Qualitätssicherung. **Vom Landwirt bis zur Ladentheke.**

QS. Ihr Prüfsystem für Lebensmittel.

Laboratory Performance Assessment

2021/1

QS Fachgesellschaft Obst-Gemüse-Kartoffeln GmbH

Report

Test Material B

Pesticide Residues in Peach

June 2021

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1. Scope of proficiency testing (PT)

This proficiency testing was run by QS Fachgesellschaft Obst-Gemüse-Kartoffeln GmbH. This proficiency testing represents an ability test. It is used to be an external quality assurance and shows on each testing field the performance/ability of the laboratory.

This report refers to the performance assessment of laboratories which analysed test material "B". Each laboratory received a sample of peach with eight spiked pesticides. 32 participants reported results and were evaluated.

The evaluation and reporting was subcontracted to DRRR GmbH.

2. Test items and homogeneity

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 "B" was distributed to thirty-two (32) participants.

The test material for this performance assessment was prepared by the GLP department of LUFA Speyer, Germany. The homogeneity test was performed by the AGES (Österreichische Agentur für Gesundheit und Ernährungssicherheit).

The material was distributed into labelled bottles with at least 250 g in each. The bottles were stored at - 20 °C in the dark until distribution.

The following pesticides were spiked to give the approximate final concentrations:

- 90 µg/kg azadirachtin,
- 30 µg/kg fenbuconazole,
- 100 µg/kg flonicamid TFNG,
- 80 µg/kg imidacloprid,
- 50 µg/kg matrine,
- 70 µg/kg tau-fluvalinate,
- 90 µg/kg tetraconazole,
- 600 µg/kg mancozeb (calculated as CS2)

2.1 Summary of results of homogeneity testing

The homogeneity test was carried out at all requested parameters to get information about the principal suitability of the material for this proficiency testing. If the standard deviation ($s_{material}$) of the material homogeneity is smaller than 30 % of a test statistic the samples are classified as homogenous. For this laboratory performance assessment the double variation coefficient [%] according to Horwitz (HORWITZ, W. (1982): Evaluation of analytical methods used for regulation of foods and drugs. Anal Chem 54 (1): 67A-76A) converted to absolute values was used as the test statistic.

The homogeneity test was carried out at 10 samples in double determination acc. to ISO 13528 (Intern. Harmonised Protocol).

The homogeneity test was subcontracted by:
AGES (Österreichische Agentur für Gesundheit und Ernährungssicherheit), Austria.

In the following table the evaluation of the homogeneity test is shown:

parameter	spiked level *[µg/kg]*	mean (homogeneity test) *[µg/kg]*	recovery (homogeneity test) *[%]*	standard deviation of the material homogeneity ($s_{material}$) *[µg/kg]*	test statistic *[µg/kg]*	evaluation of the homogeneity test ($s_{material}$ < 30 % test statistic)
azadirachtin	90	88	98	2	41	**homogenous**
fenbuconazole	30	28	94	0	13	**homogenous**
flonicamid TFNG	100	94	94	0	43	**homogenous**
imidacloprid	80	79	99	2	37	**homogenous**
matrine	50	47	94	2	22	**homogenous**
tau-fluvalinate	70	68	97	1	33	**homogenous**
tetraconazole	90	90	100	0	41	**homogenous**
mancozeb (calculated as CS2)	600	582	97	39	202	**homogenous**

3. Requested testing parameters

The participants were requested to report which pesticides the samples had been analysed for. The results had to be reported without consideration of the recovery. 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.

The laboratories were requested to identify and quantify the following eight pesticides.

- azadirachtin
- fenbuconazole
- flonicamid TFNG
- imidacloprid
- matrine
- tau-fluvalinate
- tetraconazole
- mancozeb (calculated as CS2)

4. Criteria of assessment and test items

The test materials for the Laboratory Performance Assessment 2021/1 of QS Fachgesellschaft Obst-Gemüse-Kartoffeln GmbH were prepared by the GLP department of LUFA Speyer to provide individual test material tailored to QS needs.

