lach : bruns

Bundesverband
Naturkost Naturwaren

Laboratory Performance Assessment

Analysis of spiked Pesticides in Tomato Passata

Report

June 2020

Summary

The laboratory performance assessment related to pesticides and contaminants in tomato passata was designed and organised by Lach & Bruns in June 2020 on behalf of BNN e.V. (Bundesverband Naturkost Naturwaren, Berlin, Germany).

The test material consists of tomato passata (thus: homogenate), which was spiked with 6 analytes (4 pesticides, 1 metabolite, 1 contaminant):

Chlorate, Perchlorate, Ethephon, Glufosinate, AMPA and Phosphonic acid.

The entire batch of tomato passata was thoroughly mixed and the test material samples were distributed to thirty-six (36) participants across seven (7) European countries (Belgium, France, Germany, Greece, Italy, the Netherlands, and Spain). Information with respect to the identity of spiked analytes was provided to the laboratories in an instruction letter for analysis.

All participants handed in their results before the deadline on June 26th 2020 and were considered for evaluation.

The performance assessment considers the following test criteria:

- No *false positive results*.

- Correct *identification* of the polar analytes with an assigned or spiked value above 0,01 mg/kg (in total 6 analytes). Thus, no false negative results, except an analyte is not within the analytical scope of the particular laboratory. This is mainly related to Glufosinate and AMPA, as 5 and 3 laboratories resp. do not cover these analytes within their scope.

- Correct *quantification* of polar pesticides / contaminants (Perchlorate) in terms of accuracy (trueness) or comparability (Phosphonic acid only).

- Reasonable reporting limits.

Based on these criteria, the laboratories were marked with one out of 4 marks, ranging from "*excellent*" to "*good*" to "*acceptable*" to "*insufficient*". Laboratories with an excellent (20) or good (8) result clearly satisfy the quality standards of the BNN, while labs with an acceptable (4) result satisfy these standards with some deficiency. Laboratories evaluated to have provided insufficient (4) results do not satisfy the expected requirements of the BNN.

Assessment of quantification

Analytical results within 70% - 120% of the spike or, in case of Phosphonic acid, a z-score of |z| ≤ 2 are considered satisfying for the assessment of the correct quantification of the analytes.

The overall performance is summarised as:

Pesticide	Spiked value [mg/kg]	Number of results	Correct quantification
Chlorate	0,035	36	30 out of 36 (%)
Perchlorate	0,045	36	33 out of 36 (97 %)
Ethephon	0,025	36	33 out of 36 (100 %)
Glufosinate	0,030	31[*1]	27 out of 31 (97 %)
AMPA	0,022	33[*2]	30 out of 33 (92 %)
Phosphonic acid	0,0862 (AV)	33[*3]	31 out of 33 (94 %)

AV = assigned value
[*1] Some labs (5 in total) do not cover this substance in their scope of analysis
[*2] Some labs (3 in total) do not cover this substance in their scope of analysis
[*3] Two labs did not report any result while one reported < 0,1 mg/kg, which could not be included in the z-score analysis.

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1. Test material preparation and design

The laboratory performance assessment was designed to verify the analytical competence related to BNN module-combination "A1 (pesticides) – B1/B2 (fruits and vegetables, fresh or processed.

Organic tomato passata was used for preparation of the test material. A sub-sample was taken and analysed to ensure that no incurred residues of pesticides (> 0,01 mg/kg) are present. The analysis of the sub-sample only showed positive levels of Phosphonic acid above the reporting limit of 0,01 mg/kg. Due to this, Phosphonic acid was evaluated applying the comparability criterion - as opposed to the accuracy (trueness) criterion.

The passata was spiked with a solution of six analytes to give the final concentrations of 0,035 mg/kg Chlorate, 0,045 mg/kg Perchlorate, 0,025 mg/kg Ethephon, 0,030 mg/kg Glufosinate and 0,022 mg/kg AMPA. 0,060 mg/kg Phosphonic acid also were spiked to the test material, to which the incurred residue seems to have added another 0,0262 mg/kg to give an assigned value (AV) of 0,0862 mg/kg.

Stirring was continued after spiking in order to ensure the homogeneity of the test material. Additional subsamples were prepared in addition to the test samples for the verification of the spiked amount of analytes and for the stability testing.
The test materials were stored at -18°C in the dark until distribution.

The sample, which was used for the stability testing was bottled and sealed with a lid. The bottle was stored at room temperature before stability testing was initiated. The stability testing was performed after the last participant reported the results. Results showed sufficient stability across the entire test period.

The samples were sent to the participants 22nd June 2020 thus arriving 23rd/24th June at the participating laboratories. Reporting of results was scheduled for 26th June 2020. Sending conditions were as follows: The test samples were stored in Styrofoam boxes and cooled with dry ice.

Design of BNN tomato passata test material and homogeneity testing

Analyte	Spiked level (mg/kg)	Homogeneity test: average results (mg/kg)	spike level recovery rate (in %)
AMPA	0,022	0,024	109
Chlorate	0,035	0,031	89
Ethephon	0,025	0,018	72
Glufosinate	0,030	0,028	93
Perchlorate	0,045	0,057	127
Phosphonic acid (PhAc)	0,060 plus additional incurred PhAc	0,066	-/-

Remark: Results of Phosphonic acid showed homogeneity within the test material. However, as the spiked level could not be considered for the accuracy (trueness) criterion, the reference for the assessment is the assigned value and not the spiked level.

2. Statistical evaluation of results

Accuracy (trueness) criterion

The trueness criterion considers the correct quantification of the actual analyte concentration in the sample. The trueness of the results is assessed as the coverage of the spiked level in %. The coverage of the spiked level is calculated according to the equation below:

$$coverage\ of\ the\ spiked\ level = \frac{x}{sl} * 100$$

(x = reported result; sl = spiked level)

Accepted range:

Results, which correspond to a recovery of 70 to 120 % of the spiked level, are considered satisfying in this laboratory performance assessment.

Comparability criterion

The comparability of results is evaluated according to the z-score model based on an assigned value and the target standard deviation (acc. to Horwitz).

Assigned value

The assigned value x_{pt} is the robust mean, which is derived from the results of the participants according to ISO13528, Algorithm A [1]. The winsorisation algorithm is applied to minimise the influence of outliers.

The assigned values are subject to commercial rounding and are presented with an accuracy of three significant figures.

z-score

The z-score is derived of the result *xi* of each participant, the assigned value *xpt* and the target standard deviation according to Horwitz *σH2*,[2]:

$$z - score = \frac{x_i - x_{pt}}{\sigma_H}$$

Analytical results with a z-score of |z| ≤ 2 are considered satisfying for the assessment of the correct quantification of the pesticides.

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[1] Statistical methods for use in proficiency testing by interlaboratory comparison. ISO 13528:2015. Corrected version 2016-10-15.

[2] Horwitz W. Evaluation of Analytical Methods Used for Regulation of Foods and Drugs. Anal Chem. 1982;54(1):67A–76A.

3. Results

The laboratories received the test samples without prior announcement. Upon receipt of the test sample, the laboratories were informed about the test, the type of test material and the scope of the test by an enclosed instruction letter. The laboratories were requested to analyse for "polar pesticides" and related metabolites resp. contaminants covered by the QuPPe approach and/or additional single residue methods.

