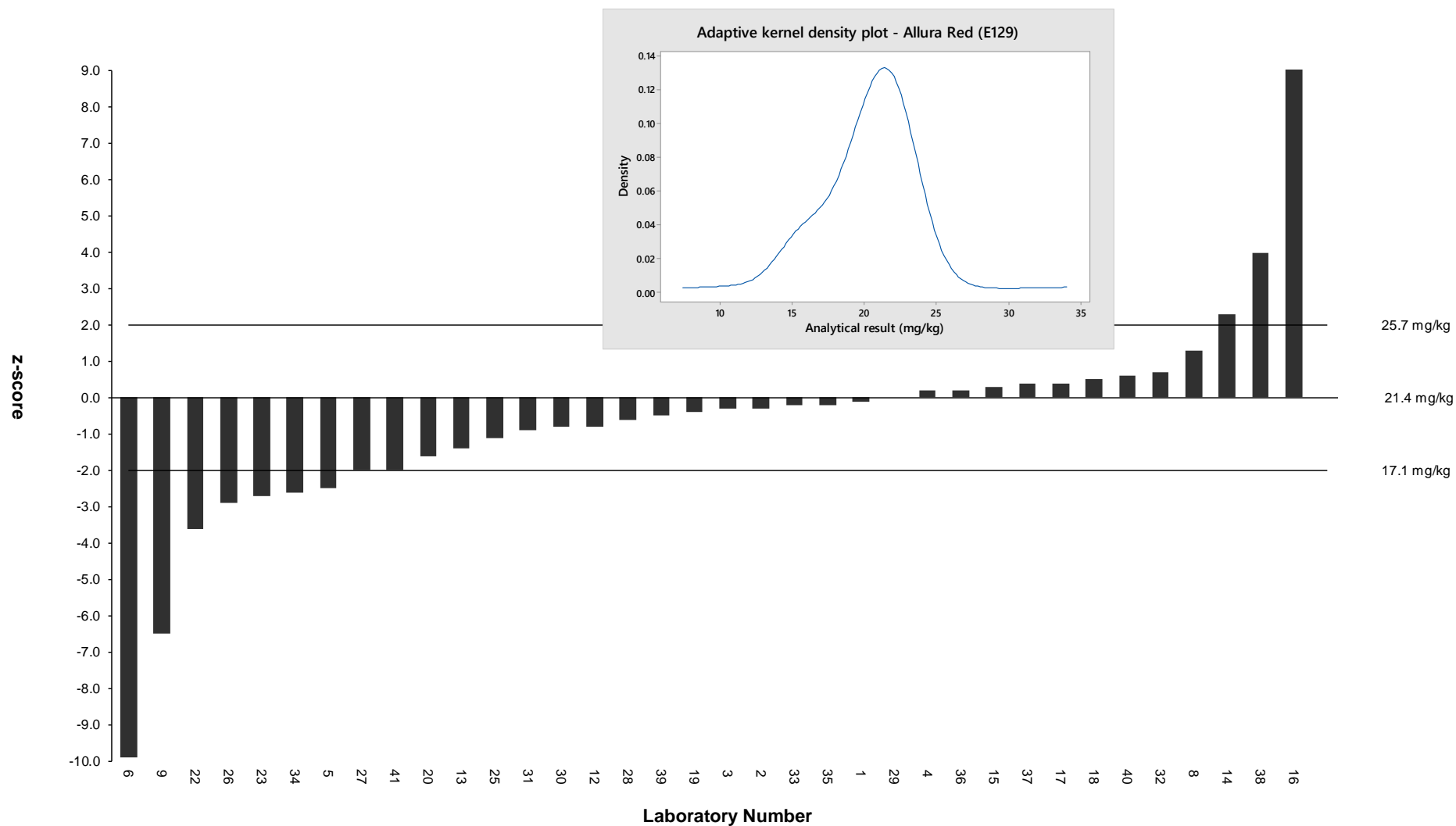| analyte	number of scores where $|z| \leq 2$	total number of scores	% $|z| \leq 2$
Allura Red (E129)	26	36	72
Carmoisine (E122)	26	34	76
Ponceau 4R (E124)	27	34	79
Sunset Yellow FCF (E110)	27	35	77

