

EUPT BF1 (2022)

1st European Proficiency Test on Pesticides in Infant Formula

Final Report

Pesticides in Infant Formula

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**European Union Reference Laboratory for Pesticides in Food of Animal
Origin and Commodities with High Fat Content**

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1 European Proficiency Test

Regulation 2017/625/EU¹ lays down the general tasks and duties of the EU Reference Laboratories (EURLs) for Food, Feed and Animal Health including the organisation of comparative tests. These proficiency tests are carried out on an annual basis and aim to improve the quality, accuracy and comparability of the analytical results generated by EU Member States within the frame of the EU coordinated control and national monitoring programmes. At the same time laboratories can assess their analytical performance and scope and make a comparison with other participating laboratories, which will hopefully encourage additional efforts for improvement.

According to Article 28 of Regulation 396/2005/EC on maximum residue levels of pesticides in or on food and feed of plant and animal origin², all laboratories analysing samples for the official controls of pesticide residues shall participate in the European Union Proficiency Tests (EUPTs) for pesticide residues on behalf of DG-SANTE³ as long as the scopes of the EUPT and the laboratory overlap.

The EURL for Pesticides in Food of Animal Origin and Commodities with High Fat Content (EURL AO) has so far conducted 17 EUPTs within the above mentioned framework.

DG-SANTE will have access to all data of EUPTs including the lab-code/lab-name key. The same will apply for all NRLs and the laboratories belonging to their own network. The results of this EUPT will be further presented to the European Commission Standing Committee for Animal Health and the Food Chain.

1.1 Basic Information and Time Schedule

EUPT AO BF1 was performed using infant formula obtained from local supermarkets as test matrix. The test items contain 10 pesticides from the list of obliged analytes and five voluntary pesticides (**Table 1**). All pesticides were spiked in 10 kg of infant formula by using two certified solutions. The spiked material was mixed for 24 h using an overhead shaker and finally sieved to remove clumps. Selected test items were analysed in duplicate to test for homogeneity. In addition, stability testing was performed at the time of sample shipment and after the deadline for submission of results.

1 Regulation (EU) 2017/625 of the European Parliament and of the Council on official controls performed and other official activities to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection. Published in Official Journal of the European Union L95/1 of 07.04.2017
2 Regulation (EC) No 396/2005, published in Official Journal of the European Union L70 of 16.03.2005
3 DG SANTE — Directorate-General for Health and Food Safety

Table 1: Overview of 10 mandatory and 5 voluntary analytes present in the infant formula for EUPT AO BF1.

Mandatory pesticides			
Disulfoton-sulfoxide	Terbufos	Diazinon	Cadusafos
Hexachlorobenzene (HCB)	Fipronil-sulfone	Heptachlor	Endrin
Chlordane, gamma(trans)-	Nitrofen		
Voluntary pesticides			
Chlorfenvinphos	BAC-C12	BAC-C14	Chlorate
Fluquinconazole			

Participating laboratories were provided with approximately 50 g portions of the prepared infant formula (test item). In addition, a ‘blank’ test item (approximately 50 g portions) was provided by EURL-AO. The participants were asked to determine and report the concentrations of any pesticide from the target lists detected at concentrations above the minimum required reporting level (MRRL). The distribution of the test items to the participants was randomly. The participation fee for all NRLs and OfLs was 180 Euro (360 Euro for laboratories from third countries) to cover the costs of shipping and handling of the test items.

52 laboratories from 24 different countries registered for participation in EUPT AO BF1. To one laboratory the sample could not be delivered within the stipulated deadline. Another laboratory did not submit results due to technical problems with the GC-MS instrument. One laboratory has withdrawn its registration and one laboratories did not register in the webtool.

46 out of 50 registered laboratories submitted their results. All participating labs are located in the European Union, one laboratory is from an EFTA country (**Table 2**). 43 results were considered for evaluation. Three labs (lab code 18, 37 and 40) reported results on ready to eat basis and were therefore excluded from evaluation.

Table 2: Number of participating laboratories in PT AO BF1 and their represented countries.

Austria	1	Norway	1	The Netherlands	2
Croatia	1	France	1	Poland	1
Cyprus	1	Germany	12	Slovakia	1
Czech Republic	3	Greece	2	Slovenia	1
Denmark	2	Hungary	2	Spain	5
Estonia	1	Italy	2	Sweden	1
Finland	2	Latvia	1		
European Union	19	EFTA	1	3rd Countries	0
Candidate countries	0				

1.2 Target Pesticide Lists and MRRL Values

For EUPT AO BF1 a mandatory and a voluntary target pesticide list was provided. 44 pesticides listed in **Table 3** were mandatory, while the analysis of 32 pesticides listed in **Table 4** was voluntary. For each pesticide and metabolite included in the residue definitions, a MRRL based on the ready to eat product was set. These MRRL values are the levels that laboratories were expected to achieve. The values were established by the organiser and confirmed by the EURL Scientific Committee. To avoid problems in reporting it was decided to report all results on the powder provided. This was stated in the specific protocol and confirmed by the Scientific Committee. Nevertheless, three laboratories reported their result on ready to eat basis with a factor < 8, resulting in “nd” reported by one lab in almost all cases. The laboratories will receive a statement with theoretical z-scores of their results, but they are not included in statistical evaluation.

Table 3: List of mandatory analytes of EUPT AO BF1. MRRL-values are given in mg/kg ready to eat product*.

Analyte	MRRL [mg/kg]	Analyte	MRRL [mg/kg]
Aldrin	0.002	Endosulfansulfate	0.002
Cadusafos	0.003	Endrin	0.0004
Chlordane, alpha (cis)-	0.001	Ethoprophos	0.004
Chlordane, gamma (trans)-	0.001	Fensulfothion	0.002
Chlorpyrifos	0.005	Fensulfothion-oxon	0.002
Chlorpyrifos-methyl	0.005	Fensulfothion-oxon-sulfone	0.002
Cyhalotrin, lambda	0.005	Fensulfothion-sulfone	0.002
DDD, p,p-	0.003	Fipronil	0.002
DDE, p,p-	0.003	Fipronil-sulfone	0.002
DDT, o,p-	0.003	HCH, alpha-	0.005
DDT, p,p-	0.003	HCH, beta-	0.005
Demeton-S-methyl	0.002	HCH, gamma- (Lindane)	0.005
Demeton-S-methyl-sulfone	0.002	Heptachlor	0.001
Demeton-S-methyl-sulfoxid	0.005	Heptachlorepoxyd, cis-	0.001
Diazinon	0.002	Heptachlorepoxyd, trans-	0.001
Dieldrin	0.002	Hexachlorobenzene (HCB)	0.002
Dimethoate	0.001	Nitrofen	0.002
Disulfoton	0.001	Omethoate	0.002
Disulfoton-sulfoxid	0.001	Oxychlordane	0.001
Disulfoton-sulfon	0.003	Terbufos	0.002
Endosulfan, alpha-	0.003	Terbufos-sulfone	0.002
Endosulfan, beta-	0.003	Terbufos-sulfoxide	0.002

Table 4: List of voluntary analytes of EUPT AO BF1. MRRL-values are given in mg/kg ready to eat product*.

