



FAPAS[®] Report 02194

Malachite Green in Oily Fish

September-October 2012

NOT CONTROLLED WHEN PRINTED

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

Since 2006 all FAPAS reports have been distributed as Adobe® Certified Document Services (CDS) Adobe® PDF documents [1].

The use of Adobe® CDS allows the PDF files to certify that the author of the report is FAPAS and that the document has not been altered in anyway. A blue ribbon and information bar indicates this validation when the document is opened using Adobe® Reader v7 or later.

Hard copies of FAPAS reports can never incorporate this level of integrity and consequently when a FAPAS report is printed a watermark, stating that printed copies are not controlled, appears on every page.

End users of FAPAS reports should ensure that either the opened PDF file displays a valid FAPAS digital signature or that the content of any hard copy exactly matches the content of a PDF file that displays a valid FAPAS digital signature.

QUALITY SYSTEMS

FAPAS® is accredited by UKAS as complying with the requirements of ISO/IEC 17043:2010 [2].

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



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.

© Crown Copyright 2012

SUMMARY

1. The test material for FAPAS® proficiency test 02194 was dispatched in September 2012. Each participant received an oily fish test material to be analysed for leucomalachite green and total malachite green (measured as malachite green + leucomalachite green).
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 µg/kg	number of scores, $ z \leq 2$	total number of scores	% $ z \leq 2$
leucomalachite green	2.46	51	58	88
total malachite green	4.75	52	62	84

4. Surplus test materials are available for sale, see APPENDIX II.

NOT CONTROLLED WHEN PRINTED

CONTENTS

1. INTRODUCTION	5
1.1. Proficiency Testing	5
2. TEST MATERIAL	5
2.1. Preparation	5
2.2. Homogeneity	5
2.3. Dispatch	5
3. RESULTS	5
4. STATISTICAL EVALUATION OF RESULTS	6
4.1. Calculation of the Assigned Value, x_a	6
4.2. Standard Deviation for Proficiency, σ_p	6
4.3. Individual z-Scores	6
5. INTERPRETATION OF SCORES	7
6. REFERENCES	7
TABLES	
Table 1: Results and z-Scores	8
Table 2: Participants' Comments	11
Table 3: Assigned Values and Standard Deviations for Proficiency	11
Table 4: Number and Percentage of z-Scores where $ z \leq 2$	11
FIGURES	
Figure 1: z-Scores for Leucomalachite Green	12
Figure 2: z-Scores for Total Malachite Green	13
APPENDICES	
APPENDIX I: Analytical Methods Used by Participants	14
APPENDIX II: FAPAS SecureWeb, Protocol and Contact Details	23

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® 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 by blending together blank samples of Atlantic Salmon (*Salmo Salar*) muscle and Atlantic Salmon muscle incurred with malachite green.

Samples were stored at -20°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 values.

2.3. Dispatch

The start date was 04 September 2012. Test materials were sent to 71 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the level of leucomalachite green and total malachite green (malachite green + leucomalachite green) present in the test material, in µg/kg, corrected for recovery.
- Report your method of recovery correction: internal standard, recovery percentage, matrix-extracted calibration or standard addition.
- Please report CCβ (CCbeta), the 'detection capability' 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 64 participants (90%) before the closing date for this test, 04 October 2012.

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 each analyte. The assigned values were then used in combination with the standard deviation for proficiency, σ_p , to calculate a z-score for each result. The procedure follows that recommended in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [7].

Further details on the procedure followed can be found in the relevant protocols [3, 4].

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) results where neither the use of an internal standard nor a recovery percentage nor a matrix-extracted calibration curve was reported (as these were considered to be uncorrected for recovery),
- ii) qualitative or semi-quantitative results, e.g. <1500, positive.

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

The assigned values for both analytes are shown in Table 3.

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 both analytes, σ_p was derived from the appropriate form of the Horwitz equation [8].

The values for σ_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 σ_p = the standard deviation for proficiency.

Participants' z-scores for both analytes are given in Table 1 and shown as histograms in Figures 1–2. 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 all analytes 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 [7].

