



5th Laboratory Performance Assessment

QS Fachgesellschaft–Obst-Gemüse-Kartoffeln GmbH

in co-operation with

Lach & Bruns, Consultant Chemists

Report

Test Material B

Code 54

Pesticide Residues in Lettuce Purée (Salad)

September 2008

Summary

The Test Materials for the 5th Laboratory Performance Assessment of QS Fachgesellschaft Obst-Gemüse-Kartoffeln GmbH were prepared by the GLP department of LUFA Speyer in summer / autumn 2008 to provide individual Test Material tailored to QS needs. For the first time QS introduced Test Material with incurred pesticides thus the lettuce has been treated with three pesticides in total during the agricultural production.

QS also required two different materials in terms of spiked pesticides. Thus, the two Test Materials - which contained each eight pesticides - differed in the levels of spiked pesticides: Three pesticides were identically spiked to Test Material "A" and "B". The additional two pesticides differed between the two Test Materials related to their spiked levels. Test Material "A" was distributed to thirty nine (39) participants (respectively test material "B" to the other 39 participating laboratories) across eight (8) countries. The analytical results of each kind of Test Material are assessed separately.

This report refers to the performance assessment of laboratories which analysed Test Material "B". Each laboratory received 100 g lettuce purée with incurred and spiked pesticides. 39 participants (of Test Material "B") kept the term for the submission of results.

The laboratories were requested to identify and quantify 8 from a list of 184 possible pesticides. These were Azoxystrobin (incurred), alpha-Cypermethrin (incurred), Dimethoat (spiked), lambda-Cyhalothrin (spiked), Iprodion (spiked) and Propamocarb (spiked). The pesticides Propyzamid (incurred) and Spinosad (spiked) were present in the Test Material at low levels ($\leq 10 \mu\text{g}/\text{kg}$). The results 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 six pesticides, see also table 5 to 10, p. 13-18)
- No *false positive* results are reported (see also table 2, p.9)
- 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 six pesticides > 20 $\mu\text{g}/\text{kg}$	36	39	92
correctly identified AND reported satisfactory results for all six pesticides	15	39	38

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.

The performance assessment results are summarised in the following table:

analyte	spiked value µg/kg	assigned value \hat{X} , µg/kg	number of satisfactory z-scores $z \leq 2 $	total number of scores	satisfactory %
Azoxystrobin	incurred residue	36	37	39	95
alpha- Cypermethrin	incurred residue	76	36	38	95
Dimethoat	25	26	36	36	100
lambda- Cyhalothrin	250	204	36	39	92

The applied robust statistics assume that the distribution of the analytical results is roughly normal. However, the distribution of *Iprodion* and *Propamocarb* 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, p9). Due to the inappropriate statistical evaluation of *Iprodion* and *Propamocarb*, 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 table 9 and 10: satisfactory results are marked with "v").

analyte	spiked level, µg/kg	number of satisfactory results (70-120% of the spiked level)	total number of scores	satisfactory %
Iprodion	6.000	28	39	72
Propamocarb	4.000	25	39	64

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1. Test Material Preparation and Design

The material for this Performance Assessment was supplied 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. 10-12.

Azoxystrobin, alpha-Cypermethrin and Propyzamid were applied during the agricultural production of the lettuce and therefore are incurred pesticides. The corresponding results of the homogeneity testing are presented in table 4, p. 10-12.

The following pesticides were spiked to give the approximate final concentrations: 25 µg/kg Dimethoat, 250 µg/kg lambda-Cyhalothrin, 6.000 µg/kg Iprodion, 4.000 µg/kg Propamocarb and 10 µg/kg Spinosad.

The lettuce 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 lettuce 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 B are presented in tables 5 to 12; P: 13-20.

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.

The z-scores are given in Tables 5 to 8. Iprodion and Propamocarb are assessed separately in Tables 9 and 10. Propyzamid and Spinosad have not been considered for the performance assessment. The results are presented in table 11 and 12 for information only.

Further approaches are presented in table 2 and table 3.

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 , $\mu\text{g/kg}$
Azoxystrobin	39	36	0,80	5	Horwitz	8
Alpha-Cypermethrin	38	76	2,43	15	Horwitz	18
Dimethoat	36	26	0,67	4	Horwitz	5
Lambda-Cyhalothrin	39	204	5,92	37	Horwitz	42
Iprodion	39	4.672	181,75	1.135	Horwitz	593
Propamocarb	39	3.248	156,77	979	Horwitz	435
<i>Propyzamid*</i>	<i>No statistics applied</i>					
<i>Spinosad*</i>	<i>33</i>	<i>11</i>	<i>0,52</i>	<i>3</i>	<i>Horwitz</i>	<i>3</i>

**italics indicate for information only*

Table 1: Assigned Values and Target Standard Deviations

N	O	N	E

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

criteria	number of satisfactory participants	total number participants	satisfactory %
correctly identified all six pesticides	36	39	92
correctly identified AND reported satisfactory results for all six pesticides	16	39	41

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

Homogeneity Data for Lettuce Homogenate Sample (CVUA Stuttgart)

	Azoxystrobin			Dimethoat			Spinosad		
	1.Aufarbeitung	2. Aufarbeitung	MW	1.Aufarbeitung	2. Aufarbeitung	MW	1.Aufarbeitung	2. Aufarbeitung	MW
QS A - 17	0,035	0,035	0,035	0,023	0,020	0,022	0,015	0,011	0,013
QS A - 25	0,032	0,034	0,033	0,024	0,023	0,024	0,010	0,010	0,010
QS A - 39	0,033	0,034	0,034	0,024	0,023	0,023	0,011	0,010	0,010
QS A - 43	0,032	0,031	0,031	0,024	0,024	0,024	0,011	0,010	0,011
QS A - 59	0,035	0,037	0,036	0,026	0,024	0,025	0,012	0,011	0,011
QS A - 61	0,032	0,035	0,033	0,023	0,023	0,023	0,010	0,010	0,010
QS A - 77	0,032	0,034	0,033	0,024	0,024	0,024	0,010	0,010	0,010
QS A - 83	0,034	0,035	0,034	0,021	0,023	0,022	0,011	0,010	0,011
QS A - 95	0,031	0,035	0,033	0,023	0,023	0,023	0,009	0,010	0,010
QS A - 105	0,032	0,033	0,033	0,024	0,022	0,023	0,011	0,010	0,010
QS B - 18	0,031	0,032	0,031	0,024	0,023	0,023	0,011	0,009	0,010
QS B - 26	0,033	0,030	0,032	0,024	0,023	0,024	0,011	0,010	0,010
QS B - 40	0,030	0,031	0,031	0,024	0,024	0,024	0,010	0,010	0,010
QS B - 44	0,031	0,031	0,031	0,024	0,023	0,024	0,011	0,010	0,010
QS B - 60	0,031	0,031	0,031	0,024	0,023	0,024	0,010	0,011	0,010
QS B - 66	0,031	0,032	0,032	0,023	0,023	0,023	0,010	0,011	0,010
QS B - 78	0,030	0,030	0,030	0,023	0,022	0,022	0,010	0,010	0,010
QS B - 84	0,032	0,030	0,031	0,023	0,024	0,023	0,010	0,010	0,010
QS B - 96	0,030	0,034	0,032	0,024	0,022	0,023	0,010	0,010	0,010
QS B - 106	0,030	0,031	0,030	0,024	0,023	0,023	0,010	0,011	0,010
Wiederfindung			0,104			0,1			0,103
Wiederfindung %			104,3			100,0			102,5
Mittelwert	0,032	0,033	0,032	0,024	0,023	0,023	0,011	0,010	0,010
Standard-abweichung	0,0016	0,0021	0,0016	0,0009	0,0008	0,0007	0,0012	0,0005	0,0007
Variation in %	4,9	6,4	5,1	4,0	3,7	3,2	11,0	4,6	6,5
Minimum	0,030	0,030	0,030	0,021	0,020	0,022	0,009	0,009	0,010
Maximum	0,035	0,037	0,036	0,026	0,024	0,025	0,015	0,011	0,013