Analyte	MRRL [mg/kg]	Analyte	MRRL [mg/kg]
BAC-C8 (expressed as chloride salt)	0.005	Fenbuconazole	0.005
BAC-C10 (expressed as chloride salt)	0.005	Fluquinconazole	0.005
BAC-C12 (expressed as chloride salt)	0.005	Flusilazole	0.005
BAC-C14 (expressed as chloride salt)	0.005	Isofenphos	0.005
BAC-C16 (expressed as chloride salt)	0.005	Mecarbam	0.005
BAC-C18 (expressed as chloride salt)	0.005	Methamidophos	0.005
DDAC-C8 (expressed as chloride salt)	0.005	Methidathion	0.005
DDAC-C10 (expressed as chloride salt)	0.005	Phorate	0.002
DDAC-C12 (expressed as chloride salt)	0.005	Phorate sulfone	0.002
Chlorate (expressed as anion)	0.005	Phorate sulfoxide	0.002
Carbofuran-3-hydroxy	0.001	Phorate oxone	0.002
Chlorfenvinphos	0.005	Phorate oxone sulfone	0.002
Ethion	0.005	Phorate oxone sulfoxide	0.002
Fenamiphos	0.002	Perchlorate (expressed as anion)	0.005
Fenamiphos sulfone	0.002	Tebuconazole	0.005

*The calculation factor from powder to ready to eat product is 8 (MRRL ready to eat value x 8)

1.3 Statistical Treatment of the Results

The EURL Scientific Committees decisions regarding the statistical treatments involved in the evaluations performed on the results reported for this proficiency test are of importance. They are listed in the following chapters 1.3.1 - 1.3.8.

1.3.1 One step evaluation of the data and omission of results

According to experiences gained from previous EUPTs, the results of laboratories deviate significantly from the median. If the number of results reported is limited, especially for voluntary analytes, the calculated statistical parameters, especially the median, could have been influenced by outlying results. Starting in 2014, the robust mean x^* calculated according algorithm A was introduced for evaluating the results of all EUPTs. In comparison with the median the robust mean x^* is less influenced by deviating results. Thus, there is no need to introduce a second evaluation of the robust mean after the omission of results from laboratories with z-scores > 5.0 . Only results reported by EU and EFTA countries are taken into account to derive the assigned value (results reported by third countries and candidate states are excluded from the estimation).

1.3.2 Robust mean according Algorithm A - assigned value

Starting in 2014, the robust mean x^* calculated according to algorithm A was introduced for evaluating the results of all EUPTs. The robust mean x^* was used as the estimation of the assigned value. In comparison with the median the robust mean x^* is less influenced by deviating results. Since there are no traceable reference values available, the assigned values in this PT were calculated as robust mean x^* calculated according to algorithm A of the results of the participants. Therefore, the assigned values are traceable to the results of the participants, only.

1.3.3 Robust standard deviation according Algorithm A

Additionally, for all pesticides the robust standard deviations s^* according algorithm A were calculated. The algorithm A minimises the influence of outlying results and provides good estimations of the standard deviation.

1.3.4 Target standard deviation (from European Proficiency Tests for pesticides)

In the range of ± 1 standard deviations there will be 68.3% of the values used for the calculations. From the results of ten previous European Proficiency Tests on pesticides in vegetable or fruit matrices, a fit-for-purpose relative target standard deviation (FFP RSD; %_{SEUPT}) of 25 % was estimated.

1.3.5 Evaluation - z-scores

The z-score is calculated from the fit for purpose standard deviation as follows:

$$z = (m - x^*) / s_{EUPT}$$

with:

Variable	Description
z	Value of the z-score
m	Result of the laboratory
x*	Robust Mean according Algorithm A
s _{EUPT}	Fit-for-purpose deviation from previous European Proficiency Tests

The z-score therefore is a factor of the fit for purpose standard deviation by which the laboratory result differs from the assigned value. Therefore, the value of the z-score can be used to assess the analytical results:

Range	Evaluation
0 – 2.0	The analysis fulfils the requirements – acceptable (at the normal distribution and the level of confidence 95%)
> 2.0 - < 3.0	The analysis should be checked – questionable
≥ 3.0	The analysis does not fulfil the requirements – unacceptable (at the level of confidence 99.7 %)

Any z-score values of $z > 5.0$ have been reported as “5”.

1.3.6 z-Scores for false negative results

Results for pesticides reported by the laboratories as “analysed” but without reporting numerical values (although they were spiked by the organiser to the test item and were detected by the majority of participants at or above the MRRL) have been considered as false negative results. Results reported as < RL (RL = reporting limit of the laboratory) will be considered as not detected and will also be judged as false negatives if the assigned value of the analyte is at or above the MRRL. For false negative results, z-scores have been calculated

- using the MRRL value in cases where the RL of the lab was higher than or equal to the MRRL;
- using the RL value in cases where the RL of the lab was lower than the MRRL.

According to the General Protocol, z-scores for false negatives will be set at -3.5 if the calculated z-scores are > -3 (still questionable) to underline that these are unacceptable results. This rule has to be applied for nitrofen, hexachlorobenzene (HCB), endrin, disulfoton-sulfoxide, trans-chlordane, fluquinconazole, chlorfenvinphos, BAC-C12, BAC-C14 and cadusafos.

1.3.7 False positive results

Results reported for pesticides included in the target pesticide list, but not spiked to the test item nor detected by the organiser (even after a repeated analysis with lower detection limits) were assigned as false positive results - if they were reported at concentrations at or above the MRRL value as stipulated by the organiser. No z-scores were calculated for these results. Results reported below the MRRL have been disregarded. These results have not been considered to be false positives.

1.3.8 Category A and B classification

Laboratories that 1.) were able to analyse for at least 90% of the compulsory pesticides on the target list, 2.) have correctly detected and quantified at least 90% of the pesticides present in the test material and 3.) reported no false positives had demonstrated “sufficient scope” and were therefore classified into category A. For the 90% criterion the number of pesticides, that needed to be correctly analysed to have sufficient scope, will be calculated by multiplying the number of mandatory pesticides from the Target Pesticides List by 0.9 and rounded to the nearest whole number with 0.5 decimals being rounded downwards.

1.4 Website for submitting results of EUPT AO BF1

Since EUPT AO 09 (2014) a web based system was used for EUPT data submission. This system was replaced in 2019 called “webtool”. For EUPT AO BF1, 6 labs (lab codes 45, 46, 48, 49, 50, 51) did not select their analytical scope within the deadline (12 August 2022). As a result, the whole scope for voluntary analytes was selected as target scope automatically by the webtool.

1.5 Observations during evaluation of the data

Laboratories reporting on ready to eat basis

Three laboratories reported their results on ready to eat basis and not on the powder provided. The scientific committee decided to exclude these results from evaluation of EUPT AO BF1. The results of the laboratories are given in the following **Table 5** with the data corrected by EURL AO and the expected z-scores. The laboratories will receive a certificate explaining the situation for their quality control.

Table 5: Results of laboratories that reported on ready to eat basis.

