



7th Laboratory Performance Assessment

QS Fachgesellschaft – Obst-Gemüse-Kartoffeln GmbH

in co-operation with

Lach & Bruns, Consultant Chemists

Report

Test Material A

Pesticide Residues in Parsley Purée

September-November 2009

Summary

The Test Materials for the 7th Laboratory Performance Assessment of QS Fachgesellschaft Obst-Gemüse-Kartoffeln GmbH were prepared by the GLP department of LUFA Speyer in summer / autumn 2009 to provide individual Test Material tailored to QS needs. For the second time QS introduced Test Material with incurred pesticides thus the Parsley has been treated with three pesticides in total during the agricultural production.

QS also required four different materials in terms of spiked pesticides. Thus, the four Test Materials differed in the number of spiked pesticides as well as in the levels of spiked pesticides. Test Material "A" was distributed to twenty four (24) participants.

This report refers to the performance assessment of laboratories which analysed Test Material "A". Each laboratory received minimum 100 g Parsley purée with incurred and spiked pesticides. 24 participants (of test material "A") kept the term for the submission of results.

The laboratories were requested to identify and quantify 8 resp. 9 pesticides. These were Biphenyl (spiked), Chlorthal-dimethyl (spiked), Cyromazine (spiked), Difenconazole (incurred), Etridiazole (spiked), Fenitrothion (spiked), Pirimicarb (incurred), and Tolclofomethyl (spiked). The pesticide Metalaxyl (incurred) was present in the Test Material at low levels ($\leq 10 \mu\text{g}/\text{kg}$). The results of Metalaxyl are provided for information only in order to evaluate the laboratories' analytical performances at the low concentration levels and they are not considered for the assessment of the participants performances.

The performance assessment considers the following aspects:

- No *false negative* results are reported (thus identification of all eight pesticides, see also table 6 to 9, p. 11-14)
- No *false positive* results are reported (see also table 2, p. 7)
- Correct quantification (either assessed by application of the z-score model or the 70 -120% recovery criteria – see also explanation next page)

The overall performance assessment results are summarised in the following table:

criteria	number of satisfactory participants	total number participants	satisfactory %
correctly identified all eight pesticides > 20 $\mu\text{g}/\text{kg}$	6	24	25
correctly identified AND reported satisfactory results for all eight pesticides	5	24	21

Assessment of quantification:

The z-scores were determined applying FAPAS[®] method ^{1, 2, 3)}. The assigned values (\hat{X}) for the pesticide concentrations were calculated using Huber's algorithm. The target standard deviation (σ_p) was calculated using the appropriate form of Horwitz equation⁴⁾. Results with $z \leq |2|$ are regarded as satisfactory. The applied robust statistics require a normal distribution of the analytical results. Outliers were identified according to the Grubbs test.

The performance assessment results are summarised in the following tables:

analyte	spiked value µg/kg	assigned value \hat{X} , µg/kg	number of satisfactory z-scores $z \leq 2 $	total number of scores	satisfactory %
Pirimicarb + P-desmethyl	incurred residue	615	22	23	96
Difenoconazole	incurred residue	1678	22	23	96
Tolclofos-methyl	120	107	22	23	96

The applied robust statistics assume that the distribution of the analytical results is roughly normal. However, the distribution of *Biphenyl*, *Chlorthal-dimethyl*, *Cyromazine*, *Etridiazole* and *Fenitrothion* results are atypical and the application of robust statistics gives misleading results. The standard deviations of the corresponding assigned values deviate considerably from the target standard deviations (see table 1, p. 7). Due to the inappropriate statistical evaluation of these pesticides, another type of evaluation of the analytical results was applied. The obligatory method performance data of the *recovery* (70 – 120%)⁶⁾ was used to evaluate the laboratories' performances. Therefore, analytical results that lie between 70 and 120 per cent of the spiked levels are considered satisfactory (see also tables 7 to 9: satisfactory results are marked with "accept").

analyte	spiked level, µg/kg	number of satisfactory results (70-120% of the spiked level)	total number of scores	satisfactory %
Biphenyl	70	12	18	67
Etridiazol	90	6	14	43
Fenitrothion	50	10	19	53
Chlorthal-dimethyl	500	17	24	71
Cyromazine	100	6	14	43

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1. Test Material Preparation and Design

The material for this Performance Assessment was prepared by the GLP department of LUFA Speyer. The homogeneity tests were performed by the CVUA Stuttgart (European reference Laboratory for Residues of Pesticides). The results of homogeneity testing are presented in table 4, p. 8-10.

Difenoconazole, Metalaxyl and Pirimicarb were applied during the agricultural production of the Parsley and therefore are incurred pesticides. The following pesticides were spiked to give the approximate final concentrations: 70 µg/kg Biphenyl, 500 µg/kg Chlorthal-dimethyl, 100 µg/kg Cyromazine, 90 µg/kg Etridiazole, 50 µg/kg Fenitrothion and 120 µg/kg Tolclofos-methyl.

The Parsley purée was distributed into labelled bottles with at least 100 g in each. The bottles were stored at -20 °C in the dark until distribution.

2. Results

The participants were requested to report which pesticides the parsley purée had been analysed for. The results had to be reported without consideration of the recovery. The limit of quantification (LOQ) had to be specified for all pesticides.

Each laboratory was given a number (laboratory code). The results of all participants analysing Test Material A are presented in tables 7 to 9 (p. 11-14).

3. Statistical Evaluation of Results

The statistical procedure applied for this performance assessment is internationally accepted and used in inter-laboratory test series to achieve transparent and fair results. The method used follows recommendations given in the IUPAC/ISO/AOAC International Harmonised Protocol for the Proficiency Testing of Chemical Analytical Laboratories ⁵⁾.

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

The assigned value, \hat{X} , is derived as a robust mean from the results of all participants and presents the best estimate of the true concentration of the analyte.

The influence of outliers is minimised via Huber's method.

The uncertainty (u) of the robust mean is given by

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

$\hat{\sigma}$ = the standard deviation of the robust mean (assigned value \hat{X}),

n = the number of data points used for calculation of \hat{X}

The values for \hat{X} , $\hat{\sigma}$, u and n are presented in table 1.

3.2. Target Standard Deviation for the Performance Assessment, σ_p

The target standard deviation for this laboratory test was derived from the appropriate form of the Horwitz equation. It predicts a standard deviation from a given concentration c , here is c the assigned value \hat{X} .