6. REFERENCES

- 1 Adobe Certified Document Services, http://www.adobe.com/security/partners_cds.html, accessed 08/05/2012.
- 2 ISO/IEC 17043:2010, Conformity assessment – General requirements for proficiency testing.
- 3 FAPAS, 2012, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 3, Issued January 2012.
- 4 FAPAS, 2012, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS®, Version 2, Issued January 2012.
- 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, *Official Journal*, L 221, 17.8.2002, 8–36.
- 6 ISO 11843-1: 1997, Capability of detection — Part 1: Terms and definitions.
- 7 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.
- 8 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								
	leucomalachite green assigned value 2.46 µg/kg					total malachite green assigned value 4.75 µg/kg			
	result µg/kg	int. std. added or % recovery	CCβ µg/kg	z-score	result µg/kg	int. std. added or % recovery	CCβ µg/kg	z-score	
001	#				3.700		2.0	-1.0	
002	2.7	Y		0.4	5.7	Y		0.9	
003	4.9	Y M	0.5	Q 4.5	8.3	Y M	0.5	Q 3.4	
004	2.13	Y M	0.40	-0.6	5.68	Y M	0.41	0.9	
005	1.35	M	1.0	Q -2.0	2.83	M	1.0	Q -1.8	
006	#				5.15	Y	0.5	0.4	
007	2.4	Y	0.25	Q -0.1	5.6	Y	0.25	Q 0.8	
008	1.93	Y 101.53% S	0.5	-1.0	3.01	Y 98.14% S	0.5	-1.7	
009	2.82	Y	0.5	0.7	5.02	Y	0.5	0.3	
010	2.80	Y		0.6	4.80	Y		0.1	
011	2.41	Y	1.43	-0.1	5.53	Y	1.43	0.8	
012	2.73	75%		0.5	4.84	80%		0.1	
013	2.69	Y	0.50	Q 0.4	4.80	Y	1.00	Q 0.1	
014	2.9450	Y	0.5	0.9	5.5725	Y	1	0.8	
015	2.80	Y 103.2%	0.03	Q 0.6	4.25	Y 63.2% 103.2%	0.08	Q -0.5	
016	2.7	Y S	0.5	Q 0.4	3.9	Y S	0.5	Q -0.8	
017	3.30	Y	0.20	Q 1.6	5.11	Y	0.20	Q 0.3	
018	2.5	S	0.9	0.1	7.3	S	0.95	2.4	
019	2.42	Y	0.50	Q -0.1	3.96	Y	0.50	Q -0.8	
020	2.59	Y	0.67	0.2	3.42	Y	1.00	-1.3	
021	1.28	96-100%	0.25	Q -2.2	3.06	96-100%		-1.6	
022	2.15	Y		-0.6	4.25	Y	0.5	-0.5	
023	3.29	Y M	1.655	1.5	7.06	Y M	1.414	2.2	
024	3.06	62%	0.03	1.1	4.53	75%	0.04	-0.2	
025	5.1	Y S	<1	4.9	13.9	Y S	<1	8.8	
026	1.79	M	0.15	-1.2	3.51			-1.2	
027	2.516	Y M	0.10	0.1	5.293	Y M	0.10	0.5	

= not analysed Y = internal standard used M = matrix-extracted calibration used
S = Standard addition Q = LoQ (Limit of Quantification)
z-scores outside |z| >2 are shown in **bold**, see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte								
	leucomalachite green assigned value 2.46 µg/kg					total malachite green assigned value 4.75 µg/kg			
	result µg/kg	int. std. added or % recovery	CCβ µg/kg	z-score	result µg/kg	int. std. added or % recovery	CCβ µg/kg	z-score	
028	1.9	Y	0.22	-1.0	4.88	Y	0.27	0.1	
029	2.6	Y	0.8	0.3	5.8	Y	0.9	1.0	
030	2.05	Y M	0.3	Q -0.8	3.56	Y M	0.3	Q -1.1	
031	2	M	0.3	-0.8	3	M	0.3	-1.7	
032	1.9	M	0.30	Q -1.0	2.8	M	0.30	Q -1.9	
033	2.51	Y M	0.3	Q 0.1	5.10	Y M	0.3	Q 0.3	
034	1.25	65	3.9	-2.2	1.75	72	3.9	-2.9	
035	2.55	Y 59.1%	2	Q 0.2	4.93	Y 61.3%	2	Q 0.2	
036	2.8	Y M		0.6	4.7	Y M		0.0	
037	2.54	Y M	0.3	0.1	4.78	Y M	0.6	0.0	
038	4.145	M	0.12	3.1	5.658	M	0.21, 0.12	0.9	
039	2.48	N 39.6%	1	Q 0.0	3.49	N 52.3% (Malachite Green)	2	Q -1.2	
040	2.20	Y 90-110%	0.05	-0.5	4.23	Y 90-110%	0.05	-0.5	
041	3.2	Y 92.8%	0.5	Q 1.4	8.1	Y 96.6%	1	Q 3.2	
042	2.10	Y M	0.5	Q -0.7	5.73	Y M	0.5	Q 0.9	
043	2.13	Y 100 M	0.03	-0.6	4.10	Y 100 M	0.06	-0.6	
044	2.25	N	1	Q -0.4	5.2	N	1	Q 0.4	
045	#				4.01		0.25	Q -0.7	
046	2.9	Y		0.8	9.4	Y		4.5	
047	2.44	Y	0.30	0.0	5.83			1.0	
048	2.42	Y	1	Q -0.1	4.63	Y		-0.1	
049	<1500		1300	Q	#				
050	1.30	Y 85.88	0.10	-2.1	3.00	Y 80.28	0.20	-1.7	
051	3.01	Y M	0.218	1.0	5.98	M		1.2	
052	2.14	Y	1.00	Q -0.6	6.28	Y	1.00	Q 1.5	
053	2.98	Y S	0.3	1.0	5.02	Y S	0.3	0.3	
054	1.57	Y 94.0% M	0.06	-1.6	2.47	Y 94.0% M	0.07	-2.2	