Figure 1:   z-Scores for Allura Red (E129)

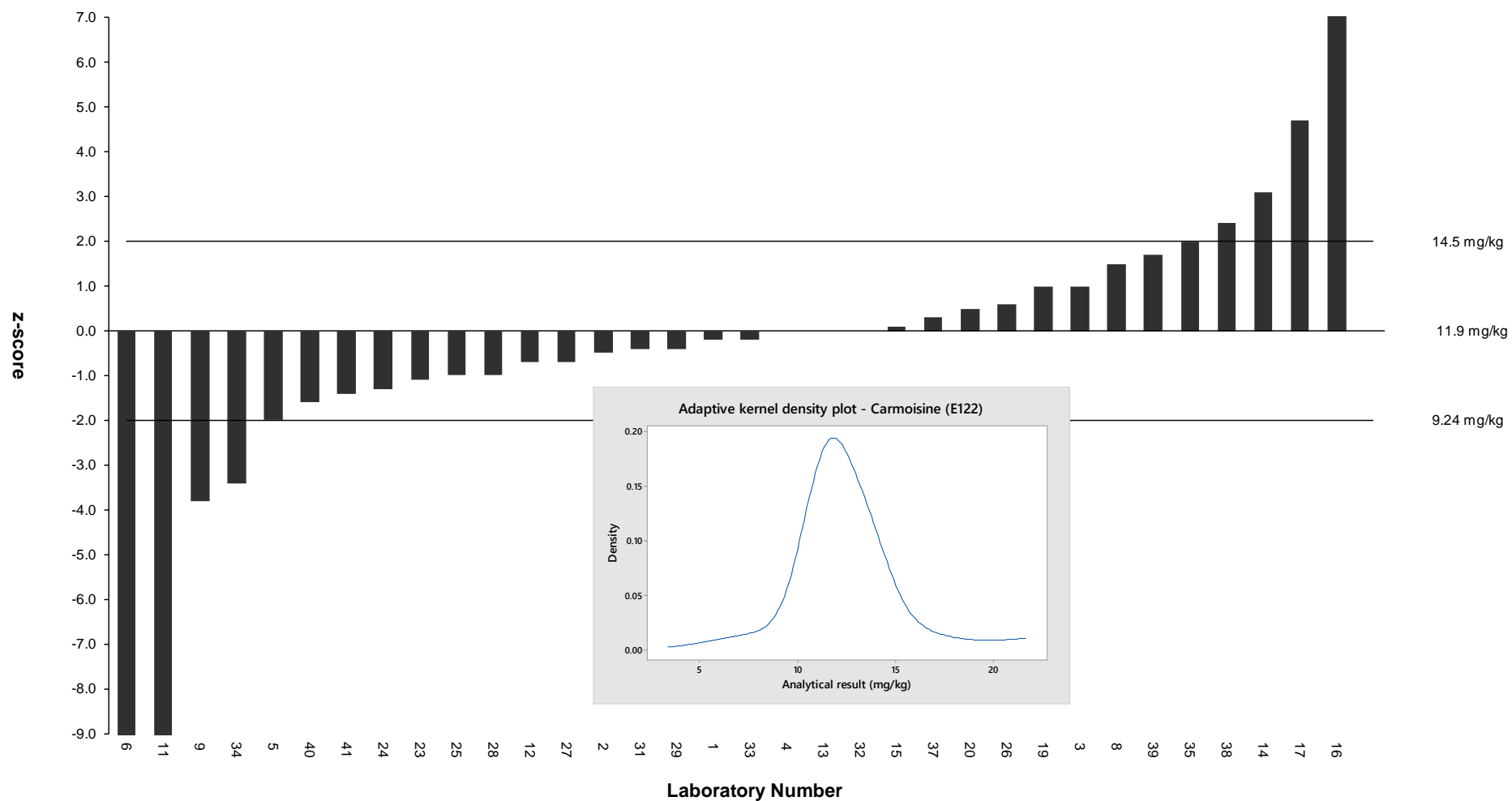insert shows a plot of the distribution of results

