FAPAS® Report 0990

Pesticide Residues in Oats

August-September 2014

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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UKAS PROFICIENCY TESTING
0009

SUMMARY

1. The test material for FAPAS® proficiency test 0990 was dispatched in August 2014. Each participant received an oat test material to be analysed for chlormequat, mepiquat and glyphosate.
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| ≤2	total number of scores	% |z| ≤2
chlormequat	282	41	53	77
mepiquat	145	43	53	81
glyphosate	954	37	42	88

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

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® proficiency testing scheme are available in our protocols [3, 4].

2. TEST MATERIAL

2.1. Preparation

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

The test material was prepared from organic oats, which were milled to a fine powder. The bulk sample was split into two batches: one for spiking and one for the blank test material.

Sub-samples were taken to screen for the possible presence of incurred residues and the remainder was stored at -20°C. No incurred residues were detected at, or above 30 µg/kg.

Chlormequat, mepiquat and glyphosate were spiked into the test material.

Samples were stored at ambient temperature 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 21 August 2014. Test materials were sent to 63 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the level of chlormequat, mepiquat and/or glyphosate present in the test material, in µg/kg and uncorrected for recovery, together with the percentage (%) recovery and limit of quantification (LoQ).

Results were submitted by 61 participants (97%) before the closing date for this test, 25 September 2014.

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 [5].

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 no percentage recovery was reported,

ii) results whose recovery was outside the range 60-140% [6],

iii) results where no limit of quantification was reported.

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

The assigned values for all 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 all analytes, σ_p was derived from the appropriate form of the Horwitz equation [7].

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 all analytes are given in Table 1 and are shown as histograms in Figures 1–3. 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 [5].

6. REFERENCES

1 Adobe Certified Document Services, http://www.adobe.com/misc/pki/cds_cp.html, accessed 14/05/2014.

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

3 FAPAS, 2014, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 4, Issued May 2014.

4 FAPAS, 2014, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS®, Version 3, Issued May 2014.

5 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.

6 Method Validation and Quality Control Procedures for Pesticide Residue Analysis in Food and Feed, Document No. SANCO/12495/2011.

7 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											
	chlormequat assigned value 282 µg/kg				mepiquat assigned value 145 µg/kg				glyphosate assigned value 954 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
001	361	102	10	1.5	162	101	10	0.6	#			
002	#				#				1200	100	50	1.6
003	300	89	10	0.3	137	97	10	-0.2	#			
004	265	89.4	10	-0.3	124	80.9	10	-0.7	#			
005	327	101	0.01	0.8	165	101	0.01	0.7	#			
006	219	93	10	-1.2	143	96	10	-0.1	875	105	20	-0.5
007	270.64	102	10	-0.2	116.47	102	10	-0.9	#			
008	300	104	7	0.3	142	100	7	-0.1	968	109	10	0.1
009	282	100	5	0.0	141	100	5	-0.1	992	100	50	0.2
010	221	108	50	-1.1	100	120	100	-1.4	#			
011	319.00	103	5	0.7	152.00	99	5	0.2	533.00	60	50	**-2.7**
012	370			1.6	130			-0.5	0			**-6.2**
013	410	93	10	**2.4**	230	109	10	**2.8**	#			
014	#				#				1055		10	0.7
015	123		10	**-2.9**	53		10	**-3.0**	1117		10	1.1
016	278	86	10	-0.1	180	101	10	1.1	#			
017	#				#				960	95	10	0.0
018	220	96	20	-1.1	120	88	20	-0.8	1130	88	20	1.1
019	33	95	10	**-4.6**	16	90	10	**-4.2**	2500	95	100	**10.1**
020	#				#				812	84.5	50	-0.9
021	#				#				930	97	50	-0.2
022	#				#				1038	98	30	0.5
023	310		5	0.5	150		5	0.2	885	116	20	-0.5
024	339	87		1.1	171	87		0.9	#			
025	294	94	5	0.2	148	84	5	0.1	1080	100	10	0.8