= not analysed Y = internal standard used N = internal standard not used
M = matrix-extracted calibration used S = Standard addition Q = LoQ (Limit of Quantification)
z-scores outside |z| >2 are shown in **bold**, see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte									
	leucomalachite green assigned value 2.46 µg/kg					total malachite green assigned value 4.75 µg/kg				
	result µg/kg	int. std. added or % recovery	CCβ µg/kg	z-score		result µg/kg	int. std. added or % recovery	CCβ µg/kg	z-score	
055	POSITIVE	Y	0.8			POSITIVE	Y	0.8		
056	2.78	73	0.25		0.6	4.3				-0.4
057	1.97	58%	2	Q	-0.9	3.28	71%	4	Q	-1.4
058	2.00	Y	1.5		-0.8	4.78	Y	1.3 (MG)		0.0
059	1.4		0.3		-2.0	4.6		0.3		-0.1
060	1.34	Y M	0.29		-2.1	1.71	Y M	0.54		-2.9
061	2.52				0.1	4.40				-0.3
062	3.43	Y 94 % M	0.2	Q	1.8	8.74	Y 94 % M	0.2	Q	3.8
063	#					4.1	Y			-0.6
064	2.17	Y M			-0.5	3.76	Y M			-0.9

= not analysed

Q = LoQ (Limit of Quantification)

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

Table 2: Participants' Comments

participant number	comments
012	Recovery Corrected
017	GB/T19857-2005 Determination of malachite green and crystal violet residues in aquatic product
022	the method: T?CBTh-LC/MS02.10
024	Recovery correction for total MG is the average of MG and LMG recoveries
031	LC/MS/MS-Ref. FDA No. 4395, Volume 23
039	Malachite green d5 and Leucomalachite green d5 are used as internal injection standard
049	Test Method: TLC; extraction SPE; Column C18 methanol; Mobile phase for TLC; Ethyl acetate-methanol-ammonia (70-30-15).
055	ONLY FOR QUALITATIVE SCREENING ANALYSIS

comments are as submitted by participants
 comments relating to CCβs or LoQs are included in Table 1

Table 3: Assigned Values and Standard Deviations for Proficiency

analyte	data points, <i>n</i>	assigned value, <i>x_a</i> , µg/kg	uncertainty, <i>u</i>	standard deviation for proficiency, <i>σ_p</i> , µg/kg	
leucomalachite green	55	2.46	0.081	Horwitz [8]	0.541
total malachite green	54	4.75	0.203	Horwitz [8]	1.044

Table 4: 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$
leucomalachite green	51	58	88
total malachite green	52	62	84