Figure 2: z-Scores for Carmoisine (E122)

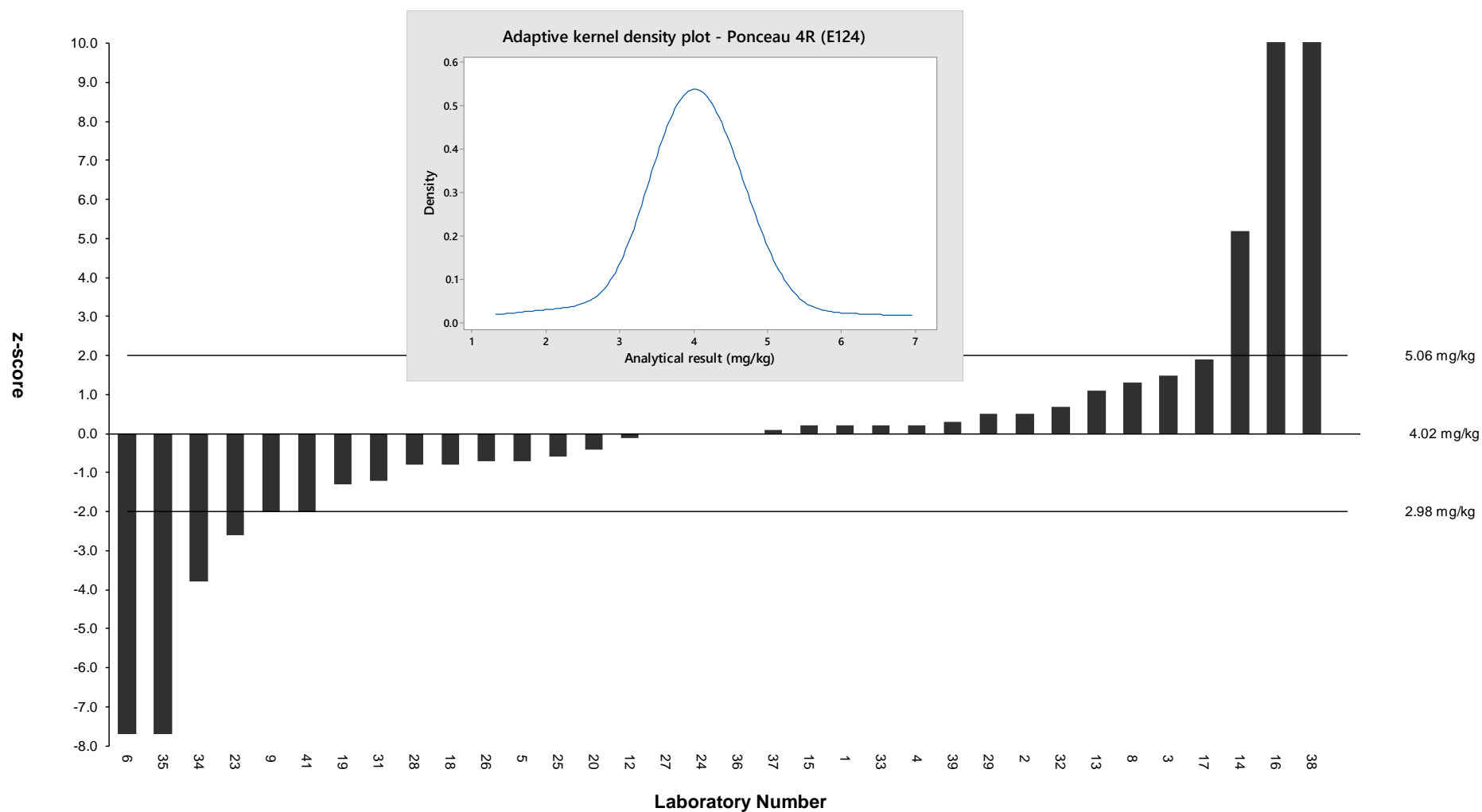insert shows a plot of the distribution of results

