



Qualitätssicherung. Vom Erzeuger bis zur Ladentheke.

Teilnahmebestätigung

Das Labor

GALAB Laboratories GmbH
Max-Planck-Straße 1
21502 Geesthacht

hat erfolgreich am **Laborkompetenztest Rückstandsmonitoring Frühjahr 2011** teilgenommen.

Der Laborcode lautet: 92.
Der Laborkompetenztest wurde durchgeführt von der QS Fachgesellschaft Obst-Gemüse-Kartoffeln GmbH.

Bonn, 15. Juni 2011

Dr. Hermann-Josef Nienhoff
Geschäftsführer



QS. Ihr Prüfsystem
für Lebensmittel.



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QS Laborkompetenztest Frühjahr 2011

Bewertungsschema Laborkompetenztest Frühjahr 2011

Bestanden

alle 6 Wirkstoffe identifiziert

und

mindestens 4 Wirkstoffe mit akzeptablem Ergebnis quantifiziert
(70-120% des dotierten Gehaltes)

und

kein falsch positiver Wirkstoff identifiziert

Nicht Bestanden*

nicht alle 6 Wirkstoffe identifiziert

oder

weniger als 4 Wirkstoffe mit akzeptablem Ergebnis quantifiziert
(70-120% des dotierten Gehaltes)

oder

einen oder mehrere falsch positive Wirkstoffe

*keine erfolgreiche Teilnahme, Stellungnahme und Maßnahmenplan sind QS
vorzulegen.



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Laboratory Performance Assessment
Spring 2011

QS Fachgesellschaft – Obst-Gemüse-Kartoffeln GmbH

Report

Test Material C

Pesticide Residues in Leek Purée

April 2011

Summary

The Test Materials for the 2011 spring Laboratory Performance Assessment of QS Fachgesellschaft Obst-Gemüse-Kartoffeln GmbH were prepared by the GLP department of LUFA Speyer in March 2011 to provide individual Test Material tailored to QS needs.

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

This report refers to the performance assessment of laboratories which analysed Test Material "C". Each laboratory received minimum 100 g leek purée with six spiked pesticides. All 24 participants kept the term for the submission of results.

The laboratories were requested to identify and quantify 6 pesticides. These were Boscalid, Cypermethrin [alpha], Epoxiconazol, Famoxadon, Fluazifop-P and Iprodion.

The performance assessment considers the following test criteria:

- No *false negative* results are reported (thus identification of all six pesticides, see also tables 2 to 4, pp. 8-10).
- No *false positive* results are reported (see also p. 5).
- Correct quantification related to the 70 - 120% recovery criteria.

The overall performance assessment results of the competence test (Material C) are summarised in the following table:

QS criteria	number of satisfactory participants	total number of participants	satisfactory (%)
correctly identified all six pesticides > 20 µg/kg	14	24	58
correctly identified AND reported satisfactory results for all six pesticides	5	24	21

Assessment of quantification:

As announced in the letter of invitation, the assessment of quantification was exactly defined: Analytical results between 70 and 120% of the spiked levels have been considered satisfying.

analyte	spiked level (µg/kg)	number of satisfactory results (70-120% of the spiked level)	total number of participants	satisfactory (%)
Boscalid	90	11	24	46
Cypermethrin [alpha]	120	20	24	83
Epoxiconazol	50	19	24	79
Famoxadon	500	19	24	79
Fluazifop-P	45	11	24	46
Iprodion	40	18	24	75

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1. Test Material Preparation and Design

The Test Material for this Performance Assessment was prepared by the GLP department of LUFA Speyer. The homogeneity tests were performed by the LAVES Oldenburg, Germany. The results of homogeneity testing are presented in table 1a and b, pp. 6-7.

The following pesticides were spiked to give the approximate final concentrations: 90 µg/kg Boscalid, 120 µg/kg Cypermethrin [alpha], 50 µg/kg Epoxiconazol, 500 µg/kg Famoxadon, 45 µg/kg Fluazifop-P, and 40 µg/kg Iprodion.

The Test Material is based on Leek labelled as organic. Before using the Leek homogenate for spiking, it has been analysed and no levels of pesticides have been detected with a limit of detection at 10 µg/kg. The Leek 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 the pesticides the Leek purée had been analysed for. The results had to be reported without consideration of the recovery unless reported differently by the participants. The participants were asked to report also recovery rates for the quantified pesticides.

The limit of quantification (LOQ) had to be specified for all sought and found pesticides.

Each laboratory was given a number (laboratory code). The results of all participants analysing Test Material C are presented in tables 1 to 3 (pp. 6-8) and subsequently in figures 1 to 6 (pp. 9-14).

The term "n.r. = not reported" applies for tables 1 to 3.