There are three forms of the Horwitz equation for different analyte concentrations:

- 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%

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

$c = \hat{X}$, expressed as a dimensionless mass ratio, e. g. 1 ppm $\equiv 10^{-6}$ or % $\equiv 10^{-2}$
 $mr =$ expressed as a dimensionless mass ratio, e. g. 1 ppm $\equiv 10^{-6}$ or % $\equiv 10^{-2}$

3.3. Calculation of the Individual z-Scores

The z-scores for each participant and each analyte were calculated as:

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

where x = the reported result

\hat{X} = the assigned value

σ_p = the target value of the standard deviation

If a participant did not identify one or more residues, these results were set equal to zero and the z-scores were calculated accordingly.

4. References

- 1) FAPAS[®], 2002, *Protocol for the Food Analysis Performance Assessment Scheme Organisation and Analysis of Data*, 6th edition.
- 2) Lowthian, P.J. and Thompson, M., 2002, Bump-hunting for the proficiency tester-searching for multimodality, *Analyst*, **127**, 1359-1364.
- 3) Analytical Methods Committee, 1989, Robust statistics – How not to reject outliers Part 1. Basic concepts, *Analyst*, **114**, 1693-1697.
- 4) 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.
- 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 residues analysis in food and feed*, Document No. SANCO/2007/3131, 31/October/2007 (Supersedes Document No. SANCO/10232/2006)

Assigned Values and Target Standard Deviations						
analyte	assigned value				target standard deviation	
	data points, <i>n</i>	robust mean, <i>X</i> , $\mu\text{g/kg}$	uncertainty, <i>u</i>	robust standard deviation, σ	derived from	σ_p , $\mu\text{g/kg}$
Biphenyl	18	59	4,01	17	Horwitz	13
Chlorthal-dimethyl	23	448	19,60	94	Horwitz	81
Cyromazine	14	101	8,82	33	Horwitz	22
Difenoconazole	23	1678	43,58	209	Horwitz	248
Etridiazole	14	84	7,75	29	Horwitz	18
Fenitrothion	19	49	3,44	15	Horwitz	11
<i>Metalaxyl*</i>	<i>No statistics applied</i>					
<i>Pirimicarb + P-desmethyl</i>	23	615	22,10	106	Horwitz	106
<i>Tolclofos-methyl</i>	23	107	4,38	21	Horwitz	24

* italics indicate for information only

Table 1: Assigned Values and Target Standard Deviations

laboratory code	pesticide residue	result ($\mu\text{g/kg}$)	recovery (%)	LoQ ($\mu\text{g/kg}$)
55	Mepanipyrim	170	105	10
61	Mepanipyrim	102	79,4	10
75	Ethoxyquin	250		
	Chinamethoate	420		50

Table 2: Additional Pesticide Residues Reported > 10 $\mu\text{g/kg}$

criteria	number of satisfactory participants	total number participants	satisfactory %
correctly identified all eight pesticides	6	24	25
correctly identified AND reported satisfactory results for all eight pesticides	5	24	21

Table 3: Number and Percentage of Participants Correctly Identifying and Reporting Satisfactory Results for all assessed Pesticides

test material	Biphenyl		MW	Wiederfindung %
	1.Aufarbeitung	2. Aufarbeitung		
PT A Nr.: 1	0,059	0,071	0,065	101
PT A Nr.: 6	0,062	0,063	0,063	
PT A Nr.: 11	0,067	0,060	0,064	Standardabweichung
PT A Nr.: 16	0,049	0,057	0,053	0,0064
PT A Nr.: 21	0,052	0,055	0,054	
PT A Nr.: 26	0,061	0,083	0,072	Variation in %
PT A Nr.: 31	0,058	0,052	0,055	10,8
PT A Nr.: 36	0,057	0,055	0,056	
PT A Nr.: 41	0,059	0,054	0,057	
PT A Nr.: 46	0,055	0,052	0,054	
mean	0,059		0,059	

Chlorthal-dimethyl		MW	Wiederfindung %
1.Aufarbeitung	2. Aufarbeitung		
0,47	0,53	0,5	97
0,53	0,51	0,52	
0,52	0,50	0,51	Standardabweichung
0,47	0,50	0,485	0,0172
0,47	0,48	0,475	
0,50	0,48	0,49	Variation in %
0,49	0,46	0,475	3,5
0,47	0,48	0,475	
0,47	0,47	0,47	
0,47	0,48	0,475	
0,488		0,488	

test material	Cyromazine (corrected by recovery)		MW	Wiederfindung(%)
	1.Aufarbeitung	2. Aufarbeitung		
PT A Nr.: 1	0,1089	0,111	0,110	34
PT A Nr.: 6	0,1086	0,1059	0,107	
PT A Nr.: 11	0,0985	0,1092	0,104	Standardabweichung
PT A Nr.: 16	0,0982	0,1021	0,1	0,0041
PT A Nr.: 21	0,0967	0,0988	0,098	
PT A Nr.: 26	0,0953	0,1062	0,101	Variation in %
PT A Nr.: 31	0,0961	0,1071	0,102	4
PT A Nr.: 36	0,1003	0,1148	0,108	
PT A Nr.: 41	0,1080	0,1098	0,109	
PT A Nr.: 46	0,1047	0,1071	0,106	
mean	0,104		0,104	

Difenconazole		MW	Wiederfindung %
1.Aufarbeitung	2. Aufarbeitung		
1,63	1,72	1,675	100
1,55	1,64	1,595	
1,58	1,74	1,66	Standardabweichung
1,59	1,74	1,665	0,0396
1,57	1,75	1,66	
1,54	1,7	1,62	Variation in %
1,53	1,67	1,6	2,4
1,49	1,67	1,58	
1,56	1,61	1,585	
1,55	1,6	1,575	
1,622		1,622	

Table 4(1): Homogeneity Data for Parsley Homogenate Sample (CVUA Stuttgart)

test material	Etridiazol		MW	Wiederfindung %
	1.Aufarbeitung	2. Aufarbeitung		
PT A Nr.: 1	0,11	0,11	0,111	88
PT A Nr.: 6	0,11	0,093	0,102	
PT A Nr.: 11	0,11	0,086	0,098	Standardabweichung
PT A Nr.: 16	0,1	0,09	0,095	0,0085
PT A Nr.: 21	0,1	0,092	0,096	
PT A Nr.: 26	0,11	0,11	0,112	Variation in %
PT A Nr.: 31	0,11	0,078	0,094	8,8
PT A Nr.: 36	0,1	0,084	0,092	
PT A Nr.: 41	0,095	0,077	0,086	
PT A Nr.: 46	0,1	0,079	0,09	
mean	0,098		0,098	