Thirty-six (36) participants across seven (7) European countries (Belgium, France, Germany, Greece, Italy, the Netherlands, and Spain) took part in the laboratory performance assessment. All participants handed in their results in time and were considered for evaluation. Each laboratory was given a randomly selected identifier, hereinafter referred to as laboratory code.

The laboratories were to report all sought and found pesticides, the reporting limits (RL) as well as the scope of the applied analytical methods.

A summary of the overall performance of the labs is provided in table 2. A more detailed evaluation of the results of the participants is presented in tables 3 to 8 and in figures 1 to 6.

A number of laboratories reported findings of Glyphosate below the reporting limit. These are however not listed here due to lack of significance for this test.

Additionally, the performance of the participants has been evaluated based on the BNN requirements using the following criteria:

- No *false positive results*.

- Correct *identification* of the polar analytes with an assigned or spiked value above 0,01 mg/kg (in total 6 analytes). Thus, no false negative results, except a pesticide is not within the analytical scope of the particular laboratory. This is mainly related to Glufosinate and AMPA, as 5 and 3 laboratories resp. do not cover these analytes within their scope.

- Correct *quantification* of polar pesticides / contaminants (Perchlorate) in terms of accuracy (trueness) or comparability (Phosphonic acid only).

- Reasonable reporting limits.

The performance is evaluated as:

excellent: all criteria fulfilled (6 out of 6 results in conformity with BNN criteria);

good: single slight errors (min. 5 out of 6 results in conformity with BNN criteria) OR all criteria fulfilled but one or more substances out of scope;

acceptable: two slight errors (more than 50% correct: min. 4 out of 6 resp. 2 out of 3 results in conformity with BNN criteria) OR single extreme errors (f. ex. more than 200 % recovery of spike);

insufficient: 50% or more erroneous reporting AND/OR several extreme errors.

Results in detail

- Apart from Phosphonic acid all substances were successfully identified by all laboratories, unless the substance was not included in the scope of the method employed.

- AMPA was successfully identified by 33 out of 36 labs, while 3 did not have it included in their scope of analysis. For Glufosinate this number was 5, while the other 31 labs successfully identified the substance.

- Chlorate was correctly quantified 30 times, with 36 results being submitted, Perchlorate and Ethephon were successfully quantified 33 out of 36 times. Glufosinate was successfully quantified in 27 out of 31 results, while AMPA was successfully quantified 30 out of 33 times.

- In the case of Phosphonic acid, this substance was identified 34 times, with one result being noted as < 0,1 mg/kg (RL). Two labs did not report on Phosphonic acid. Out of 33 evaluated results 31 passed the comparability criterion. Lab 32 (< 0,1 mg/kg) was not classified to have a false-negative result, as they detected Phosphonic acid at < 0,1 mg/kg. In case of Lab 10, the minimum of the accepted range (0,0483 mg/kg) is very close to the reporting limit (0,05 mg/kg). Therefore, a detection was to be reasonably expected here - but not reported. Consequently, the laboratory was evaluated to have a false negative result.

   Excellent results (20, sorted by lab code):

- Lab 1 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 9% and a z-score of 0,7.

- Lab 3 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 6% and a z-score of -0,2.

- Lab 5 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 5% and a z-score of 0,7.

- Lab 6 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 8% and a z-score of 0,1.

- Lab 7 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 13% and a z-score of 1,3.

- Lab 8 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 9% and a z-score of -0,2.

- Lab 11 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 9% and a z-score of -0,4.

- Lab 13 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 4% and a z-score of -0,2.

- Lab 15 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 7% and a z-score of 1,8.

- Lab 16 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 5% and a z-score of 0,1.

- Lab 17 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 16% and a z-score of 0,1.

- Lab 18 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 8% and a z-score of -1,0.

- Lab 23 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 12% and a z-score of -0,1.

- Lab 26 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 7% and a z-score of 0,6.

- Lab 27 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 7% and a z-score of -0,6.

- Lab 28 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 9% and a z-score of 0,1.

- Lab 31 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 6% and a z-score of -0,6.

- Lab 34 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 19% and a z-score of 0,6.

- Lab 35 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 9% and a z-score of -1,3.

- Lab 36 successfully passed the criteria for all 6 substances with an average deviation from the spiked value of 5% and a z-score of 0,4.

Good results (8, sorted by lab code):

- Lab 12 passed 4 of the 5 trueness criteria with an average deviation from the spiked value of 11% and passed the comparability criterion with a z-score of -0,9.

- Lab 19 passed 4 of the 5 trueness criteria with an average deviation from the spiked value of 17% and passed the comparability criterion with a z-score of -0,1.

- Lab 20 successfully passed the criterion for all 4 substances included in their scope with an average deviation from the spiked value of 8% and a z-score of 0,7.

- Lab 21 passed 4 of the 5 trueness criteria with an average deviation from the spiked value of 8% and passed the comparability criterion with a z-score of 0,4.

- Lab 25 passed 4 of the 5 trueness criteria with an average deviation from the spiked value of 12% and passed the comparability criterion with a z-score of 1,3.

- Lab 29 passed 4 of the 5 trueness criteria with an average deviation from the spiked value of 15% and passed the comparability criterion with a z-score of -0,6.

- Lab 30 successfully passed all 5 trueness criteria with an average deviation from the spiked value of 11% but failed to pass the comparability criterion with a z-score of 2,3.

- Lab 32 successfully passed all 4 trueness criteria for the substances included in their scope with an average deviation from the spiked value of 1%, but was excluded from z-score analysis, as the reporting limit is too high related to the assigned value and to BNN requirements.

Acceptable results (4, sorted by lab code):

- Lab 2 passed 4 of the 5 trueness criteria with an average deviation from the spiked value of 35% and passed the comparability criterion with a z-score of -0,8. The reported result of Glufosinate is 243% of the spike and thus evaluated as a strong error.

- Lab 9 passed 4 of the 5 trueness criteria with an average deviation from the spiked value of 16% and failed to pass the comparability criterion with a z-score of -2,3.

- Lab 22 passed 3 of the 5 trueness criteria with an average deviation from the spiked value of 18% and passed the comparability criterion with a z-score of 0,7.

- Lab 33 passed 3 of the 5 trueness criteria with an average deviation from the spiked value of 15% and passed the comparability criterion with a z-score of 0,2.

Insufficient results (4, sorted by lab code):

- Lab 4 passed 1 of the 3 trueness criteria related to the analytes included in their scope with an average deviation from the spiked value of 22%. The lab passed the comparability criterion with a z-score of -1,0. This overall result is not satisfactory.

- Lab 10 passed 3 of the 4 trueness criteria related to the analytes included in their scope with an average deviation from the spiked value of 15%. The lab failed to identify Phosphonic acid, meaning they are the only lab with a false-negative result in the test. This overall result is not satisfactory.

- Lab 14 passed 2 of the 3 trueness criteria related to the analytes included in their scope with an average deviation from the spiked value of 18%. The lab was excluded from z-score analysis, as the reporting limit is too high related to the assigned value and to BNN requirements. This overall result is not satisfactory.

- Lab 24 passed 1 of the 5 trueness criteria with an average deviation from the spiked value of 56%. The lab passed the comparability criterion with a z-score of -1,4. This overall result is not satisfactory.