	Lab 37			Lab 18			Lab 40		
	Concentration ready to eat [mg/kg]	Concentration calculated on powder by EURL AO [mg/kg]	z-score	Concentration ready to eat [mg/kg]	Concentration calculated on powder by EURL AO [mg/kg]	z-score	Concentration ready to eat [mg/kg]	Concentration calculated on powder by EURL AO [mg/kg]	z-score
Cadusafos	0.00897	0.072	-0.4	0.00848	0.068	-0.6	0.0105	0.084	0.2
Chlordane-trans	0.00574	0.046	0.0	0.00606	0.048	0.2	0.0055	0.044	-0.2
Diazinon	0.0191	0.153	-0.3	0.0196	0.157	-1.1	0.0224	0.179	0.3
Disulfoton-Sulfoxide	0.00643	0.051	-0.2	na			0.00727	0.058	0.4
Endrin	0.0018	0.014	0.2	nd			0.00167	0.013	-0.1
Fipronil-Sulfone	0.0191	0.153	0.6	0.0181	0.145	0.3	0.0181	0.145	0.3
Heptachlor	0.0099	0.079	0.3	0.00925	0.074	0.0	0.00512	0.041	-1.8
Hexachlorobenzene	0.00785	0.063	-0.1	0.00693	0.055	-0.6	0.008	0.064	0.0
Nitrofen	0.00864	0.069	-0.7	0.00822	0.066	-0.8	0.0122	0.098	0.7
Terbufos	0.01389	0.111	-0.4	0.00709	0.057	-2.1	0.0155	0.124	0.1

Chlorate

In EUPT AO BF1 only 12 numerical values of chlorate were reported. Due to the fact that this number is quite low and the uncertainty of the robust mean is high, the Scientific Committee decided to evaluate chlorate for information, only. As a consequence, the results should not be used for quality control purposes of the laboratories.

2 Results of EUPT AO BF1

The evaluation of EUPT AO BF1 followed the “General protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed”, the norm ISO EN 13528:2015 and “The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories: Pure & Applied Chemistry 78, 145-196 (2006)”. Relevant statistical data including z-scores were calculated.

In total, 430 results for 10 spiked mandatory pesticides were submitted. Thereof, 419 results (97%) were reported as analysed and 11 results (3%) were reported as not analysed (Table 6).

Table 6: The total number of spiked pesticides, the numbers of spiked pesticides analysed and the number of spiked pesticides reported as “not analysed” are presented. Chlorate is not included in this overview as the result is evaluated for information, only.

Pesticides	Total number #1	Analysed #2	Non analysed #3	Percentage of „non analysed“ #4
Mandatory (n = 24)	430	419	11	2.6%
Voluntary (n = 4)	172	126	46	26.7%

#1 Expected (maximum) number of results

#2 Number of results reported as “analysed”

#3 Number of results reported as “not analysed”

#4 Ratio of the “not analysed” pesticide to the expected number of results with calculations for “all” pesticides and separated between mandatory and voluntary pesticides

In the following chapters 2.1 and 2.2 an overview of the individual results of each laboratory is given. A legend for the chapters is shown in Table 7.

Table 7: Legend for the tables “Overview of individual results” chapter 2.1 and 2.2

Abbreviation	Description
Number of detects	Number of pesticides quantified in the test item, differentiated between mandatory and voluntary pesticides (printed in green if ≥ 90 % of the mandatory pesticides present)
Number of analysed	Number of pesticides analysed in the test item, differentiated between mandatory and voluntary pesticides (printed in green if ≥ 90 % of the mandatory pesticides analysed)
Total scope	Number of mandatory and voluntary pesticides analysed in the test item
5	z-Scores higher than 5.0 were limited to 5, printed in red.
False negatives	Number of results reported for pesticides reported as “n.d.” Z-Scores calculated using the <ul style="list-style-type: none"> • MRRL value in cases RL of lab were higher than, or equal to the MRRL value, • the RL in case where the RL value of the lab was below the MRRL value. Z-Scores for false negatives will be fixed at -3.5 if the calculated z-scores are > -3 False negative results were included in the calculations of AZ ²
False positives	Number of results considered as false positives
AZ ²	Average of the squared z-scores (category A laboratories, only) “B” in case the laboratory is categorized into the category B
na	Results reported as “not analysed”. It is printed in red.
Chlorate	Chlorate is evaluated for information, only: see chapter 1.5; the pesticide is therefore not counted for number of detects, number of analysed and total scope

2.1 Mandatory analytes: Overview of individual results (z-scores)

Lab code	Cadusafos	Chlordane-trans	Diazinon	Disulfoton-sulfoxide	Endrin	Firpronil-sulfone
2	-0.3	-1.0	0.8	0.0	0.2	0.2
3	-0.2	-0.1	1.7	0.9	0.8	-3.5
5	0.0	0.2	0.1	0.0	-3.5	0.1
6	0.2	0.7	-0.4	-0.3	-0.7	0.5
7	-1.0	-0.2	-0.7	-1.2	-0.7	-0.9
8	-0.6	na	-0.6	-0.4	-1.1	-0.3
9	0.2	-1.7	0.2	-1.0	-1.0	-0.3
10	0.2	0.3	-0.1	-0.6	0.0	-0.2
11	-0.3	-0.2	-0.4	-0.6	-0.4	-2.1
12	0.0	-1.4	0.1	0.1	0.8	0.1
13	1.0	-0.3	-0.7	0.1	-0.7	0.1
14	0.5	0.4	0.4	-0.5	-0.4	0.0
15	-0.3	-0.3	-0.2	-0.6	-2.1	-0.2
16	0.4	1.9	0.4	0.8	2.1	-0.2
17	0.1	-0.2	-0.1	0.0	0.2	-0.1
19	0.4	0.6	0.9	0.4	-0.2	0.4
20	-0.2	0.5	-0.6	0.3	-0.2	0.0
21	-1.4	0.5	-0.9	0.7	1.4	0.9
22	1.6	0.9	-0.4	0.2	-0.8	-0.2

Lab code	Cadusafos	Chlordane-trans	Diazinon	Disulfoton-sulfoxide	Endrin	Firpronil-sulfone
23	-0.1	-0.4	1.9	-0.4	0.8	0.3
24	-1.0	-0.3	-0.6	na	-0.5	-1.5
25	0.6	0.2	0.7	-0.4	0.8	0.2
26	0.5	-0.9	-1.1	na	1.1	-0.9
27	-0.5	-0.6	-0.2	-0.2	0.6	0.0
28	0.9	0.6	0.1	0.2	0.2	1.4
29	0.3	-1.6	0.5	0.1	-1.3	-0.6
30	-0.3	0.0	1.2	2.6	0.0	0.0
31	0.2	-0.2	0.4	0.0	0.4	0.3
32	na	0.2	-0.1	na	0.5	-0.2
33	-0.5	-0.1	-1.2	0.1	-0.3	0.2
34	0.4	0.1	-0.5	0.3	-0.1	-0.5
35	0.2	0.7	0.4	1.0	0.6	0.3
36	-0.1	0.9	-0.6	0.1	1.1	0.0
38	0.5	0.3	-0.5	-0.2	-1.0	0.2
39	0.1	0.1	0.1	0.5	0.2	-0.1
42	-0.7	na	2.1	-0.5	1.3	0.5
44	-3.5	-0.5	0.9	0.3	-0.3	1.0
45	0.4	1.1	0.1	na	1.3	0.2
46	-3.5	-0.5	-3.7	-3.5	-0.9	-3.5
48	0.2	-0.2	0.2	0.4	-1.0	-0.1
49	-0.7	-3.5	-0.3	-0.6	-0.4	1.9
50	-0.3	-3.5	-0.5	-0.5	-3.5	-0.4
51	-0.1	-0.2	0.1	0.4	-0.4	-0.1