	Propamocarb				Propyzamid			Lambda-Cyhalothrin		
	1. Aufarbeitung	2. Aufarbeitung	MW		1. Aufarbeitung	2. Aufarbeitung	MW	1. Aufarbeitung	2. Aufarbeitung	MW
QS A - 17	5,30	4,53	4,916		0,0045	0,0042	0,004	0,262	0,252	0,257
QS A - 25	5,20	4,94	5,070		0,0043	0,0039	0,004	0,281	0,289	0,285
QS A - 39	5,00	4,81	4,901		0,0042	0,0038	0,004	0,279	0,284	0,282
QS A - 43	3,93	5,23	4,578		0,0040	0,0035	0,004	0,22	0,269	0,245
QS A - 59	5,45	5,05	5,250		0,0036	0,0039	0,004	0,313	0,278	0,296
QS A - 61	5,05	4,81	4,930		0,0043	0,0040	0,004	0,263	0,288	0,276
QS A - 77	5,15	5,02	5,083		0,0039	0,0042	0,004	0,296	0,322	0,309
QS A - 83	4,38	5,05	4,715		0,0044	0,0040	0,004	0,264	0,287	0,276
QS A - 95	5,05	4,90	4,974		0,0042	0,0038	0,004	0,245	0,238	0,242
QS A - 105	5,04	4,99	5,013		0,0041	0,0040	0,004	0,279	0,292	0,286
QS B - 18	2,81	2,81	2,808		0,0039	0,0039	0,004	0,253	0,264	0,259
QS B - 26	2,83	2,68	2,753		0,0038	0,0034	0,004	0,252	0,262	0,257
QS B - 40	2,76	2,68	2,719		0,0042	0,0039	0,004	0,261	0,264	0,263
QS B - 44	2,89	2,62	2,755		0,0040	0,0036	0,004	0,263	0,265	0,264
QS B - 60	2,93	2,73	2,831		0,0043	0,0040	0,004	0,247	0,254	0,251
QS B - 66	2,68	2,45	2,561		0,0037	0,0035	0,004	0,252	0,255	0,254
QS B - 78	2,67	2,59	2,631		0,0043	0,0038	0,004	0,255	0,25	0,253
QS B - 84	2,61	2,62	2,614		0,0042	0,0037	0,004	0,256	0,263	0,260
QS B - 96	2,71	2,58	2,643		0,0039	0,0042	0,004	0,237	0,252	0,245
QS B - 106	2,69	2,69	2,685		0,0039	0,0037	0,004	0,234	0,261	0,248
Wiederfindung			0,076				0,108			0,105
Wiederfindung %			75,6				108,0			105,0
Mittelwert	4,954	4,932	4,943	Block A	0,004	0,004	0,004	0,261	0,269	0,265
Standard-abweichung	0,4558	0,1873	0,1897		0,0002	0,0002	0,0002	0,0214	0,0195	0,0186
Variation in %	9,2	3,8	3,8		5,9	5,9	4,8	8,2	7,3	7,0
Minimum	3,930	4,533	4,578		0,004	0,003	0,004	0,220	0,238	0,242
Maximum	5,450	5,225	5,250		0,004	0,004	0,004	0,313	0,322	0,309

Mittelwert	2,756	2,644	2,700	Block B
Standard-abweichung	0,1061	0,0974	0,0881	
Variation in %	3,9	3,7	3,3	
Minimum	2,605	2,448	2,561	
Maximum	2,933	2,808	2,831	

	Alphamethrin			Iprodion		
	1. Aufarbeitung	2. Aufarbeitung	MW	1. Aufarbeitung	2. Aufarbeitung	MW
QS A - 17	0,092	0,094	0,093	0,68	0,8	0,800
QS A - 25	0,093	0,096	0,095	1	0,95	0,975
QS A - 39	0,092	0,103	0,098	1	0,9	0,950
QS A - 43	0,08	0,087	0,084	0,99	0,95	0,970
QS A - 59	0,095	0,09	0,093	1,1	1	1,050
QS A - 61	0,088	0,1	0,094	1,03	0,95	0,990
QS A - 77	0,096	0,101	0,099	1,05	0,98	1,015
QS A - 83	0,099	0,093	0,096	0,91	0,95	0,930
QS A - 95	0,082	0,078	0,080	1,07	0,95	1,010
QS A - 105	0,092	0,097	0,095	1,05	0,95	1,000
QS B - 18	0,084	0,085	0,085	5,85	5,8	5,825
QS B - 26	0,085	0,081	0,083	5,95	5,65	5,800
QS B - 40	0,085	0,083	0,084	5,95	5,85	5,900
QS B - 44	0,083	0,085	0,084	6,1	5,8	5,950
QS B - 60	0,079	0,081	0,080	6,25	5,55	5,900
QS B - 66	0,084	0,08	0,082	6,2	5,75	5,975
QS B - 78	0,084	0,079	0,082	6,1	5,65	5,875
QS B - 84	0,084	0,084	0,084	6,1	6	6,050
QS B - 96	0,075	0,084	0,080	6,3	5,6	5,950
QS B - 106	0,073	0,081	0,077	6,1	5,6	5,850
Wiederfindung						
Wiederfindung %						
Mittelwert	0,086	0,088	0,087	0,988	0,938	0,969
Standardabweichung	0,0070	0,0080	0,0070	0,1202	0,0547	0,0685
Variation in %	8,1	9,1	8,0	12,2	5,8	7,1
Minimum	0,073	0,078	0,077	0,680	0,800	0,800
Maximum	0,099	0,103	0,099	1,100	1,000	1,050

