

SUMMARY

1. The test material for FAPAS[®] proficiency test 1238 was dispatched in September 2009. Each participant received a sunflower oil test material to analyse for diisobutyl phthalate, di-n-butyl phthalate and diisononyl phthalate. Participants also received a sample of blank sunflower oil that was used to prepare the test material. In total, 47 sets of test materials were distributed to participants in 19 countries and, of these, 33 participants, i.e. 70 %, returned results within the time-scale demanded by the Scheme.
2. The assigned values (\hat{X}) were calculated from the most appropriate measure of central tendency of participants' results [1, 2].
3. The target standard deviation (σ_p), for each analyte was calculated by using the appropriate form of the Horwitz equation [3], and in conjunction with the assigned value (\hat{X}) was used to derive a z-score for participants' results. z-Scores are considered satisfactory if $|z| \leq 2$.
4. For diisononyl phthalate it was not possible to generate an assigned value due to bi-modality of the results and therefore no z-scores were calculated, (see section 4.1).
5. Results for this proficiency test are summarised as follows:

analyte	assigned value \hat{X} , mg/kg	number of satisfactory scores $ z \leq 2$	total number of scores	satisfactory %
diisobutyl phthalate	1.16	26	29	90
di-n-butyl phthalate	1.17	23	30	77
diisononyl phthalate	NA	NA	NA	NA

NA - no assigned value or z-scores calculated, (see section 4.1).

6. Surplus test materials are available for sale, see APPENDIX III.
7. Whereas this Report has been produced in good faith and in accordance with best industry practice, neither the Food and Environment Research Agency nor the Secretary of State for Environment, Food and Rural Affairs accepts any liability whatsoever as to the application or use of the information contained therein.

CONTENTS

1. INTRODUCTION	4
1.1. Proficiency Testing	4
1.2. Phthalates	4
2. TEST MATERIAL	5
2.1. Preparation	5
2.2. Homogeneity	5
2.3. Distribution	5
3. RESULTS	5
4. STATISTICAL EVALUATION OF RESULTS	6
4.1. Calculation of the Assigned Value, \hat{X}	6
4.2. Target Standard Deviation for the Test, σ_p	7
4.3. Individual z-Scores	7
5. REFERENCES	8

TABLES

Table 1: Results and z-Scores for Diisobutyl Phthalate in Sunflower Oil Test Material	9
Table 2: Results and z-Scores for Di-n-butyl Phthalate in Sunflower Oil Test Material	11
Table 3: Results for Diisononyl Phthalate in Sunflower Oil Test Material	13
Table 4: Assigned Values and Target Standard Deviations	15
Table 5: Number and Percentage of Satisfactory z-Scores	15

FIGURES

Figure 1: z-Scores for Diisobutyl Phthalate (1.16 mg/kg) in Sunflower Oil Test Material	16
Figure 2: z-Scores for Di-n-butyl Phthalate (1.17 mg/kg) in Sunflower Oil Test Material	17
Figure 3: Adaptive Kernel Density Plot for Diisononyl Phthalate in Sunflower Oil Test Material	18

APPENDICES

APPENDIX I: Homogeneity Data for Sunflower Oil Test Material	19
APPENDIX II: Analytical Methods Used by Participants	20
APPENDIX III: FAPAS [®] SecureWeb, Reports and Protocol	32

1. INTRODUCTION

1.1. Proficiency Testing

The demand for independent proof of competence from regulatory bodies and customers means that proficiency testing is relevant to all laboratories testing food and feed for quality and safety in every country. Hence, it is a requirement of accreditation to ISO 17025 [4] that the laboratory takes part in a proficiency testing scheme, if a suitable scheme exists. Further, for laboratories entrusted with the official control of food and feeds, Article 12 of EU Regulation (EC) 882/2004 [5] requires such laboratories to be assessed and accredited in accordance with ISO 17025, i.e. proficiency testing is a legal requirement for these laboratories. Thus, together with the use of validated methods, proficiency testing is an essential element of laboratory quality assurance.

The analysis of an external quality check sample as part of a laboratory's routine procedures provides objective standards for individual laboratories to perform against and permits them to compare their analytical results with those from other laboratories. Such standards and comparisons can go beyond the actual chemical analysis. For example, the ability to report results in specified units and within a given time scale are important aspects of quality. Hence, participants in FAPAS[®] who submit results after the closing date of a proficiency test are only included in the statistical evaluation if there are extenuating circumstances.

It is important to understand the statistical limitations of this external means of quality assessment when gauging the competence of a laboratory. The results of a typical chemical analysis will be normally distributed. That is to say, the majority of results will be centred on a mean value but, inevitably, some results will lie at the extremes of the distribution. The statistics of a normal distribution mean that about 95% of data points will lie between a z-score of -2 and +2. Performance in a FAPAS[®] proficiency test, therefore, is considered 'satisfactory' if a participant's z-score lies within this range. It follows that if a participant's z-score lies outside $|z| > 2$ there is about a 1 in 20 chance that their result is in fact an acceptable result from the extreme of the distribution. If a participant's z-score lies outside $|z| > 3$ the chance that their result is actually acceptable is only about 1 in 300.

Full details of the FAPAS[®] proficiency testing scheme is available via our protocols [6, 7].