The sample should be analysed according to the QS control plan (multi method, dithianon, matrine). The test material was not spiked with dithianon. No laboratory reported a positive result. Therefore, the reported results are not included in the evaluation of the laboratory performance assessment and the report.

The validation of the multi method screening result for the parameter matrine could be subcontracted as a single method to a QS authorized laboratory. If screening and single method result were available from one laboratory, the single method result was used for the evaluation of the test result. The results were evaluated independently, whether the determination was performed by the participant themselves or subcontracted. According to QS specifications, testing for dithiocarbamates (DTC) may not be subcontracted, but must be performed by the participant itself.

All three test materials naturally contained a small amount of DEET (diethyltoluamide) with < 5 µg/kg. Some participants reported traces of DEET.

The performance assessment considers the following test criteria:

- No false negative results are reported (thus identification of all eight pesticides)
- No false positive results are reported
- Correct quantification of at least six pesticides related to the recovery criteria. Analytical results between 70 % and 120% of the spiked levels have been considered satisfying.

5. Demonstration of the results

Each laboratory was given a number (laboratory code).

A summarized evaluation of the laboratory performance is shown in table 1 to 4 according to the test criteria.

The results of all participants analysing test material "B" are presented in tables 5 to 13. Each table follows a diagram with the individual results in ascending order. The diagrams include the spiked level and the accepted range of the respective pesticide.

6. Remarks and comments

- The labs 33 and 36 reported DEET < LoQ (5 µg/kg).
- The labs 38 and 59 reported DEET < LoQ.
- The lab 50 reported DEET < LoQ (4 µg/kg).
- The lab 50 reported a result for imidacloprid (result: 80µg/kg, recovery 80%, LoQ 10 µg/kg) after the deadline. Therefore, it cannot be considered in the evaluation.
- The lab 50 reported, that the analysis for dithiocarbamates (result: 410µg/kg, recovery 76%, LoQ 50 µg/kg) was subcontracted. Subcontracting is not possible for this according to the QS specifications, which is why the result cannot be considered in the evaluation.
- The lab 63: The laboratory did not want to participate in the proficiency test with the analysis for dithiocarbamates. For this laboratory, the analysis for dithiocarbamates is not included in the evaluation.

7. Results, tables and graphics

7.1 Summary

Table 1: Summarized laboratory rating in test material B

QS criteria	number of participants	percent of participants
passed	28	88
failed	4	13
total	32	100

Table 2: Number of satisfactory participants in test material B

QS criteria	number of satisfactory participants	number of participants	satisfactory %
all analyts identified	30	32	94
all analyts identified and reported in acceptable results	10	32	31
no false positive results	32	32	100

7.2 Rating of performance

Table 3: Rating

laboratory code	**criterion 1** all analytes identified	**criterion 2** number ofacceptable results	**criterion 3** number of false positive results	**rating**
32	yes	7	0	**passed**
33	yes	7	0	**passed**
34	yes	8	0	**passed**
35	yes	7	0	**passed**
36	yes	8	0	**passed**
37	yes	8	0	**passed**
38	yes	7	0	**passed**
39	yes	8	0	**passed**
40	yes	7	0	**passed**
41	yes	7	0	**passed**
42	yes	7	0	**passed**
43	yes	6	0	**passed**
44	yes	7	0	**passed**
45	yes	8	0	**passed**
46	yes	8	0	**passed**
47	yes	7	0	**passed**
48	yes	7	0	**passed**
49	yes	8	0	**passed**
50	no	3	0	**failed**
51	yes	8	0	**passed**
52	yes	4	0	**failed**
53	yes	8	0	**passed**
54	yes	7	0	**passed**
55	yes	5	0	**failed**
56	yes	7	0	**passed**
57	yes	6	0	**passed**
58	yes	6	0	**passed**
59	yes	7	0	**passed**
60	yes	8	0	**passed**
61	yes	7	0	**passed**
62	yes	7	0	**passed**
63	no	5	0	**failed**