Block A

Mittelwert
Standardabweichung
Variation in %
Minimum
Maximum

6,090	5,725	5,908
0,1410	0,1399	0,0755
2,3	2,4	1,3
5,850	5,550	5,800
6,300	6,000	6,050

Block B

Table 4: Results of Homogeneity Tests by CVUA Stuttgart

laboratory code	Azoxystrobin – incurred residue (assigned value 36 µg/kg)			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	z-score
4	34	91	10	-0,2
6	64	81	10	3,6
8	33,8	108	10	-0,2
10	33	113	10	-0,3
12	34	103	5	-0,2
14	33	98	10	-0,3
16	28	84	10	-1,0
24	31	95	10	-0,6
28	35,1	95,5	10	-0,1
30	39	89	10	0,4
32	33,8	89	5	-0,2
34	36	83	10	0,0
38	35	75	10	-0,1
42	36	92	10	0,0
46	36	107,4	10	0,0
48	37	116	10	0,2
50	39	99	10	0,4
54	39	83	5	0,4
56	30	100	10	-0,7
58	30	< 90	10	-0,7
64	42	88	10	0,8
66	210	87	10	22,2
68	40	-	10	0,5
70	47	103	10	1,4
72	35	94	5	-0,1
74	40	93	10	0,5
76	28	116	10	-1,0
80	37,8	100	10	0,3
82	31	95	10	-0,6
86	34	98,6	10	-0,2
88	47	102	10	1,4
90	22	92	10	-1,7
92	35	103	10	-0,1
94	31,7	93	10	-0,5
98	29	95	10	-0,9
100	33	90	10	-0,3
102	42	< 90	10	0,8
104	45	95	5	1,2
108	38	106	10	0,3

Table 5: Results and z-Scores for Azoxystrobin in Lettuce Purée Test Material B

laboratory code	alpha-Cypermethrin – incurred residue (assigned value 76 µg/kg)			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	z-score
4	115	94	10	2,3
6	n.d.	-	10	-4,5
8	69	100	50	-0,4
10	78	115	10	0,1
12	97	88	10	1,2
14	63	89	10	-0,8
16	66	81	30	-0,6
24	57	97	10	-1,1
28	87	105	10	0,6
30	88	90	10	0,7
32	80,6	97	5	0,3
34	65	104	10	-0,7
38	69	72	10	-0,4
42	72	89	10	-0,3
46	72,4	72,3	10	-0,2
48	64	106	10	-0,7
50	78,5	84,8	10	0,1
54	92	110	5	0,9
56	50	50	10	-1,6
58	96	< 90	10	1,2
64	63	91	10	-0,8
66	40	90	10	-2,2
68	90	-	10	0,8
70	99	103	10	1,4
72	74	81	10	-0,1
74	60	98	10	-1,0
76	60,5	106	10	-0,9
80	91,3	100	10	0,9
82	80	91	10	0,2
86	72	109,5	30	-0,3
88	83	96	10	0,4
90	62	91	10	-0,8
92	91	103	10	0,9
94	61,5	88	10	-0,9
98	86	90	5	0,6
100	79	90	10	0,2
102	99,5	< 90	10	1,4
104	79	95	5	0,2
108	72	98	10	-0,3

Table 6: Results and z-Scores for alpha-Cypermethrin in Lettuce Purée Test Material B

laboratory code	Dimethoat – spiked pesticide (assigned value 26 µg/kg; spiked level 25 µg/kg)			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	z-score
4	26	85	10	0,1
6	n.d.	-	10	-4,5
8	27,9	94	10	0,4
10	23	95	5	-0,5
12	25	81	5	-0,1
14	28	95	10	0,4
16	23	124	10	-0,5
24	27	93	10	0,3
28	27	93	10	0,3
30	24	90	10	-0,3
32	29,8	108	10	0,7
34	31	90	10	1,0
38	22	81	10	-0,6
42	26	91	10	0,1
46	< 20	70,2	20* (10)	-4,5
48	n.d.	-	10	-4,5
50	27,9	107	10	0,4
54	23	101	5	-0,5
56	25	100	10	-0,1
58	28	< 90	10	0,4
64	23	85	10	-0,5
66	27	91	10	0,3
68	27	-	10	0,3
70	24	97	10	-0,3
72	29,8	79	5	0,7
74	31	94	10	1,0
76	22	116	10	-0,6
80	26	89	10	0,1
82	30	90	10	0,8
86	21	90,2	10	-0,8
88	23	90	10	-0,5
90	18	89	10	-1,3
92	22	114	10	-0,6
94	22,9	97	10	-0,5
98	20	95	5	-1,0
100	29	90	10	0,6
102	31,3	< 90	10	1,0
104	21	93	10	-0,8
108	28	89	5	0,4

*: LoQ of 10µg/kg were documented in the QS approval

Table 7: Results and z-Scores for Dimethoat in Lettuce Purée Test Material B

laboratory code	lambda-Cyhalothrin – spiked pesticide (assigned value 204 µg/kg; spiked level 250 µg/kg)			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	z-score
4	230	89	10	0,6
6	77	112	10	-3,1
8	190	88	20	-0,3
10	218	101	5	0,3
12	225	106	10	0,5
14	188	95	10	-0,4
16	177	81	10	-0,6
24	199	98	10	-0,1
28	228	99	10	0,6
30	232	91	10	0,7
32	362	78	5	3,8
34	185	85	10	-0,4
38	196	90	10	-0,2
42	210	95	10	0,2
46	186,2	99,9	10	-0,4
48	264	116	10	1,5
50	201,8	144	10	0,0
54	216	110	5	0,3
56	150	100	10	-1,3
58	188	< 90	10	-0,4
64	204	82	10	0,0
66	150	77	10	-1,3
68	250	-	10	1,1
70	242	103	10	0,9
72	203	92	5	0,0
74	150	94	10	-1,3
76	114	89	10	-2,2
80	229,9	95	10	0,6
82	175	94	10	-0,7
86	177	91	10	-0,6
88	211	97	10	0,2
90	146	74	10	-1,4
92	265	110	10	1,5
94	182	93	10	-0,5
98	172	95	10	-0,8
100	243	90	10	1,0
102	191	< 90	10	-0,3
104	269	94	10	1,6
108	257	102	5	1,3