None participant reported a false positive result above or equal 10 µg/kg.

laboratory code	Boscalid spiked level: 90 µg/kg				Cypermethrin [alpha] spiked level: 120 µg/kg			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 63-108	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 84-144
30	76	98	10	yes	126	105	10	yes
41	166	105	10	no	140	112	10	yes
43	46	96	10	no	88	84	10	yes
44	95	95	10	yes	95	92	10	yes
46	90	89	10	yes	125	91	25	yes
48	60	70-120	10	no	96	70-120	10	yes
54	146	86	10	no	133	101	10	yes
57	83	86	1	yes	120	103	5	yes
58	120	81	20	no	120	95	10	yes
59	60	84	10	no	106	91	10	yes
60	49	95	10	no	97	94	10	yes
61	87	76	10	yes	122	85	10	yes
62	60	97	10	no	97	98	10	yes
67	180	120	5	no	138	103	10	yes
68	120		10	no	229		10	no
71	84	98	10	yes	96	99	5	yes
79	95	90	10	yes	115	90	10	yes
86	58		10	no	40		10	no
88	95	90	10	yes	120	90	10	yes
89				n.r.	108	100	10	yes
92	90	95	10	yes	100	95	5	yes
93	153	86	10	no	150	90	10	no
97	95,3	97	10	yes	145	92	10	no
99	82	90	10	yes	106	96	10	yes

Table 1: Results for Boscalid and Cypermethrin [alpha] in Leek Purée Test Material C

	Epoxiconazol spiked level: 50 µg/kg				Famoxadon spiked level: 500 µg/kg			
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 35-60	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 350-600
30	44	102	10	yes	466	85	10	yes
41	52	88	10	yes	577	115	10	yes
43	56	87	10	yes	423	65	10	yes
44	48	98	10	yes	360	103	10	yes
46	54	78	10	yes	463	89	10	yes
48	42	70-120	10	yes	420	70-120	10	yes
54	67	98	10	no	495	97	10	yes
57	56	98	5	yes	382	87	5	yes
58	55	80	10	yes	450	75	50	yes
59	44	83	10	yes	430	83	10	yes
60	37	94	10	yes	425	103	10	yes
61	53	90	10	yes				n.r.
62	36	70	50	yes	460	68	50	yes
67	86	107	10	no	582	100	10	yes
68	60		10	yes	654		10	no
71	48	97	20	yes	502	101	10	yes
79	50	90	10	yes	700	90	10	no
86				n.r.	160		20	no
88	60	95	10	yes	540	94	10	yes
89	46	105	10	yes	446	90	10	yes
92	50	95	10	yes	360	95	10	yes
93	82	87	10	no	500	95	10	yes
97	47,6	104	10	yes	536	105	10	yes
99	61	95	10	no	718	79	10	no

Table 2: Results for Epoxiconazol and Famoxadon in Leek Purée Test Material C

laboratory code	Fluazifop-P spiked level: 45 µg/kg				Iprodion spiked level: 40 µg/kg			
	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 31,5-54	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 28-48
30				n.r.	33	95	10	yes
41	56	96	10	no	53	83	10	no
43				n.r.	28	86	10	yes
44	42	90	10	yes	36	102	10	yes
46	45	89	10	yes	42	79	10	yes
48	60	70-120	10	no	42	70-120	10	yes
54	37	90	10	yes	40	85	10	yes
57	37	93	5	yes	44	85	10	yes
58	39	108	10	yes	45	96	10	yes
59				n.r.	34	105	10	yes
60				n.r.	32	97	10	yes
61	44	79	10	yes	36	96	10	yes
62				n.r.	22	73	10	no
67	66	103	10	no	55	95	10	no
68				n.r.	72		10	no
71	36	94	5	yes	36	96	10	yes
79	75	90	10	no	45	90	10	yes
86				n.r.	146		10	no
88	35	80	10	yes				n.r.
89				n.r.	32	110	10	yes
92	45	95	10	yes	36	95	10	yes
93	48	87	10	yes	48	111	10	yes
97	49,8	92	10	yes	42,7	89	10	yes
99	55	86	10	no	48	88	10	yes