Table 4: Number of satisfactory results in test material B

analyte	number of satisfactory results	total number of correct identification	satisfactory (%)
azadirachtin	27	32	84
fenbuconazole	29	32	91
flonicamid TFNG	31	32	97
imidacloprid	27	31	87
matrine	28	31	90
tau-fluvalinate	26	32	81
tetraconazole	31	32	97
mancozeb (calculated as CS2)	21	30	70

7.3 Results of Azadirachtin

Table 5: Results for azadirachtin in test material B

azadirachtin - spiked level: 90 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 63-108
32	62	102	10	**no**
33	78	91	10	**yes**
34	80	89	10	**yes**
35	75	100	20	**yes**
36	80	90	10	**yes**
37	85	100	5	**yes**
38	78	96	10	**yes**
39	94,8	95	10	**yes**
40	84	100	10	**yes**
41	54	86	10	**no**
42	108	98	10	**yes**
43	82	100	10	**yes**
44	70	90	5	**yes**
45	90	95	10	**yes**
46	80	99,5	10	**yes**
47	97	110	10	**yes**
48	68	97	10	**yes**
49	92	99	10	**yes**
50	35	100	10	**no**
51	96	100	10	**yes**
52	68	101	10	**yes**
53	91	91	10	**yes**
54	110	102	20	**no**
55	64	88	10	**yes**
56	88	83	5	**yes**
57	110	76	10	**no**
58	85	105	10	**yes**
59	105	95,2	10	**yes**
60	94	100	10	**yes**
61	84	105	10	**yes**
62	93	45,7	50	**yes**
63	78	100	5	**yes**

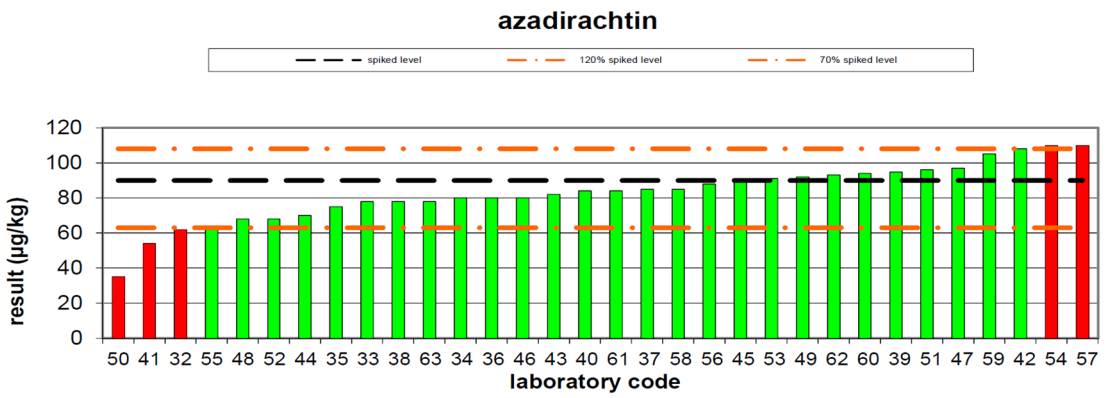

Figure 1: Assessment of azadirachtin in test material B

7.4 Results of Fenbuconazole

Table 6: Results for fenbuconazole in test material B

fenbuconazole - spiked level: 30 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 21-36
32	31	98	10	yes
33	28	92	10	yes
34	26	96	10	yes
35	28	103	10	yes
36	27	95	5	yes
37	28	105	5	yes
38	30	100	10	yes
39	33,3	102	10	yes
40	29	100	5	yes
41	22	87,8	10	yes
42	26	102	10	yes
43	33	111	10	yes
44	42	90	10	no
45	30	95	10	yes
46	32	102,8	10	yes
47	32	99	10	yes
48	27	83	10	yes
49	27	88	10	yes
50	28	88	10	yes
51	29	100	10	yes
52	20	104	10	no
53	28	93	10	yes
54	30	96,4	10	yes
55	50	93	10	no
56	31	80	5	yes
57	31	105	10	yes
58	25	90	10	yes
59	28	92	10	yes
60	28	100	10	yes
61	30	110	10	yes
62	29	98,9	10	yes
63	26	100	1	yes