Table 8: Results and z-Scores for lambda-Cyhalothrin in Lettuce Purée Test Material B

laboratory code	Iprodion – spiked pesticide (spiked level 6.000 µg/kg)			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	result within 70-120% of the spiked level
4	4670	95	10	✓
6	5743	71	10	✓
8	5000	93	50	✓
10	4820	86	10	✓
12	3601	93	10	-
14	4727	95	10	✓
16	4331	88	20	✓
24	5300	96	10	✓
28	5910	103	10	✓
30	4826	109	10	✓
32	5790	73	5	✓
34	4644	99	10	✓
38	4421	84	10	✓
42	5033	98	10	✓
46	3818	88,4	10	-
48	6769	105	10	✓
50	5270	97,3	20	✓
54	4300	84	5	✓
56	3400	100	10	-
58	6224	< 90	10	✓
64	3480	77	10	-
66	474	81	10	-
68	4800	-	10	✓
70	3600	98	10	-
72	5600	91	10	✓
74	530	96	10	-
76	2115	120	10	-
80	2892	70-100	10	-
82	4410	96	10	✓
86	5039	104,6	10	✓
88	6081	113	20	✓
90	3100	84	10	-
92	6079	107	10	✓
94	2860	84	10	-
98	4600	95	10	✓
100	6020	100	10	✓
102	5211	< 90	10	✓
104	4830	93	20	✓
108	5550	74	10	✓

Table 9: Results and Assessment for Iprodion in Lettuce Purée Test Material B

laboratory code	Propamocarb – spiked pesticide (spiked level 4.000 µg/kg)			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	result within 70-120% of the spiked level
4	3400	81	10	✓
6	1448	58	10	-
8	3200	100	50	✓
10	3370	95	5	✓
12	3160	105	5	✓
14	3070	102	10	✓
16	3371	118	50	✓
24	2123	90	10	-
28	3330	94,3	10	✓
30	4492	88	20	✓
32	4240	77	20	✓
34	2351	103	10	-
38	4303	80	10	✓
42	4201	97	10	✓
46	3447	69,3	10	✓
48	6864	60	10	-
50	3034	95	10	✓
54	5963	96	5	-
56	2000	75	50	-
58	957	< 90	10	-
64	3700	80	50	✓
66	1100	80	10	-
68	3700	-	10	✓
70	3440	94	10	✓
72	5390	87	5	-
74	4100	87	10	✓
76	846	96	10	-
80	3164	70-100	10	✓
82	3320	90	10	✓
86	3148	90,5	10	✓
88	1500	89	10	-
90	2130	89	10	-
92	2974	100	10	✓
94	2918	83	10	✓
98	2700	90	5	-
100	4490	90	10	✓
102	3192	< 90	10	✓
104	2660	93	10	-
108	4640	100	20	✓

Table 10: Results and Assessment for Propamocarb in Lettuce Purée Test Material B

laboratory code	Spinosad – spiked pesticide (assigned value 11,5 µg/kg; spiked level 10 µg/kg)			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	z-score (for information only)
4	14	78	10	1,0
6	n.d.	-	10	
8	8,4	90	10	-1,2
10	11	118	5	-0,2
12	10	94	5	-0,6
14	8	96	10	-1,4
16	9	108	10	-1,0
24	12	93	10	0,2
28	14,2	82,3	10	1,1
30	10	101	10	-0,6
32	10,4	85	5	-0,4
34	12	87	10	0,2
38	10	90	10	-0,6
42	10	93	10	-0,6
46	< 10	90,7	10	
48	20	74	10	3,4
50	12,8	90	10	0,5
54	< 5	-	5	
56	10	100	10	-0,6
58	9	< 90	5	-1,0
64	13	80	10	0,6
66	15	92	10	1,4
68	12	-	10	0,2
70	10	92	10	-0,6
72	9	108	10	-1,0
74	n.d.	-	10	
76	15	98	10	1,4
80	3,7	76	2	-3,1
82	10	72	10	-0,6
86	8,4	99,9	5	-1,2
88	11	104	10	-0,2
90	< 10	89	10	
92	10	115	10	-0,6
94	9,7	96	10	-0,7
98	7	95	5	-1,8
100	11	100	10	-0,2
102	12,2	< 90	10	0,3
104	10	92	10	-0,6
108	n.d.	-	10	

Table 11: Results for Spinosad in Lettuce Purée Test Material B (for information only)

laboratory code	Propyzamid – incurred residue			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	No z-score evaluation applied
4	ca. 5	84	10	N O z - s c o r e E v a l u a t i o n a p p l i e d
6	n.d.	-	10	
8	4,3	108	10	
10	6	117	5	
12	< 5	90	5	
14	5	85	10	
16	3,3	81	10	
24	n.d.	-	10	
28	n.d.	-	10	
30	< 10	94	10	
32	n.d.	-	5	
34	n.d.	95	10	
38	n.d.	-	10	
42	< 5	88	10	
46	< 10	88,1	10	
48	n.d.	-	10	
50	< 10	115	10	
54	< 5	-	5	
56	n.d.	-	10	
58	8	< 90	5	
64	4	89	10	
66	n.d.	-	50	
68	< 10	-	10	
70	< 10	-	10	
72	4	94	10	
74	n.d.	-	10	
76	n.d.	-	10	
80	3,8	100	2	
82	n.d.	-	10	
86	4,8	95,2	10	
88	n.d.	-	10	
90	n.d.	-	10	
92	n.d.	-	10	
94	3,7	87	10	
98	< 10	-	10	
100	n.d.	-	10	
102	< 10	-	10	
104	5	94	5	
108	6	93	5	

Table 12: Results for Propyzamid in Lettuce Purée Test Material B (for information only)