Figure 3:   z-Scores for Ponceau 4R (E124)

insert shows a plot of the distribution of results
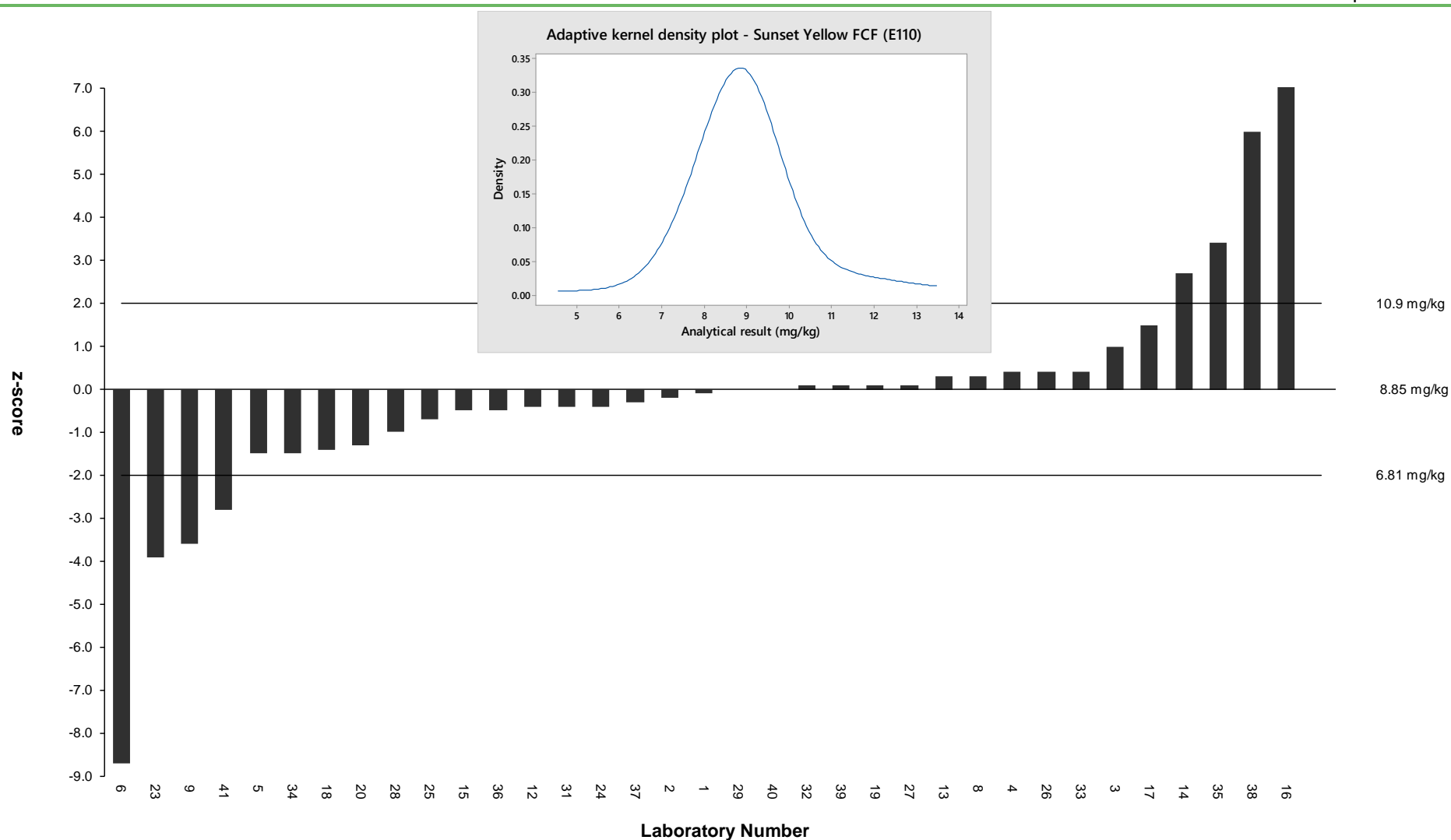
Figure 4: z-Scores for Sunset Yellow FCF (E110)