4. Overview of laboratory evaluation

Table 1: laboratory evaluation

lab code	trueness achieved / analytes reported	avg. dev. from spike [%]	z-score (phos. acid)	comparability crit. passed	Final evaluation based on reported results[2]
1	5 / 5	9	0,7	Yes	excellent
2	4 / 5	35	-0,8	Yes	acceptable
3	5 / 5	6	-0,2	Yes	excellent
4	1 / 3	22	-1,0	Yes	insufficient
5	5 / 5	5	0,7	Yes	excellent
6	5 / 5	8	0,1	Yes	excellent
7	5 / 5	13	1,3	Yes	excellent
8	5 / 5	9	-0,2	Yes	excellent
9	4 / 5	16	-2,3	No	acceptable
10	3 / 4	15	-4,5	No	insufficient
11	5 / 5	9	-0,4	Yes	excellent
12	4 / 5	11	-0,9	Yes	good
13	5 / 5	4	-0,2	Yes	excellent
14	2 / 3	18	/[1]	No	insufficient[3]
15	5 / 5	7	1,8	Yes	excellent
16	5 / 5	5	0,1	Yes	excellent
17	5 / 5	16	0,1	Yes	excellent
18	5 / 5	8	-1,0	Yes	excellent
19	4 / 5	17	-0,1	Yes	good
20	3 / 3	8	0,7	Yes	good
21	4 / 5	8	0,4	Yes	good
22	3 / 5	18	0,7	Yes	acceptable
23	5 / 5	12	-0,1	Yes	excellent
24	1 / 5	56	-1,4	Yes	insufficient
25	4 / 5	12	1,3	Yes	good
26	5 / 5	7	0,6	Yes	excellent
27	5 / 5	7	-0,6	Yes	excellent
28	5 / 5	9	0,1	Yes	excellent
29	4 / 5	15	-0,6	Yes	good
30	5 / 5	11	2,3	No	good
31	5 / 5	6	-0,6	Yes	excellent
32	4 / 4	1	/[1]	No	barely good[4]
33	3 / 5	15	0,2	Yes	acceptable
34	5 / 5	19	0,6	Yes	excellent
35	5 / 5	9	-1,3	Yes	excellent
36	5 / 5	5	0,4	Yes	excellent

[1]: RL (reporting limit) > AV (assigned value)

[2]: **excellent**: all criteria fulfilled (6 out of 6 results in conformity with BNN criteria); **good**: single slight errors (min. 5 out of 6 results in conformity with BNN criteria) OR all criteria fulfilled but one or more substances out of scope; **acceptable**: two slight errors (more than 50% correct: min. 4 out of 6 resp. 2 out of 3 results in conformity with BNN criteria) OR single extreme errors (f. ex. more than 200 % recovery of spike); **insufficient**: 50% or more erroneous reporting AND/OR several extreme errors.

[3]: Lab 14 was rated insufficient due to an unreasonably high reporting limit for Phosphonic acid and thus only two out of four analytes within their scope were reported according to BNN requirements.

[4]: Lab 32 reported Phosphonic acid as "detected, below 0,1 mg/kg". This is not considered as false-negative result.

5. Tables and figures

Table 2: Summary of the overall performance

	Chlorate		Perchlorate		Ethephon		Glufosinate		AMPA		trueness achieved / analytes reported	average deviation from spike[%]	Phosphonic acid	
	result relative to spike [%]	criterion passed	result relative to spike [%]	criterion passed	result relative to spike [%]	criterion passed	result relative to spike [%]	criterion passed	result relative to spike [%]	criterion passed			z-score	criterion passed
1	117	Yes	118	Yes	92	Yes	103	Yes	100	Yes	5 / 5	9	0,7	Yes
2	97	Yes	91	Yes	96	Yes	243	No	114	Yes	4 / 5	35	-0,8	Yes
3	103	Yes	109	Yes	96	Yes	110	Yes	95	Yes	5 / 5	6	-0,2	Yes
4	134	No	127	No	96	Yes	o. o. s.	/	o. o. s.	/	1 / 3	22	-1,0	Yes
5	103	Yes	102	Yes	100	Yes	103	Yes	118	Yes	5 / 5	5	0,7	Yes
6	97	Yes	100	Yes	108	Yes	120	Yes	91	Yes	5 / 5	8	0,1	Yes
7	103	Yes	102	Yes	80	Yes	117	Yes	77	Yes	5 / 5	13	1,3	Yes
8	109	Yes	102	Yes	104	Yes	107	Yes	77	Yes	5 / 5	9	-0,2	Yes
9	83	Yes	116	Yes	108	Yes	63	No	95	Yes	4 / 5	16	-2,3	No
10	80	Yes	102	Yes	84	Yes	o. o. s.	/	123	No	3 / 4	15	-4,5	No
11	120	Yes	109	Yes	108	Yes	103	Yes	105	Yes	5 / 5	9	-0,4	Yes
12	123	No	96	Yes	100	Yes	110	Yes	118	Yes	4 / 5	11	-0,9	Yes
13	100	Yes	100	Yes	96	Yes	117	Yes	100	Yes	5 / 5	4	-0,2	Yes
14	114	Yes	124	No	116	Yes	o. o. s.	/	o. o. s.	/	2 / 3	18	/*1	No
15	111	Yes	100	Yes	88	Yes	97	Yes	91	Yes	5 / 5	7	1,8	Yes
16	97	Yes	100	Yes	108	Yes	107	Yes	109	Yes	5 / 5	5	0,1	Yes
17	103	Yes	89	Yes	76	Yes	73	Yes	114	Yes	5 / 5	16	0,1	Yes
18	106	Yes	102	Yes	96	Yes	120	Yes	91	Yes	5 / 5	8	-1,0	Yes

Table 2 (continued):   Summary of the overall performance

	Chlorate		Perchlorate		Ethephon		Glufosinate		AMPA		trueness achieved / analytes reported	average deviation from spike[%]	Phosphonic acid	
	result relative to spike [%]	criterion passed	result relative to spike [%]	criterion passed	result relative to spike [%]	criterion passed	result relative to spike [%]	criterion passed	result relative to spike [%]	criterion passed			z-score	criterion passed
19	137	No	111	Yes	96	Yes	117	Yes	118	Yes	4 / 5	17	-0,1	Yes
20	86	Yes	107	Yes	96	Yes	o. o. s.	/	o. o. s.	/	3 / 3	8	0,7	Yes
21	100	Yes	102	Yes	104	Yes	107	Yes	127	No	4 / 5	8	0,4	Yes
22	129	No	118	Yes	108	Yes	133	No	105	Yes	3 / 5	18	0,7	Yes
23	86	Yes	89	Yes	96	Yes	120	Yes	109	Yes	5 / 5	12	-0,1	Yes
24	51	No	51	No	204	No	40	No	82	Yes	1 / 5	56	-1,4	Yes
25	111	Yes	98	Yes	132	No	103	Yes	109	Yes	4 / 5	12	1,3	Yes
26	106	Yes	113	Yes	84	Yes	100	Yes	100	Yes	5 / 5	7	0,6	Yes
27	109	Yes	100	Yes	96	Yes	117	Yes	105	Yes	5 / 5	7	-0,6	Yes
28	106	Yes	89	Yes	88	Yes	97	Yes	86	Yes	5 / 5	9	0,1	Yes
29	91	Yes	107	Yes	56	No	113	Yes	95	Yes	4 / 5	15	-0,6	Yes
30	111	Yes	98	Yes	104	Yes	120	Yes	82	Yes	5 / 5	11	2,3	No
31	89	Yes	96	Yes	92	Yes	100	Yes	105	Yes	5 / 5	6	-0,6	Yes
32	100	Yes	100	Yes	100	Yes	o. o. s.	/	95	Yes	4 / 4	1	/*1	No
33	123	No	82	Yes	108	Yes	100	Yes	127	No	3 / 5	15	0,2	Yes
34	74	Yes	84	Yes	80	Yes	120	Yes	86	Yes	5 / 5	19	0,6	Yes
35	111	Yes	98	Yes	104	Yes	120	Yes	91	Yes	5 / 5	9	-1,3	Yes
36	97	Yes	111	Yes	100	Yes	100	Yes	109	Yes	5 / 5	5	0,4	Yes

o. o. s.: Out of scope.
*1: RL (reporting limit) > assigned value

Table 3: Results of Chlorate

Lab code	result [mg/kg]	RL (reporting-limit) [mg/kg]	relative to spike [%]	relative value (satisfactory)
Chlorate				