Azoxystrobin

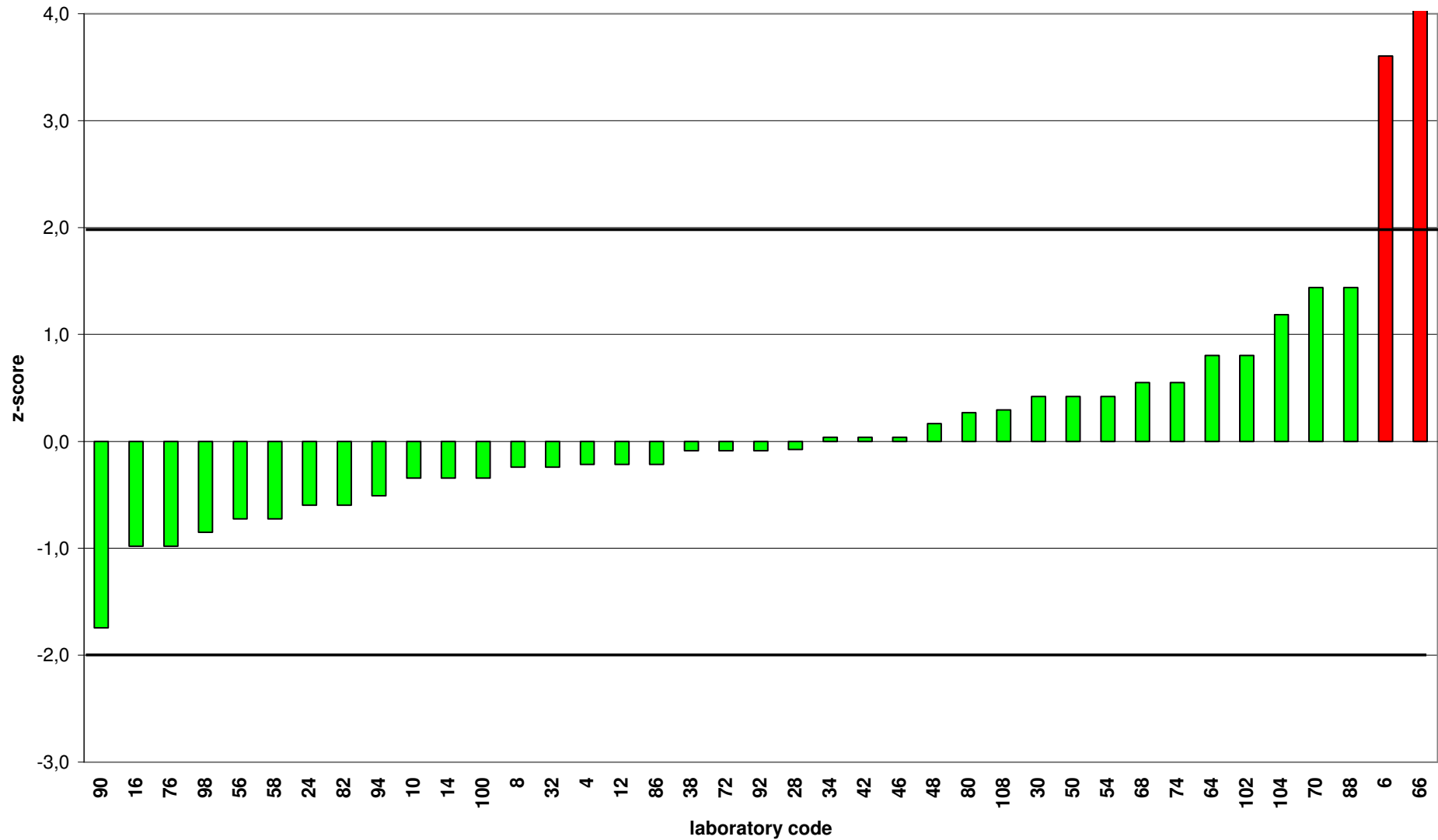


Figure 1: z-Scores for Azoxystrobin (36 µg/kg) in Lettuce Purée Test Material B

alpha-Cypermethrin

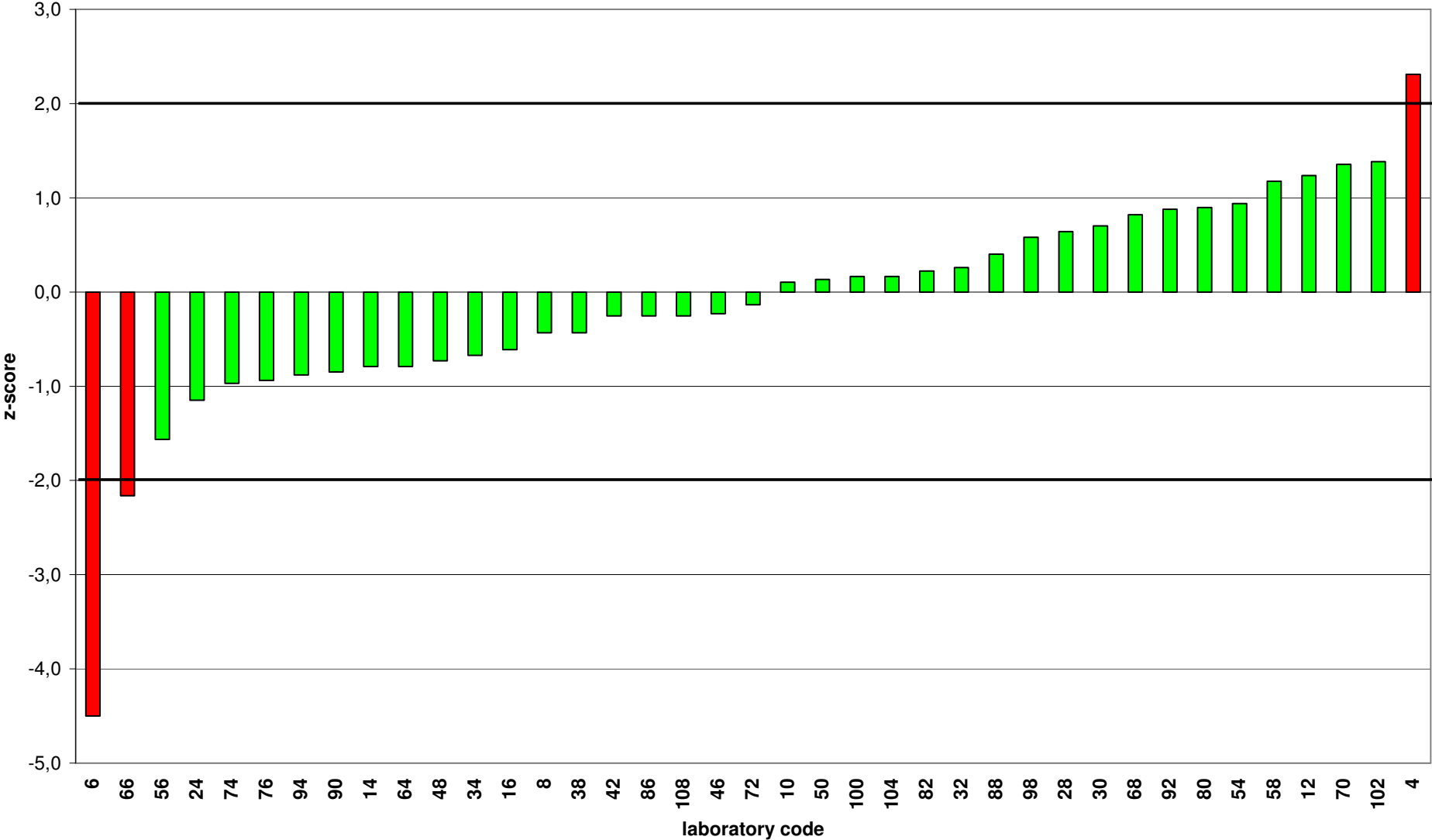


Figure 2: z-Scores for alpha-Cypermethrin (76 µg/kg) in Lettuce Purée Test Material B