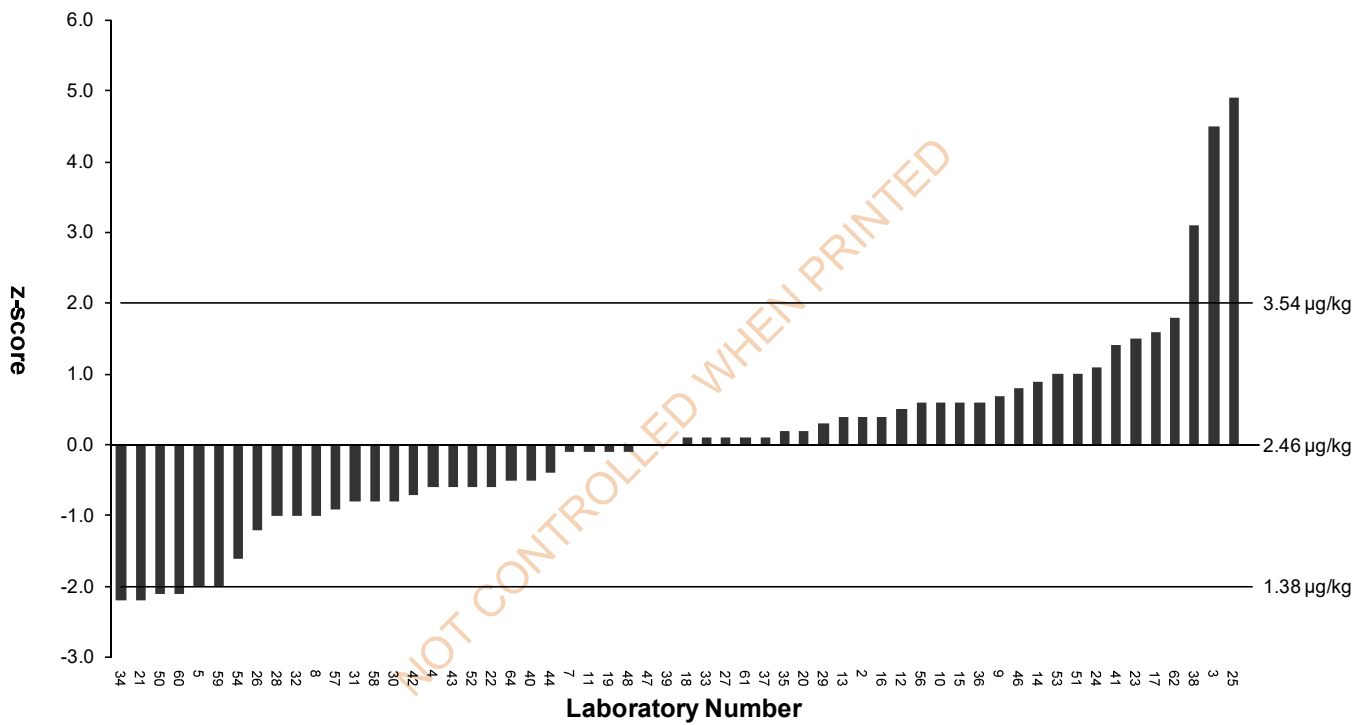


Figure 1: z-Scores for Leucomalachite Green

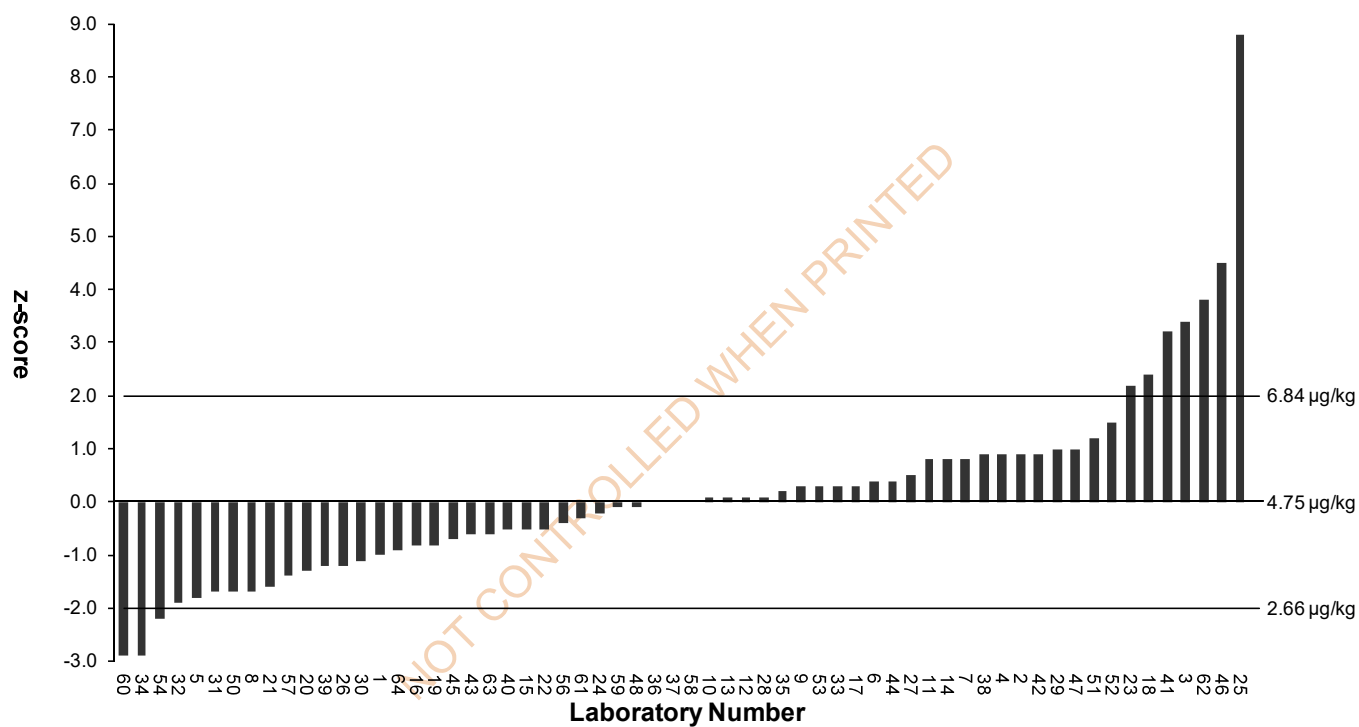


Figure 2: z-Scores for Total Malachite Green

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	003 004 006 008 010 011 012 013 014 015 016 017 018 019 021 023 024 025 029 031 032 034 037 042 043 044 046 047 048 050 051 052 053 054 055 056 057 058 059 061 064
no	001 005 007 020 026 027 028 035 038 039 040 045 049 060 062 063

Method Basis	laboratory number
International Standard	008 009 010 020 028 029 031 034 035 047 049 053 062
National Standard	007 012 013 015 016 017 019 042 054
Paper Published In An International Journal	018 021 024 026 037 038 040 044 056 057 058 060
Manufacturer/Kit Instructions/Technical Note	001 005 045
In house method	003 004 006 011 014 023 025 027 032 039 043 046 048 050 051 052 055 061 063 064

Sample Weight (g)	laboratory number
<1	048
≥1 - <2	001 003 004 005 007 008 011 019 021 023 027 028 032 038 042 050 052 054 062 063
≥2 - <5	010 013 014 015 020 024 025 026 029 031 034 037 040 043 044 047 051 053 056 057 060 061
≥5 - <10	006 012 016 017 018 035 039 045 055 058 064

Extraction Procedure	laboratory number