	Chlorate			
	spiked value [mg/kg]: 0,035		AV [mg/kg]: 0,0363 (104%)	
	Accepted range [%]: 70 - 120		Accepted range [mg/kg]: 0,0245 – 0,0420	
Lab code	**result [mg/kg]**	**RL (reporting-limit) [mg/kg]**	**relative to spike [%]**	**relative value (satisfactory)**
1	0,041	0,01	117	Yes
2	0,034	0,01	97	Yes
3	0,036	0,01	103	Yes
4	0,047	0,01	134	No
5	0,036	0,01	103	Yes
6	0,034	0,01	97	Yes
7	0,036	0,010	103	Yes
8	0,038	0,01	109	Yes
9	0,029	0,01	83	Yes
10	0,028	0,01	80	Yes
11	0,042	0,010	120	Yes
12	0,043	0,010	123	No
13	0,035	0,010	100	Yes
14	0,04	0,01	114	Yes
15	0,039	0,010	111	Yes
16	0,034	0,01	97	Yes
17	0,036	0,01	103	Yes
18	0,037	0,010	106	Yes
19	0,048	0,01	137	No
20	0,030	0,01	86	Yes
21	0,035	0,01	100	Yes
22	0,045	0,005	129	No
23	0,0300	0,01	86	Yes
24	0,018	0,01	51	No
25	0,039	0,01	111	Yes
26	0,037	0,01	106	Yes
27	0,038	0,010	109	Yes
28	0,037	0,01	106	Yes
29	0,032	0,010	91	Yes
30	0,039	0,01	111	Yes
31	0,031	0,01	89	Yes
32	0,035	0,01	100	Yes
33	0,043	0,010	123	No
34	0,026	0,01	74	Yes
35	0,039	0,01	111	Yes
36	0,034	0,01	97	Yes

Chlorate (trueness criterion) - accepted range: spike x 0,7 ≤ reported result ≤ spike x 1,2
AV = assigned value, in brackets: percentage relative to spike (data for information only)

Table 4: Results of Perchlorate

Lab code	result [mg/kg]	RL (reporting-limit) [mg/kg]	relative to spike [%]	relative value (satisfactory)
Perchlorate				
spiked value [mg/kg]: 0,045			AV [mg/kg]: 0,0458 (102%)	
Accepted range [%]: 70 - 120			Accepted range [mg/kg]: 0,0315 – 0,0540	
1	0,053	0,01	118	Yes
2	0,041	0,01	91	Yes
3	0,049	0,01	109	Yes
4	0,057	0,01	127	No
5	0,046	0,01	102	Yes
6	0,045	0,01	100	Yes
7	0,046	0,010	102	Yes
8	0,046	0,01	102	Yes
9	0,052	0,01	116	Yes
10	0,046	0,1	102	Yes
11	0,049	0,010	109	Yes
12	0,043	0,010	96	Yes
13	0,045	0,010	100	Yes
14	0,056	0,01	124	No
15	0,045	0,002	100	Yes
16	0,045	0,01	100	Yes
17	0,040	0,010	89	Yes
18	0,046	0,010	102	Yes
19	0,050	0,01	111	Yes
20	0,048	0,01	107	Yes
21	0,046	0,01	102	Yes
22	0,053	0,002	118	Yes
23	0,040	0,01	89	Yes
24	0,023	0,01	51	No
25	0,044	0,01	98	Yes
26	0,051	0,01	113	Yes
27	0,045	0,010	100	Yes
28	0,040	0,01	89	Yes
29	0,048	0,010	107	Yes
30	0,044	0,01	98	Yes
31	0,043	0,01	96	Yes
32	0,045	0,01	100	Yes
33	0,037	0,002	82	Yes
34	0,038	0,01	84	Yes
35	0,044	0,01	98	Yes
36	0,050	0,01	111	Yes

Perchlorate (trueness criterion) - accepted range: spike x 0,7 ≤ reported result ≤ spike x 1,2
AV = assigned value, in brackets: percentage relative to spike (data for information only)

Table 5: Results of Ethephon

Lab code	result [mg/kg]	RL (reporting-limit) [mg/kg]	relative to spike [%]	relative value (satisfactory)
**Ethephon**			
	spiked value [mg/kg]: 0,025			AV [mg/kg]: 0,0245 (98%)
	Accepted range [%]: 70 - 120			Accepted range [mg/kg]: 0,0175 – 0,0300
1	0,023	0,01	92	Yes
2	0,024	0,01	96	Yes
3	0,024	0,01	96	Yes
4	0,024	0,01	96	Yes
5	0,025	0,01	100	Yes
6	0,027	0,01	108	Yes
7	0,020	0,010	80	Yes
8	0,026	0,01	104	Yes
9	0,027	0,01	108	Yes
10	0,021	0,01	84	Yes
11	0,027	0,010	108	Yes
12	0,025	0,010	100	Yes
13	0,024	0,010	96	Yes
14	0,029	0,01	116	Yes
15	0,022	0,010	88	Yes
16	0,027	0,01	108	Yes
17	0,019	0,01	76	Yes
18	0,024	0,010	96	Yes
19	0,024	0,01	96	Yes
20	0,024	0,02	96	Yes
21	0,026	0,01	104	Yes
22	0,027	0,01	108	Yes
23	0,0240	0,01	96	Yes
24	0,051	0,01	204	No
25	0,033	0,01	132	No
26	0,021	0,01	84	Yes
27	0,024	0,010	96	Yes
28	0,022	0,01	88	Yes
29	0,014	0,01	56	No
30	0,026	0,01	104	Yes
31	0,023	0,01	92	Yes
32	0,025	0,01	100	Yes
33	0,027	0,010	108	Yes
34	0,020	0,01	80	Yes
35	0,026	0,01	104	Yes
36	0,025	0,01	100	Yes

Ethephon (trueness criterion) - accepted range: spike x 0,7 ≤ reported result ≤ spike x 1,2
AV = assigned value, in brackets: percentage relative to spike (data for information only)