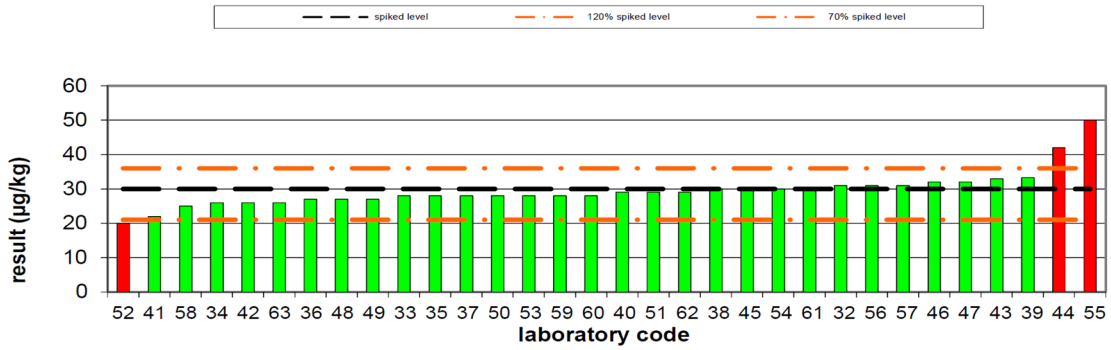

Figure 2: Assessment of fenbuconazole in test material B

7.5 Results of Flonicamid TFNG

Table 7: Results for flonicamid TFNG in test material B

flonicamid TFNG - spiked level: 100 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 70-120
32	99	96	10	**yes**
33	105	96	10	**yes**
34	90	71	5	**yes**
35	96	102	10	**yes**
36	100	90	5	**yes**
37	87	86	5	**yes**
38	107	109	10	**yes**
39	94,9	105	10	**yes**
40	119	102	10	**yes**
41	73	87,2	10	**yes**
42	106	87	10	**yes**
43	79	81	10	**yes**
44	90	90	10	**yes**
45	89	95	10	**yes**
46	110	94	10	**yes**
47	86	60	10	**yes**
48	84	87	10	**yes**
49	90	105	10	**yes**
50	77	100	10	**yes**
51	91	100	10	**yes**
52	86	92	10	**yes**
53	100	116	10	**yes**
54	96	98,6	10	**yes**
55	94	95	10	**yes**
56	91	78	5	**yes**
57	109	78	10	**yes**
58	130	103	10	**no**
59	104	87,8	10	**yes**
60	91	100	10	**yes**
61	90	90	10	**yes**
62	84	45,1	10	**yes**
63	93	100	5	**yes**