Lab code	Heptachlor	Hexachlorobenzene	Nitrofen	Terbufos
2	-0.3	0.5	0.2	0.1
3	0.4	-0.9	0.1	-2.0
5	0.5	0.4	0.8	0.0
6	0.3	0.4	0.7	-0.7
7	-0.2	-0.6	-0.8	-0.7
8	-1.6	-0.3	-0.7	na
9	-1.8	-2.7	-1.5	-0.2
10	-0.2	1.6	0.7	0.4
11	0.1	0.0	-0.1	-0.1
12	-1.2	2.3	0.5	0.3
13	-0.1	-0.1	-0.9	0.5
14	0.4	0.4	-1.0	-0.1
15	-0.4	-0.4	-0.5	-0.6
16	-0.2	-1.5	0.9	0.3
17	-0.1	-0.5	0.0	1.6
19	1.2	0.3	0.8	1.5
20	0.2	0.6	-0.4	-0.2
21	-0.9	-1.4	0.1	-3.7
22	0.3	-0.1	-0.2	-1.1

Lab code	Heptachlor	Hexachlorobenzene	Nitrofen	Terbufos
23	0.2	0.4	0.3	0.1
24	0.1	-0.4	-0.8	-0.1
25	0.4	-0.5	0.7	0.0
26	-0.7	-1.0	-1.4	na
27	0.2	0.3	0.3	-0.3
28	0.7	0.5	0.8	-0.4
29	-1.6	-1.8	-0.5	-0.1
30	0.6	0.2	0.7	1.7
31	0.1	-0.8	0.4	na
32	0.6	-0.1	-3.5	na
33	0.0	-0.2	-1.3	-1.2
34	-0.2	-0.2	-0.4	0.6
35	0.5	0.3	1.0	0.7
36	0.6	0.9	0.6	0.4
38	0.3	0.9	-0.8	0.9
39	0.6	0.3	-0.2	0.1
42	2.3	1.7	1.9	-0.2
44	0.0	2.0	0.7	-3.5
45	0.7	0.8	0.8	0.8
46	-0.1	-0.4	-3.5	-3.5
48	-0.4	-0.3	-0.1	-0.3
49	-1.0	-0.1	-0.5	0.0
50	-0.5	-0.5	-0.2	-0.3
51	-0.7	-0.8	-0.6	-2.3

Lab code	Number of mandatory pesticides detected	Number of mandatory pesticides analysed	False negatives	False positives concentration in [mg/kg]	AZ ²
2	10	44			0.21
3	9	40	1		2.15
5	9	40	1		1.35
6	10	44			0.26
7	10	42			0.58
8	8	33			B
9	10	44			1.80
10	10	44			0.38
11	10	41			0.51
12	10	44			0.99
13	10	43			0.32
14	10	42			0.24
15	10	44			0.59
16	10	44			1.25
17	10	42			0.31
19	10	39			0.61

Lab code	Number of mandatory pesticides detected	Number of mandatory pesticides analysed	False negatives	False positives concentration in [mg/kg]	AZ ²
20	10	44			0.14
21	9	44	1		2.27
22	10	44			0.54
23	10	44			0.51
24	9	37			B
25	10	41			0.27
26	8	32			B
27	10	43			0.13
28	10	42			0.46
29	10	44			1.09
30	10	42			1.22
31	9	39			0.15
32	6	32	1		B
33	10	41			0.50
34	10	44			0.13
35	10	37			B
36	10	44			0.40
38	10	39			0.40
39	10	44			0.09
42	9	40			2.11
44	8	44	2		B
45	9	32			B
46	4	44	6		B
48	10	44			0.16
49	9	44	1	Cis-Chlordane (0.034 mg/kg)	B
50	8	44	2		B
51	10	44			0.74

2.2 Voluntary analytes: Overview of individual results (z-scores)

Lab code	BAC-C12	BAC-C14	Chlorfenvinphos	Fluquinconazole	Chlorate	Number of voluntary detected
2	-0.7	-0.6	0.2	0.2	na	4
3	na	na	-2.0	na	na	1
5	na	na	0.3	-0.1	-0.1	3
6	1.2	0.9	0.4	-0.1	na	4
7	na	na	-1.2	-0.5	na	2
8	na	na	-0.3	-2.4	na	2
9	-0.8	-1.3	-0.2	-0.1	-0.1	5
10	na	na	0.4	0.1	na	2

Lab code	BAC-C12	BAC-C14	Chlorfenvinphos	Fluquinconazole	Chlorate	Number of voluntary detected
11	na	na	-0.1	-0.4	na	2
12	0.6	0.5	0.4	1.0	0.1	5
13	na	na	0.2	-0.1	na	2
14	0.2	0.6	0.0	-0.1	-0.3	5
15	na	na	-0.1	-0.2	na	2
16	0.9	0.7	0.9	1.1	na	4
17	na	na	-0.2	0.4	na	2
19	0.4	-0.7	0.8	na	-2.7	4
20	na	na	-0.6	0.1	na	2
21	0.6	0.5	-0.3	0.1	na	4
22	na	na	0.5	-0.3	na	2
23	-0.6	-0.5	0.3	-0.5	0.1	5
24	na	na	-1.1	-0.6	na	2
25	na	na	1.0	na	-3.7	2
26	0.1	-0.2	-0.8	-1.8	na	4
27	-0.3	-0.4	0.2	-0.3	na	4
28	1.8	0.9	0.6	0.5	1.5	5
29	-0.1	0.0	0.5	0.3	0.1	5
30	na	na	0.8	0.1	na	2
31	na	na	na	na	na	0
32	na	na	na	na	na	0
33	-0.4	1.0	-1.7	-1.9	na	4
34	-0.5	-0.6	0.0	0.0	5.0	5
35	na	na	0.2	1.0	na	2
36	0.0	0.4	-0.3	0.4	0.4	5
38	na	na	0.3	0.3	na	2
39	-0.2	-0.4	-0.5	0.0	na	4
42	-3.5	-3.5	-0.5	-0.6	-3.7	5
44	-3.5	-3.5	0.7	0.7	-3.7	5
45	na	na	0.4	na	na	1
46	-3.5	-3.5	-3.5	-3.7	-3.7	5
48	-0.7	-0.7	0.2	0.4	-1.0	5
49	-3.5	-3.5	-3.5	-3.7	-3.7	5
50	-3.5	-3.5	-0.6	0.9	-3.7	5
51	-0.6	-0.2	-0.9	-0.7	-0.6	5

