Laboratory Performance Assessment

2023/1

QS Fachgesellschaft Obst-Gemüse-Kartoffeln GmbH

Report

Test Material B

Pesticide Residues in Kohlrabi (tuber without leaves)

June 2023

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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 kohlrabi (tuber without leaves) with eight spiked pesticides. 34 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-four (34) 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), Austria.

The material was distributed into labelled bottles with at least 200 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:

- 80 µg/kg Cyantraniliprole
- 50 µg/kg Fluazinam
- 40 µg/kg Metobromuron
- 40 µg/kg Omethoat
- 320 µg/kg Pendimethalin
- 50 µg/kg Pirimicarb
- 30 µg/kg Spirotetramat-enol
- 50 µg/kg Chlorate

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. The test statistic is usually calculated of all previous proficiency testings with this material type determining the relative mean standard deviation as well as extended uncertainty of the relative mean standard deviation of all previous proficiency testings with this material type. The relative values are referred to absolute values related to the actual value of the material and are added to obtain the test statistic. When this rule could not be followed it will be proved if the higher variance is usual for this material type. The test statistic for the evaluation of the homogeneity test of test material "B" was calculated with the results from this proficiency testing round (Laboratory Performance Assessment 2023/1), because no previous proficiency testing data is available for the requested parameter matrix combination.

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

The homogeneity tests are 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)
Fluazinam	30	21	69	2	6	**homogenous**
Fluopyram	60	62	103	2	12	**homogenous**
Metobromuron	40	40	101	1	4	**homogenous**
Omethoat	50	52	104	0	10	**homogenous**
Pendimethalin	280	274	98	10	48	**homogenous**
Pirimicarb	40	38	96	1	4	**homogenous**
Spirotetramat-enol	50	46	93	3	11	**homogenous**
Chlorate	70	71	102	4	15	**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.

- Cyantraniliprole
- Fluopyram
- Metobromuron
- Omethoat
- Pendimethalin
- Pirimicarb
- Spirotetramat-enol
- Chlorate

4. Criteria of assessment and test items

The test materials for the Laboratory Performance Assessment 2023/1 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 specifications of the control plan of the QS guideline "Residue monitoring fruit, vegetables, potatoes", i.e. multi-method. In addition, the analysis for Chlorate (single method) was instructed. The analysis of the parameter Chlorate could be subcontracted to a QS authorized laboratory. The results were evaluated independently, whether the determination was performed by the participant themselves or subcontracted.

The results of the sum parameter Spirotetramat and Spirotetramat-enol (sum of), expressed as Spirotetramat® are presented informatively. The metabolite Spirotetramat-enol was evaluated.

All three test materials naturally contained a small amount of Spinosad (Spinosyn A, Spinosyn D) with< 3 µg/kg. Some laboratories also report the active substance (see point 6).

All three test materials naturally contained a small amount of Perchlorate with < 3 µg/kg. Some laboratories also report the active substance (see point 6).

Some laboratories report Spirotetramat-keto-hydroxy in traces (see point 6). The reference laboratory confirms the traces in the stability test. The active substance Fluazinam could only be

evaluated qualitatively. The homogeneity tests already showed after 3 weeks a loss of 30 % of the spiked amount.

The active substance Omethoat could only be evaluated qualitatively. In contrast to the homogeneity test and the results of the participants, the stability test, performed after submission of all participant results, showed a degradation between 20 and 26 % in the different test materials. Thus, the results did not meet the quality specifications for the test design for a quantitative evaluation (recovery ≤ 10 percent of the spiked value).

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 four pesticides related to the recovery criteria of the quantitative evaluations. 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 with evaluated results 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

Lab 43 reported traces of Spinosad and Spirotetramat-keto-hydroxy.

Lab 46 reported traces of Spirotetramat. Spirotetramat is part of the sum-residue definition of Spirotetramat.

Lab 47 reported a detection of Perchlorate: result: 3 µg/kg; rec. 100%; LoQ: 1 µg/kg.