cold solvent extraction at atmospheric pressure	003 004 006 013 015 020 044 045 052 054 055 057 061 064
maceration/homogenisation	001 035 038 056
shaking	010 012 025 028 029 031 037 040 042 047 051 053 058
sonicate/ultrasonic bath	011 019 032
vortex mix	005 007 008 014 017 021 023 026 027 034 046 048 050 062 063

Extraction Procedure (continued)	laboratory number
homogenise with solvent	043
Polytron followed by rotation mixing	024
rotative stirrer	060
shake with solvent/sonicate - ultrasonic bath	039
SPE	049
ultra turrax	018

Extraction Solvent Components	laboratory number
acetic acid	052
acetonitrile	003 004 007 008 010 011 013 014 015 016 017 019 020 021 023 024 025 026 027 028 029 031 032 034 035 037 038 039 040 042 043 044 045 046 047 048 051 052 053 054 055 056 057 058 060 061 062 063 064
ammonia	049
ammonium acetate	018
buffer	003 046
dichloromethane	024 028 039 040 056 058 063
diethyl ether	009
ethyl acetate	001 049
hydroxylamine hydrochloride	004 018 053 060 062
magnesium sulphate	060
Mcllvaine buffer	005 006 007 025 050
methanol	005 006 049 050
perchloric acid	024 040 055
phosphate buffer	042 063
p-toluenesulfonic acid	018
TMPD solution	005
trichloroacetic acid	054
water	052 061 062