Table 3: Results for Fluazifop-P and Iprodion in Leek Purée Test Material C

Boscalid

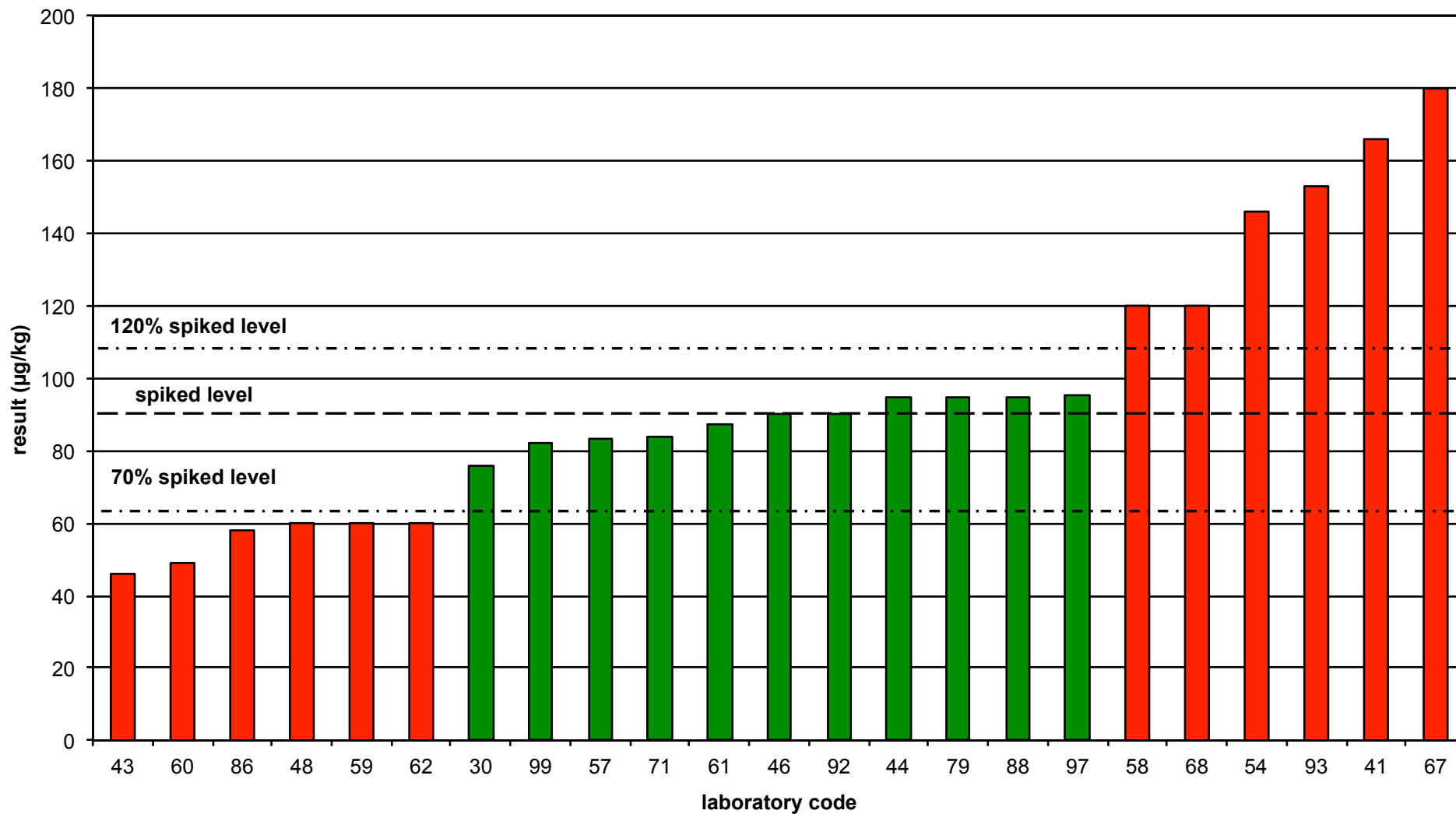


Figure 1: Assessment of Boscalid (spiked level: 90 $\mu\text{g/kg}$) in Leek Purée Test Material C

Cypermethrin [alpha]