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte											
	chlormequat assigned value 282 µg/kg				mepiquat assigned value 145 µg/kg				glyphosate assigned value 954 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
026	436	116	10	**2.8**	202	120	10	1.9	1045	77	40	0.6
027	265	100	10	-0.3	190	100	10	1.5	936	45	100	-0.1
028	341	93	10	1.1	150	100	10	0.2	673	97	10	-1.8
029	467	116	10	**3.4**	153	112	10	0.3	#			
030	285		10	0.1	133		10	-0.4	862		100	-0.6
031	319.5	102	20	0.7	144.4	102	20	0.0	904.2	100	50	-0.3
032	280		10	0.0	140		10	-0.1	1000		10	0.3
033	386		0.010	1.9	183		0.010	1.2	1014		0.020	0.4
034	362	86	50	1.5	178	80	50	1.1	#			
035	#				#				810	110	500	-0.9
036	247	88	10	-0.6	151	78	10	0.2	1080	106	50	0.8
037	284		5	0.0	137		5	-0.2	840		10	-0.7
038	250	100	20	-0.6	123	107	20	-0.7	972	85	20	0.1
039	410	50-150	20	**2.4**	230	50-150	20	**2.8**	250	50-150	20	**-4.6**
040	158	100	5	**-2.3**	83	100	5	-2.0	818	100	10	-0.9
041	246	100	0.010	-0.7	178	100	0.010	1.1	#			
042	230.6	121.1	0.01	-0.9	135.4	121.6	0.01	-0.3	#			
043	280	85	5	0.0	110	98	5	-1.1	860	95	10	-0.6
044	139	87	10	**-2.6**	42	98	10	**-3.3**	#			
045	286.0	80.5	10	0.1	145.4	89.7	10	0.0	901.7	84.9	10	-0.3
046	152.83	80	50	**-2.4**	78.75	80	50	**-2.1**	1079.7	80	100	0.8
047	231	108	10	-0.9	131	103	10	-0.4	1081	103	10	0.8
048	286	97	10	0.1	232	94	10	**2.8**	845	87	10	-0.7
049	345.59	93	50	1.2	160.73	91	50	0.5	#			
050	260	86	0.010	-0.4	140	78	0.010	-0.1	970	105	0.050	0.1

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte											
	chlormequat assigned value 282 µg/kg				mepiquat assigned value 145 µg/kg				glyphosate assigned value 954 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
051	220	105	10	-1.1	153	96	10	0.3	805	90	100	-1.0
052	472	89		**3.5**	264	78		**3.9**	#			
053	370		10	1.6	180		10	1.1	930		50	-0.2
054	480	98	10	**3.6**	240	96	10	**3.1**	#			
055	314.33	89.30	10	0.6	117.43	107	10	-0.9	919.40	89.4	10	-0.2
056	440	75		**2.9**	436	93		**9.4**	673	94		-1.8
057	369	91	10	1.6	187	92	10	1.4	#			
058	#				#				160			**-5.2**
059	284	95	10	0.0	145	95	10	0.0	1042	95	10	0.6
060	170	93	10	-2.0	95	83	10	-1.6	#			
061	333	92	10	0.9	155	105	10	0.3	1008	89	10	0.3

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 2: Participants' Comments

participant number	comments
002	A labelled internal standard was used, added before extraction, so an 'automatic' correction for recovery took place
010	detected as Chloride
011	no blank correction of chlormequat (Blank < 5µg/kg)
031	please note corrected & reported as chlormequat cation & mepiquat cation
039	Only screening semi/qualitative analysis
042	Levels of chlormequat and mepiquat are uncorrected for recovery and are not blank corrected.
059	also found in the blank material: chlormequat 5µg/kg

comments are as submitted by participants

Table 3: Assigned Values and Standard Deviations for Proficiency

analyte	data points, n	assigned value, x_a, µg/kg	uncertainty, u	standard deviation for proficiency, σ_p, µg/kg	
chlormequat	41	282	11.8	Horwitz [7]	54.5
mepiquat	41	145	5.74	Horwitz [7]	31.0
glyphosate	30	954	24.5	Horwitz [7]	154

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

analyte	number of scores where |z| ≤2	total number of scores	% |z| ≤2
chlormequat	41	53	77
mepiquat	43	53	81
glyphosate	37	42	88