Table 6: Results of Glufosinate

	Glufosinate			
	spiked value [mg/kg]: 0,030		AV [mg/kg]: 0,0326 (109%)	
	Accepted range [%]: 70 - 120		Accepted range [mg/kg]: 0,0210 – 0,0360	
Lab code	**result [mg/kg]**	**RL (reporting-limit) [mg/kg]**	**relative to spike [%]**	**relative value (satisfactory)**
1	0,031	0,01	103	Yes
2	0,073	0,01	243	No
3	0,033	0,01	110	Yes
4	/	oos	/	/
5	0,031	0,01	103	Yes
6	0,036	0,01	120	Yes
7	0,035	0,010	117	Yes
8	0,032	0,01	107	Yes
9	0,019	0,01	63	No
10	/	oos	/	/
11	0,031	0,010	103	Yes
12	0,033	0,010	110	Yes
13	0,035	0,010	117	Yes
14	/	oos	/	/
15	0,029	0,010	97	Yes
16	0,032	0,01	107	Yes
17	0,022	0,01	73	Yes
18	0,036	0,01	120	Yes
19	0,035	0,01	117	Yes
20	/	oos	/	/
21	0,032	0,1	107	Yes
22	0,040	0,01	133	No
23	0,036	0,01	120	Yes
24	0,012	0,01	40	No
25	0,031	0,01	103	Yes
26	0,030	0,01	100	Yes
27	0,035	0,010	117	Yes
28	0,029	0,01	97	Yes
29	0,034	0,010	113	Yes
30	0,036	0,01	120	Yes
31	0,030	0,01	100	Yes
32	/	oos	/	/
33	0,030	0,010	100	Yes
34	0,036	0,01	120	Yes
35	0,036	0,01	120	Yes
36	0,030	0,01	100	Yes

Glufosinate (trueness criterion) - accepted range: spike x 0,7 ≤ reported result ≤ spike x 1,2
oos: out of scope; AV = assigned value, in brackets: percentage relative to spike (data for information only)

Table 7: Results of AMPA

Lab code	result [mg/kg]	RL (reporting-limit) [mg/kg]	relative to spike [%]	relative value (satisfactory)
	AMPA			
	spiked value [mg/kg]: 0,022		AV [mg/kg]: 0,0223 (101%)	
	Accepted range [%]: 70 - 120		Accepted range [mg/kg]: 0,0154 – 0,0264	
1	0,022	0,01	100	Yes
2	0,025	0,01	114	Yes
3	0,0210	0,01	95	Yes
4	/	oos	/	/
5	0,026	0,01	118	Yes
6	0,02	0,01	91	Yes
7	0,017	0,010	77	Yes
8	0,017	0,01	77	Yes
9	0,021	0,01	95	Yes
10	0,027	0,02	123	No
11	0,023	0,010	105	Yes
12	0,026	0,010	118	Yes
13	0,022	0,010	100	Yes
14	/	oos	/	/
15	0,020	0,010	91	Yes
16	0,024	0,01	109	Yes
17	0,025	0,01	114	Yes
18	0,020	0,010	91	Yes
19	0,026	0,01	118	Yes
20	/	oos	/	/
21	0,028	0,01	127	No
22	0,023	0,01	105	Yes
23	0,024	0,01	109	Yes
24	0,018	0,01	82	Yes
25	0,024	0,01	109	Yes
26	0,022	0,01	100	Yes
27	0,023	0,010	105	Yes
28	0,019	0,01	86	Yes
29	0,021	0,010	95	Yes
30	0,018	0,01	82	Yes
31	0,023	0,01	105	Yes
32	0,021	0,02	95	Yes
33	0,028	0,010	127	No
34	0,019	0,01	86	Yes
35	0,020	0,01	91	Yes
36	0,024	0,01	109	Yes

AMPA (trueness criterion) - accepted range: spike x 0,7 ≤ reported result ≤ spike x 1,2
oos: out of scope; AV = assigned value, in brackets: percentage relative to spike; data for information only

Table 8: Results of Phosphonic acid

Lab code	result [mg/kg]	RL (reporting-limit) [mg/kg]	z-score	z-score |z| ≤ 2 (satisfactory)
**Phosphonic acid**			
	Assigned value [mg/kg]		0,0862	
	Accepted range [mg/kg]	0,0483	-	0,1242
1	0,10	0,10	0,7	Yes
2	0,072	0,05	-0,8	Yes
3	0,082	0,01	-0,2	Yes
4	0,068	0,025	-1,0	Yes
5	0,099	0,01	0,7	Yes
6	0,088	0,05	0,1	Yes
7	0,11	0,010	1,3	Yes
8	0,082	0,01	-0,2	Yes
9	0,042	0,01	-2,3	No
10	n.d.	0,05	-4,5	No
11	0,078	0,010	-0,4	Yes
12	0,070	0,010	-0,9	Yes
13	0,083	0,010	-0,2	Yes
14	n.d.	0,1	/*1	No
15	0,12	0,10	1,8	Yes
16	0,089	0,05	0,1	Yes
17	0,0890	0,01 (Fosetyl-Al)	0,1	Yes
18	0,067	0,05	-1,0	Yes
19	0,085	0,01	-0,1	Yes
20	0,100	0,05	0,7	Yes
21	0,094	0,01	0,4	Yes
22	0,10	0,01	0,7	Yes
23	0,084	0,01	-0,1	Yes
24	0,059	0,01	-1,4	Yes
25	0,11	0,1	1,3	Yes
26	0,097	0,01	0,6	Yes
27	0,075	0,050	-0,6	Yes
28	0,088	0,01	0,1	Yes
29	0,075	0,050	-0,6	Yes
30	0,129	0,05	2,3	No
31	0,074	0,01	-0,6	Yes
32	<0,1	0,1	/*2	No
33	0,090	0,010	0,2	Yes
34	0,097	0,01	0,6	Yes
35	0,062	0,05	-1,3	Yes
36	0,093	0,01	0,4	Yes