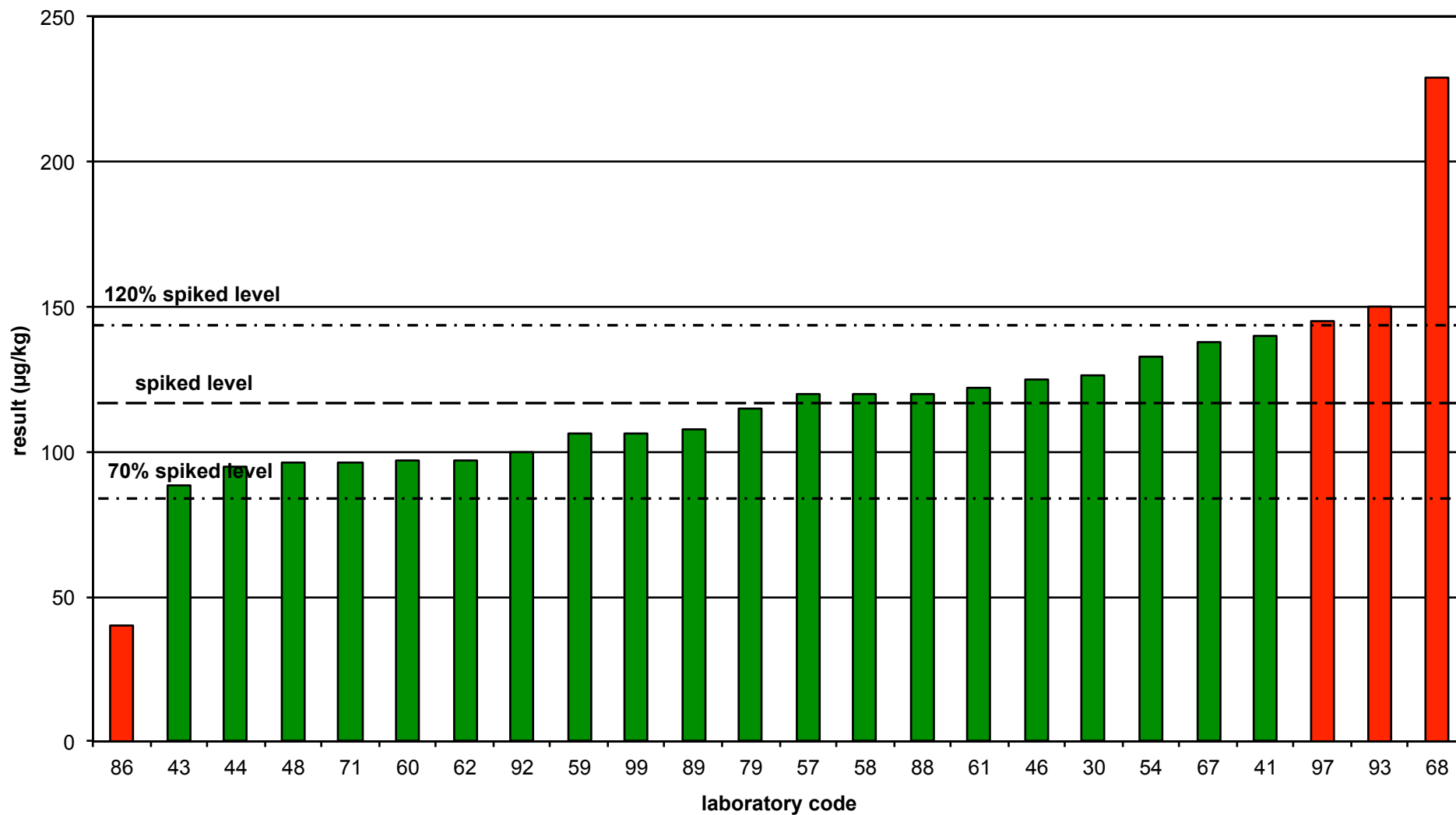


Figure 2: Assessment of Cypermethrin [alpha] (spiked level: 120 µg/kg) in Leek Purée Test Material C