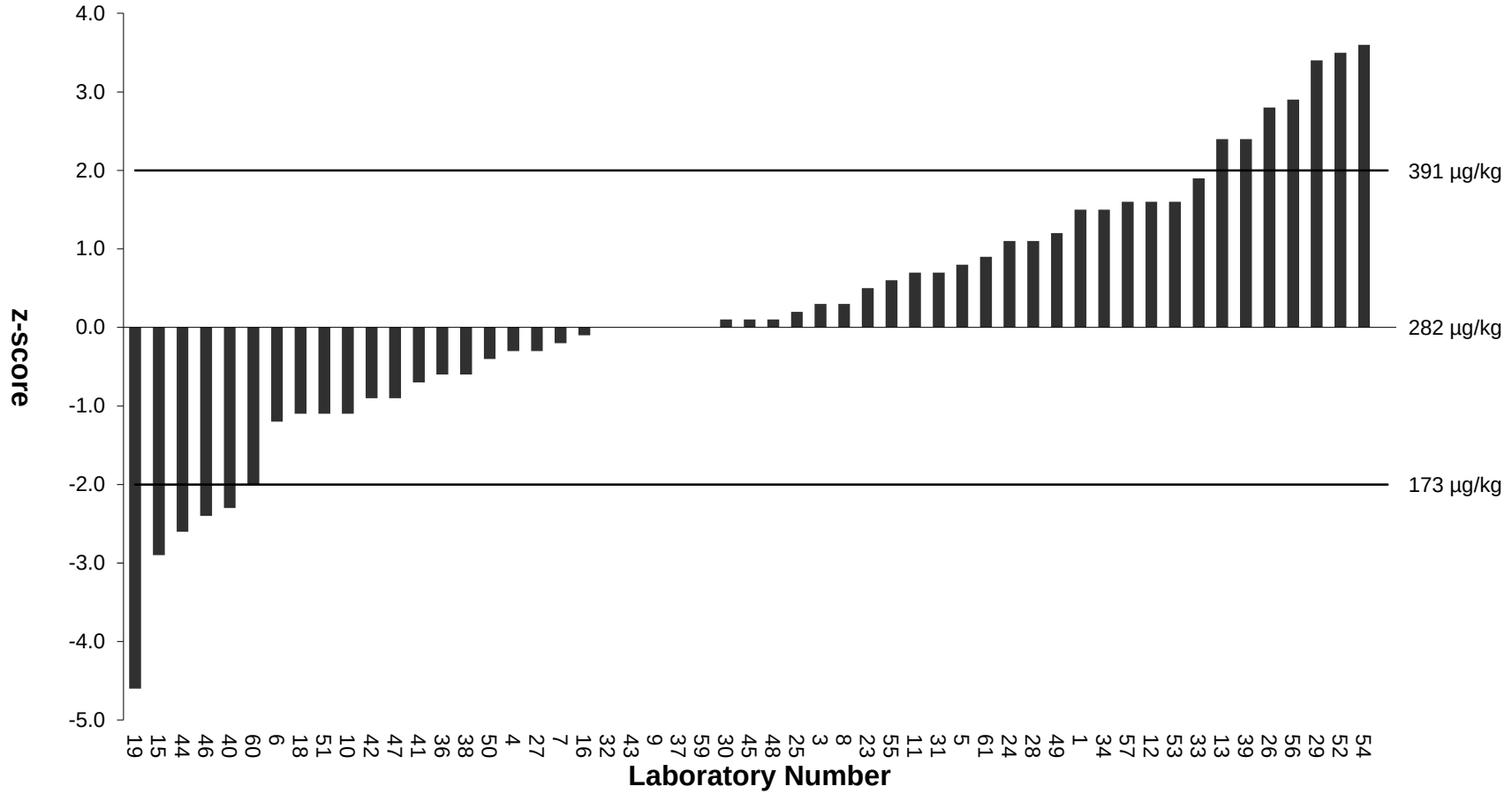

Figure 1: z-Scores for chlormequat

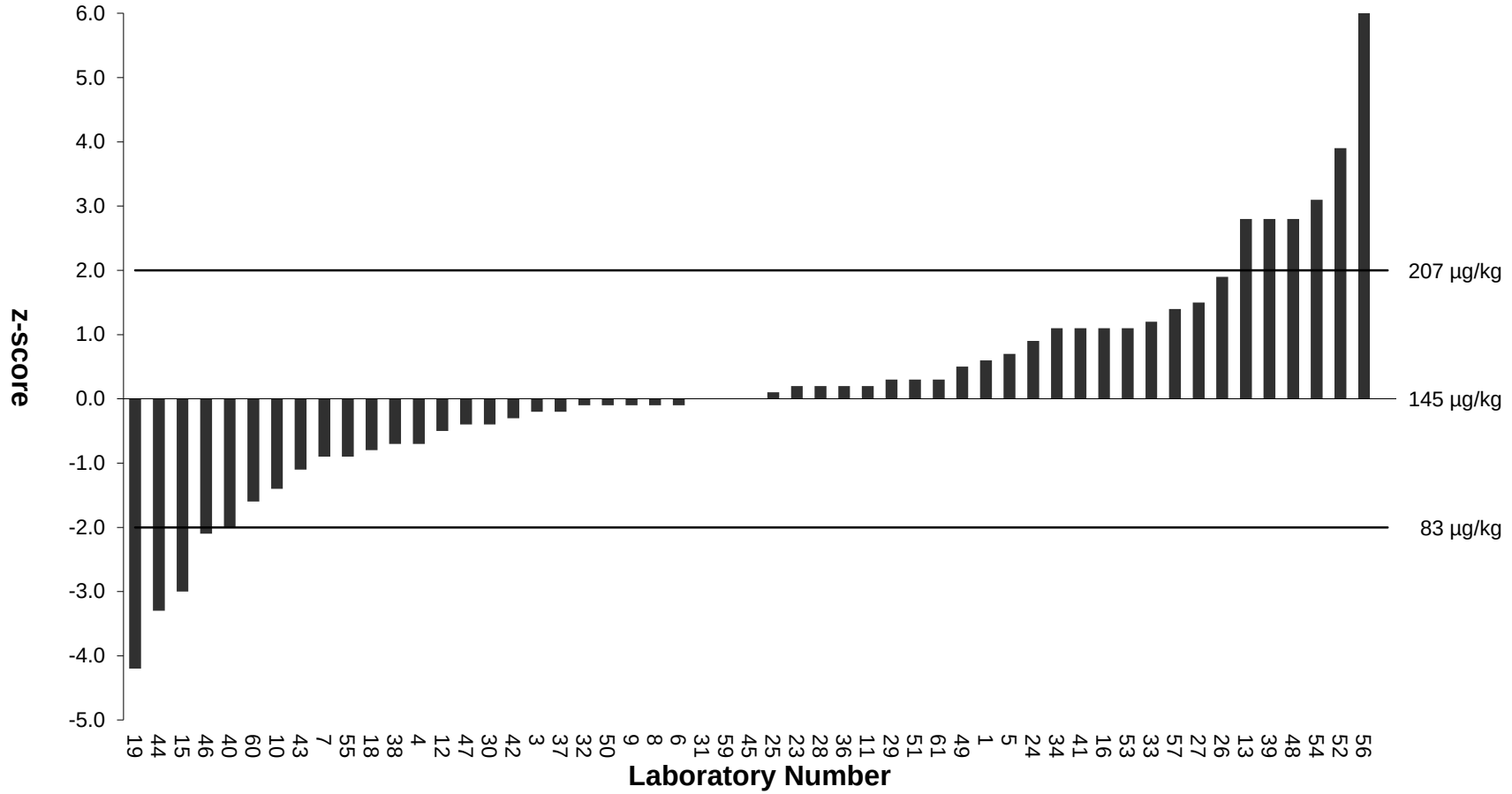

Figure 2: z-Scores for mepiquat

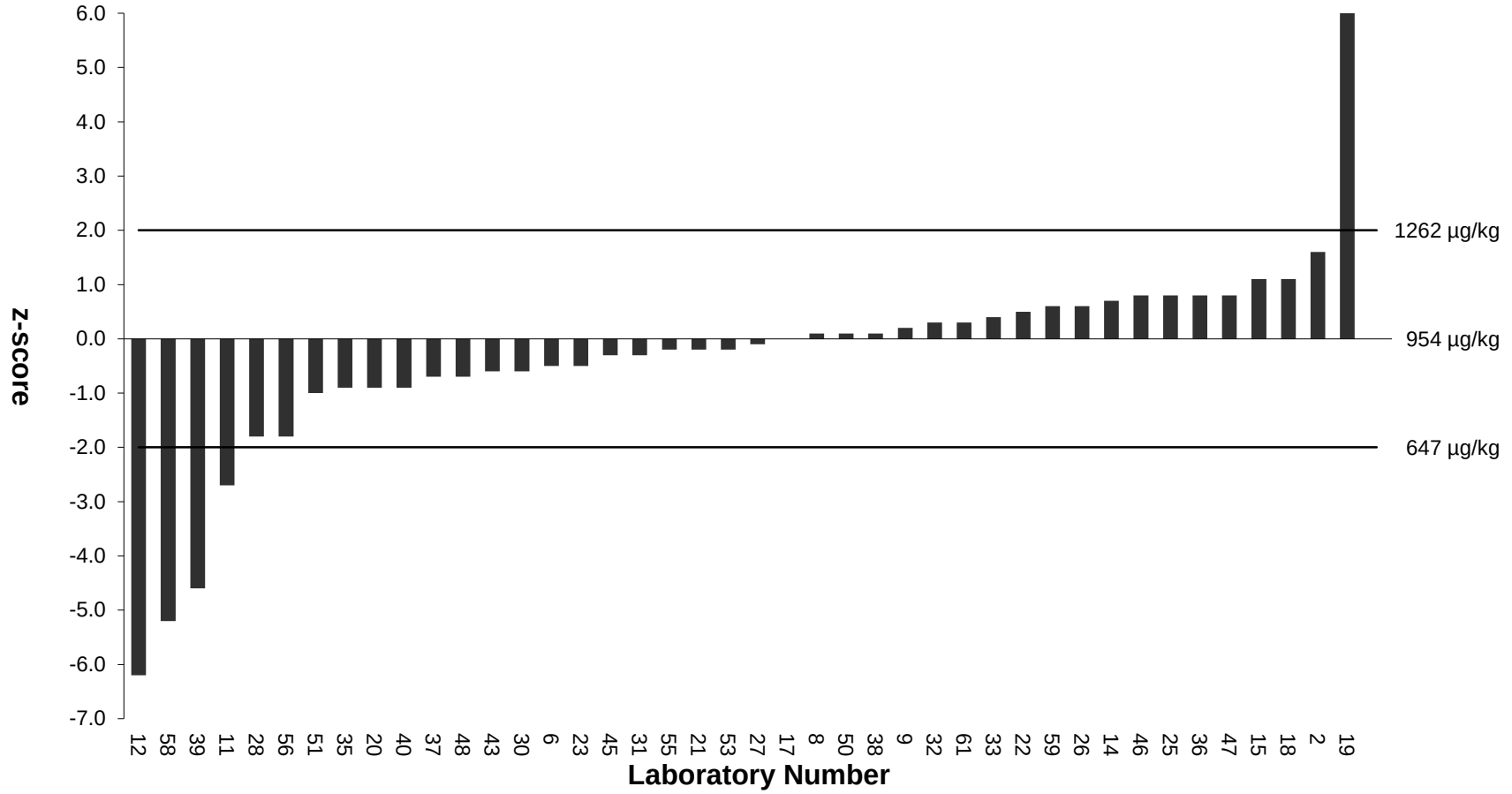

Figure 3: z-Scores for glyphosate

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.

Method Used Accredited	laboratory number
yes	001 004 005 006 010 013 015 016 017 019 023 026 027 028 030 031 032 033 037 039 040 041 044 046 047 051 052 054 055 057 059 061
no	002 011 020 021 022 034 035 036 038 045 048 056 058

Method Based On	laboratory number
International Standard	004 006 011 013 015 016 023 026 028 031 033 037 039 040 045 054 057 058
National Standard	010 030 047 052 055 061
Paper Published In An International Journal	001 020 021 027 035 038 051 056
Manufacturer/Kit Instructions/Technical Note	005
In house method	002 017 019 022 034 036 041 044 046 048 059

Percentage Recovery Measured in Same Analytical Batch as Test Material	laboratory number
yes	001 002 005 006 010 011 013 016 017 019 020 021 022 023 026 027 031 032 035 036 038 039 040 044 045 047 048 051 055 056 057 059
no	004 015 034 041 046 052 054 058 061

Stage the Spike was Added	laboratory number
prior to extraction	001 002 005 006 010 011 013 016 017 019 020 021 022 023 026 027 031 032 035 038 039 040 044 046 047 048 051 052 054 055 056 057 059 061
prior to clean up	045
prior to instrument measurement	036