1.2. Phthalates

Phthalates are organic chemicals produced from oil that are mainly used as plasticizers. The most widely used phthalates are di-2-ethylhexyl phthalate, diisodecyl phthalate and diisononyl phthalate. Phthalates are also frequently used in food packaging, childrens toys, nail polish, fishing lures, adhesives and paint pigments.

2. TEST MATERIAL

2.1. Preparation

The test material was prepared by a laboratory contracted to do so by FAPAS[®].

Sunflower oil was mixed and split into two portions. The first portion was measured into glass bottles (50 mL) and used for the blank material. The second portion was spiked with diisobutyl phthalate, di-n-butyl phthalate and diisononyl phthalate to achieve nominal concentrations of 1 mg/kg, 1 mg/kg and 10 mg/kg respectively. The spiked oil was mixed for a further 2 hours and approximately 50 ml was transferred to glass serum bottles. The samples were stored at ambient temperature before dispatch.

2.2. Homogeneity

Ten randomly selected test materials were analysed in duplicate for all analytes by a laboratory contracted to do so by FAPAS[®]. The results, together with their statistical evaluation [8], are given in APPENDIX I. These data show sufficient homogeneity and are not included in the subsequent calculation of the assigned value.

The statistical tests initially check the data for any widely discrepant pairs using Cochran's test. If found, such data are removed. Thereafter the remaining data are subject to analysis of variance (ANOVA) to estimate the sampling and analytical variances.

2.3. Distribution

The dispatch date was 10 September 2009. Each participant received an individually numbered oil test material and a blank oil packed in a padded envelope, together with a covering letter, instructions for electronic submission and the results form for participants with no internet access.

3. RESULTS

Participants were required to report their data in mg/kg, corrected for recovery. Participants were advised to calculate results based on the true mass of simulant analysed and not to make the conventional assumption that the density of food simulants is 1. Participants were required to report if their results were corrected for recovery and to state their percentage recovery if this was applicable. Results were submitted by 33 participants before the closing date for this test, 14 October 2009.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported concentrations are given in Tables 1-3.

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

4. STATISTICAL EVALUATION OF RESULTS

The object of the statistical procedure employed is to obtain a simple and transparent result, which the participant and other interested parties can readily appreciate. The procedure follows that recommended in the IUPAC/ISO/AOAC International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories [9].

4.1. Calculation of the Assigned Value, \hat{X}

The assigned value, \hat{X} , i.e. the best estimate of the true concentration of each analyte, was set as the consensus of the results submitted by participants. The procedure used to derive this consensus involved where necessary:

- Removing non valid data,
 - i) results reported as approximately 10, 100 or 1000 x greater or smaller than the majority of submitted results (as these were considered to be reporting errors).
 - ii) results reported as semi quantitative.
- Minimising the influence of outliers by the use of a robust statistical procedure to derive the mean [2].
- Comparing the mean derived from a robust statistical procedure [2] with any modes found by a procedure to identify multimodality ('bump-hunting') [1].
- Considering the normality (Kolmogorov-Smirnov test), or otherwise, of the distribution of the selected results.
- Assessing the uncertainty (u) of the mode and the robust mean. For the mode u was taken to be directly equivalent to the standard error of the mode. For the robust mean:

$$u = \frac{\hat{\sigma}}{\sqrt{n}}$$

where $\hat{\sigma}$ = the standard deviation of the robust mean [2].

NB this is NOT the target standard deviation for the test (σ_p)

and n = the number of data points used to calculate the robust mean.

The scrutiny of the results for diisobutyl phthalate and di-n-butyl phthalate was straightforward. The robust mean was considered to be the most appropriate measure of central tendency of participants' results and was therefore used to set the assigned values.

For diisononyl phthalate (DINP), investigation showed the distribution of results to be multi-modal, with two major modes of approximately equal proportion. The IUPAC name of DINP is 1,2-benzenedicarboxylic acid, di-C8-10 branched alkylesters, C9 rich; meaning that it is a mixture of isomers and homologues. Further investigation showed that DINP is described by two CAS numbers and these are 68515-48-0 and 28553-12-0. When samples of these two were purchased and compared by analysis using GC-MS, by a laboratory contracted to do so by FAPAS[®], both the selected ion chromatograms and the total ion chromatograms revealed a difference in their isomer profiles and a difference in their response factors. A large majority of participants used GC-MS to test for DINP. If these participants used one or other of these two DINPs as calibration standards this could account for the bimodal distribution seen.

Due to the empirical nature of these data for this analyte, it was not possible to calculate an assigned value, nor issue z-scores. Results are given for information in Table 3.

The robust means used to set the assigned values for diisobutyl phthalate and di-n-butyl phthalate, together with u , n and $\hat{\sigma}$ are shown in Table 4.

4.2. Target Standard Deviation for the Test, σ_p

The value of σ_p determines the limits of satisfactory performance in a FAPAS® proficiency test. It is set at a value that reflects best practice for the analyses in question. The standard deviation of reproducibility found in collaborative trials is generally considered an appropriate indicator of the best agreement that can be obtained between laboratories. However, not all analyses have been characterised in this manner. In such cases, the predictive models of the appropriate form of the Horwitz equation [3] are valuable indicators of best practice.