Phosphonic acid (comparability criterion): Accepted range z-score of |z| ≤ 2   /   n.d. = not detected
*1: Lab 14 reported no result (n.d.). The reporting limit is not as low as necessary to identify and quantify the related level of Phosphonic acid in the test sample.
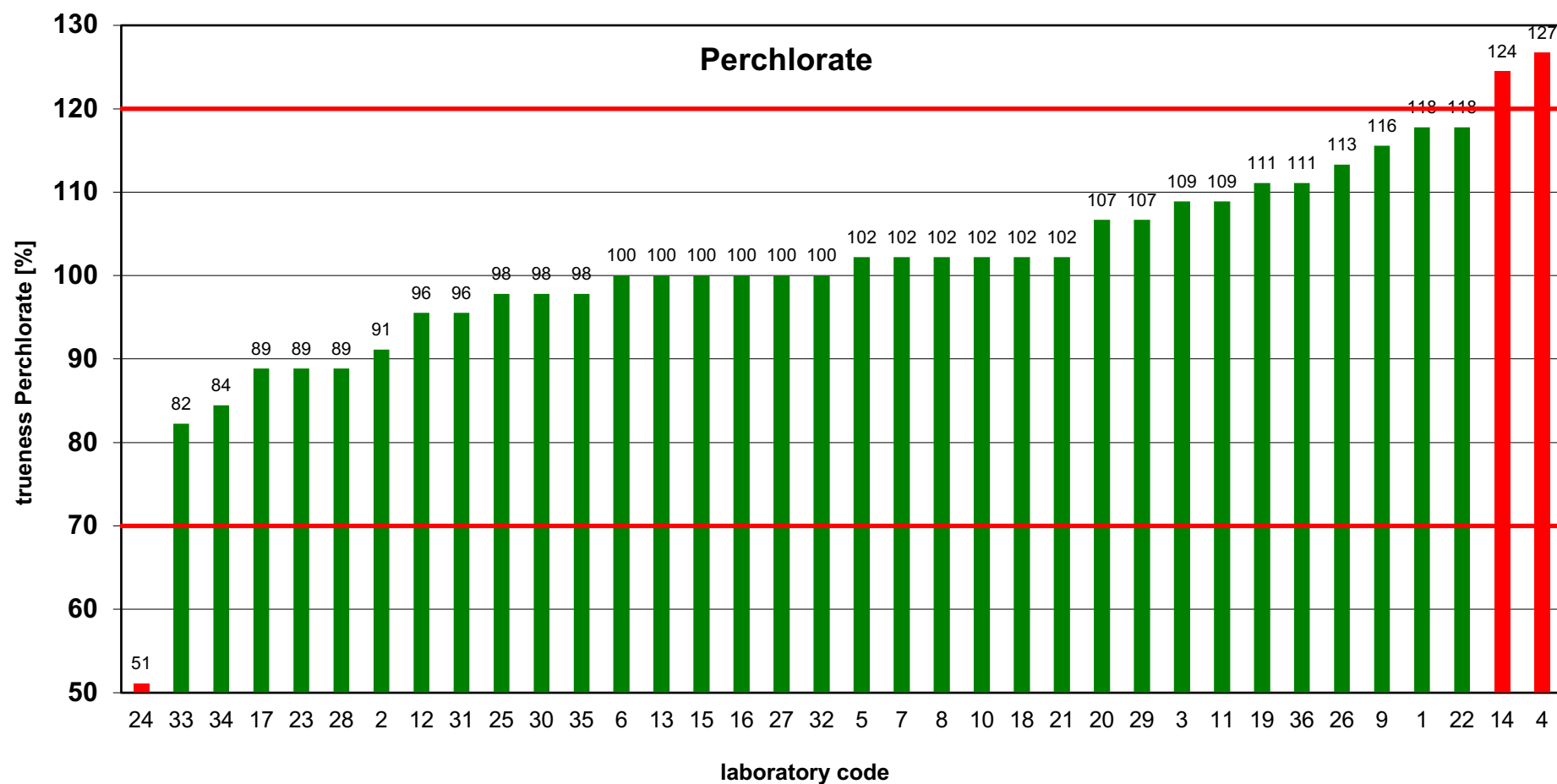*2: Lab 32 reported "detected, < 0,1 mg/kg". The reporting limit is not as low as necessary to quantify the related level of Phosphonic acid in the test sample.
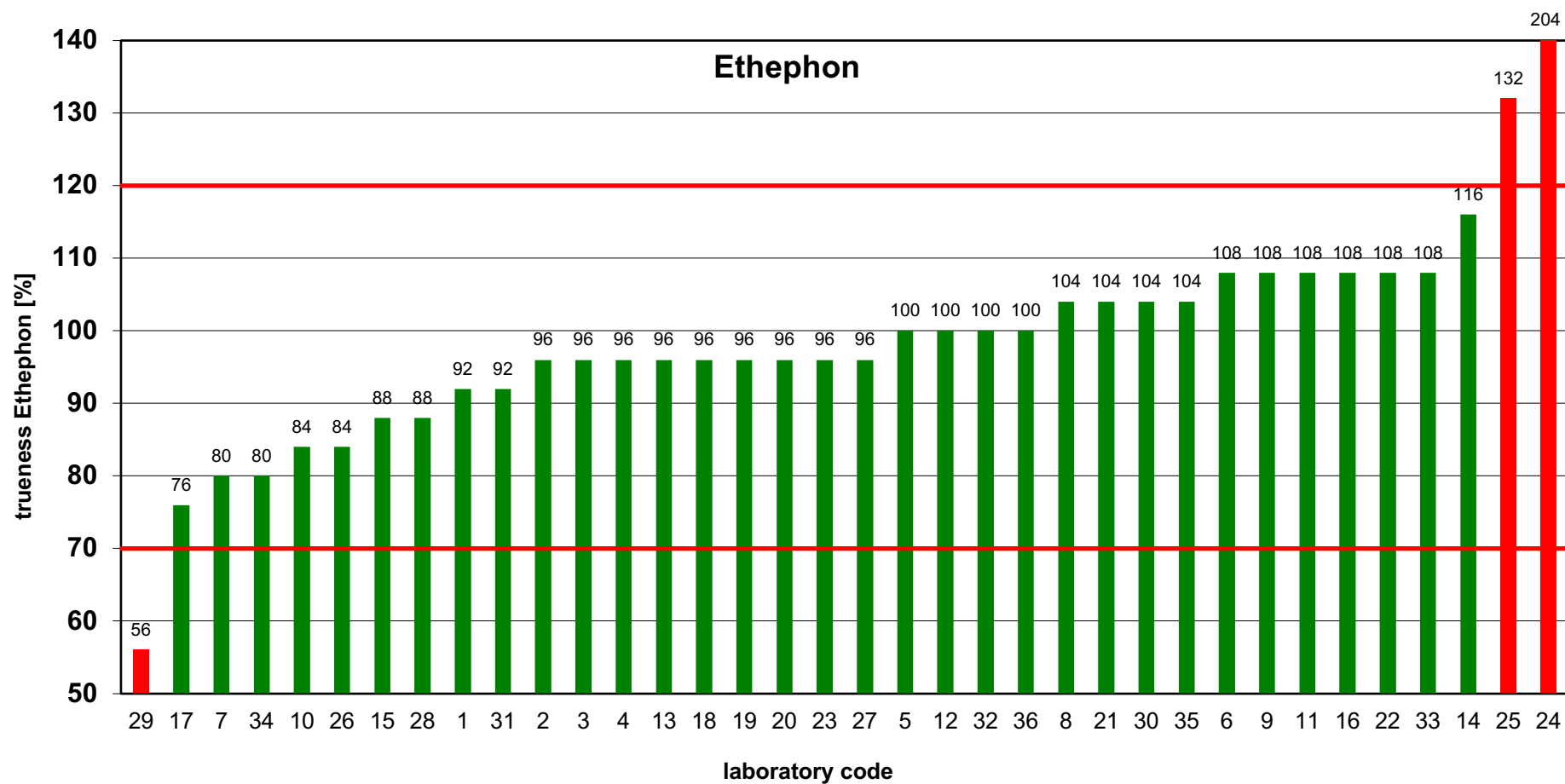
Figure 1: Assessment of Chlorate (trueness)
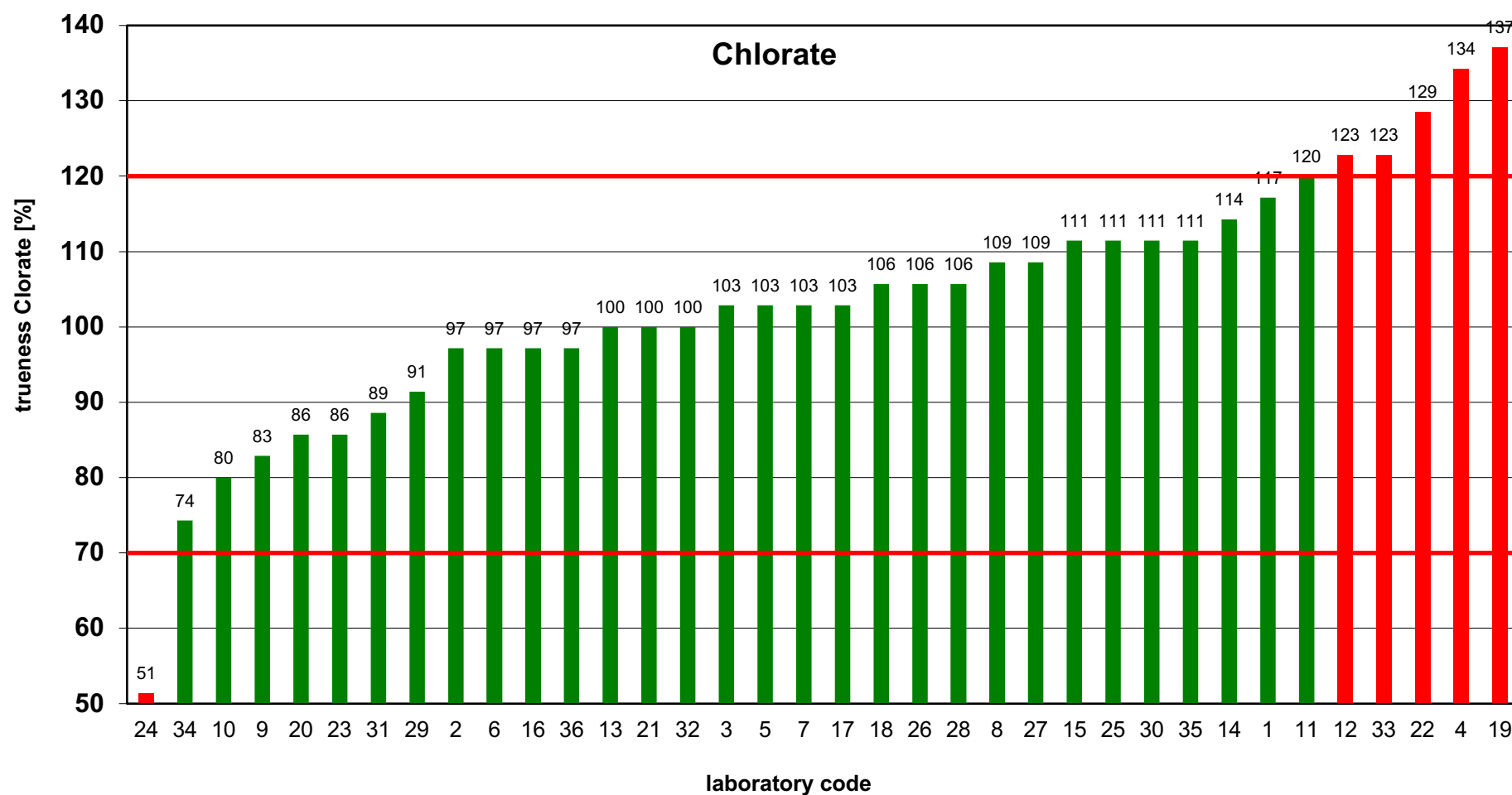
Green: satisfactory results, red: dissatisfactory results