Epoxiconazol

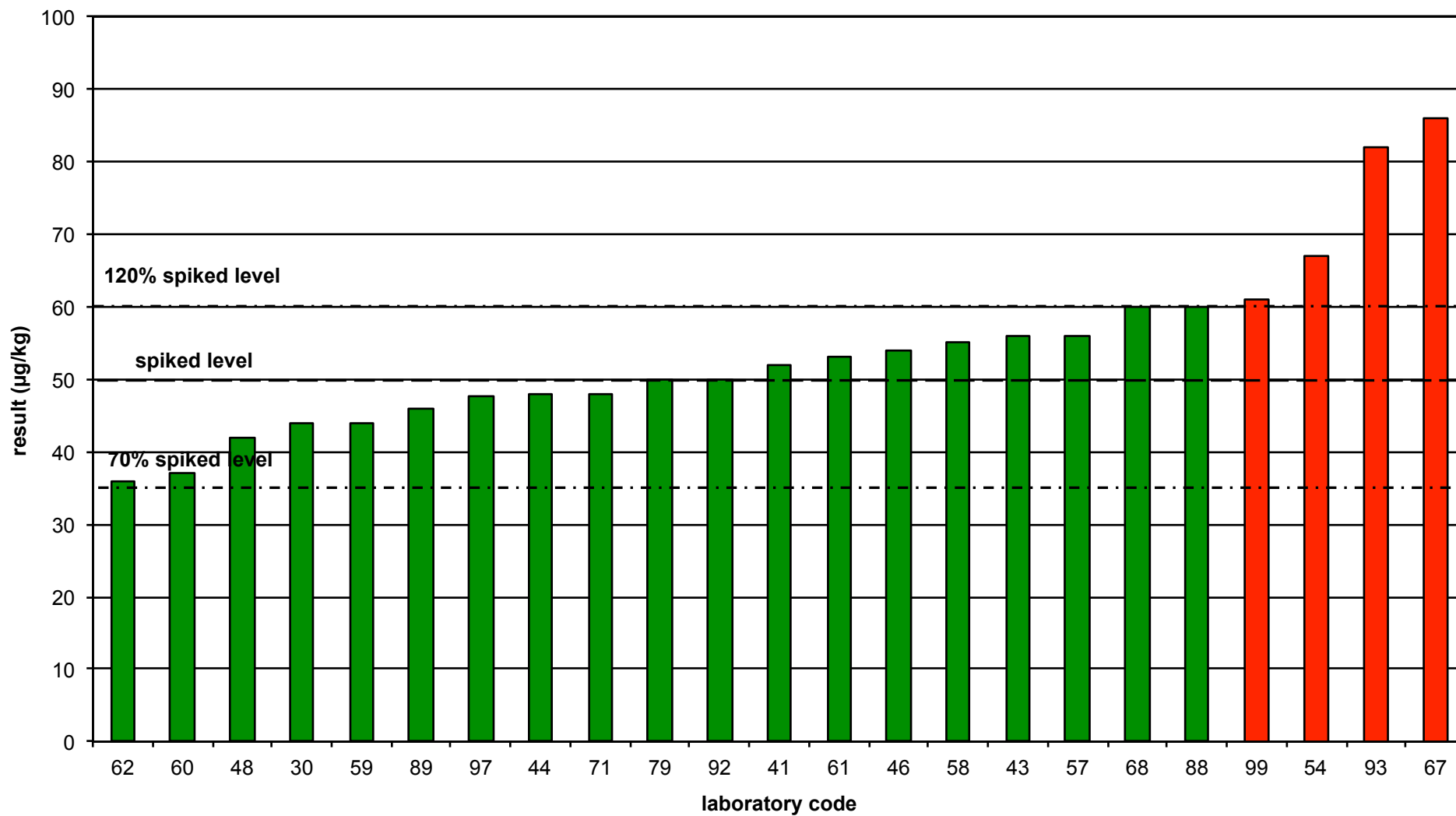


Figure 3: Assessment of Epoxiconazol (spiked level: 50 µg/kg) in Leek Purée Test Material C