Lab 49 reported trace levels of Spirotetramat-keto-hydroxy.

Lab 58: The laboratory's results were removed from the evaluation because it did not pay the participation fee despite multiple requests.

Lab 70 did not submit any results. The test is evaluated as failed.

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	29	85
failed	5	15
total	34	100

Table 2: Number of satisfactory participants in test material B

QS criteria	number of satisfactory participants	number of participants	satisfactory %
all Analyt identified	31	34	91
all analyts identified and reported in acceptable results	17	34	50
no false positive results	34	34	100

7.2 Rating of performance

Table 3: Rating

laboratory code	**criterion 1** all analytes identified	**criterion 2** number of acceptable results of the quantitative evaluation	**criterion 3** number of false positive results	**rating**
37	yes	6	0	**passed**
38	yes	5	0	**passed**
39	yes	5	0	**passed**
40	yes	5	0	**passed**
41	yes	6	0	**passed**
42	yes	5	0	**passed**
43	yes	6	0	**passed**
44	yes	6	0	**passed**
45	yes	5	0	**passed**
46	yes	6	0	**passed**
47	yes	5	0	**passed**
48	yes	4	0	**passed**
49	yes	6	0	**passed**
50	yes	6	0	**passed**
51	yes	6	0	**passed**
52	no	4	0	**failed**
53	no	1	0	**failed**
54	yes	5	0	**passed**
55	yes	6	0	**passed**
56	yes	6	0	**passed**
57	yes	6	0	**passed**
59	yes	6	0	**passed**
60	yes	5	0	**passed**
61	yes	1	0	**failed**
62	yes	5	0	**passed**
63	yes	6	0	**passed**
64	yes	6	0	**passed**
65	yes	6	0	**passed**
66	yes	6	0	**passed**
67	yes	3	0	**failed**
68	yes	5	0	**passed**
69	yes	4	0	**passed**
70	no	0	0	**failed**
71	yes	6	0	**passed**

Table 4: Number of satisfactory results in test material B

analyte	number of satisfactory results	total number of correct identification	satisfactory (%)
Cyantraniliprole	25	32	78
Fluazinam	34	31	110
Metobromuron	29	33	88
Omethoat	33	33	100
Pendimethalin	29	33	88
Pirimicarb	32	33	97
Spirotetramat-enol	28	32	88
Chlorate	26	33	79

7.3 Results of Cyantraniliprole

Table 5: Results for Cyantraniliprole in test material B

Cyantraniliprole - spiked level: 80 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 56-96
37	89	95	10	yes
38	100	87	10	no
39	79	105	10	yes
40	105	94	10	no
41	90	80	10	yes
42	62	92	10	yes
43	84	95	10	yes
44	89	114	10	yes
45	98	90	10	no
46	85	99	10	yes
47	78	95.5	5	yes
48	98	110	10	no
49	93	98	10	yes
50	84	86	10	yes
51	74	90	10	yes
52	82.78	85	10	yes
53				no
54	86	96	10	yes
55	80	102	10	yes
56	84	86	10	yes
57	96	99	10	yes
59	74	94	5	yes
60	89	98	10	yes
61	120	95	5	no
62	90	107	10	yes
63	73	90	5	yes
64	68	102	10	yes
65	87	93	10	yes
66	93	102	10	yes
67	52.7	95	10	no
68	97	86	10	no
69	80	92	5	yes
70				no
71	78	90	10	yes