insert shows a plot of the distribution of results

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. Text that appears as unreadable symbols are derived from entries made using non-Western characters.

Allura Red (E129)

Source of Standards	laboratory number
Dr Ehrenstorfer	012 015 016 020 029
Merck	011 036
Sigma/Aldrich	001 002 006 008 009 013 014 017 023 026 032 035 039
Wako	025
Warner Jenkinson	003
Beijing Haibin Hongmeng Standard Material Co., Ltd	018
Institute of Leather Industry	004
ROHA-IDACOL	030

Wavelength (absorbance)(nm)	laboratory number
245	036
254	012
254 nm	025
480	018
500	020 030
503 nm	009
506	008
510	017
510 nm	029
515	001 026
515 nm	023
518	039
519	015
520	002 011 013 014 016
520, 480 and 430nm	003
520nm	004
530	035

Carmoisine (E122)

Source of Standards	laboratory number
Dr Ehrenstorfer	012 015 023 029
Fluka	020
Sigma/Aldrich	001 002 006 008 009 014 017 025 032 039
Institute of Leather Industry	004
LGC	016 035
Pfaltz & Bauer	013
ROHA-IDACOL	030

Wavelength (absorbance)(nm)	laboratory number
254	012
254 m	025
500	020 030
510	017
510 nm	029
515	001 026
516	024
518	039
519	015
520	002 013 014 016
520, 480 and 430nm	003
520nm	004
523 nm	009
526	008
530	035

Ponceau 4R (E124)

Source of Standards	laboratory number
Acros	006
Dr Ehrenstorfer	012 015 016 029
Merck	011 036
Sigma/Aldrich	001 002 008 009 014 020 023 032 039
Micotox	017
Beijing Haibin Hongmeng Standard Material Co., Ltd	018
Institute of Leather Industry	004
TCI	013

Wavelength (absorbance)(nm)	laboratory number
245	036
254	012
430	011
480	018
500	020
503 nm	009
506	008
507	024
510	017 032
510 nm	029
515	001 026
515 nm	023
518	039
519	015
520	002 013 014 016
520, 480 and 430nm	003
520nm	004

Sunset Yellow FCF (E110)

Source of Standards	laboratory number
Dr Ehrenstorfer	012 015 016 029
Merck	011 036
Sigma/Aldrich	001 002 006 008 009 013 014 017 020 023 032 035 039
Beijing Haibin Hongmeng Standard Material Co., Ltd	018
Institute of Leather Industry	004
ROHA-IDACOL	030

Wavelength (absorbance)(nm)	laboratory number
245	036
254	012
415	015
430	035
435	002
480	017 018
480nm	004
482	024
485	001
485 nm	009
486	014
490	011 013 016
500	020 030
506	008
510	032
510 nm	029
515	026
515 nm	023
518	039
520, 480 and 430nm	003

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 [7, 8] 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 Claire Williamson (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

Fapas®

Fera Science Ltd (Fera)
York Biotech Campus
Sand Hutton
York
YO41 1LZ
UK

Tel: +44 (0)1904 462100

info@fapas.com

fapas.com