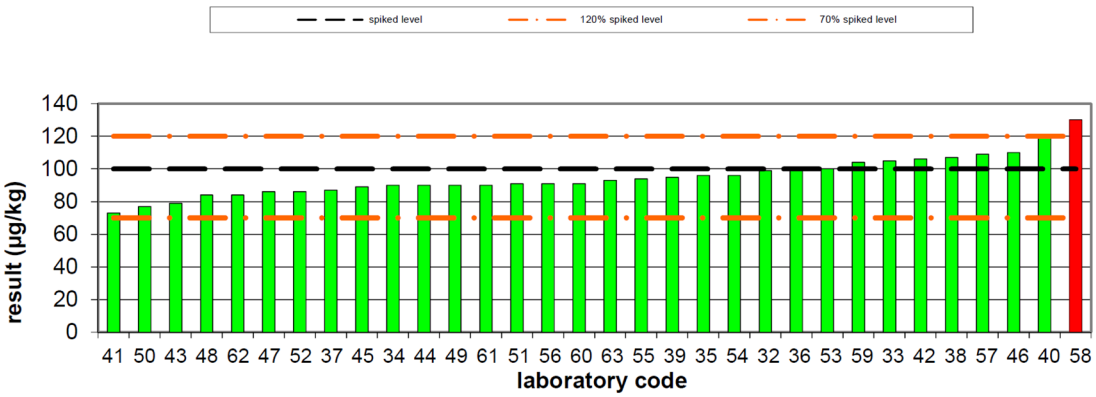

Figure 3: Assessment of flonicamid TFNG in test material B

Table 8: Results for flonicamid (sum of flonicamid, TFNA and TFNG expressed as flonicamid) in test material B

flonicamid (sum of flonicamid, TFNA and TFNG expressed as flonicamid)				
laboratory code	result (µg/kg) *only informative*	recovery (%)	LoQ (µg/kg)	evaluation
32	91			not part of the evaluation
33	97			
34	83			
35	89			
36				
37				
38				
39	87,7			
40	110			
41				
42	98			
43	73			
44	83			
45	82			
46				
47	79			
48	78			
49	83			
50	71			
51				
52	79			
53	92,4			
54				
55				
56	84			
57				
58	120			
59				
60				
61	83			
62				
63				

7.6 Results of Imidacloprid

Table 9: Results for imidacloprid in test material B

imidacloprid - spiked level: 80 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 56-96
32	87	97	10	yes
33	84	93	10	yes
34	76	85	10	yes
35	86	101	10	yes
36	70	92	5	yes
37	90	100	5	yes
38	93	94	10	yes
39	92,9	96	10	yes
40	108	89,8	10	no
41	76	89	10	yes
42	74	108	10	yes
43	96	117	10	yes
44	80	90	5	yes
45	77	95	10	yes
46	85	100,6	10	yes
47	92	93	10	yes
48	62	84	10	yes
49	75	107	10	yes
50				no
51	92	100	10	yes
52	53	91	10	no
53	92	89	10	yes
54	79	99	10	yes
55	99	87	10	no
56	120	87	5	no
57	79	88	10	yes
58	58	95	10	yes
59	78	92,2	10	yes
60	93	100	10	yes
61	90	100	10	yes
62	89	89,7	10	yes
63	72	100	1	yes