In the absence of appropriate collaborative trial data, σ_p was derived from the appropriate form of the Horwitz equation [3]. This equation predicts a standard deviation from a given concentration, c , and requires c to be expressed as a dimensionless mass ratio, e.g. 1 ppm $\equiv 10^{-6}$ or % $\equiv 10^{-2}$. It follows therefore that to express the dimensionless standard deviation predicted by the equation in the original concentration units it must be divided by the relevant mass ratio:

- i) for analyte concentrations <120 ppb

$$\sigma_p = \frac{0.22c}{mr}$$

- ii) for analyte concentrations ≥ 120 ppb and $\leq 13.8\%$

$$\sigma_p = \frac{0.02c^{0.8495}}{mr}$$

- iii) for analyte concentrations >13.8%

$$\sigma_p = \frac{0.01c^{0.5}}{mr}$$

where, in all three cases, c = concentration, i.e. the assigned value, \hat{X} , expressed as a dimensionless mass ratio, e.g. 1 ppm $\equiv 10^{-6}$ or % $\equiv 10^{-2}$
 and mr = dimensionless mass ratio, e.g. 1 ppm $\equiv 10^{-6}$ or % $\equiv 10^{-2}$.

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

4.3. Individual z-Scores

Participants' z-scores were calculated as:

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

where x = the participant's reported result
 \hat{X} = the assigned value
 and σ_p = the target standard deviation.

Participants' z-scores for diisobutyl phthalate and di-n-butyl phthalate are given in Table 1 and 2, and shown as histograms in Figures 1 and 2. The kernel density plot showing the modality for diisononyl phthalate is shown in Figure 3.

The number and percentage of z-scores in the satisfactory range, $|z| \leq 2$, for diisobutyl phthalate and di-n-butyl phthalate are given in Table 5.

z-Scores were not calculated for diisononyl phthalate as it was not possible to calculate an assigned value.

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 target standard deviations prior to their publication in Table 4.

5. REFERENCES

- 1 Lowthian, P.J. and Thompson, M., 2002, Bump-hunting for the proficiency tester-searching for multimodality, *Analyst*, **127**, 1359-1364.
- 2 Analytical Methods Committee, 1989, Robust Statistics – How not to reject outliers Part 1. Basic Concepts, *Analyst*, **114**, 1693-1697.
- 3 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.
- 4 ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories.
- 5 Regulation (EC) 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules, *Official Journal*, **L 165**, 30/04/2004, 0001-0141.
- 6 FAPAS[®], 2009, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Revision 2009, Version 1, Issued November 2009.
- 7 FAPAS[®], 2009, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS[®], Revision 2009, Version 1, Issued November 2009.
- 8 Fearn, T. and Thompson, M., 2001, A new test for sufficient homogeneity, *Analyst*, **126**, 1414-1417.
- 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(1)**, 145–196.

Table 1: Results and z-Scores for Diisobutyl Phthalate in Sunflower Oil Test Material

laboratory number	analyte			
	diisobutyl phthalate			
	assigned value 1.16 mg/kg			
	result mg/kg	corrected for recovery	% recovery	z-score
001	1.3	no	99	0.8
002	0.99	Yes	see comments*	-1.0
003	3.0	no	-	10.1
004	1.044	Yes	94.3	-0.7
005	1.12	no		-0.2
006	1.3	no	85	0.8
007	1.20	No		0.2
008	1.18	Yes		0.1
009	1.15	No		-0.1
010	1.210	Yes	95	0.3
011	1.22	No		0.3
012	1.29	No	100.17	0.7
013	1.18	No		0.1
014	1.09	No	NA	-0.4
015	1.12	No		-0.2
016	1.33	yes	94	0.9
017	0.90	No		-1.4
018				
019	1.750	Yes		3.2
020	12.75	NO		63.7

z-scores outside the satisfactory range, i.e. $|z| > 2$, are shown in **bold**

*participant comment – Matrix matched calibration curve.

Table 1 (continued): Results and z-Scores for Diisobutyl Phthalate in Sunflower Oil Test Material

laboratory number	analyte			
	diisobutyl phthalate			
	assigned value 1.16 mg/kg			
	result mg/kg	corrected for recovery	% recovery	z-score
021	1.3	NO		0.8
022	1.52	no		2.0
023	1.17	yes		0.0
024	1.2	No	89	0.2
025	0.90	Yes	76	-1.4
026				
027				
028	0.85	YES	60	-1.7
029	0.968	no		-1.1
030	0.797	No	80	-2.0
031	1.1	No	100	-0.3
032	<10	No	89	
033	1.2	No		0.2

z-scores outside the satisfactory range, i.e. $|z| > 2$, are shown in **bold**

Table 2: Results and z-Scores for Di-n-butyl Phthalate in Sunflower Oil Test Material

laboratory number	analyte			
	di-n-butyl phthalate			
	assigned value 1.17 mg/kg			
	result mg/kg	corrected for recovery	% recovery	z-score
001	1.2	no	91	0.2
002	1.05	Yes	see comments*	-0.7
003	3.6	no	-	13.3
004	1.156	Yes	94.3	-0.1
005	1.00	no		-0.9
006	n.n.	no	82	
007	1.22	No		0.3
008	1.29	Yes		0.7
009	1.10	No		-0.4
010	1.240	Yes	95	0.4
011	1.30	No		0.7
012	1.34	No	94.77	0.9
013	1.08	No		-0.5
014	1.01	No	NA	-0.9
015	1.09	No		-0.4
016	2.46	yes	96	7.1
017	0.87	No		-1.6
018				
019	1.654	Yes		2.7
020	5.26	NO		22.4

z-scores outside the satisfactory range, i.e. $|z| > 2$, are shown in **bold**

*participant comment – Matrix matched calibration curve.