Concentration of Spike (µg/kg)	laboratory number
≥1 - <5	004
≥10 - <25	016 019 026 045 048 051
≥25 - <50	001 031 036 046 052
≥50 - <100	002 010 011 013 017 027 032 038 040 044 056 057

Concentration of Spike (µg/kg) (continued)	laboratory number
≥100	005 006 015 020 021 022 023 035 039 047 054 055 059 061

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	002 005 011 013 016 017 019 020 021 022 023 026 034 038 039 041 044 045 047 051 052 054 055 057 059
test material provided	006 010 027 032 035 036 056 061
In house Blank	001
organic oats	015

Calibration	laboratory number
standard addition	011 015 033 041 046 052 054 059 061
matrix-matched	002 004 006 013 016 022 026 031 032 034 035 036 038 039 045 047 054 056 058
solvent	005 017 020 023 027 030 037 039 040 057
multi-level	001 005 010 015 017 019 021 023 026 027 028 034 040 044 046 047 048 051 054 055

Internal Standard Added	laboratory number
yes	002 005 006 011 015 016 017 020 023 026 027 030 031 032 033 035 036 037 039 040 044 045 047 051 052 054 055 056 057 058 059
no	001 004 010 013 019 021 022 028 034 038 041 046 048 061

Internal Standard Used	laboratory number
atrazin d5 or dimethoate d6	044
Bromequat	039
Chlormequat-D4	005 006 015 016
Mepiquat-D3	005 006 011 016 030 036 051 054
glyphosate (13C, 15N)	006 011 015 023 030 031 055
Chlormequat Chloride d4	011 027 030 031 036 054 055
Chlormequat D9	051
chlormequat isotope	026
Glyphosate C13	017 020 035
isotope labelled	033 040 047
PCB 198	002

Internal Standard Used (continued)	laboratory number
PCB 31, Sulfotep	052
TPP	052 059
TRIS	057

GC Method: Sample Weight (g)	laboratory number
≥1 - <5	002 010 022 044 061
≥5 - <10	019 028 038
≥10 - <20	015 026 031 052
≥20 - <50	021 057
LC MSMS	054

GC Method: Extraction Solvent Components	laboratory number
acetonitrile	002 010 026 028 051 052 057 061
ethyl acetate	019
methanol	031
H2O	022
LC MSMS	054
Water	021

GC Method: Extraction pH Adjusted	laboratory number
yes	015 023 031 051 056 059 061
no	001 002 004 006 010 016 019 021 022 026 028 030 036 038 041 044 052 054 057

GC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	021 031 038
QuEChERS	002 010 019 026 044 051 052 054 061
liquid-liquid partition	057
Ion exchange	022
shaking	038

GC Method: Sample Clean-up Technique	laboratory number
none	019 028 052 061
solid phase extraction (SPE) (column/cartridge)	021 022 031 038 057
solid phase extraction (SPE) (dispersive)	002 010 026 044 051 054

GC Method: SPE Sorbent Type	laboratory number
PSA	010 026 051 054
Mixed Mode	044
Cation Exchange	021
Florisil	022
Envicarb	010
Resin	038

GC Method: GC Column Packing	laboratory number
50% methyl 50% phenyl polysiloxane	031 044
65% methyl 35% phenyl polysiloxane	022 038
95% methyl 5% phenyl polysiloxane	001 010 019 021 026 051 052 061

GC Method: GC Detector Type	laboratory number
MS	022 026 038 052 054 057
MS-MS	010 019 021 031 044 051 061

LC Method: Sample Weight (g)	laboratory number
≥1 - <5	002 010 015 017 027 032 034 035 037 038 040 041 044 045 047 051 055 057 061
≥5 - <10	001 004 011 013 019 028 030 033 036 039 048 052 054 056 059
≥10 - <20	006 016 022 026 031
≥20 - <50	020
<1	046

LC Method: Extraction Solvent Components	laboratory number
acetonitrile	010 019 022 026 027 040 041 044 046 052 054 057 061
dichloromethane	020 035
methanol	001 004 006 011 013 015 016 027 030 031 032 033 036 037 038 039 040 046 047 048 051 055 056 059
MeOH/Water 1:1 with 1% HAc	002
water	001 017 020 033 034 038 039 040 044 045 047
formic acid	038 039