Sample Work Up	laboratory number
centrifuge	004 005 006 007 008 011 012 013 014 015 016 017 018 019 021 023 024 025 026 028 029 031 037 038 039 040 042 043 045 047 050 051 053 054 056 057 058 060 061 062 064
defatted with hexane	039 055

Sample Work Up (continued)	laboratory number
evaporate	001 010 013 020 039 058
filter	027 035
pH adjustment	044 052
none	003

Sample Clean-up Technique	laboratory number
filter	015 026 027 037 040 053 062
liquid/liquid extraction	003 011 020 025 028 035 038 039 047 057 061
solid phase extraction (SPE) (column/cartridge)	005 007 008 010 012 013 014 015 016 017 018 019 024 025 034 035 040 042 043 044 049 050 054 055 056 057 058 061 063 064
solid phase extraction (SPE) (dispersive)	045
QuEChERS	031
none	001 004 021 023 029 052 060

SPE Sorbent Type	laboratory number
alumina	013 015 017 018 045
Bond Elut AccuCAT	055
C18	024 034 040 043 049 056
MCX	005 007 008 010 014 019 042 044 050 054
Oasis HLB	063
SCX	006 016 025 061 064
Alumina and PRS	058
InertSep PRS	035
LB-1	012
PRS	057

Calibrations	laboratory number
solvent	019 052 055 056 057
matrix-matched	003 004 005 011 013 020 023 024 026 027 028 029 031 032 037 038 042 043 047 050 053 054 058 060 062 063 064
single-level	005 006 012 055
multi-level	004 007 009 010 013 014 015 017 019 020 021 024 028 035 037 039 040 045 046 048 051 052 053 057 060 061
standard addition	008 016 018 025 052 053

Type of Internal Standard Added	laboratory number
stable isotope labelled analogue	003 004 006 007 008 010 011 013 014 015 016 017 019 020 021 023 025 027 028 029 035 037 039 040 042 043 046 047 048 050 051 052 053 054 055 058 060 062 063
structural analogue	005 064
none	001 012 024 038 044 056 057 061

Method of Determination	laboratory number
ELISA	001 045
HPLC	003 004 005 006 008 010 011 012 013 014 016 017 018 019 020 021 024 025 026 027 028 031 034 035 037 038 039 040 042 043 044 046 047 048 051 053 054 055 056 057 058 059 060 061 062 063 064
LC-MS/MS	007 023 050 009 015
TLC	049
Witega	029

HPLC Column Packing	laboratory number
C18	003 004 005 006 007 008 010 011 012 013 014 015 016 017 018 019 020 021 023 025 026 027 028 029 031 034 035 037 038 039 040 042 043 046 047 049 050 051 053 054 055 058 062 063 064
C8	056 060
cyano	057
PFP	052
phenyl-hexyl	061

Mobile Phase Components	laboratory number
acetate buffer	035
acetic acid	003 004 009 016 017 043 051 053
acetonitrile	004 006 008 013 014 015 016 017 018 019 020 024 025 026 029 031 034 035 037 038 039 040 042 046 047 050 051 052 053 054 055 057 058 060 061 062 063
ammonia	049
ammonium acetate	004 006 007 008 010 013 014 015 016 017 018 019 023 025 026 027 034 037 038 042 043 046 047 050 051 053 054 055 056 058 060 062

Mobile Phase Components (continued)	laboratory number
ammonium formate	020 024 028 040 044
ethyl acetate	049
formic acid	005 008 014 020 021 027 029 031 037 039 042 051 052 054 063
methanol	003 005 007 013 021 028 049 056 063
sodium acetate	057
water	005 006 007 008 011 013 024 031 039 040 042 051 052 053 061 063 064

HPLC Post-Column Derivatisation	laboratory number
lead oxide	057
none	003 005 006 007 008 010 012 013 015 016 017 019 020 021 023 024 025 026 035 038 040 042 047 050 054 055 056 058 060 061 062 063 064

HPLC Detector Type	laboratory number
Diode Array Detector	057
fluorescence	018 061
MS-MS	003 004 005 006 007 008 010 011 013 014 015 016 017 019 020 021 023 024 025 026 027 028 029 031 034 035 037 038 039 040 042 043 044 046 047 050 051 052 053 054 055 056 058 060 062 063 064
VWD	012