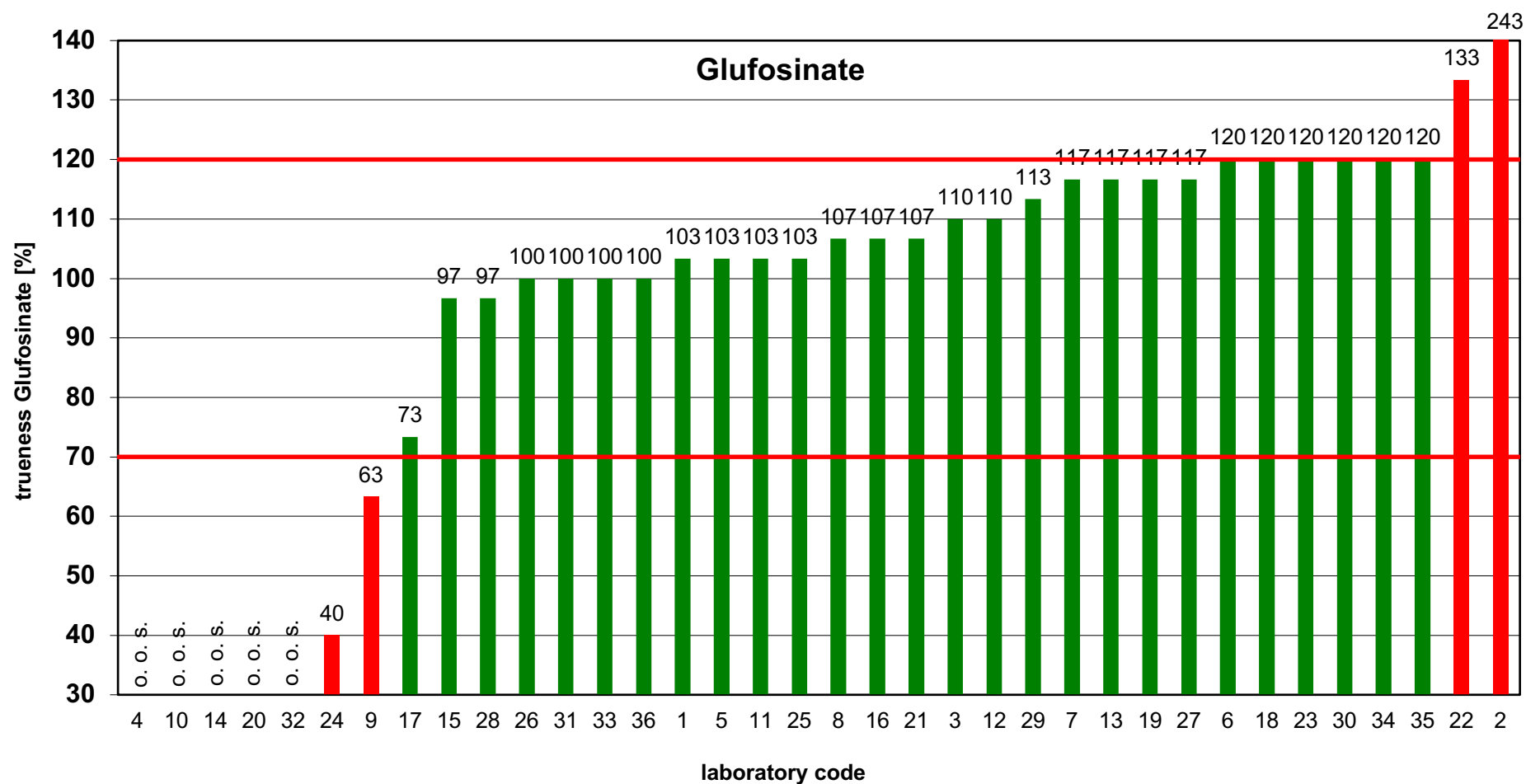*Figure 2: Assessment of Perchlorate (trueness)*