Fenitrothion		MW	Wiederfindung %
1.Aufarbeitung	2. Aufarbeitung		
0,054	0,054	0,054	94
0,057	0,054	0,056	
0,056	0,052	0,054	Standardabweichung
0,05	0,051	0,051	0,0026
0,049	0,05	0,05	
0,05	0,049	0,05	Variation in %
0,051	0,048	0,05	5
0,049	0,049	0,049	
0,049	0,048	0,049	
0,049	0,049	0,049	
0,451		0,051	

test material	Pirimicarb		MW	Wiederfindung %
	1.Aufarbeitung	2. Aufarbeitung		
PT A Nr.: 1	0,61	0,69	0,650	91
PT A Nr.: 6	0,64	0,63	0,635	
PT A Nr.: 11	0,65	0,62	0,635	Standardabweichung
PT A Nr.: 16	0,59	0,63	0,610	0,0171
PT A Nr.: 21	0,59	0,62	0,605	
PT A Nr.: 26	0,64	0,63	0,635	Variation in %
PT A Nr.: 31	0,63	0,61	0,620	2,8
PT A Nr.: 36	0,59	0,62	0,605	
PT A Nr.: 41	0,61	0,59	0,600	
PT A Nr.: 46	0,61	0,61	0,610	
mean	0,621		0,621	

Pirimicarb-desmethyl		MW	Wiederfindung %
1.Aufarbeitung	2. Aufarbeitung		
0,046	0,051	0,049	n.d.
0,049	0,049	0,049	
0,053	0,053	0,053	Standardabweichung
0,045	0,050	0,048	0,02
0,045	0,051	0,048	
0,049	0,050	0,050	Variation in %
0,050	0,053	0,052	3,9
0,050	0,054	0,052	
0,046	0,055	0,051	
0,048	0,057	0,053	
0,050		0,050	

Table 4(2): Homogeneity Data for Parsley Homogenate Sample (CVUA Stuttgart)

test material	Tolclofos		MW	Wiederfindung %
	1.Aufarbeitung	2. Aufarbeitung		
PT A Nr.: 1	0,115	0,108	0,112	103
PT A Nr.: 6	0,107	0,106	0,107	
PT A Nr.: 11	0,113	0,108	0,111	Standardabweichung
PT A Nr.: 16	0,105	0,108	0,107	0,0032
PT A Nr.: 21	0,102	0,104	0,103	
PT A Nr.: 26	0,103	0,112	0,108	Variation in %
PT A Nr.: 31	0,102	0,105	0,104	3
PT A Nr.: 36	0,103	0,107	0,105	
PT A Nr.: 41	0,103	0,103	0,103	
PT A Nr.: 46	0,0992	0,105	0,102	
mean	0,106		0,106	

Table 4(3): Homogeneity Data for Parsley Homogenate Sample (CVUA Stuttgart)

laboratory code	Metalaxyl		
	result (µg/kg)	recovery (%)	LoQ (µg/kg)
1	8	92	
9	7	95	10
13	11	102	
14	8,6	97	
18	9	100	5
19	7,5		
27	16	95	
36	<10	89,1	10
37	7,51	83	
53	<10.		
68	10	98,8	

Table 5: Results for Metalaxyl in Parsley Purée Test Material A (for information only)

laboratory code	Pirimicarb + P-desmethyl (assigned value 615 µg/kg)				Difenoconazole (assigned value 1678 µg/kg)			
	result (µg/kg)	recovery (%) Pirimicarb/ P.desmethyl	LoQ (µg/kg)	z-score	result (µg/kg)	recovery (%)	LoQ (µg/kg)	z-score
1	550	89		-0,6	1475	102		-0,8
7	552	79/72	10	-0,6	1446	107	10	-0,9
9	698	95	5	0,8	1550	95	10	-0,5
13	670	117/111		0,5	1700	100		0,1
14	853,4	97/98		2,2	1715	97		0,2
17	504	90	10	-1,1	1628	114	10	-0,2
18	681	100	10	0,6	1430	100	10	-1,0
19	417	91/98		-1,9	1698	113		0,1
27	617	91		0,0	2500	81		3,3
28	641	105	10	0,2	1481	90	10	-0,8
29	490	85/92		-1,2	2160	103		1,9
32	734	100/90	20	1,1	1701	118		0,1
36	19655	78,7	10	Outlier*	1388	60,6	10	-1,2
37	684,48	90/91		0,7	1722,67	85		0,2
40	726	75	10	1,0	1989	90	10	1,3
42	570	97	10	-0,4	1995	88	10	1,3
48	530	90	10	-0,8	1500	80	10	-0,7
53	675	91/102	10	0,6	1778	94	10	0,4
55	493	76/75	10	-1,2	1637	76	10	-0,2
61	569	94,2	10	-0,4	1700	104	10	0,1
65	630			0,1	1920			1,0
68	706,1	102,6/100,9		0,9	1784	95,7		0,4
71	725,8	105/88		1,0	1499,1	93		-0,7
75	420			-1,8	350			Outlier*

* according to Grubbs

Table 6: Results and z-Scores for sum of Pirimicarb / Pirimicarb-desmethyl and Difenoconazole in Parsley Purée Test Material A

laboratory code	Biphenyl assigned value 59 µg/kg / spiked level: 70 µg/kg				Etridiazole assigned value 84 µg/kg / spiked level: 90 µg/kg			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	70-120%	result (µg/kg)	recovery (%)	LoQ (µg/kg)	70-120%
1	n.r.			-	60	110		-
7	62	107	20	accept	84	78	20	accept
9	63	95	10	accept	65	95	10	accept
13	100	106		-	110	79		-
14	70,6	89		accept	n.r.			-
17	39	102	10	-	47	79	10	-
18	66	100	10	accept	78	100	10	accept
19	41,6	125		-	n.r.			-
27	60	90		accept	n.r.			-
28	53	98	10	accept	77	83		accept
29	78	93		accept	101	106		accept
32	80	73		accept	110	110		-
36	20	83,5	10	-	n.r.			-
37	n.r.			-	n.r.			-
40	n.r.			-	n.r.			-
42	71	112	10	accept	55	95	10	-
48	50	90	10	accept	50	90	10	-
53	69	81	10	accept	94	73	10	accept
55	43	83	5	-	129	80	10	-
61	n.r.			-	n.r.			-
65	66			accept	118			-
68	n.r.			-	n.r.			-
71	16,9	81		-	n.r.			-
75	n.r.			-	n.r.			-