2.3 Summary of submitted results and comments

Lab code	Number of voluntary pesticides analysed	Number of pesticides analysed	Comments submitted via webtool
2	27	71	
3	13	53	
5	19	59	
6	26	70	
7	16	58	
8	18	51	
9	32	76	
10	8	52	
11	15	56	
12	32	76	
13	20	63	
14	32	74	
15	13	57	
16	21	65	
17	8	50	
19	22	61	<p>The sample was not refrigerated, on the outside of the box there was no indication that it should be stored cooled, therefore the sample was kept in sample reception at room temperature until registration, at least 12 hours (around 30°C).</p> <p>Fluquinconazole tested but not included in scope (Lab error). The Lab concentration is 0.133 mg/kg (the result is recovery corrected, 61%). GC-MSMS(QQQ).</p> <p>The blank cannot be analyzed for the quaternary ammonium method.</p>
20	10	54	
21	21	65	
22	14	58	
23	8	52	
24	7	44	
25	13	54	
26	18	50	
27	15	58	
28	29	71	
29	31	75	
30	14	56	
31	0	39	
32	7	39	
33	19	60	
34	32	76	

Lab code	Number of voluntary pesticides analysed	Number of pesticides analysed	Comments submitted via webtool
35	15	52	
36	30	74	
38	16	55	
39	28	72	
42	32	72	
44	32	76	
45	3	35	
46	32	76	
48	32	76	
49	32	76	
50	32	76	The box arrived broked and the blank was also broked. We can use it because there have not been loses, but just to let know you.
51	32	76	

2.4 Overview of the results

The robust mean and relative robust standard deviation for EU and EFTA labs for all spiked pesticides is summarised in **Table 8**. The number of results includes the number of acceptable, questionable, unacceptable and not analysed results for all labs participating in PT AO BF1. The number of false negative results as well as the ratio of the robust mean to the spiked concentration is listed additionally in **Table 8**.

Table 8: Summary of results of spiked pesticides. The results for chlorate are for information only.

Analyte	Robust mean X* [mg/kg]	Robust RSD (FFP- σ_{pt})	Number of results	Acceptable	Questionable	Unacceptable	False negatives	Not analysed	Spike value [mg/kg]	Ratio X* / spike value	Acceptable	Questionable	Unacceptable
Cadusafos	0.0804	13.0%	42	40	0	2	2	1	0.090	89%	95.2%	0.0%	4.8%
Chlordane, gamma(trans)-	0.0461	16.0%	41	39	0	2	2	2	0.054	85%	95.1%	0.0%	4.9%
Diazinon	0.1649	17.0%	43	41	1	1	1	0	0.195	85%	95.3%	2.3%	2.3%
Disulfoton-sulfoxide	0.0531	13.6%	39	37	1	1	1	4	0.060	89%	94.9%	2.6%	2.6%
Endrin	0.0133	22.3%	43	39	2	2	2	0	0.015	89%	90.7%	4.7%	4.7%
Fipronil-sulfone	0.1335	10.3%	43	40	1	2	2	0	0.150	89%	93.0%	2.3%	4.7%
Heptachlor	0.0739	15.4%	43	42	1	0	0	0	0.090	82%	97.7%	2.3%	0.0%
Hexachlorobenzene (HCB)	0.0643	20.0%	43	41	2	0	0	0	0.080	80%	95.3%	4.7%	0.0%
Nitrofen	0.0834	19.8%	43	41	0	2	2	0	0.110	76%	95.3%	0.0%	4.7%
Terbufos	0.1211	16.1%	39	35	1	3	3	4	0.150	81%	89.7%	2.6%	7.7%
BAC-C12 (expressed as chloride salt)	0.2010	17.3%	24	19	0	5	5	19	0.260	77%	79.2%	0.0%	20.8%
BAC-C14 (expressed as chloride salt)	0.1713	18.7%	24	19	0	5	5	19	0.220	78%	79.2%	0.0%	20.8%
Chlorate (expressed as anion)	0.1460	24.0%	18	10	1	7	6	25	0.190	77%	55.6%	5.6%	38.9%
Chlorfenvinphos	0.1553	16.2%	41	39	0	2	2	2	0.180	86%	95.1%	0.0%	4.9%
Fluquinconazole	0.1234	14.3%	37	34	1	2	2	6	0.140	88%	91.9%	2.7%	5.4%

2.5 Distribution of z-scores and AZ²

2.5.1 Mandatory pesticides

The distribution of z-scores and AZ² for mandatory pesticides of all labs participating in EUPT AO BF1 is shown in **Table 9** and **Table 10**.

Table 9: Number of acceptable ($|z| \leq 2.0$), questionable ($2.0 < |z| < 3.0$) and unacceptable ($|z| \geq 3.0$) results for all mandatory pesticides.

Range of absolute z-scores	Number of z-scores	Percentage of all z-scores
0 – 2.0	395	94.3%
>2.0 - < 3.0	9	2.1%
≥3.0	15	3.6%

Table 10: Number of good ($AZ^2 \leq 2.0$), satisfactory ($2.0 < AZ^2 < 3.0$) and unsatisfactory ($AZ^2 \geq 3.0$) results for all mandatory pesticides.

Range of AZ ²	Number of laboratories in Cat A (33 of 43)	Percentage of laboratories in Cat A (77% of the total)
0 – 2.0	30	91%
>2.0 - < 3.0	3	9%
≥3.0 - 5	0	0%

2.5.2 Voluntary pesticides

The distribution of z-scores for voluntary pesticides (excluding chlorate) of all labs participating in EUPT AO BF1 is shown in **Table 11**.

Table 11: Number of acceptable ($|z| \leq 2.0$), questionable ($2.0 < |z| < 3.0$) and unacceptable ($|z| \geq 3.0$) results for all voluntary pesticides

Range of absolute z-scores	Number of z-scores	Percentage of all z-scores
0 – 2.0	111	88.1%
>2.0 - < 3.0	1	0.8%
≥3.0	14	11.1%

2.6 Dispersion of results

The dispersion of results pictured by the relative robust standard deviation of the spiked pesticides is shown in **Figure 1**.