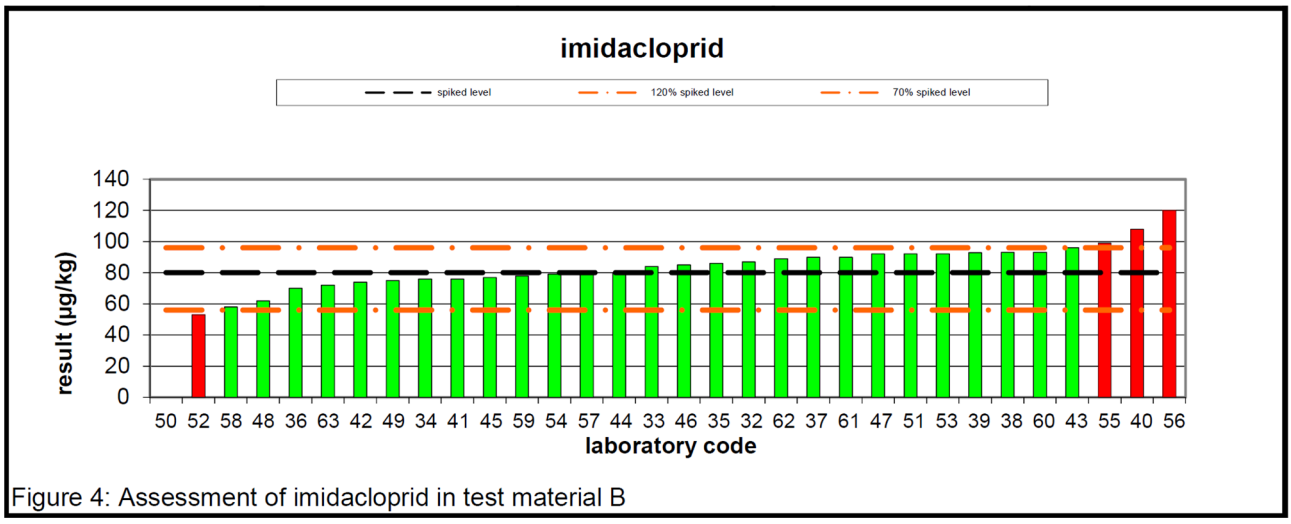

Figure 4: Assessment of imidacloprid in test material B

7.7 Results of Matrine

Table 10: Results for matrine in test material B

matrine - spiked level: 50 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 35-60
32	48	95	10	yes
33	54	93	10	yes
34	50	110	10	yes
35	53	106	10	yes
36	50	90	10	yes
37	42	82	5	yes
38	67	92	10	no
39	37,3	87	10	yes
40	53	76,3	10	yes
41	40	74,5	10	yes
42	45	117	10	yes
43	43	101	10	yes
44	48	90	10	yes
45	57	95	10	yes
46	52	105,4	10	yes
47 *	71	96	10	no
48	51	108	10	yes
49	44	82	10	yes
50	32	100	50	no
51	42	100	10	yes
52	39	97	10	yes
53	45	89	10	yes
54	38	75	10	yes
55	45	94	10	yes
56	45	82	10	yes
57	50	90	10	yes
58	41	95	10	yes
59	50	90	10	yes
60	43	100	10	yes
61	53	90	10	yes
62	48	60,7	10	yes
63				no