Table 2 (continued): Results and z-Scores for Di-n-butyl Phthalate in Sunflower Oil Test Material

laboratory number	analyte			
	di-n-butyl phthalate			
	assigned value 1.17 mg/kg			
	result mg/kg	corrected for recovery	% recovery	z-score
021	1.0	NO		-0.9
022	1.26	no		0.5
023	1.12	yes		-0.3
024	1.2	No	92	0.2
025	0.98	Yes	76	-1.0
026	1.14	yes	123	-0.2
027	3.6	no	102	13.3
028	0.55	YES	60	-3.4
029	1.16	no		-0.1
030	0.757	No	85	-2.3
031	0.88	No	100	-1.6
032	<10	No	93	
033	1.1	No		-0.4

z-scores outside the satisfactory range, i.e. $|z| > 2$, are shown in **bold**

Table 3: Results for Diisononyl Phthalate in Sunflower Oil
 Test Material

laboratory number	analyte		
	diisononyl phthalate		
	<i>no assigned value calculated</i>		
	result mg/kg	corrected for recovery	% recovery
001	12	no	110
002	5.61	Yes	see comments*
003	26.1	no	-
004	5.979	Yes	94.3
005			
006	8.3	no	50
007	5.81	No	
008	3.69	Yes	
009	<20	No	
010	6.500	Yes	95
011	7.86	No	
012	7.49	Yes	84.4
013	5.64	No	
014	9.37	No	NA
015	9.2	No	
016	5.14	yes	90
017	4.87	No	
018	71.7	Yes	152
019	9.07	Yes	
020	3.59	NO	

*participant comment – Matrix matched calibration curve.

Table 3 (continued): Results for Diisononyl Phthalate in Sunflower Oil Test Material

laboratory number	analyte		
	diisononyl phthalate		
	<i>no assigned value calculated</i>		
	result mg/kg	corrected for recovery	% recovery
021	<4	NO	
022	8.34	no	
023	10.2	yes	
024	8.5	No	81
025			
026	1.88	yes	70
027			
028			
029	10.3	no	
030	0	No	
031	3.3	No	100
032	<10	No	
033	11.6	No	

Table 4: Assigned Values and Target Standard Deviations

analyte	assigned value				target standard deviation	
	data points, n	robust mean, \hat{X} , mg/kg	robust standard deviation, $\hat{\sigma}$	uncertainty, u	derived from	σ_p
diisobutyl phthalate	28	1.16	0.19	0.0355	Horwitz*	0.182
di-n-butyl phthalate	30	1.17	0.25	0.0499	Horwitz*	0.183

*see page 7 for appropriate form of the Horwitz equation

Table 5: Number and Percentage of Satisfactory z-Scores

analyte	number of satisfactory scores $ z \leq 2$	total number of scores	satisfactory %
diisobutyl phthalate	26	29	90
di-n-butyl phthalate	23	30	77