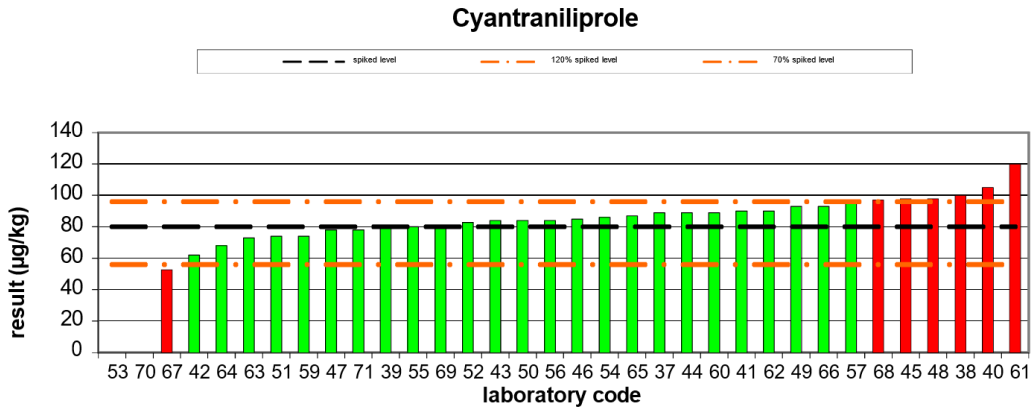

Figure 1: Assessment of Cyantraniliprole in test material B

7.4 Results of Fluazinam

Table 6: Results for Fluazinam in test material B

Fluazinam - spiked level: 50 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): > 0
37	24	109	10	yes
38	20	80	10	yes
39	24	90	10	yes
40	25	90	10	yes
41	23	94	10	yes
42	27	87	10	yes
43	25	95	10	yes
44	27	103	5	yes
45	26	83	10	yes
46	27	96	10	yes
47	20	95.5	5	yes
48	23	81	10	yes
49	26	88	10	yes
50	29	94	10	yes
51	19	80	10	yes
52				no
53				no
54	21	111	10	yes
55	28	98	10	yes
56	23	96	10	yes
57	22	100	10	yes
59	24	115	5	yes
60	23	86	10	yes
61	150	85	5	yes
62	22	73	10	yes
63	19	94	5	yes
64	21	100	10	yes
65	24	87	10	yes
66	23	98	10	yes
67	122.97	95	10	yes
68	35	96	10	yes
69	42	110	5	yes
70				no
71	14	100	10	yes

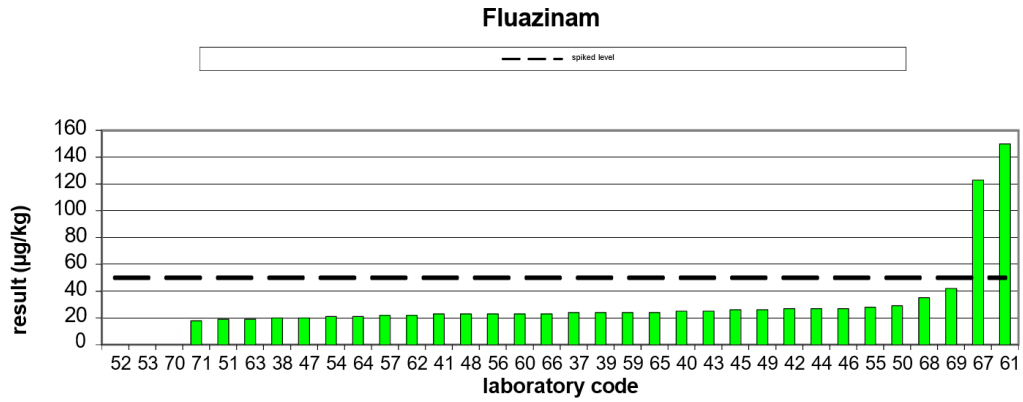

Figure 2: Assessment of Fluazinam in test material B

7.5 Results of Metobromuron

Table 7: Results for Metobromuron in test material B

Metobromuron - spiked level: 40 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 28-48
37	44	114	10	yes
38	35	85	10	yes
39	43	100	10	yes
40	42	92	10	yes
41	42	85	10	yes
42	44	94	10	yes
43	38	95	10	yes
44	47	107	5	yes
45	42	91	10	yes
46	34	108	10	yes
47	40	95.5	5	yes
48	50	107	10	no
49	39	99	10	yes
50	46	96	10	yes
51	40	90	10	yes
52	38.89	105	10	yes
53	75	87	10	no
54	50	101	10	no
55	41	93	10	yes
56	36	103	10	yes
57	42	90	10	yes
59	38	95	5	yes
60	37	100	10	yes
61	70	80	5	no
62	38	103	5	yes
63	37	95	5	yes
64	40	107	10	yes
65	37	86	10	yes
66	40	100	10	yes
67	39.24	95	10	yes
68	42	89	10	yes
69	46	100	5	yes
70				no
71	36	95	10	yes