*The analysis was subcontracted

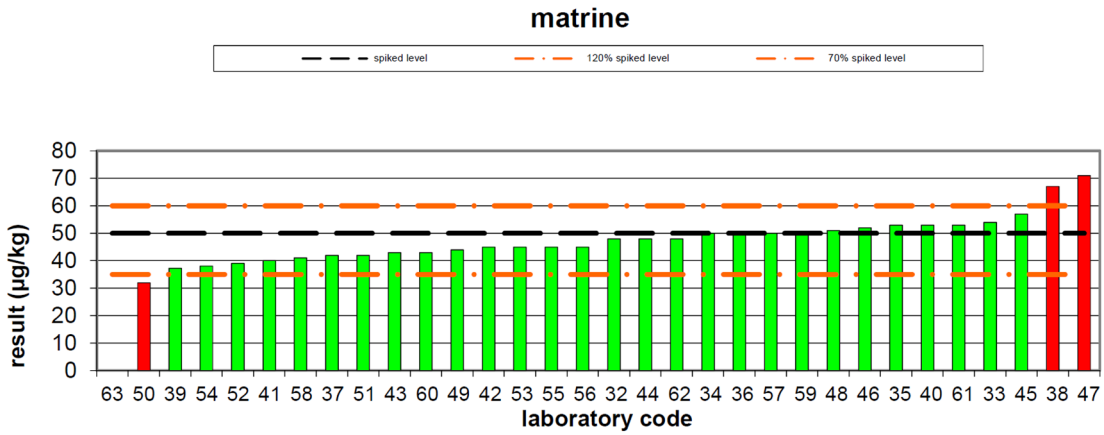

Figure 5: Assessment of matrine in test material B

7.8 Results of Tau-Fluvalinate

Table 11: Results for tau-fluvalinate in test material B

tau-fluvalinate - spiked level: 70 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 49-84
32	70	103	10	yes
33	54	92	10	yes
34	57	97	10	yes
35	60	102	10	yes
36	65	94	5	yes
37	70	100	5	yes
38	67	91	10	yes
39	83,7	104	10	yes
40	72	87	10	yes
41	67	98	10	yes
42	42	96	5	no
43	47	96	10	no
44	82	90	10	yes
45	78	95	10	yes
46	70	100,8	10	yes
47	71	108	10	yes
48	46	100	10	no
49	71	78	10	yes
50	60	94	10	yes
51	65	100	10	yes
52	23	90	10	no
53	72	88	10	yes
54	63	102	10	yes
55	111	79	10	no
56	65	88	5	yes
57	67	98	10	yes
58	54	99	10	yes
59	71	92,5	10	yes
60	66	100	10	yes
61	58	100	10	yes
62	72	73,1	10	yes
63	44	100	1	no

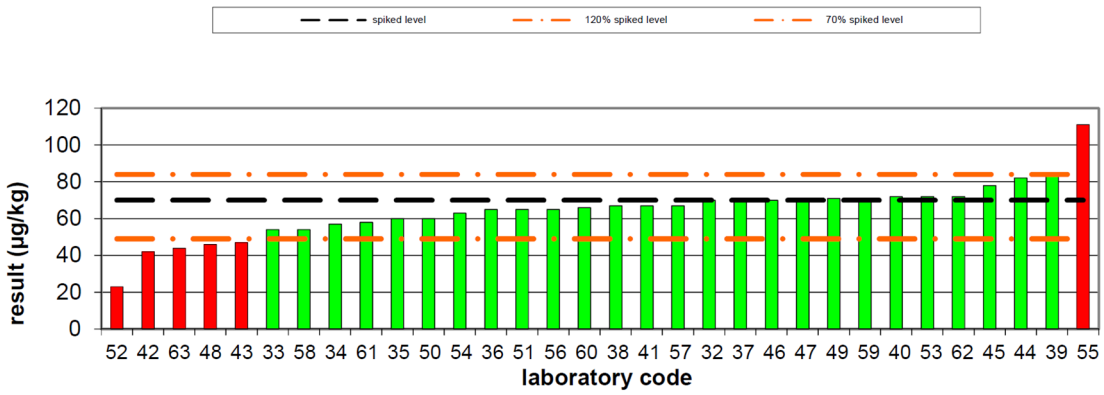

Figure 6: Assessment of tau-fluvalinate in test material B

7.9 Results of Tetraconazole

Table 12: Results for tetraconazole in test material B

tetraconazole - spiked level: 90 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 63-108
32	82	99	10	yes
33	84	94	10	yes
34	70	96	10	yes
35	84	99	10	yes
36	80	93	5	yes
37	81	95	5	yes
38	93	100	10	yes
39	93,4	87	10	yes
40	90	98,1	10	yes
41	81	88,4	10	yes
42	73	92	5	yes
43	83	102	10	yes
44	80	90	10	yes
45	84	95	10	yes
46	93	103,7	10	yes
47	89	99	10	yes
48	83	88	10	yes
49	80	76	10	yes
50	61	108	10	no
51	82	100	10	yes
52	70	79	10	yes
53	74	112	10	yes
54	75	100,3	10	yes
55	78	103	10	yes
56	72	76	5	yes
57	91	109	10	yes
58	72	93	10	yes
59	92	90,7	10	yes
60	80	100	10	yes
61	74	95	10	yes
62	77	78,1	10	yes
63	71	100	1	yes