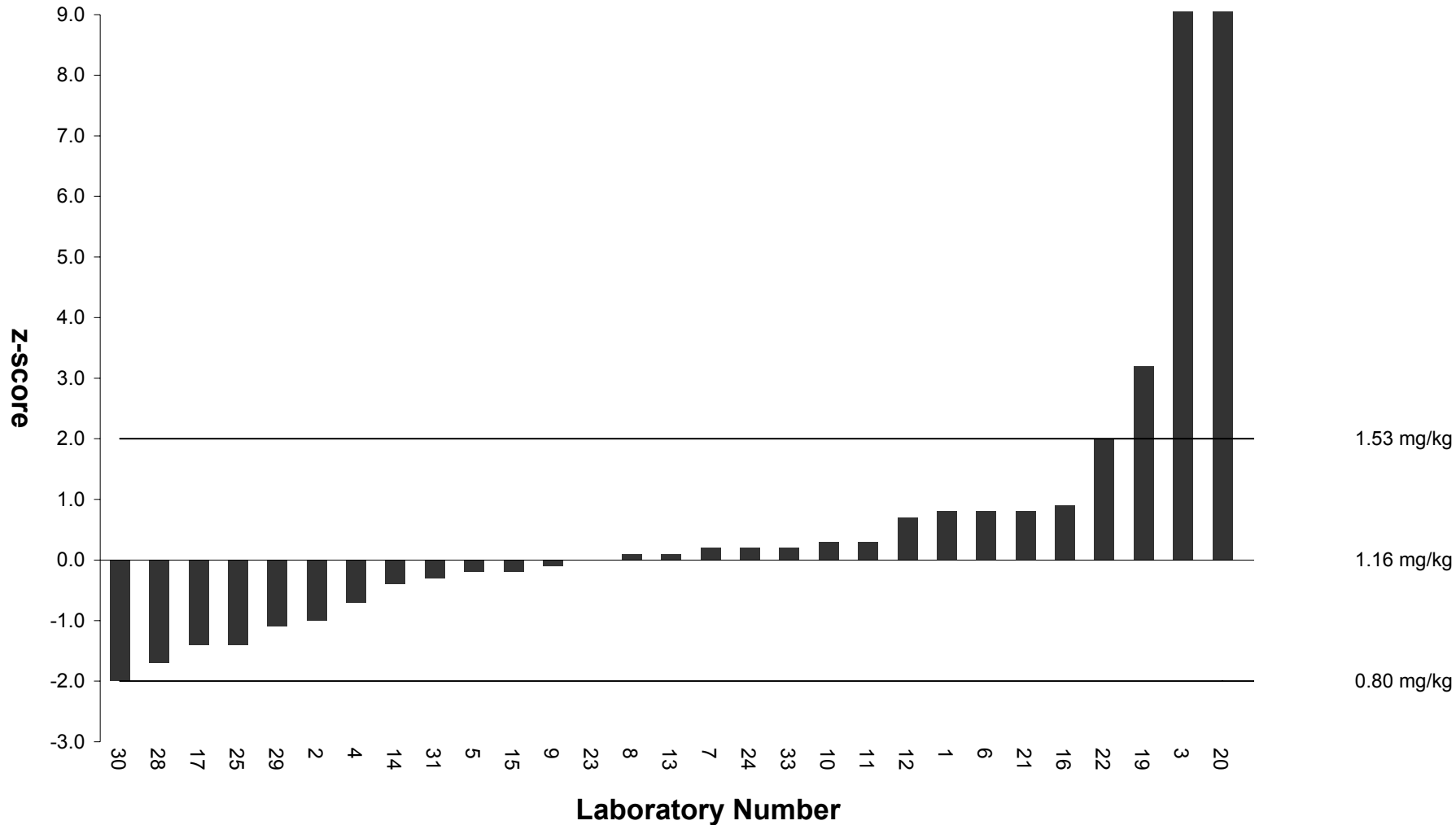


Figure 1: z-Scores for Diisobutyl Phthalate (1.16 mg/kg) in Sunflower Oil Test Material

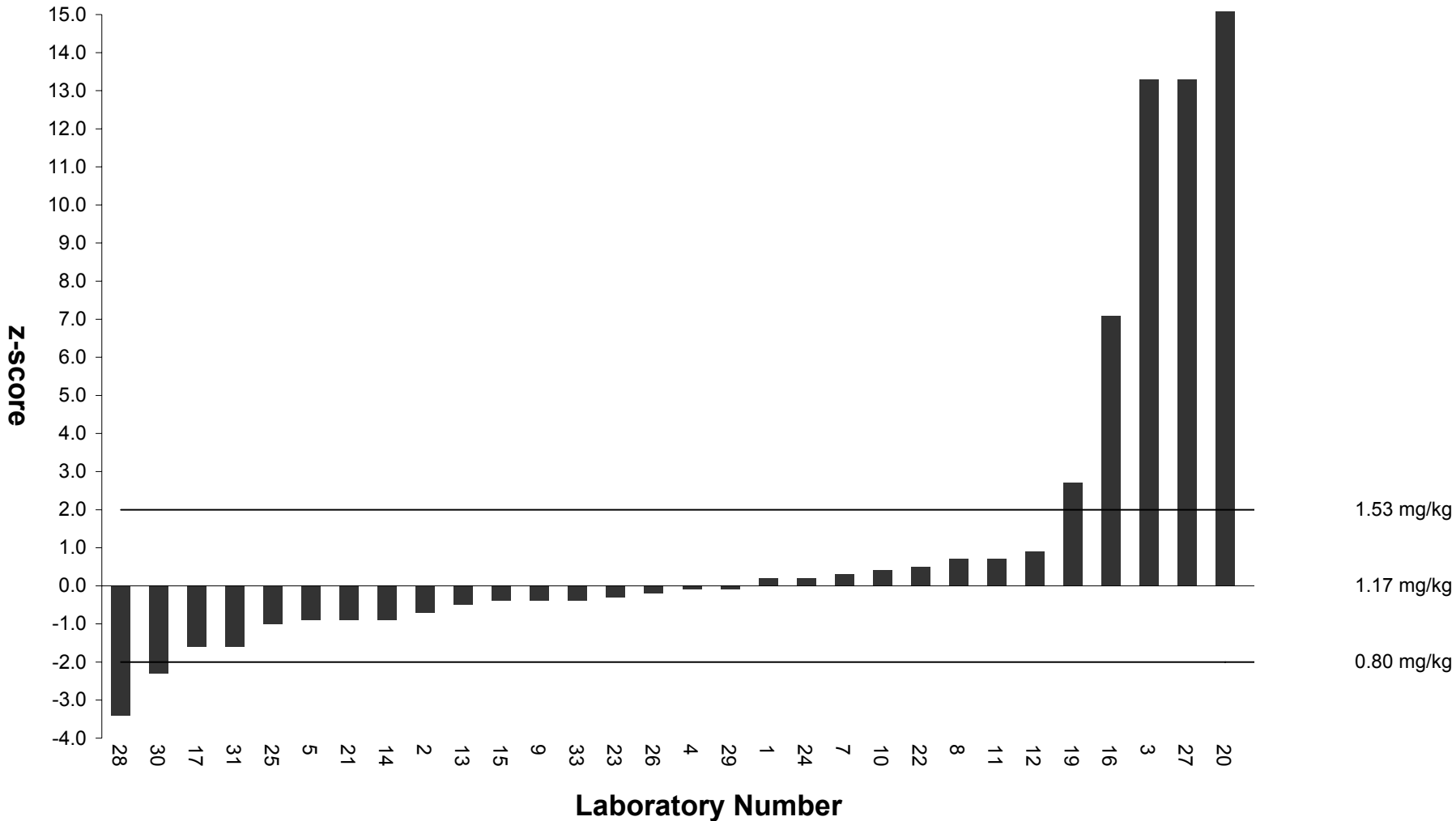


Figure 2: z-Scores for Di-n-butyl Phthalate (1.17 mg/kg) in Sunflower Oil Test Material