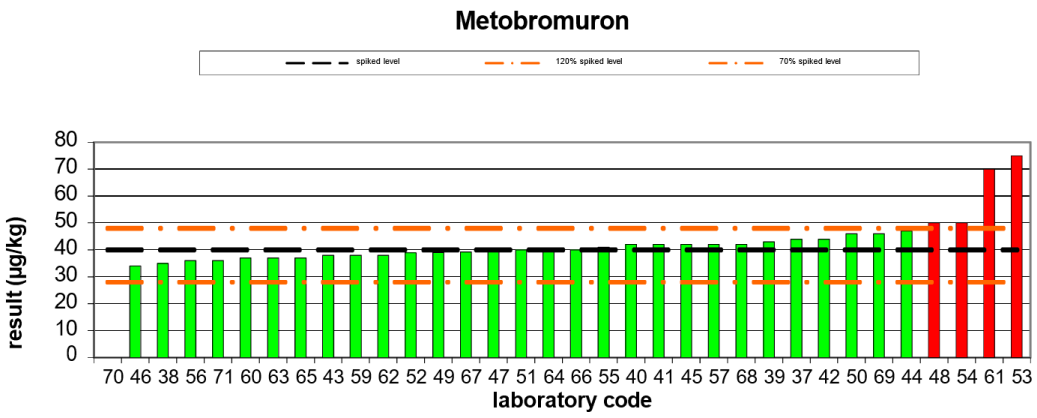

Figure 3: Assessment of Metobromuron in test material B

7.6 Results of Omethoat

Table 8: Results for Omethoat in test material B

Omethoat - spiked level: 40 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 28-48
37	41	85	10	yes
38	34	80	10	yes
39	39	95	10	yes
40	42	94	10	yes
41	35	95	10	yes
42	41	86	10	yes
43	38	95	10	yes
44	41	114	5	yes
45	44	43	10	yes
46	31	107	10	yes
47	36	86,5	5	yes
48	44	100	10	yes
49	46	105	10	yes
50	43	90	10	yes
51	42	90	10	yes
52	37.95	89	10	yes
53	138	119	10	yes
54	33	95	10	yes
55	38	91	10	yes
56	35	94	10	yes
57	41	100	10	yes
59	36	94	5	yes
60	36	110	10	yes
61	30	96	5	yes
62	30	115	5	yes
63	32	91	5	yes
64	42	105	10	yes
65	38	73	10	yes
66	40	95	10	yes
67	35.18	95	10	yes
68	43	100	10	yes
69	36	100	5	yes
70				no
71	42	105	10	yes