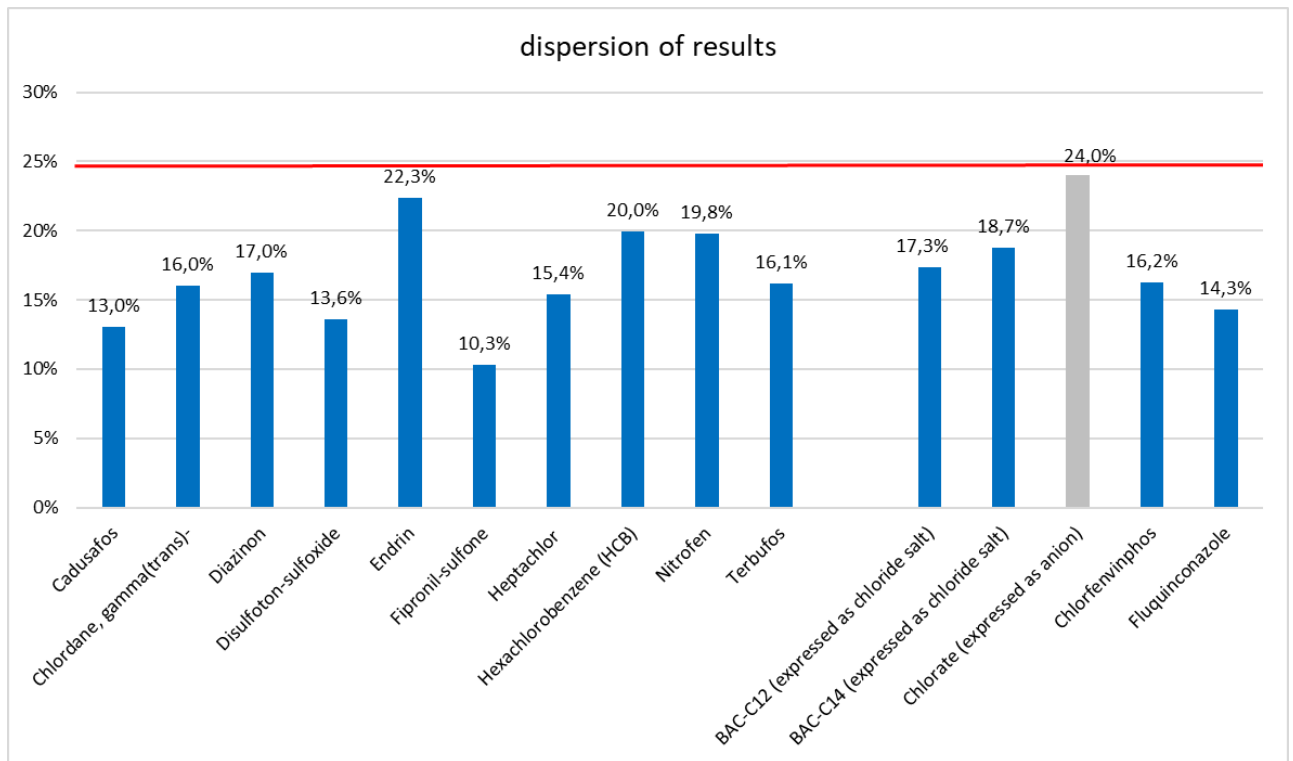


Figure 1: Relative robust standard deviation of 10 mandatory and 5 voluntary pesticides. The dispersion of results for chlorate is given for information only.

3 Results for Spiked Analytes

In the following chapters 3.1 and 3.2 an overview of the results of each laboratory is given. A legend for the chapters is shown in **Table 12**.

Table 12: Legend for the tables in chapter 3.1 and 3.2.

Abbreviation	Description
Lab number	code of the participating laboratory during the evaluation of the PT
Result	reported result from the laboratory [mg/kg]
Deviation	deviation of the reported result from the robust mean (assigned value) [mg/kg]
z-Scores (EUPT)	z-scores, calculated using the “fit-for-purpose standard deviation” for EUPTs (25 % of the robust mean)
Red figures	<ul style="list-style-type: none"> • false negative results (nd) • z-scores for false negative results calculated using <ul style="list-style-type: none"> ○ MRRL, if RL of the lab \geq MRRL ○ RL of the lab, if RL < MRRL • Z-Scores for false negatives will be fixed at -3.5 if the calculated z-scores are > -3
5 in red colour	z-Scores greater than 5.0, limited to 5 according to the protocol of EUPTs

3.1 Mandatory analytes

3.1.1 Cadusafos and trans-Chlordane

Lab code	Cadusafos [mg/kg]			trans-Chlordane [mg/kg]		
	Result	Deviation	z-Scores (EUPT)	Result	Deviation	z-Scores (EUPT)
2	0.0750	-0.0054	-0.3	0.0350	-0.0111	-1.0
3	0.0770	-0.0034	-0.2	0.0450	-0.0011	-0.1
5	0.0794	-0.0010	0.0	0.0481	0.0020	0.2
6	0.0840	0.0036	0.2	0.0540	0.0079	0.7
7	0.0600	-0.0204	-1.0	0.0440	-0.0021	-0.2
8	0.0680	-0.0124	-0.6			
9	0.0850	0.0046	0.2	0.0260	-0.0201	-1.7
10	0.0837	0.0033	0.2	0.0496	0.0035	0.3
11	0.0745	-0.0059	-0.3	0.0440	-0.0021	-0.2
12	0.0797	-0.0007	0.0	0.0299	-0.0162	-1.4
13	0.1010	0.0206	1.0	0.0421	-0.0040	-0.3
14	0.0905	0.0101	0.5	0.0505	0.0044	0.4
15	0.0753	-0.0051	-0.3	0.0424	-0.0037	-0.3
16	0.0880	0.0076	0.4	0.0682	0.0221	1.9
17	0.0823	0.0019	0.1	0.0437	-0.0024	-0.2
19	0.0890	0.0086	0.4	0.0533	0.0072	0.6
20	0.0761	-0.0043	-0.2	0.0520	0.0059	0.5
21	0.0524	-0.0280	-1.4	0.0514	0.0053	0.5
22	0.1120	0.0316	1.6	0.0567	0.0106	0.9
23	0.0780	-0.0024	-0.1	0.0410	-0.0051	-0.4
24	0.0604	-0.0200	-1.0	0.0432	-0.0029	-0.3
25	0.0930	0.0126	0.6	0.0480	0.0019	0.2
26	0.0897	0.0093	0.5	0.0354	-0.0107	-0.9
27	0.0700	-0.0104	-0.5	0.0395	-0.0066	-0.6
28	0.0980	0.0176	0.9	0.0530	0.0069	0.6
29	0.0870	0.0066	0.3	0.0280	-0.0181	-1.6
30	0.0752	-0.0052	-0.3	0.0458	-0.0003	0.0
31	0.0845	0.0041	0.2	0.0440	-0.0021	-0.2
32				0.0480	0.0019	0.2
33	0.0698	-0.0106	-0.5	0.0453	-0.0008	-0.1
34	0.0890	0.0086	0.4	0.0478	0.0017	0.1
35	0.0851	0.0047	0.2	0.0540	0.0079	0.7
36	0.0780	-0.0024	-0.1	0.0560	0.0099	0.9
38	0.0898	0.0094	0.5	0.0491	0.0030	0.3
39	0.0816	0.0012	0.1	0.0476	0.0015	0.1
42	0.0661	-0.0143	-0.7			
44	nd	-0.0564	-3.5	0.0409	-0.0052	-0.5
45	0.0885	0.0081	0.4	0.0586	0.0125	1.1
46	nd	-0.0564	-3.5	0.0401	-0.0060	-0.5
48	0.0840	0.0036	0.2	0.0433	-0.0028	-0.2
49	0.0660	-0.0144	-0.7	nd	-0.0381	-3.5
50	0.0740	-0.0064	-0.3	nd	-0.0381	-3.5
51	0.0780	-0.0024	-0.1	0.0440	-0.0021	-0.2