Dimethoat

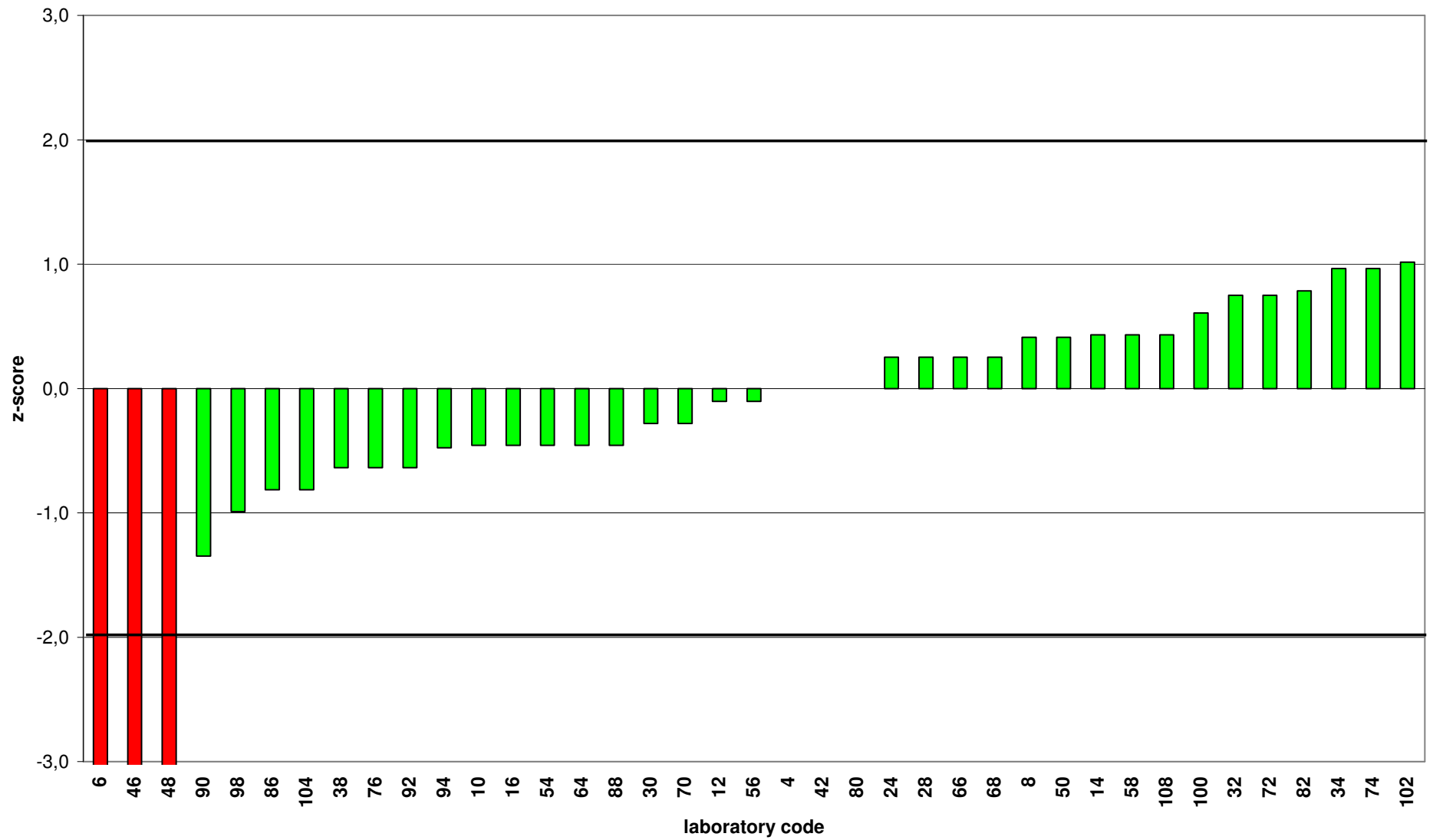


Figure 3: z-Scores for Dimethoat (26 µg/kg) in Lettuce Purée Test Material B

lambda-Cyhalothrin

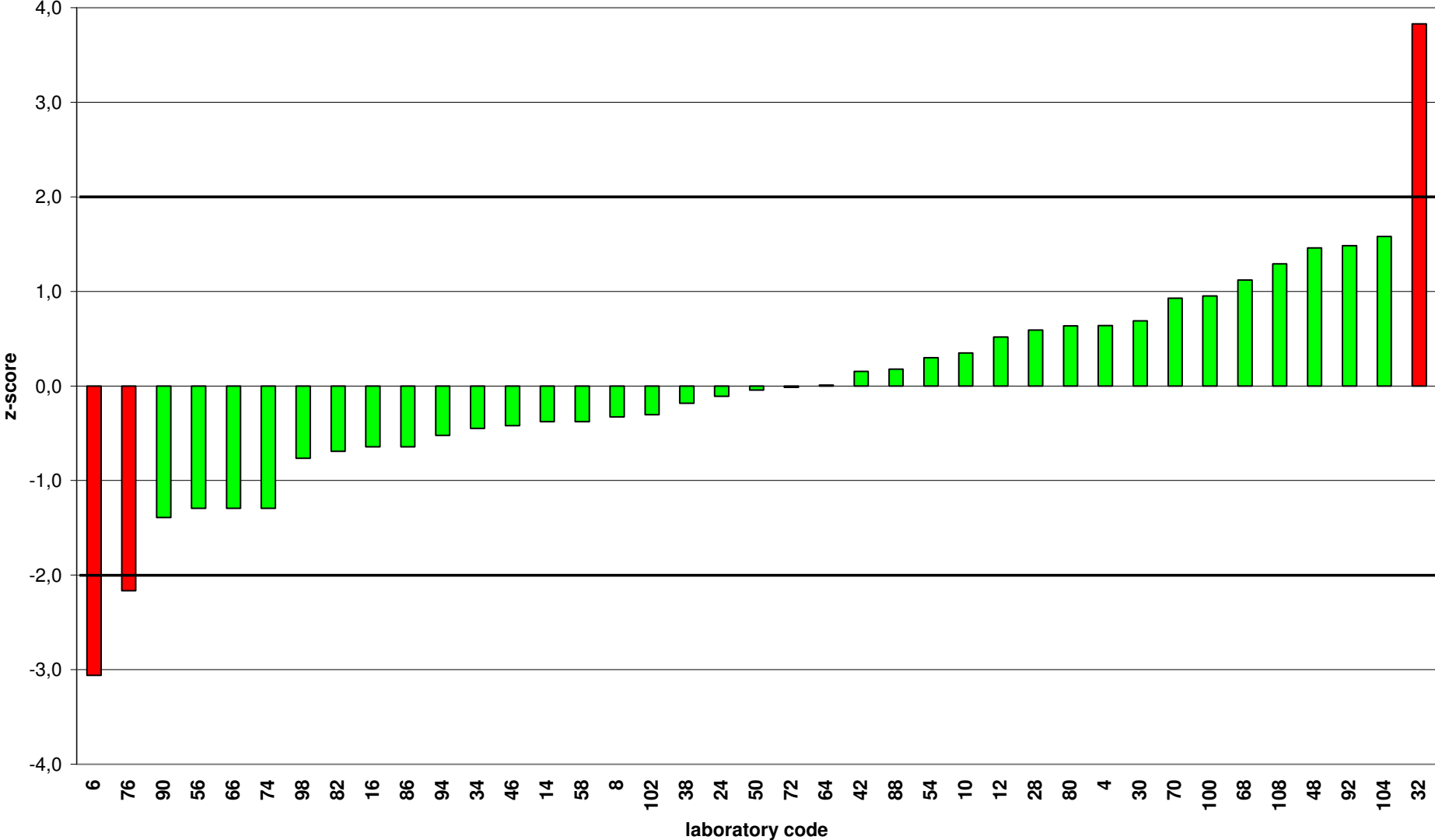


Figure 4: z-Scores for lambda-Cyhalothrin (204 µg/kg) in Lettuce Purée Test Material B