Table 7: Results and Assessment for Biphenyl and Etridiazole in Parsley Purée Test Material A

laboratory code	Fenitrothion assigned value 49 µg/kg / spiked level: 50 µg/kg				Chlorthal-dimethyl assigned value 448 µg/kg / spiked level: 500 µg/kg			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	70-120%	result (µg/kg)	recovery (%)	LoQ (µg/kg)	70-120%
1	47	121		accept	620	95		-
7	44	95	10	accept	443	109	10	accept
9	55	95	5	accept	505	95	5	accept
13	67	103		-	650	99		-
14	71,5	89		-	653	90		-
17	40	91	10	accept	363	106	10	accept
18	45	100	10	accept	438	100	10	accept
19	n.r.			-	393	111		accept
27	65	89		-	452	80		accept
28	44	116		accept	416	107		accept
29	43	95		accept	458	89		accept
32	190	110		-	480	95		accept
36	26	108,8	10	-	56	48	10	-
37	49,94	88		accept	458,07	90		accept
40	63	83	10	-	572	85	10	accept
42	70	100	10	-	483	100	10	accept
48	45	90	5	accept	370	90	10	accept
53	40	105		accept	518	87		accept
55	32	106	10	-	378	78	10	accept
61	n.r.			-	230	98	10	-
65	n.r.			-	512			accept
68	n.r.			-	371	102,3		accept
71	29,7	94		-	269,4	90		-
75	n.r.			-	320			-

Table 8: Results and Assessment for Fenitrothion and Chlorthal-dimethyl in Parsley Purée Test Material A

laboratory code	Cyromazine assigned value 101 µg/kg / spiked level: 100 µg/kg				Tolclofos-methyl assigned value 107 µg/kg / spiked level: 120 µg/kg			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	70-120%	result (µg/kg)	recovery (%)	LoQ (µg/kg)	z-score
1	185	90		-	101	100		-0,3
7	n.r.			-	50	83	10	-2,4
9	105	95	10	accept	111	95	5	0,2
13	63	119		-	150	117		1,8
14	n.r.			-	142	90		1,5
17	n.r.			-	85	111	10	-0,9
18	88	100	10	accept	109	100	10	0,1
19	102	22		accept	65,1	95		-1,8
27	36	96		-	130	110		1,0
28	96	46		accept	105	107		-0,1
29	n.r.			-	110	73		0,1
32	n.r.			-	109	81		0,1
36	128	15,7	10	-	67	114,5	10	-1,7
37	n.r.			-	95,34	84		-0,5
40	126	70	10	-	147	85	10	1,7
42	n.r.			-	113	102	10	0,2
48	81	75	5	accept	95	90	5	-0,5
53	102	78		accept	123	89		0,7
55	n.r.			-	123	88	10	0,7
61	n.r.			-	96	105	10	-0,5
65	136			-	129			0,9
68	129	96,3		-	106	91,6		-0,1
71	60	84		-	85,3	74		-0,9
75	n.r.			-	n.r.			-4,5

Table 9: Results and Assessment / z-Scores for Cyromazine and Tolclofos-methyl in Parsley Purée Test Material A

Pirimicarb (sum)