Famoxadon

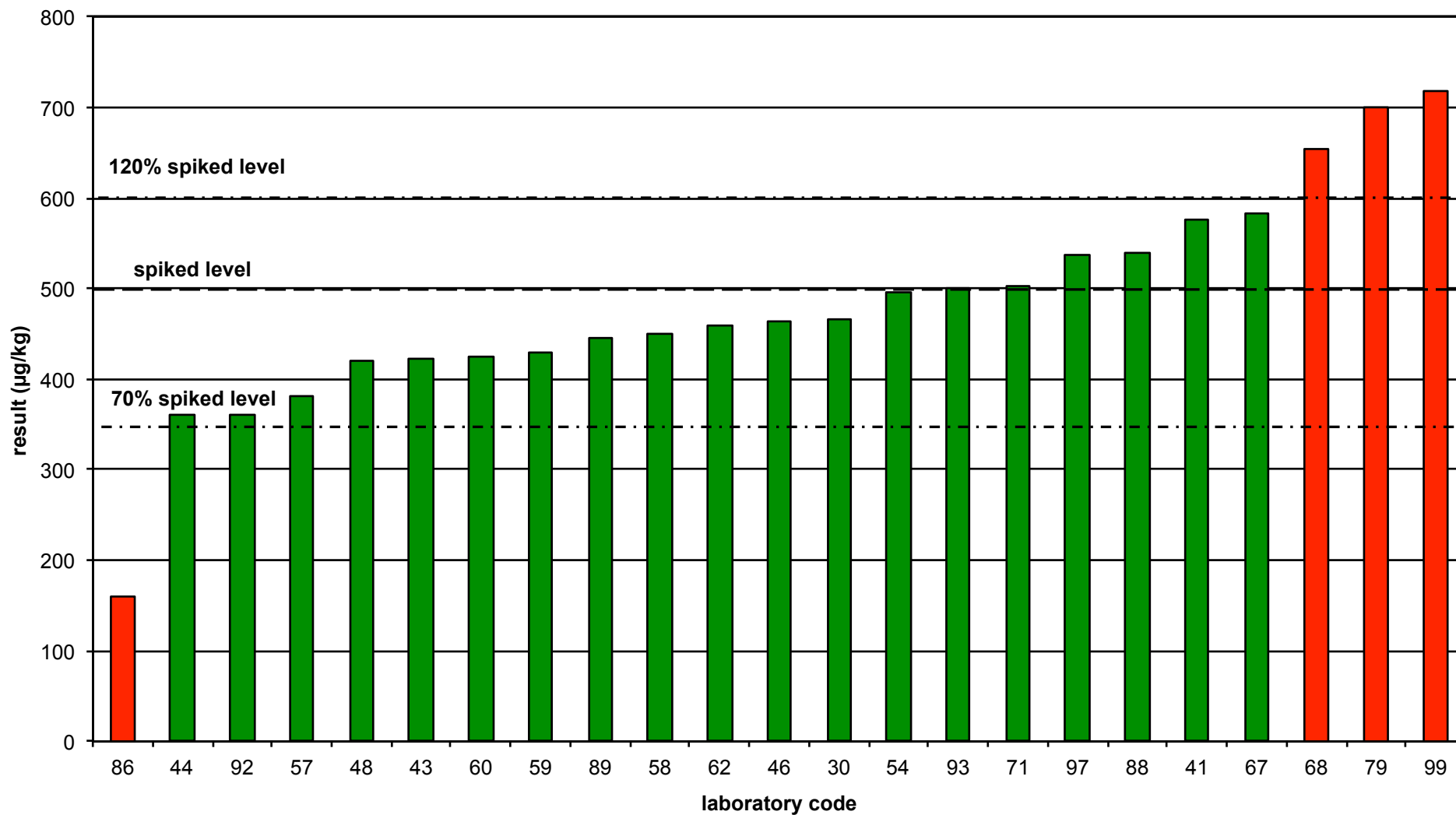


Figure 4: Assessment of Famoxadon (spiked level: 500 µg/kg) in Leek Purée Test Material C

Fluazifop-P

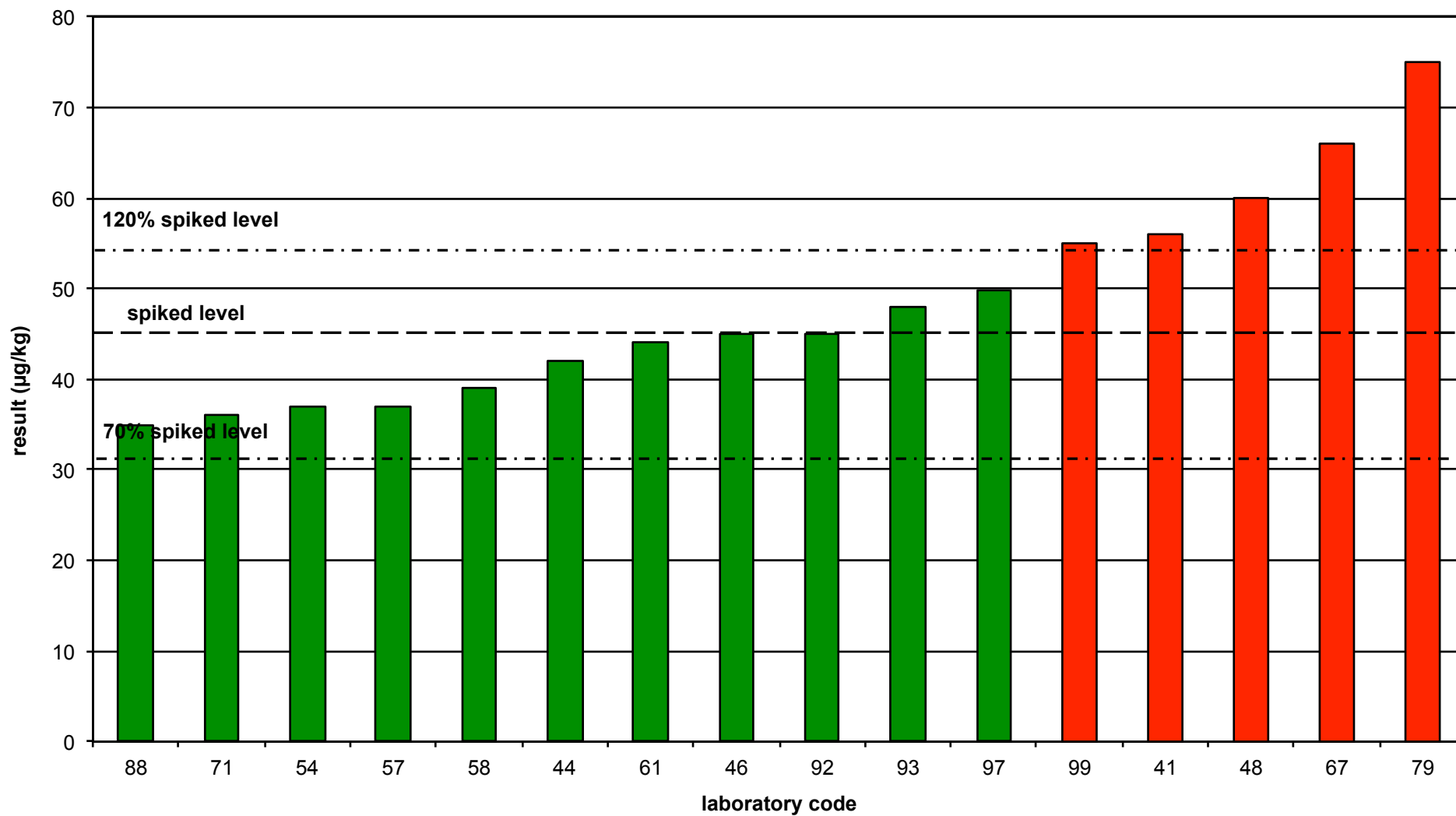


Figure 5: Assessment of Fluazifop-P (spiked level: 45 µg/kg) in Leek Purée Test Material C