Green: satisfactory results, red: dissatisfactory results

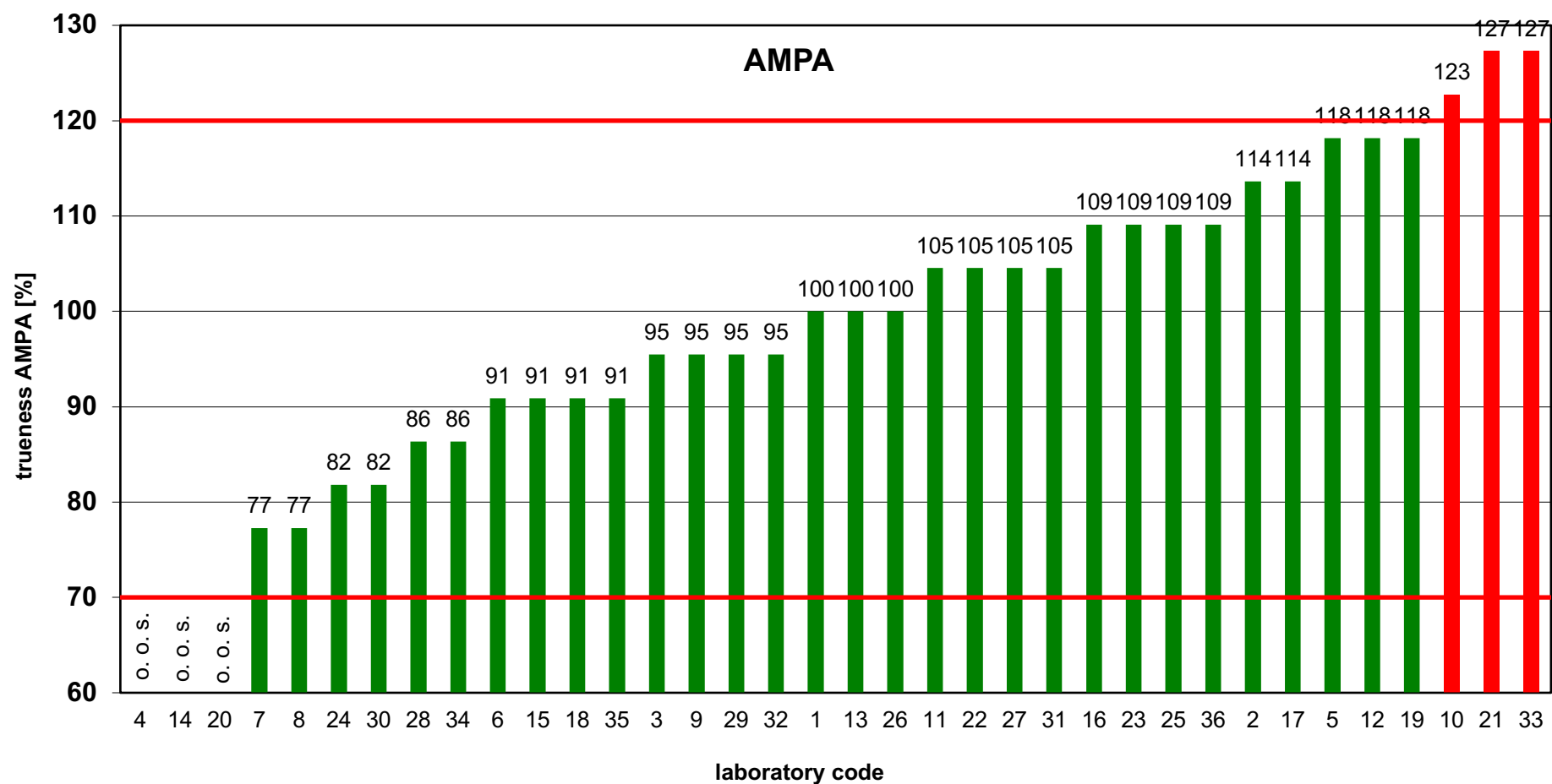*Figure 3: Assessment of Ethephon (trueness)*

Green: satisfactory results, red: dissatisfactory results

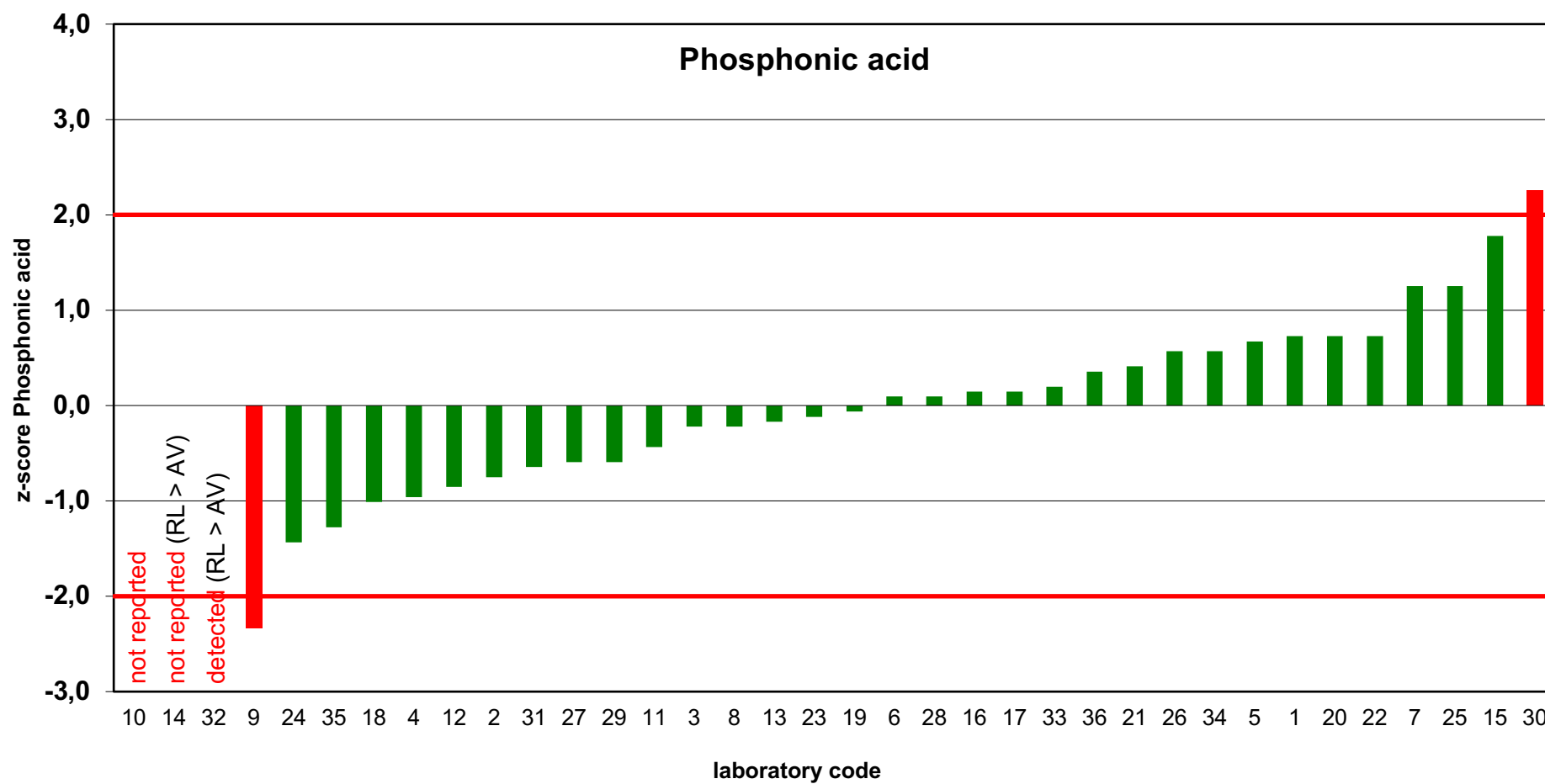*Figure 4: Assessment of Glufosinate (trueness)*

Green: satisfactory results, red: dissatisfactory results

Figure 5: Assessment of AMPA (trueness)

Green: satisfactory results, red: dissatisfactory results

Figure 6: Assessment of Phosphonic acid (comparability)

Green: satisfactory results, red: dissatisfactory results