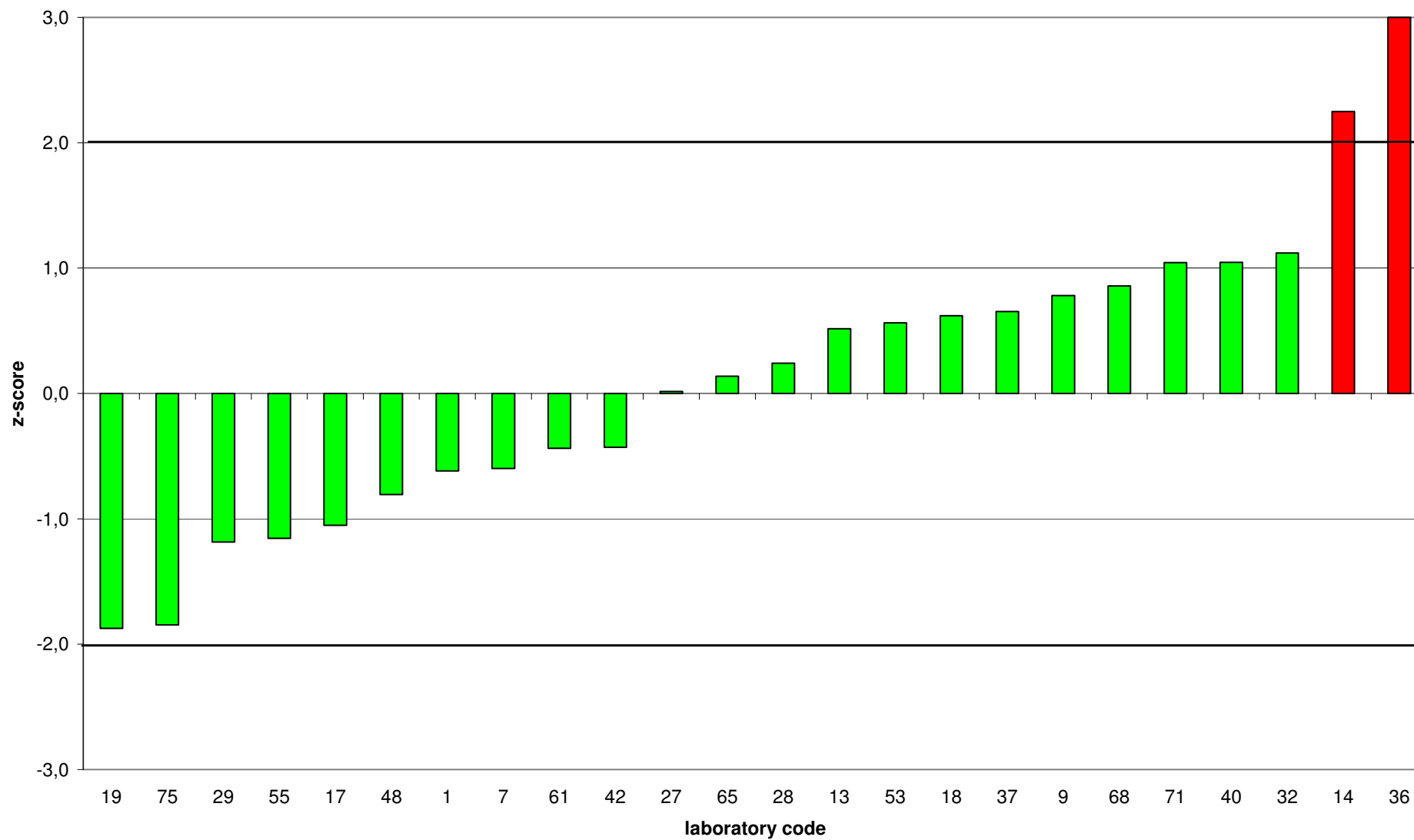


Figure 1: z-Scores for sum of Pirimicarb / Pirimicarb-desmethyl (615 µg/kg) in Parsley Purée Test Material A

Difenoconazole

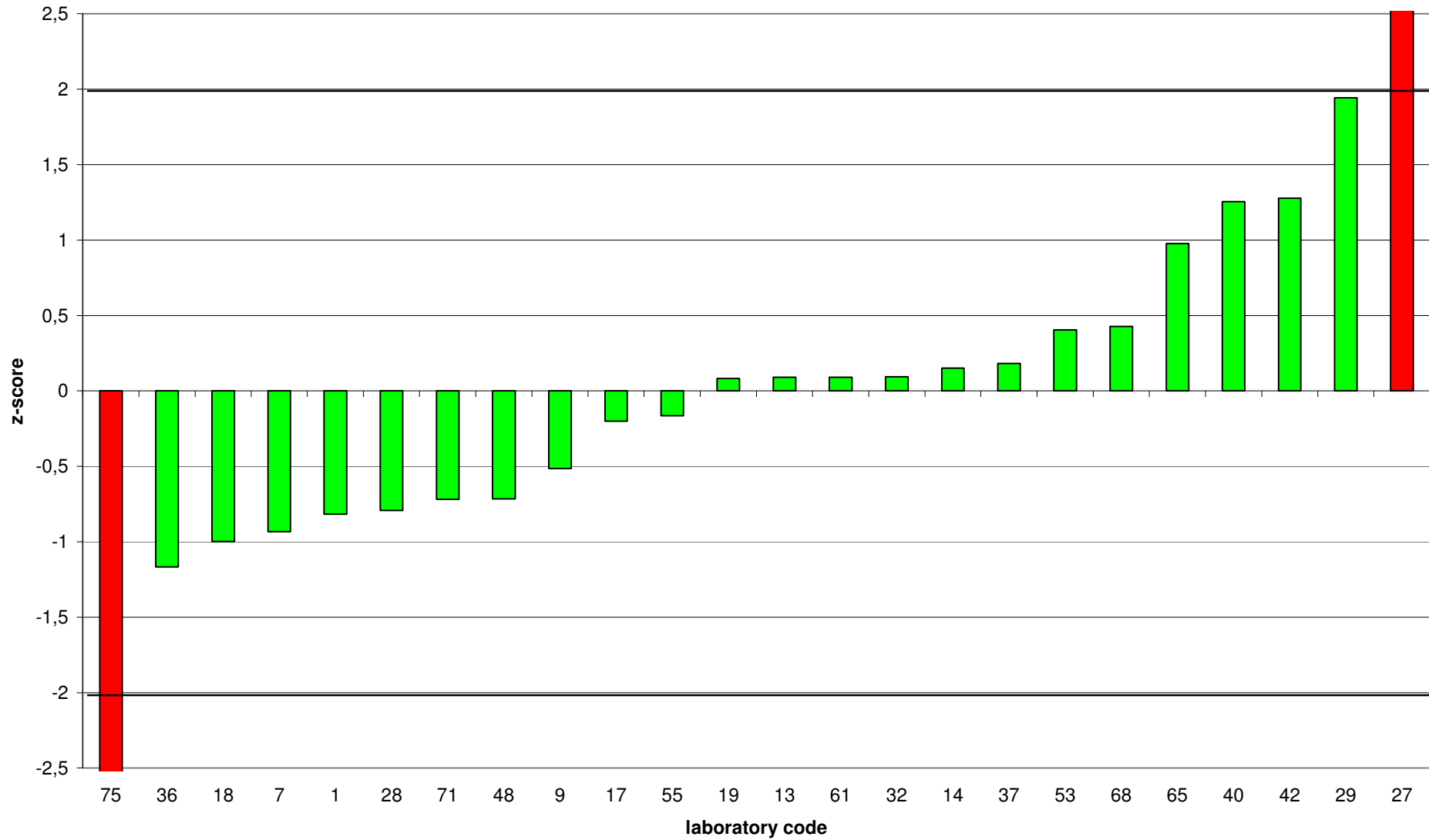


Figure 2: z-Scores for Difenoconazole (1678 µg/kg) in Parsley Purée Test Material A