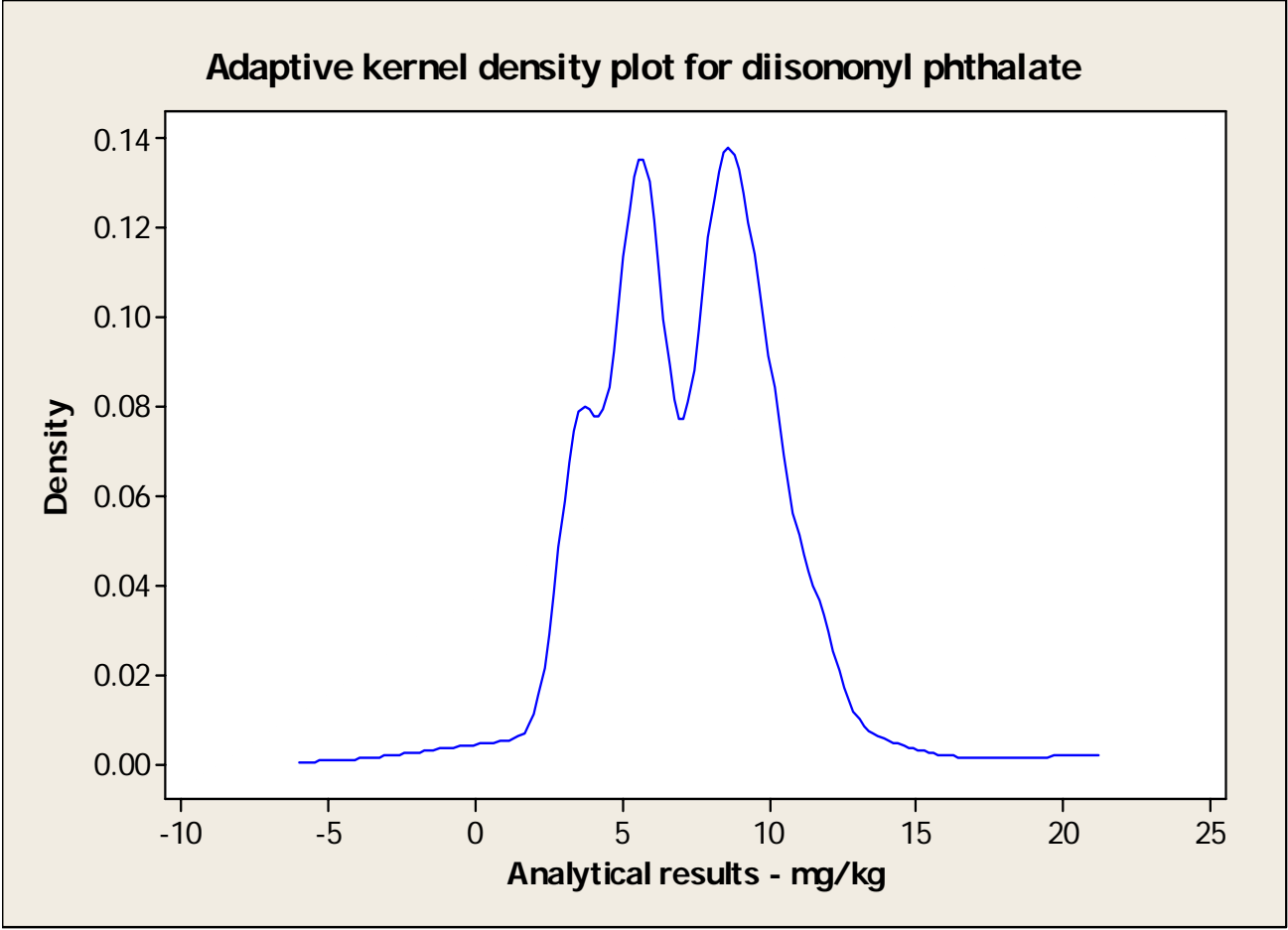


Figure 3: Adaptive Kernel Density Plot for Diisononyl Phthalate in Sunflower Oil Test Material

APPENDIX I: Homogeneity Data for Sunflower Oil Test Material

sample identity	analyte					
	diisobutyl phthalate mg/kg		di-n-butyl phthalate mg/kg		diisononyl phthalate mg/kg	
	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2
1	1.2	1.2	1.3	1.3	10.5	10.5
2	1.2	1.2	1.3	1.4	9.4	10.5
3	1.2	1.2	1.4	1.4	11.2	10.7
4	1.2	1.3	1.4	1.4	10.0	10.3
5	1.3	1.2	1.4	1.4	10.5	10.8
6	1.2	1.2	1.4	1.4	10.3	10.5
7	1.2	1.2	1.4	1.3	10.4	11.9
8	1.2	1.2	1.3	1.3	11.7	11.2
9	1.2	1.2	1.4	1.3	10.5	11.8
10	1.2	1.2	1.4	1.3	11.2	10.7
<i>mean, n</i>	1.2	20	1.4	20	10.7	20
origin of target sd (σ_p)	Horwitz	original	Horwitz	original	Horwitz	original
abs. target sd (σ_p) & as RSD%	0.19	15.5	0.21	15.3	1.20	11.2
s_{an}	0.032		0.045		0.553	
s_{sam}^2	0		0.00056		0.0782	
σ_{all}^2	0.0032		0.0039		0.130	
<i>critical</i>	0.0070		0.0093		0.553	
$s_{sam}^2 < \text{critical?}$	ACCEPT		ACCEPT		ACCEPT	

APPENDIX II: Analytical Methods Used by Participants

Notes:

- 1) Participants' methods are tabulated according to the information submitted electronically, but some responses may have been combined or edited for clarity.