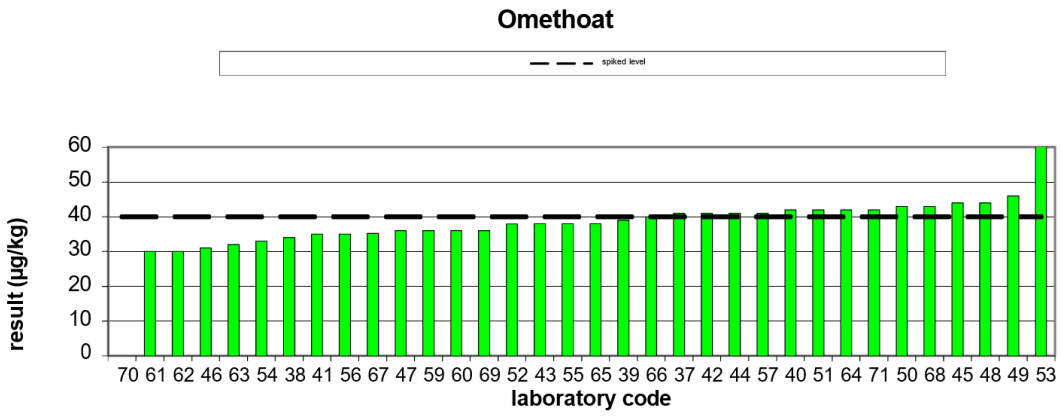

Figure 4: Assessment of Omethoat in test material B

7.7 Results of Pendimethalin

Table 9: Results for Pendimethalin in test material B

Pendimethalin - spiked level: 320 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 224-384
37	280	96	10	yes
38	240	86.17	10	yes
39	247	88	10	yes
40	300	96	10	yes
41	244	120	10	yes
42	290	90	10	yes
43	280	95	10	yes
44	261	98	5	yes
45	262	109	10	yes
46	274	92	10	yes
47	250	103.4	5	yes
48	260	114	10	yes
49	266	98	10	yes
50	352	108	10	yes
51	300	90	10	yes
52	219.79	81	5	no
53	250	103	10	yes
54	284	74	10	yes
55	290	93	10	yes
56	268	89	10	yes
57	274	100	10	yes
59	275	102	5	yes
60	210	115	10	no
61	150	105	5	no
62	285	82	5	yes
63	270	88	5	yes
64	293	100	10	yes
65	231	88	10	yes
66	280	95	10	yes
67	658.87	95	10	no
68	293	117	10	yes
69	312	100	10	yes
70				no
71	228	95	10	yes

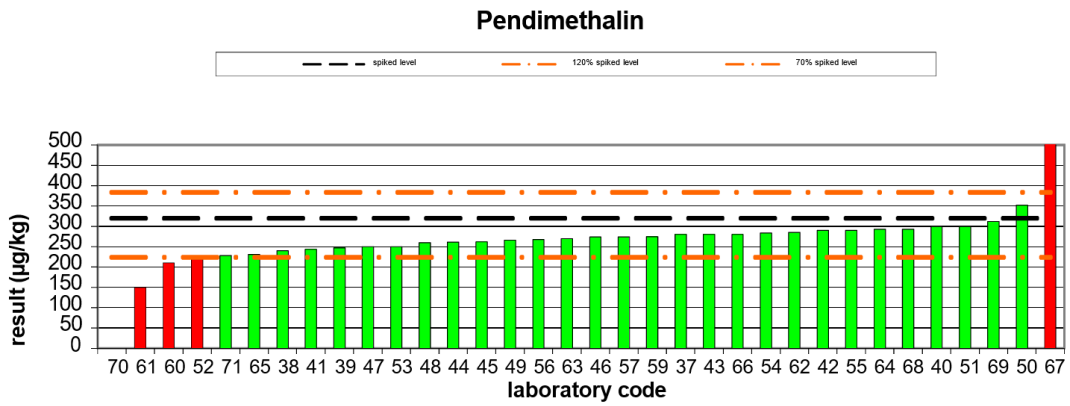

Figure 5: Assessment of Pendimethalin in test material B

7.8 Results of Pirimicarb

Table 10: Results for Pirimicarb in test material B

Pirimicarb - spiked level: 50 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 35-60
37	46	97	10	yes
38	45	88	10	yes
39	52	104	10	yes
40	50	95	10	yes
41	54	113	10	yes
42	50	91	10	yes
43	47	95	10	yes
44	52	113	5	yes
45	53	92	10	yes
46	47	102	10	yes
47	48	96.2	5	yes
48	52	110	10	yes
49	47	98	10	yes
50	53	94	10	yes
51	44	80	10	yes
52	42.56	103	10	yes
53	105	113	10	no
54	52	73	10	yes
55	46	97	10	yes
56	48	101	10	yes
57	49	100	10	yes
59	47	96	5	yes
60	48	103	10	yes
61	35	100	5	yes
62	44	72	5	yes
63	45	89	5	yes
64	57	111	10	yes
65	44	84	10	yes
66	50	93	10	yes
67	43.86	95	10	yes
68	50	100	10	yes
69	57	94	5	yes
70				no
71	48	95	10	yes