Biphenyl

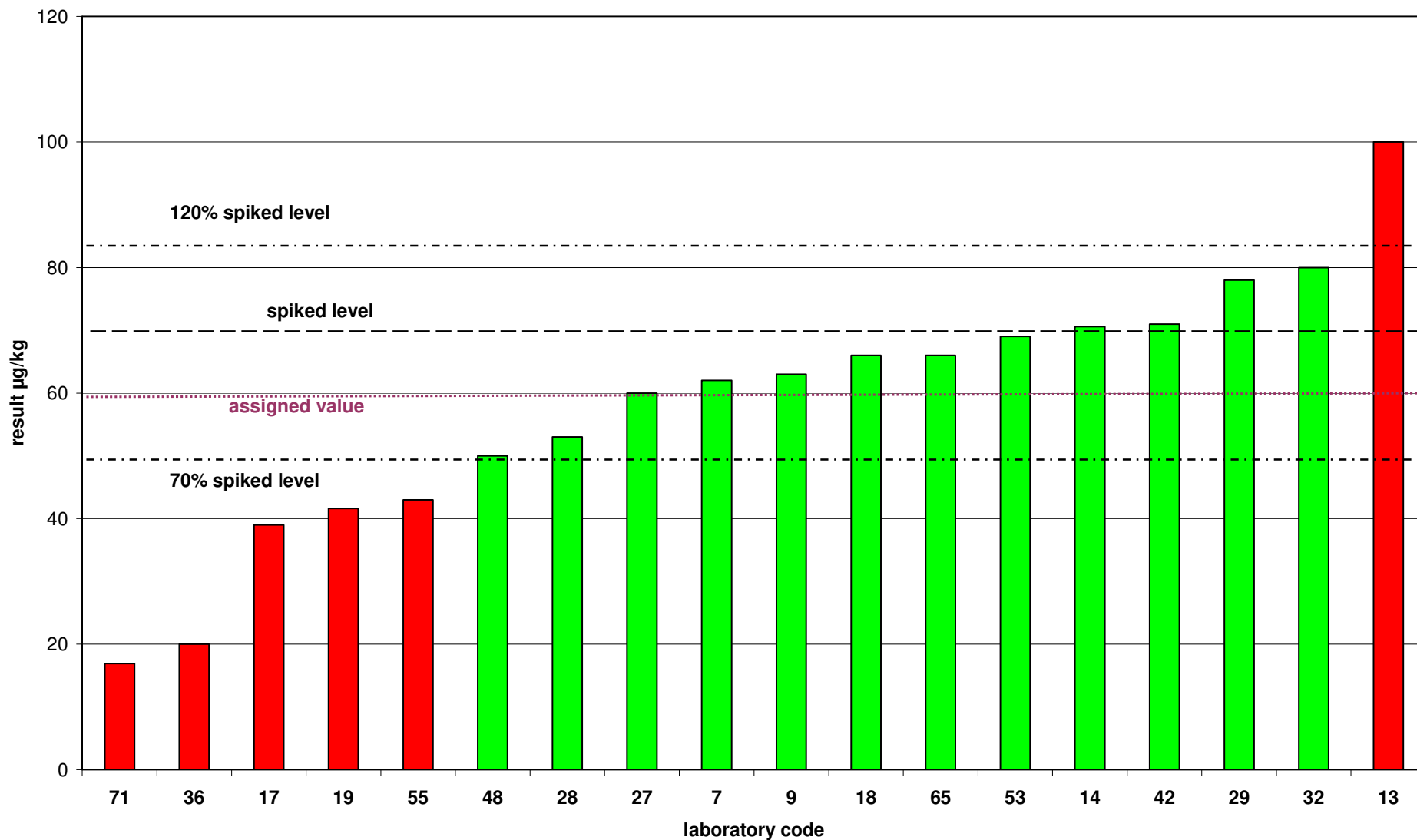


Figure 3: Assessment of Biphenyl (spiked level: 70 µg/kg) in Parsley Purée Test Material A

Etridiazole

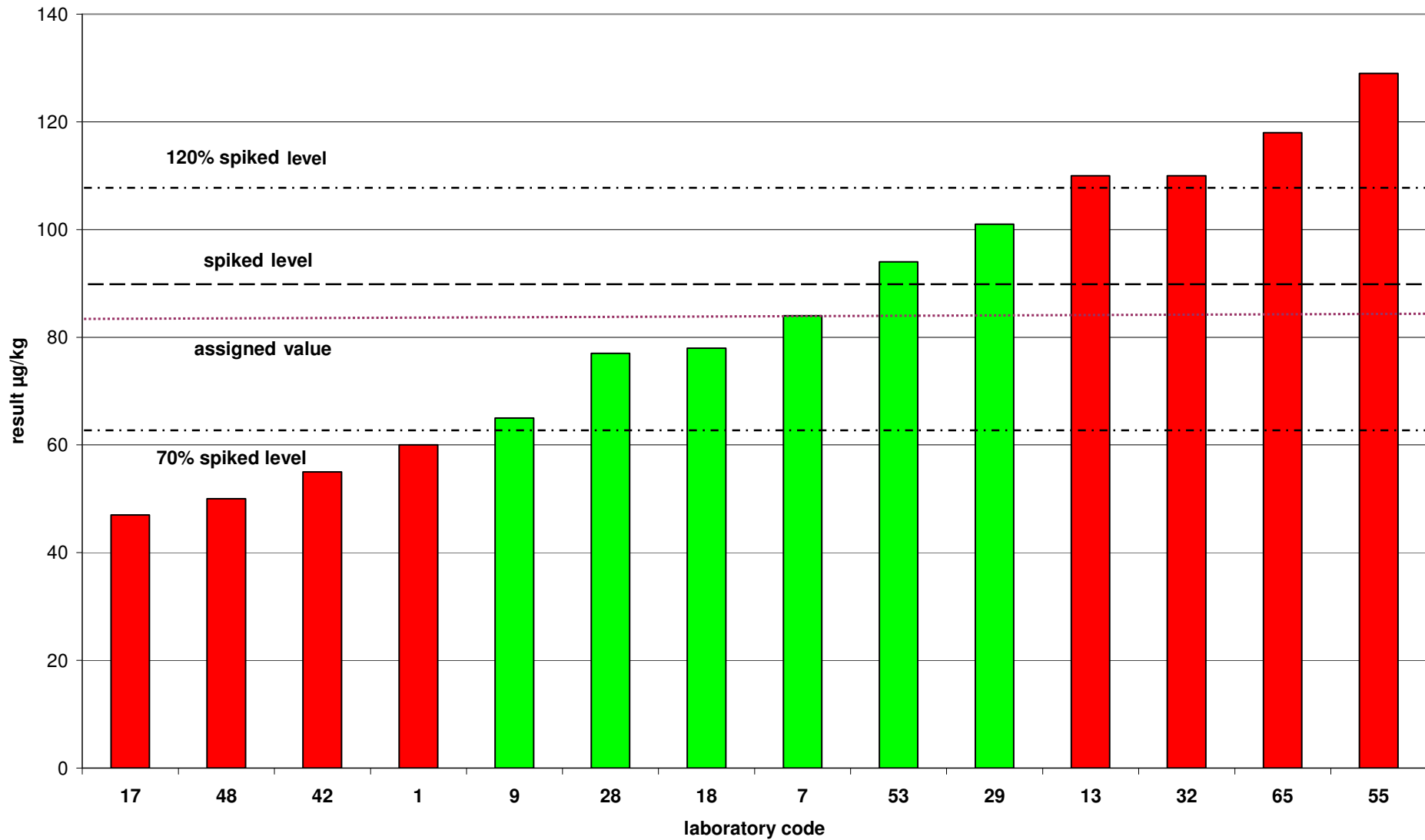


Figure 4: Assessment of Etridiazole (spiked level: 90 µg/kg) in Parsley Purée Test Material A