Di-isobutyl phthalate

Accredited Method Used	laboratory number
yes	006 007 010 016 019 024 031
no	001 002 003 004 008 009 011 012 013 014 015 022 023 025 028 029 032 033

Sample Weight (g)	laboratory number
<1	008 031
≥1 - <2	003 004 006 007 010 016 019 022 023 024 025 029 033
≥2 - <5	002 005 009 013 020 028 032
≥5 - <10	012 014 015
≥10 - <25	011

Extraction Solvent	laboratory number
acetonitrile	002 003 004 011 012 013 015 022 023 024 028 033
cyclohexane	005 006 008 010
dichloromethane	025 032
ethyl acetate	005 006 008 010
hexane	016 029 031
isopropanole	004
t-butylmethylether	001
water	004

Sample Clean-up Technique	laboratory number
extraction	003 020 023 024
GPC / HPGPC	005 006 008 010 016 019 025
liquid / liquid extraction	029
silica column	013
solid phase extraction (SPE) (column/cartridge)	004 011
none	001 002 012 014 022 028 031 032 033

SPE Column Type	laboratory number
alumina	015
C18	004
LC-florisil	011
silica	006 013

GC Column Type	laboratory number
capillary	001 002 003 005 006 007 008 010 011 013 014 015 022 024 025 028 029 031 032 033
narrowbore	016 023

GC Column Packing	laboratory number
100% methyl polysiloxane	024
95% methyl 5% phenyl polysiloxane	002 006 008 010 011 013 014 015 016 019 022 023 025 028 029 031 032 033
HP5	003
HT-8	001

GC Injection Volume (µL)	laboratory number
≥1 - <2	001 002 003 005 006 010 013 014 015 016 019 022 023 024 025 028 029 031 032 033
≥2 - <5	011
≥5 - <10	008

GC Injection Mode	laboratory number
PTV	008 031
split	013 032
splitless	001 002 003 005 006 010 011 014 015 016 019 022 023 028 029 033

GC Detector	laboratory number
ITD	029
MSD	001 002 005 006 008 010 013 014 015 016 019 022 023 024 025 028 031 032 033
MS-MS	003 011

HPLC Column Packing	laboratory number
C18	004 012 020 024

Used HPLC Guard Column	laboratory number
yes	012 024
no	004 016 020

Mobile Phase Programme	laboratory number
isocratic	020
gradient	004 012 024

Mobile Phase Components	laboratory number
acetic acid	024
acetonitrile	020 024
ammonium formate	004
methanol	004 012
water	012 020

HPLC Column Temperature (°C)	laboratory number
>ambient - <50	004 012 020 024

HPLC Injection Volume (µL)	laboratory number
≥5 - <10	012 020
≥10 - <25	004 024

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	004 024
≥0.25 - <0.75	012 020

HPLC Pre Column Derivatisation	laboratory number
none	004 012

HPLC Post Column Derivatisation	laboratory number
none	004 012

HPLC Detector Type	laboratory number
MS	012
MS-MS	004 024

Di-n-butyl phthalate

Accredited Method Used	laboratory number
yes	006 010 016 024 031
no	001 002 003 004 008 009 011 012 013 014 015 022 023 025 026 027 028 029 032 033

Sample Weight (g)	laboratory number
<1	008 031
≥1 - <2	003 004 006 010 016 022 023 024 025 026 029 033
≥2 - <5	002 005 009 013 020 028 032
≥5 - <10	012 014 015
≥10 - <25	011

Extraction Solvent	laboratory number
acetonitrile	002 003 004 011 012 013 015 022 023 024 028 033
cyclohexane	005 006 008 010 026
dichloromethane	025 032
ethyl acetate	005 006 008 010 026
hexane	016 027 029 031
isopropanole	004
t-butylmethylether	001
water	004

Sample Clean-up Technique	laboratory number
extraction	003 020 023 024
florisil column	011
GPC / HPGPC	005 006 008 010 016 025 026
liquid / liquid extraction	029
silica column	013
solid phase extraction (SPE) (column/cartridge)	004 027
none	001 002 012 014 022 028 031 032 033

SPE Column Type	laboratory number
alumina	015
C18	004
florisil	027
LC-florisil	011
silica	006 013

GC Column Type	laboratory number
capillary	001 002 003 005 006 008 010 011 013 014 015 022 024 025 026 027 028 029 031 032 033
narrowbore	016 023

GC Column Packing	laboratory number
100% methyl polysiloxane	024
95% methyl 5% phenyl polysiloxane	002 006 008 010 011 013 014 015 016 022 023 025 026 027 028 029 031 032 033
HP5	003
HT-8	001

GC Injection Volume (µL)	laboratory number
≥1 - <2	001 002 003 005 006 010 013 014 015 016 022 023 024 025 026 027 028 029 031 032 033
≥2 - <5	011
≥5 - <10	008