Results	Cadusafos		trans-Chlordane	
Minimum required reporting level (MRRL) [mg/kg] powder provided	0.024		0.008	
Number of results	42		41	
Not analysed	1		2	
Not detected - false negatives	2		2	
Spike level [mg/kg] (x* / spike value) or from homogeneity	0.090	89.3%	0.054	85.4%
Minimum value [mg/kg]	0.0524		0.0260	
Median [mg/kg]	0.0807		0.0453	
Robust mean x* [mg/kg]	0.0804		0.0461	
Uncertainty of the robust mean [mg/kg] (coefficient of variation)	0.0021	2.6%	0.0015	3.2%
Average/mean [mg/kg]	0.0805		0.0459	
Maximum value [mg/kg]	0.1120		0.0682	
Standard deviation [mg/kg] (coefficient of variation)	0.0115	14.3%	0.0083	18.1%
Standard deviation (EUPT) [mg/kg] (coefficient of variation)	0.0201		0.0115	
Robust standard deviation s* [mg/kg] (coefficient of variation)	0.0105	13.0%	0.0074	16.0%
Acceptable results (percentage)	40	95.2%	39	95.1%
Questionable results (percentage)	0	0%	0	0%
Unacceptable results (percentage)	2	4.8%	2	4.9%

3.1.2 Diazinon and Disulfoton-sulfoxide

Lab code	Diazinon [mg/kg]			Disulfoton-sulfoxide [mg/kg]		
	Result	Deviation	z-Scores (EUPT)	Result	Deviation	z-Scores (EUPT)
2	0.0750	-0.0054	-0.3	0.0350	-0.0111	-1.0
3	0.0770	-0.0034	-0.2	0.0450	-0.0011	-0.1
5	0.0794	-0.0010	0.0	0.0481	0.0020	0.2
6	0.0840	0.0036	0.2	0.0540	0.0079	0.7
7	0.0600	-0.0204	-1.0	0.0440	-0.0021	-0.2
8	0.0680	-0.0124	-0.6			
9	0.0850	0.0046	0.2	0.0260	-0.0201	-1.7
10	0.0837	0.0033	0.2	0.0496	0.0035	0.3
11	0.0745	-0.0059	-0.3	0.0440	-0.0021	-0.2
12	0.0797	-0.0007	0.0	0.0299	-0.0162	-1.4
13	0.1010	0.0206	1.0	0.0421	-0.0040	-0.3
14	0.0905	0.0101	0.5	0.0505	0.0044	0.4
15	0.0753	-0.0051	-0.3	0.0424	-0.0037	-0.3
16	0.0880	0.0076	0.4	0.0682	0.0221	1.9
17	0.0823	0.0019	0.1	0.0437	-0.0024	-0.2
19	0.0890	0.0086	0.4	0.0533	0.0072	0.6
20	0.0761	-0.0043	-0.2	0.0520	0.0059	0.5
21	0.0524	-0.0280	-1.4	0.0514	0.0053	0.5
22	0.1120	0.0316	1.6	0.0567	0.0106	0.9
23	0.0780	-0.0024	-0.1	0.0410	-0.0051	-0.4
24	0.0604	-0.0200	-1.0	0.0432	-0.0029	-0.3
25	0.0930	0.0126	0.6	0.0480	0.0019	0.2
26	0.0897	0.0093	0.5	0.0354	-0.0107	-0.9
27	0.0700	-0.0104	-0.5	0.0395	-0.0066	-0.6
28	0.0980	0.0176	0.9	0.0530	0.0069	0.6
29	0.0870	0.0066	0.3	0.0280	-0.0181	-1.6
30	0.0752	-0.0052	-0.3	0.0458	-0.0003	0.0
31	0.0845	0.0041	0.2	0.0440	-0.0021	-0.2
32				0.0480	0.0019	0.2
33	0.0698	-0.0106	-0.5	0.0453	-0.0008	-0.1
34	0.0890	0.0086	0.4	0.0478	0.0017	0.1
35	0.0851	0.0047	0.2	0.0540	0.0079	0.7
36	0.0780	-0.0024	-0.1	0.0560	0.0099	0.9
38	0.0898	0.0094	0.5	0.0491	0.0030	0.3
39	0.0816	0.0012	0.1	0.0476	0.0015	0.1
42	0.0661	-0.0143	-0.7			
44	nd	-0.0564	-3.5	0.0409	-0.0052	-0.5
45	0.0885	0.0081	0.4	0.0586	0.0125	1.1
46	nd	-0.0564	-3.5	0.0401	-0.0060	-0.5
48	0.0840	0.0036	0.2	0.0433	-0.0028	-0.2
49	0.0660	-0.0144	-0.7	nd	-0.0381	-3.5
50	0.0740	-0.0064	-0.3	nd	-0.0381	-3.5
51	0.0780	-0.0024	-0.1	0.0440	-0.0021	-0.2

Results	Diazinon		Disulfoton-sulfoxide	
Minimum required reporting level (MRRL) [mg/kg] powder provided	0.016		0.008	
Number of results	43		39	
Not analysed	0		4	
Not detected - false negatives	1		1	
Spike level [mg/kg] (x* / spike value) or from homogeneity	0.195	84.6%	0.060	88.5%
Minimum value [mg/kg]	0.1160		0.0370	
Median [mg/kg]	0.1650		0.0538	
Robust mean x* [mg/kg]	0.1649		0.0531	
Uncertainty of the robust mean [mg/kg] (coefficient of variation)	0.0054	3.3%	0.0015	2.8%
Average/mean [mg/kg]	0.1674		0.0536	
Maximum value [mg/kg]	0.2500		0.0875	
Standard deviation [mg/kg] (coefficient of variation)	0.0311	18.8%	0.0087	16.5%
Standard deviation (EUPT) [mg/kg] (coefficient of variation)	0.0412		0.0133	
Robust standard deviation s* [mg/kg] (coefficient of variation)	0.0280	17.0%	0.0072	13.6%
Acceptable results (percentage)	41	95.3%	37	95.1%
Questionable results (percentage)	1	2.3%	1	0%
Unacceptable results (percentage)	1	2.3%	1	4.9%