Fenitrothion



Figure 5: Assessment of Fenitrothion (spiked level: 50 µg/kg) in Parsley Purée Test Material A

Chlorthal-dimethyl

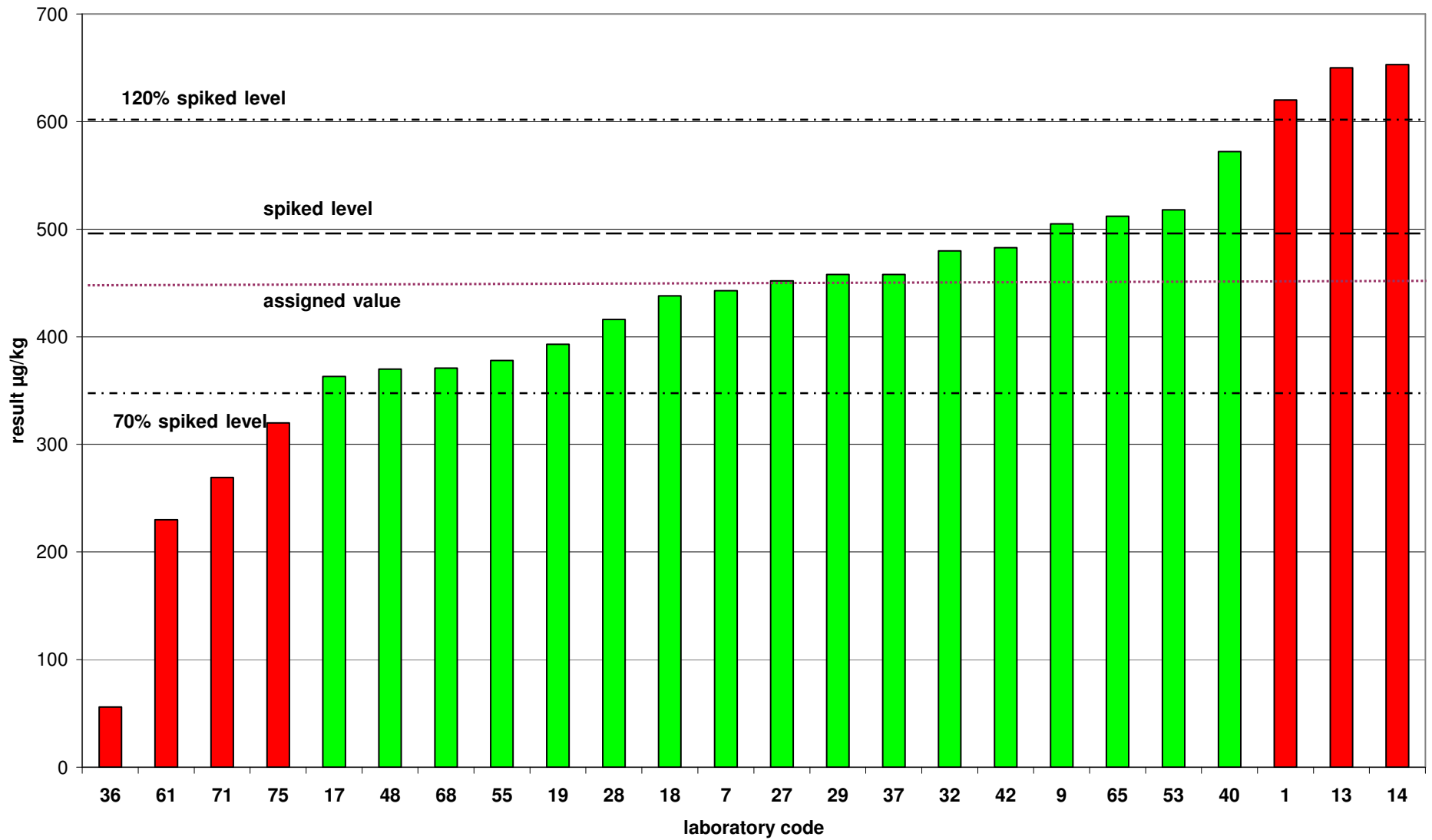


Figure 6: Assessment of Chlorthal-dimethyl (spiked level: 500 µg/kg) in Parsley Purée Test Material A