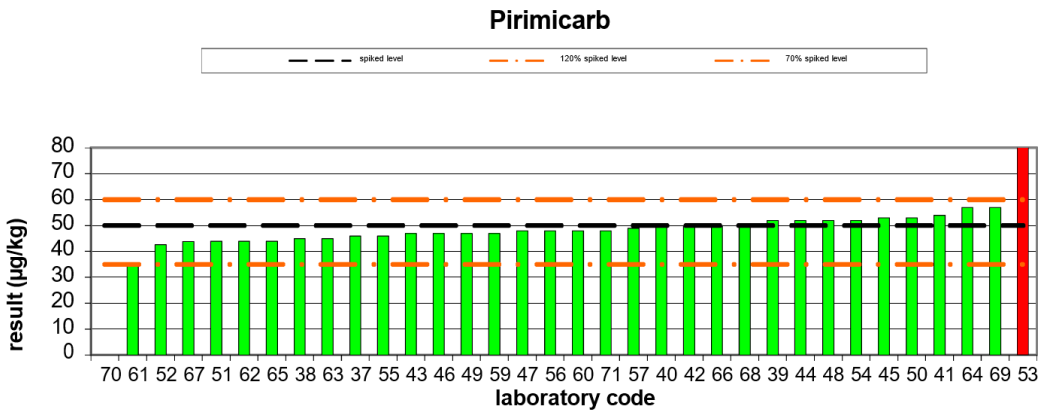

Figure 6: Assessment of Pirimicarb in test material B

7.9 Results of Spirotetramat-enol

Table 11: Results for Spirotetramat-enol in test material B

Spirotetramat-enol - spiked level: 30 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 21-36
37	27	84	10	yes
38	21	86	10	yes
39	29	105	10	yes
40	30	92	10	yes
41	28	104	10	yes
42	28	85	10	yes
43	32	95	10	yes
44	33	110	5	yes
45	27	106	10	yes
46	33	91	10	yes
47	42	95.5	5	no
48	35		10	yes
49	25	86	5	yes
50	28	102	10	yes
51	30	80	10	yes
52	47.78	95	10	no
53				no
54	35	102	10	yes
55	26	95	10	yes
56	29	96	10	yes
57	27	97	10	yes
59	23	112	10	yes
60	26	104	10	yes
61	50	90	5	no
62	21	94	10	yes
63	29	98	5	yes
64	22	100	10	yes
65	25	88	10	yes
66	21	100	10	yes
67	27.36	95	10	yes
68	31	104	10	yes
69	127	88	5	no
70				no
71	24	95	10	yes