ELISA Test Kit Name	laboratory number
Leucomalachite Green ELISA	001
Malachite Green Plate kit	045

ELISA Kit Manufacturer	laboratory number
Abraxis LLC	045
Randox	001

ELISA Antibody Description	laboratory number
no info	001 045

ELISA Standard Material	laboratory number
provided by test manufacturer	001 045

ELISA Number of Standards	laboratory number
6	001 045

ELISA Time Requirement for Testing (min) [not sample preparation]	laboratory number
≥90 - <120	045
≥120	001

ELISA Calculation of Results	laboratory number
4 parameter	001
Point to point	045

ELISA Extraction Solvent	laboratory number
acetonitrile	045
Ethyl Acetate	001

ELISA Sample Extraction (weight/volume, g/ml)	laboratory number
<0.5	001
≥2 - <5	045

ELISA Dilution Factor of Sample Preparation	laboratory number
1:4	001
1:26 - 1:50	045

Leucomalachite Green

Limit of Detection (µg/kg)	laboratory number
<0.01	012 050
≥0.01 - <0.1	015 024 027 040
≥0.1 - <1	003 007 008 010 013 014 016 017 019 020 021 023 025 026 028 029 031 032 042 043 044 047 052 058 061 062
≥1 - <10	005 035 048 056 057

CC alpha (decision limit) µg/kg	laboratory number
≥0.01 - <0.1	024 027 038 040 043 050
≥0.1 - <1	004 007 008 020 025 026 028 029 031 032 047 051 053 056 060
≥1 - <10	011 023 058

CC beta (detection capability) µg/kg	laboratory number
≥0.01 - <0.1	024 040 043 050
≥0.1 - <1	004 007 008 016 020 025 026 027 028 029 031 032 038 047 051 053 056 060
≥1 - <10	011 023 058

MS-MS Transitions Monitored ions have been rounded to whole numbers	laboratory number
leucomalachite green	
331>222	038
331>223	004 056
331>239	003 019 020 031 060 056 053 008 062 029 040 039 007 042 046 027 026 052 028 023 024 051 011 004 032 017 050 016 005 015 043 037 013 035 058 038
331>315	020 008 029 040 039
331>316	003 019 025 031 060 053 062 026 052 028 023 024 032 017 005 015 013 047 058
d5- leucomalachite green	
336>321	037

Wavelength (Absorbance)(nm)	laboratory number
257	012
618	057

Total Malachite Green

Limit of Detection (µg/kg)	laboratory number
<0.01	012
≥0.01 - <0.1	014 015 019 024 027 040 052
≥0.1 - <1	003 006 007 008 013 016 017 020 021 023 025 026 028 029 031 032 042 043 044 045 058 061 063
≥1 - <10	005 035 056 057 062

CC alpha (decision limit) µg/kg	laboratory number
≥0.01 - <0.1	024 027 038 040 043
≥0.1 - <1	004 006 007 008 020 025 026 028 029 031 032 051 053 056 060
≥1 - <10	011 023 058

CC beta (detection capability) µg/kg	laboratory number
≥0.01 - <0.1	024 040 043
≥0.1 - <1	004 006 007 008 016 020 025 026 027 028 029 031 032 038 051 053 056 060
≥1 - <10	001 011 023 058

MS-MS Transitions Monitored	laboratory number
ions have been rounded to whole numbers	
malachite green	
329>208	019 008 062 052 028 024 032 038 005 023 031 053 040 017 029 013 060 020 058 003 015
329>241	038
329>313	027 028 052 051 024 016 005 023 015 032 060 042 020 058 003 019 006 046 008 062 031 053 040 007 017 025 029 013 035 043
329>314	037
leucomalachite green	
331>223	004
331>239	058 027 026 024 011 004 032 016 005 015 043 007 029 014 042 020 062 013
331>315	029 020
331>316	025 062 058 026 024 032 005 015 013
d5-malachite green	
335>320	037

Wavelength (Absorbance)(nm)	laboratory number
600 & 257	012
618	057

NOT CONTROLLED WHEN PRINTED

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 and quality control materials.
- Freely download copies of reports (PDF file), of proficiency tests in which they have participated.

2. PROTOCOL

The Protocols [3, 4] set out how FAPAS® 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
The Food and Environment Research Agency
Sand Hutton
York
YO41 1LZ
UK

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

info@fapas.com
testmaterials@fapas.com

www.fapas.com