Cyromazine

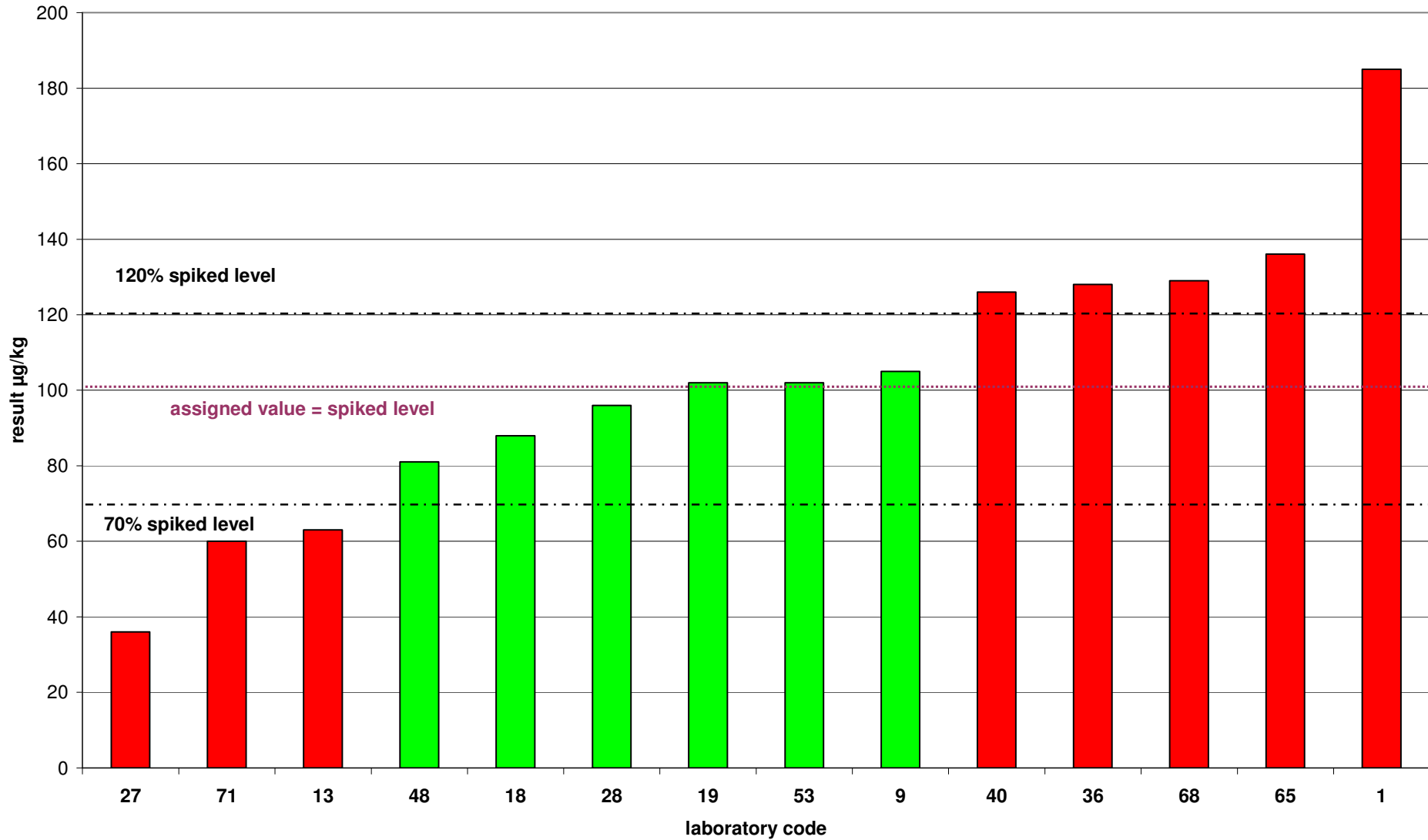


Figure 7: Assessment of Cyromazine (spiked level: 100 µg/kg) in Parsley Purée Test Material A

Tolclofos-methyl

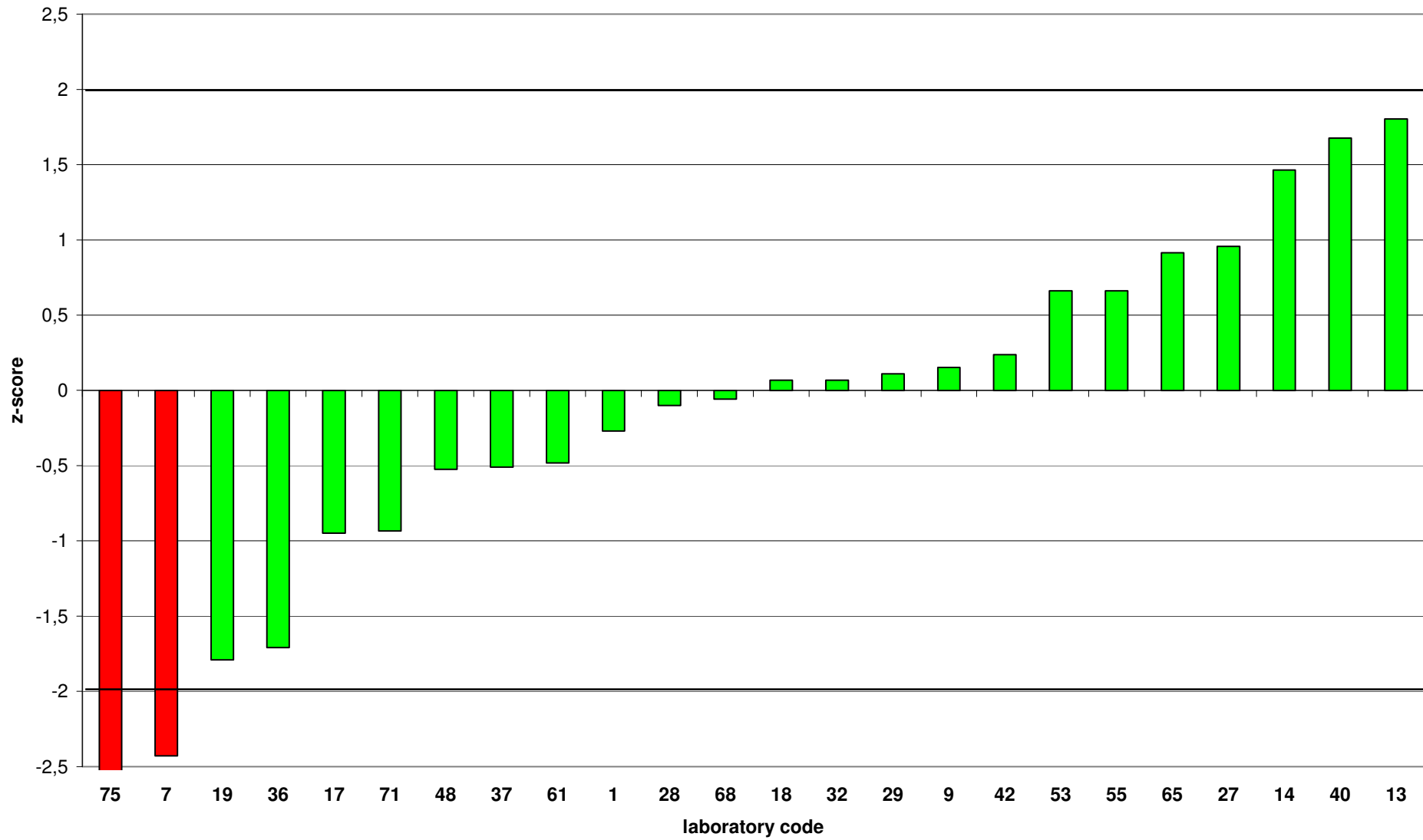


Figure 8: z-Scores for Tolclofos-methyl (107 µg/kg) in Parsley Purée Test Material A