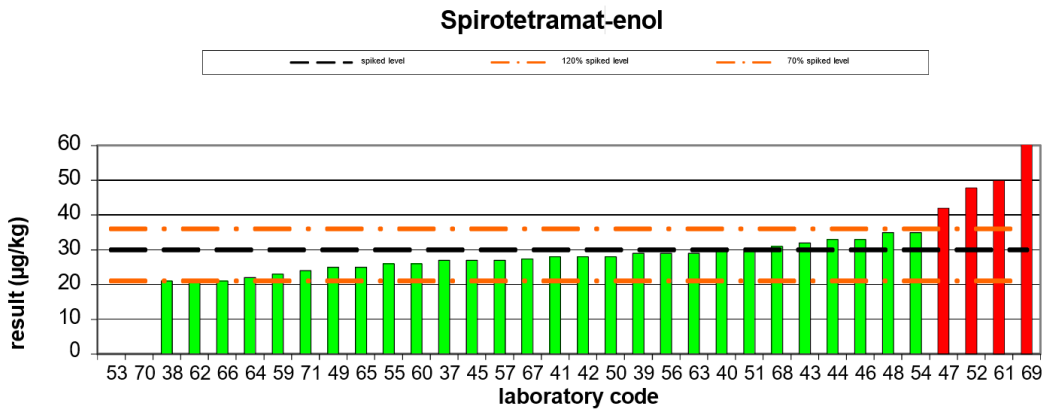

Figure 7: Assessment of Spirotetramat-enol in test material B

7.10 Results of Spirotetramat and Spirotetramat-enol

Table 12: Results for Spirotetramat and Spirotetramat-enol (sum of), expressed as Spirotetramat® in test material B

Spirotetramat and Spirotetramat-enol (sum of), expressed as Spirotetramat®				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	evaluation
37	33			not part of the evaluation
38	26			
39	36			
40	37			
41	35			
42	30			
43	40			
44	41			
45	33			
46	41			
47	52			
48	43			
49	31			
50	35			
51	37			
52	48			
53				
54	43			
55	32			
56	36			
57	33			
59	28			
60	32			
61	62			
62	26			
63	36			
64	27			
65	31			
66	26			
67	27			
68	38			
69	157			
70				
71	30			

7.11 Results of Chlorate

Table 13: Results for Chlorate in test material B

Chlorate - spiked level: 50 µg/kg				
laboratory code	result (µg/kg)	recovery (%)	LoQ (µg/kg)	accepted range (µg/kg): 35-60
37	53	119	5	yes
38	45	74	10	yes
39	71	103	10	no
40*	42	98	10	yes
41	54	120	10	yes
42	64	91	10	no
43	45	95	10	yes
44	36	110	10	yes
45	52	98	10	yes
46	45	93	10	yes
47	45	100	5	yes
48	45	97	10	yes
49	48	98	10	yes
50	51	87	10	yes
51	50	90	10	yes
52*	53	50	10	yes
53	<10		10	no
54	51	94	2	yes
55	50	96	10	yes
56	46	98	10	yes
57	48	100	10	yes
59	46	85	10	yes
60	44	89	10	yes
61	30	85	10	no
62	34	117	10	no
63	56	108	10	yes
64*	46	103	10	yes
65	50	100	10	yes
66	49	100	10	yes
67	23.45	90	10	no
68	46	103	10	yes
69	118	118	10	no
70				no
71	48	100	10	yes

* The analysis was subcontracted

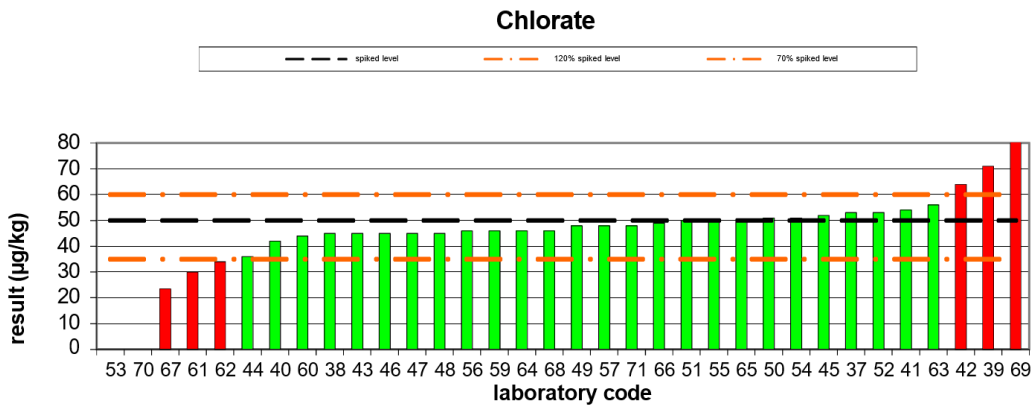

Figure 8: Assessment of Chlorate in test material B

QS Fachgesellschaft Obst-Gemüse-Kartoffeln GmbH

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