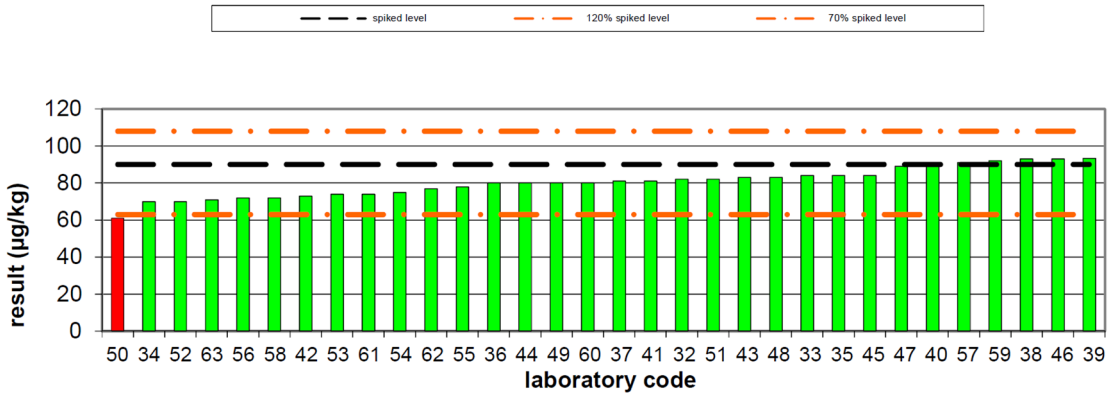

Figure 7: Assessment of tetraconazole in test material B

7.10 Results of Mancozeb (calculated as CS2)

Table 13: Results for mancozeb (calculated as CS2) in test material B

mancozeb (calculated as CS2) - spiked level: 600 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 420-720
32	420	92	10	**yes**
33	770	94	10	**no**
34	600	82	20	**yes**
35	314	98	10	**no**
36	530	92	10	**yes**
37	572	89	10	**yes**
38	630	112	10	**yes**
39	478	90	10	**yes**
40	479	82,2	10	**yes**
41	630	101	10	**yes**
42	600	109	10	**yes**
43	280	98	10	**no**
44	450	90	10	**yes**
45	720	95	10	**yes**
46	600	102	10	**yes**
47	481	72	100	**yes**
48	540	115	10	**yes**
49	490	81	50	**yes**
50				**no**
51	460	100	50	**yes**
52	860	92	10	**no**
53	495	92	10	**yes**
54	450	117,8	10	**yes**
55	448	95	50	**yes**
56	480	79	100	**yes**
57	250	81	10	**no**
58	6500	93	10	**no**
59	419	105,4	10	**no**
60	460	100	10	**yes**
61	410	90	50	**no**
62	418	100	10	**no**
63	not part of the evaluation			

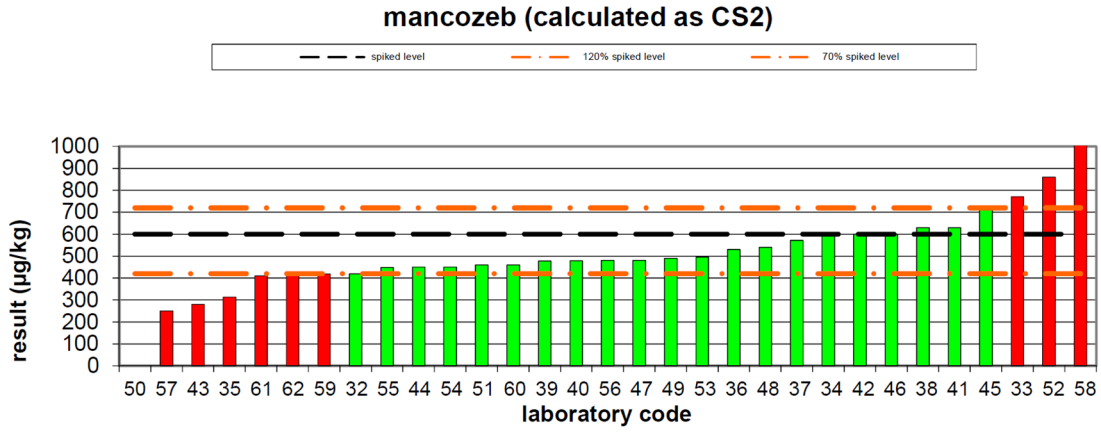

Figure 8: Assessment of mancozeb (calculated as CS2) in test material B

QS. Ihr Prüfsystem für Lebensmittel.

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