Iprodion

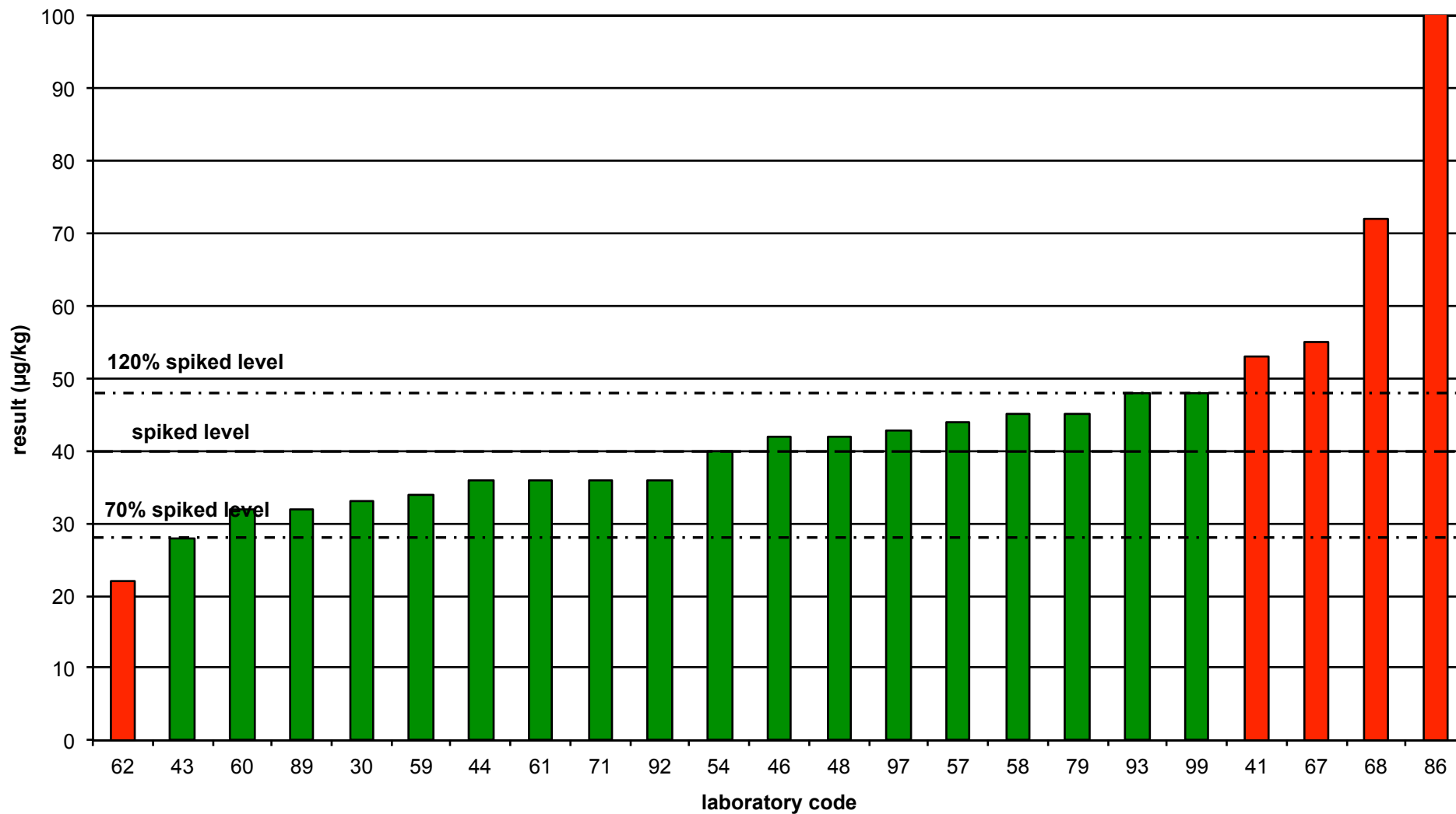


Figure 6: Assessment of Iprodion (spiked level: 40 µg/kg) in Leek Purée Test Material C

3. Homogeneity Testing

The Homogeneity Testings were performed at the LAVES (Niedersächsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit) Oldenburg, Germany. Ten randomly chosen Test Material samples were analysed in duplicate to check the corresponding homogeneity of the six spiked pesticides.