GC Injection Mode	laboratory number
PTV	008 031
split	013 026 027 032
splitless	001 002 003 005 006 010 011 014 015 016 022 023 028 029 033

GC Detector	laboratory number
FID	027
ITD	029
MSD	001 002 005 006 008 010 013 014 015 016 022 023 024 025 026 028 031 032 033
MS-MS	003 011

HPLC Column Packing	laboratory number
C18	004 012 020 024

Used HPLC Guard Column	laboratory number
yes	012 024
no	004 020

Mobile Phase Programme	laboratory number
isocratic	020
gradient	004 012 024

Mobile Phase Components	laboratory number
acetic acid	024
acetonitrile	020 024
ammonium formate	004
methanol	004 012
water	012 020

HPLC Column Temperature (°C)	laboratory number
>ambient - <50	004 012 020 024

HPLC Injection Volume (µL)	laboratory number
≥5 - <10	012 020
≥10 - <25	004 024

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	004 024
≥0.25 - <0.75	012 020

HPLC Pre Column Derivatisation	laboratory number
none	004 012

HPLC Post Column Derivatisation	laboratory number
none	004 012

HPLC Detector Type	laboratory number
MS	012
MS-MS	004 024

Di-isononyl phthalate

Accredited Method Used	laboratory number
yes	006 010 016 019 024 031
no	001 002 003 004 008 009 011 012 013 014 015 018 022 023 026 029 032 033

Sample Weight (g)	laboratory number
<1	008 031
≥1 - <2	003 004 006 010 016 019 022 023 024 026 029 033
≥2 - <5	002 009 013 020 032
≥5 - <10	012 014 015
≥10 - <25	011

Extraction Solvent	laboratory number
acetone	018
acetonitrile	002 003 004 011 012 013 015 022 023 024 033
cyclohexane	006 008 010 019 026
dichloromethane	032
ethyl acetate	006 008 010 019 026
hexane	016 018 029 031
isopropanole	004
t-butylmethylether	001
water	004

Sample Clean-up Technique	laboratory number
extraction	003 020 023 024
GPC / HPGPC	006 008 010 016 018 019 026
liquid / liquid extraction	029
silica column	013
solid phase extraction (SPE) (column/cartridge)	004 011
none	001 002 012 014 022 031 032 033

SPE Column Type	laboratory number
alumina	015
C18	004
LC-florisil	011
silica	006 013

GC Column Type	laboratory number
capillary	001 002 003 006 008 010 011 013 014 015 018 022 024 026 029 031 032 033
narrowbore	016 019 023

GC Column Packing	laboratory number
100% methyl polysiloxane	024
95% methyl 5% phenyl polysiloxane	002 006 008 010 011 013 014 015 016 019 022 023 026 029 031 032 033
HP5	003
HT-8	001
WCOT Fused Silica 25m x 0.25mm ID coating CP-SIL	018

GC Injection Volume (µL)	laboratory number
≥1 - <2	001 002 003 006 010 013 014 015 016 018 019 022 023 024 026 029 031 032 033
≥2 - <5	011
≥5 - <10	008

GC Injection Mode	laboratory number
PTV	008 031
split	013 018 026 032
splitless	001 002 003 006 010 011 014 015 016 019 022 023 029 033

GC Detector	laboratory number
FID	018
ITD	029
MSD	001 002 006 008 010 013 014 015 016 019 022 023 024 026 031 032 033
MS-MS	003 011

HPLC Column Packing	laboratory number
C18	004 012 020 024

Used HPLC Guard Column	laboratory number
yes	012 024
no	004 016 020

Mobile Phase Programme	laboratory number
isocratic	020
gradient	004 012 024

Mobile Phase Components	laboratory number
acetic acid	024
acetonitrile	020 024
methanol	004 012
water	012 020
ammonium formate	004

HPLC Column Temperature (°C)	laboratory number
>ambient - <50	004 012 020 024

HPLC Injection Volume (µL)	laboratory number
≥5 - <10	012 020
≥10 - <25	004 024

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	004 024
≥0.25 - <0.75	012 020

HPLC Pre Column Derivatisation	laboratory number
none	004 012

HPLC Post Column Derivatisation	laboratory number
none	004 012

HPLC Detector Type	laboratory number
MS	012
MS-MS	004 024

APPENDIX III: FAPAS[®] SecureWeb, Reports and Protocol

1. FAPAS[®] SECUREWEB

Access to the secure area of our web site 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 and quality control materials. , *including surplus test materials from the batch used in this proficiency test.*
- Freely download copies of reports, in Acrobat PDF format, of proficiency tests in which they have participated.

2. REPORTS

The Acrobat PDF version of this report is available to all participants as a free download from FAPAS[®] SecureWeb.

A printed and bound version of this report is priced £35 if ordered at the same time as the proficiency test or £50 if ordered subsequently.

3. PROTOCOL

The Protocols [6, 7] set out how FAPAS[®] is organised. Copies can be downloaded from our website.

4. CONTACT DETAILS

Participants with any comments or concerns about this proficiency test should contact:

FAPAS[®]

Food and Environment Research Agency
Sand Hutton, York
YO41 1LZ
UK

Tel: +44 (0)1904 462100
Fax: +44 (0)1904 462040
e-mail: info@fapas.com
testmaterials@fapas.com
web: www.fapas.com

The Food and Environment Research Agency is an ISO 9001 certified organisation.