Iprodion

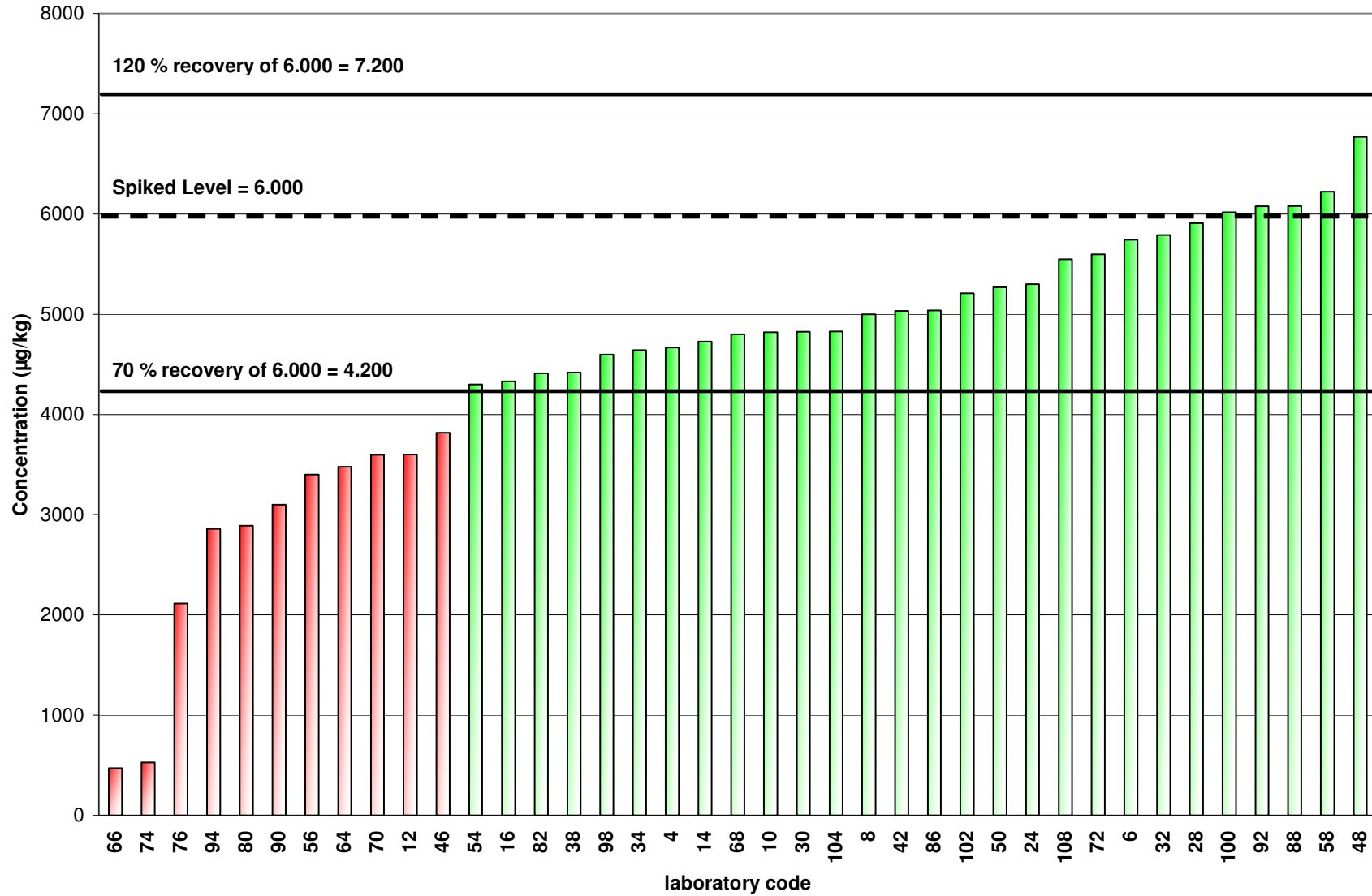


Figure 5: Assessment of Iprodion (6000 µg/kg) in Lettuce Purée Test Material B

Propamocarb

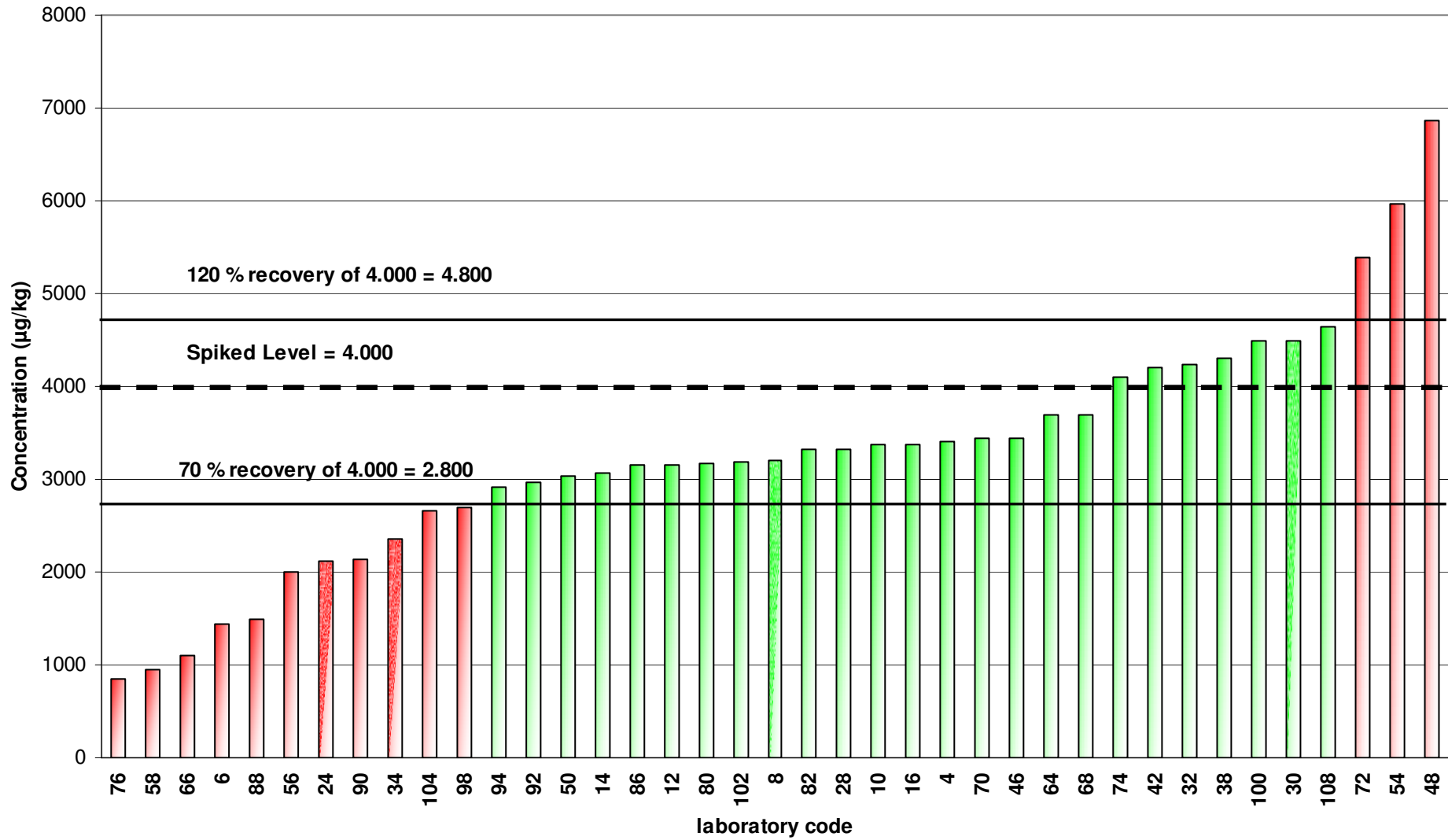