LC Method: Extraction pH Adjusted	laboratory number
yes	015 017 022 023 039 051 056 059 061
no	001 002 004 005 006 010 011 013 016 019 020 026 027 028 030 031 032 033 034 035 036 037 038 040 041 044 045 046 047 052 054 055 057

LC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	005 006 013 015 026 031 032 033 040 041 047
QuEChERS	001 010 019 022 027 040 044 045 052 054 056 057 059 061
liquid-liquid partition	004 016 020 030 035 039 055
end-over-d shaking for 30 min.	002
homogenization with Ultra-Turrax	011
Methanol Extraction	036
QuPPe method	051
shake at room temperature	038
ultrasound extraction	034
vortexed, incubation derivitization	017

LC Method: Sample Clean-up Technique	laboratory number
liquid-liquid partition	031 035
none	001 002 004 006 010 011 013 015 016 019 026 030 034 036 038 041 048 051 055 056 059 061
solid phase extraction (SPE) (column/cartridge)	005 017 020 027 044 045 047
solid phase extraction (SPE) (dispersive)	022 033 052 054 057

LC Method: SPE Sorbent Type	laboratory number
C18	022 027 046
PSA	052 057
Mixed Mode	005 044 047
anion exchange	017
HLB	020
none	038 054

LC Method: HPLC Column Packing	laboratory number
C18	001 004 006 010 016 020 022 026 027 028 031 032 038 040 045 047 048 054 057 059 061
C8	005 035
Mixed Mode	044
anion exchange	017
C18 und Ionenaustauscher Säule	030
Carbon	056
Dionex AS-11	002
HILIC	034
Hilic (Chlormequat/Mepiquat), Hypercarb (Glyphosat)	033
Hypercarb	011
Hypercarb, normal phase	051
Shodex / Hypercarb	055
sil	041

LC method: Mobile Phase Components	laboratory number
acetic acid	002 011 017 040 051
acetonitrile	001 004 006 013 015 016 017 020 030 033 034 035 038 039 041 046 051 054
ammonium acetate	006 020 030 034 040 045 054
ammonium formate	010 013 016 017 038 047 051
formic acid	022 032 034 038 046 048 051
methanol	002 005 010 011 026 027 031 032 033 040 047 048 051 055 056 059
water	002 004 005 006 010 011 015 017 019 026 027 030 032 033 034 036 037 038 039 040 041 046 047 048 051 052 055 056 061
ammonium bicarbonate	047
Ammoniumhydroxid und Citronensäureanhydrid	030
HFBA	001 005

LC Method: Detector Type	laboratory number
MS-MS	001 002 004 005 006 010 011 013 015 016 017 019 020 022 026 027 028 030 031 032 033 034 035 036 037 038 039 040 041 044 045 046 047 048 051 052 054 055 056 057 058 059 061

Chlormequat

Method Principle	laboratory number
LC	001 004 005 006 010 011 015 016 019 023 026 027 028 030 031 032 033 034 036 037 038 039 040 041 044 046 048 051 052 054 055 056 057 059 061

Identification by Mass Spectrometry	laboratory number
yes	001 004 005 006 010 011 015 016 019 023 026 027 028 030 031 032 033 034 036 037 038 039 040 041 044 046 048 051 052 054 055 056 057 061
no	059

Mepiquat

Method Principle	laboratory number
LC	001 004 005 006 010 011 015 016 019 023 026 027 028 030 031 032 033 034 036 037 038 039 040 041 044 046 048 051 052 054 055 056 057 059 061

Identification by Mass Spectrometry	laboratory number
yes	001 004 005 006 010 011 015 016 019 023 026 027 028 030 031 032 033 034 036 037 038 039 040 041 044 046 048 051 052 054 055 056 057 061
no	059

Glyphosate

Method Principle	laboratory number
GC	021 022 031 038
LC	002 006 011 015 017 019 020 023 026 027 028 030 032 033 035 036 037 039 040 046 048 051 055 056 058 059 061

Identification by Mass Spectrometry	laboratory number
yes	002 006 011 015 017 019 020 021 022 023 026 027 028 030 031 032 033 035 036 037 038 039 040 046 048 051 055 056 058 061
no	059

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.
- View charts of their z-scores obtained in previous FAPAS® proficiency tests.

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 Jennifer Leak (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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