	Boscalid			
Test Material	1.Aufarbeitung	2. Aufarbeitung	MW	Wiederfindung %
TM C Nr.: 21	0,092	0,089	0,091	109
TM C Nr.: 25	0,091	0,096	0,093	
TM C Nr.: 29	0,100	0,094	0,097	Standardabweichung
TM C Nr.: 53	0,091	0,095	0,093	0,0037
TM C Nr.: 57	0,087	0,090	0,089	
TM C Nr.: 81	0,082	0,085	0,084	Variation in %
TM C Nr.: 85	0,094	0,090	0,092	4,03
TM C Nr.: 89	0,093	0,095	0,094	
TM C Nr.: 113	0,089	0,091	0,090	Soll
TM C Nr.: 117	0,093	0,099	0,096	0,090
mean	0,092			

	Cypermethrin [alpha]			
Test Material	1.Aufarbeitung	2. Aufarbeitung	MW	Wiederfindung %
TM C Nr.: 21	0,133	0,132	0,133	116
TM C Nr.: 25	0,128	0,124	0,126	
TM C Nr.: 29	0,121	0,125	0,123	Standardabweichung
TM C Nr.: 53	0,114	0,116	0,115	0,0055
TM C Nr.: 57	0,115	0,122	0,118	
TM C Nr.: 81	0,113	0,119	0,116	Variation in %
TM C Nr.: 85	0,116	0,117	0,117	4,56
TM C Nr.: 89	0,120	0,115	0,117	
TM C Nr.: 113	0,116	0,130	0,123	Soll
TM C Nr.: 117	0,119	0,120	0,120	0,120
mean	0,121			

	Epoxiconazol			
Test Material	1.Aufarbeitung	2. Aufarbeitung	MW	Wiederfindung %
TM C Nr.: 21	0,062	0,059	0,061	114
TM C Nr.: 25	0,057	0,056	0,057	
TM C Nr.: 29	0,050	0,051	0,051	Standardabweichung
TM C Nr.: 53	0,051	0,051	0,051	0,0038
TM C Nr.: 57	0,052	0,052	0,052	
TM C Nr.: 81	0,050	0,055	0,053	Variation in %
TM C Nr.: 85	0,048	0,048	0,048	7,27
TM C Nr.: 89	0,051	0,046	0,049	
TM C Nr.: 113	0,048	0,053	0,051	Soll
TM C Nr.: 117	0,050	0,051	0,051	0,050
mean	0,052			

**Table 4a: Homogeneity Data for Leek Homogenate Samples
(provided by LAVES Oldenburg)**

	Famoxadon			
Test Material	1.Aufarbeitung	2. Aufarbeitung	MW	Wiederfindung %
TM C Nr.: 21	0,551	0,542	0,547	101
TM C Nr.: 25	0,507	0,531	0,519	
TM C Nr.: 29	0,520	0,526	0,523	Standardabweichung
TM C Nr.: 53	0,499	0,516	0,508	0,0131
TM C Nr.: 57	0,506	0,525	0,515	
TM C Nr.: 81	0,480	0,513	0,496	Variation in %
TM C Nr.: 85	0,508	0,521	0,514	2,52
TM C Nr.: 89	0,512	0,517	0,515	
TM C Nr.: 113	0,503	0,540	0,521	Soll
TM C Nr.: 117	0,529	0,525	0,527	0,500
mean	0,519			

	Fluazifop-P			
Test Material	1.Aufarbeitung	2. Aufarbeitung	MW	Wiederfindung %
TM C Nr.: 21	0,045	0,046	0,046	110
TM C Nr.: 25	0,042	0,043	0,043	
TM C Nr.: 29	0,045	0,045	0,045	Standardabweichung
TM C Nr.: 53	0,043	0,048	0,046	0,0021
TM C Nr.: 57	0,042	0,044	0,043	
TM C Nr.: 81	0,044	0,045	0,045	Variation in %
TM C Nr.: 85	0,044	0,038	0,041	4,78
TM C Nr.: 89	0,045	0,043	0,044	
TM C Nr.: 113	0,049	0,048	0,049	Soll
TM C Nr.: 117	0,048	0,045	0,047	0,045
mean	0,045			

	Iprodion			
Test Material	1.Aufarbeitung	2. Aufarbeitung	MW	Wiederfindung %
TM C Nr.: 21	0,053	0,054	0,054	112
TM C Nr.: 25	0,050	0,051	0,051	
TM C Nr.: 29	0,047	0,048	0,048	Standardabweichung
TM C Nr.: 53	0,049	0,047	0,048	0,0045
TM C Nr.: 57	0,047	0,047	0,047	
TM C Nr.: 81	0,039	0,043	0,041	Variation in %
TM C Nr.: 85	0,041	0,040	0,041	9,77
TM C Nr.: 89	0,042	0,040	0,041	
TM C Nr.: 113	0,041	0,044	0,043	Soll
TM C Nr.: 117	0,043	0,045	0,044	0,040
mean	0,046			

**Table 4b: Homogeneity Data for Leek Homogenate Samples
(provided by LAVES Oldenburg)**