Figure 6: Assessment of Propamocarb ($4000 \mu\text{g}/\text{kg}$) in Lettuce Purée Test Material B

Spinosad

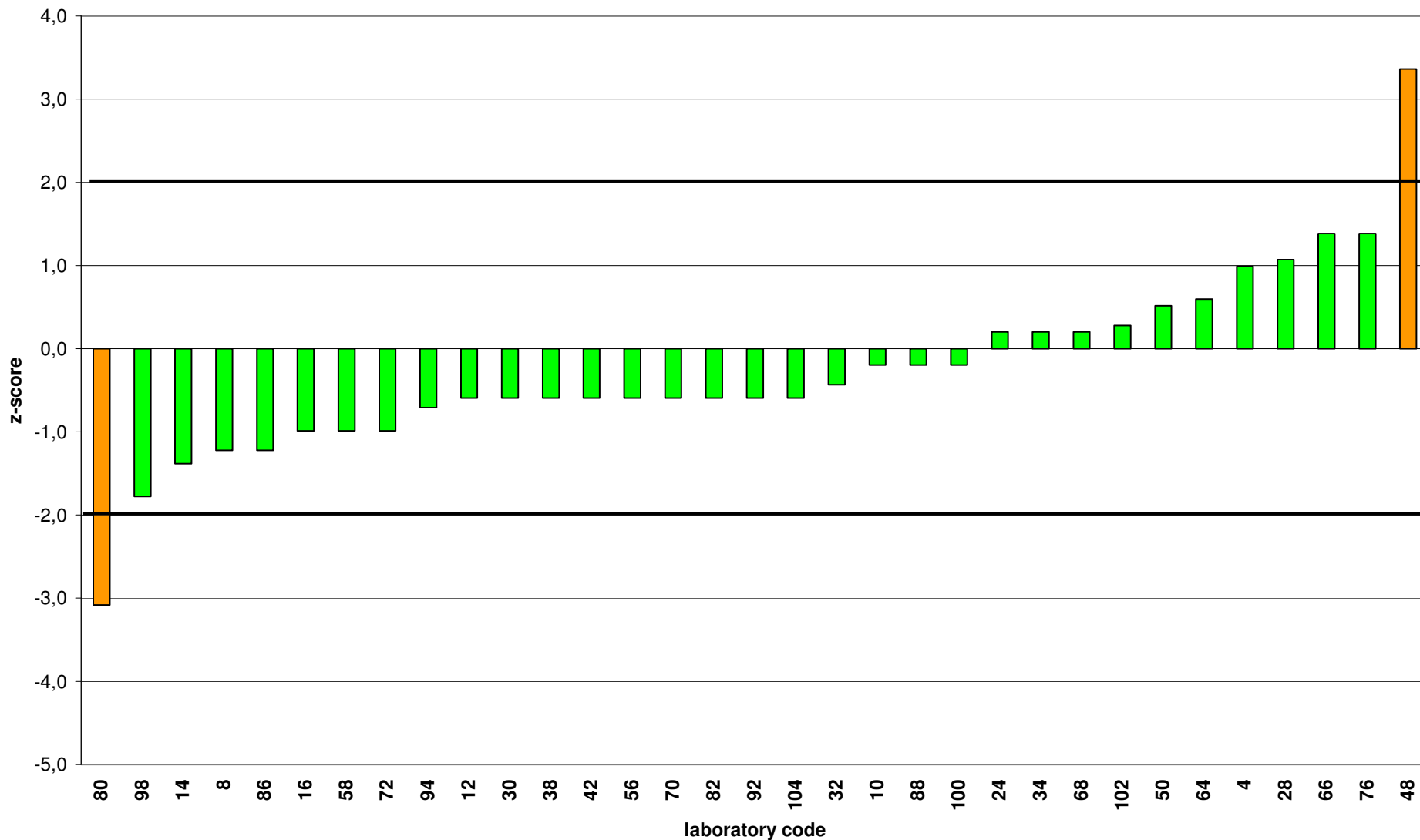


Figure 7: z-Scores for Spinosad (11,5 µg/kg) in Lettuce Purée Test Material B

For information only