3.1.3 Endrin and Fipronil-sulfone

Lab code	Endrin [mg/kg]			Fipronil-sulfone [mg/kg]		
	Result	Deviation	z-Scores (EUPT)	Result	Deviation	z-Scores (EUPT)
2	0.0140	0.0007	0.2	0.1410	0.0075	0.2
3	0.0160	0.0027	0.8	nd	-0.1175	-3.5
5	nd	-0.0101	-3.5	0.1370	0.0035	0.1
6	0.0110	-0.0023	-0.7	0.1500	0.0165	0.5
7	0.0110	-0.0023	-0.7	0.1040	-0.0295	-0.9
8	0.0096	-0.0037	-1.1	0.1230	-0.0105	-0.3
9	0.0100	-0.0033	-1.0	0.1240	-0.0095	-0.3
10	0.0132	-0.0001	0.0	0.1260	-0.0075	-0.2
11	0.0120	-0.0013	-0.4	0.0649	-0.0686	-2.1
12	0.0158	0.0025	0.8	0.1370	0.0035	0.1
13	0.0110	-0.0023	-0.7	0.1370	0.0035	0.1
14	0.0120	-0.0013	-0.4	0.1330	-0.0005	0.0
15	0.0063	-0.0070	-2.1	0.1270	-0.0065	-0.2
16	0.0204	0.0071	2.1	0.1260	-0.0075	-0.2
17	0.0140	0.0007	0.2	0.1310	-0.0025	-0.1
19	0.0125	-0.0008	-0.2	0.1470	0.0135	0.4
20	0.0125	-0.0008	-0.2	0.1320	-0.0015	0.0
21	0.0181	0.0048	1.4	0.1630	0.0295	0.9
22	0.0106	-0.0027	-0.8	0.1280	-0.0055	-0.2
23	0.0160	0.0027	0.8	0.1420	0.0085	0.3
24	0.0116	-0.0017	-0.5	0.0825	-0.0510	-1.5
25	0.0160	0.0027	0.8	0.1400	0.0065	0.2
26	0.0171	0.0038	1.1	0.1050	-0.0285	-0.9
27	0.0152	0.0019	0.6	0.1340	0.0005	0.0
28	0.0140	0.0007	0.2	0.1800	0.0465	1.4
29	0.0090	-0.0043	-1.3	0.1150	-0.0185	-0.6
30	0.0134	0.0001	0.0	0.1330	-0.0005	0.0
31	0.0147	0.0014	0.4	0.1420	0.0085	0.3
32	0.0150	0.0017	0.5	0.1270	-0.0065	-0.2
33	0.0124	-0.0009	-0.3	0.1400	0.0065	0.2
34	0.0131	-0.0002	-0.1	0.1180	-0.0155	-0.5
35	0.0152	0.0019	0.6	0.1440	0.0105	0.3
36	0.0170	0.0037	1.1	0.1330	-0.0005	0.0
38	0.0101	-0.0032	-1.0	0.1400	0.0065	0.2
39	0.0139	0.0006	0.2	0.1290	-0.0045	-0.1
42	0.0176	0.0043	1.3	0.1490	0.0155	0.5
44	0.0124	-0.0009	-0.3	0.1670	0.0335	1.0
45	0.0176	0.0043	1.3	0.1410	0.0075	0.2
46	0.0104	-0.0029	-0.9	nd	-0.1175	-3.5
48	0.0100	-0.0033	-1.0	0.1290	-0.0045	-0.1
49	0.0120	-0.0013	-0.4	0.1980	0.0645	1.9
50	nd	-0.0101	-3.5	0.1200	-0.0135	-0.4
51	0.0120	-0.0013	-0.4	0.1290	-0.0045	-0.1

Results	Endrin		Fipronil-sulfone	
Minimum required reporting level (MRRL) [mg/kg] powder provided	0.0032		0.016	
Number of results	43		43	
Not analysed	0		0	
Not detected - false negatives	2		2	
Spike level [mg/kg] (x* / spike value) or from homogeneity	0.015	88.7%	0.150	88.5%
Minimum value [mg/kg]	0.0063		0.0649	
Median [mg/kg]	0.0131		0.1330	
Robust mean x* [mg/kg]	0.0133		0.1335	
Uncertainty of the robust mean [mg/kg] (coefficient of variation)	0.0006	4.4%	0.0027	2.8%
Average/mean [mg/kg]	0.0133		0.1334	
Maximum value [mg/kg]	0.0204		0.1980	
Standard deviation [mg/kg] (coefficient of variation)	0.0029	21.8%	0.0222	16.5%
Standard deviation (EUPT) [mg/kg] (coefficient of variation)	0.0033		0.0334	
Robust standard deviation s* [mg/kg] (coefficient of variation)	0.0030	22.3%	0.0137	13.6%
Acceptable results (percentage)	39	90.7%	40	93.0%
Questionable results (percentage)	2	4.7%	1	2.3%
Unacceptable results (percentage)	2	4.7%	2	4.7%

3.1.4 Heptachlor and Hexachlorobenzene (HCB)

Lab code	Heptachlor [mg/kg]			Hexachlorobenzene [mg/kg]		
	Result	Deviation	z-Scores (EUPT)	Result	Deviation	z-Scores (EUPT)
2	0.0690	-0.0049	-0.3	0.0720	0.0077	0.5
3	0.0810	0.0071	0.4	0.0500	-0.0143	-0.9
5	0.0835	0.0096	0.5	0.0710	0.0067	0.4
6	0.0790	0.0051	0.3	0.0700	0.0057	0.4
7	0.0700	-0.0039	-0.2	0.0550	-0.0093	-0.6
8	0.0438	-0.0301	-1.6	0.0597	-0.0046	-0.3
9	0.0400	-0.0339	-1.8	0.0210	-0.0433	-2.7
10	0.0693	-0.0046	-0.2	0.0901	0.0258	1.6
11	0.0750	0.0011	0.1	0.0641	-0.0002	0.0
12	0.0518	-0.0221	-1.2	0.1020	0.0377	2.3
13	0.0715	-0.0024	-0.1	0.0631	-0.0012	-0.1
14	0.0820	0.0081	0.4	0.0700	0.0057	0.4
15	0.0672	-0.0067	-0.4	0.0583	-0.0060	-0.4
16	0.0700	-0.0039	-0.2	0.0397	-0.0246	-1.5
17	0.0713	-0.0026	-0.1	0.0565	-0.0078	-0.5
19	0.0954	0.0215	1.2	0.0691	0.0048	0.3
20	0.0775	0.0036	0.2	0.0740	0.0097	0.6
21	0.0581	-0.0158	-0.9	0.0422	-0.0221	-1.4
22	0.0795	0.0056	0.3	0.0628	-0.0015	-0.1
23	0.0780	0.0041	0.2	0.0710	0.0067	0.4
24	0.0758	0.0019	0.1	0.0571	-0.0072	-0.4
25	0.0820	0.0081	0.4	0.0570	-0.0073	-0.5
26	0.0610	-0.0129	-0.7	0.0488	-0.0155	-1.0
27	0.0773	0.0034	0.2	0.0695	0.0052	0.3
28	0.0860	0.0121	0.7	0.0720	0.0077	0.5
29	0.0450	-0.0289	-1.6	0.0350	-0.0293	-1.8
30	0.0844	0.0105	0.6	0.0674	0.0031	0.2
31	0.0760	0.0021	0.1	0.0510	-0.0133	-0.8
32	0.0850	0.0111	0.6	0.0630	-0.0013	-0.1
33	0.0747	0.0008	0.0	0.0608	-0.0035	-0.2
34	0.0710	-0.0029	-0.2	0.0618	-0.0025	-0.2
35	0.0827	0.0088	0.5	0.0698	0.0055	0.3
36	0.0850	0.0111	0.6	0.0780	0.0137	0.9
38	0.0802	0.0063	0.3	0.0790	0.0147	0.9
39	0.0856	0.0117	0.6	0.0686	0.0043	0.3
42	0.1170	0.0431	2.3	0.0919	0.0276	1.7
44	0.0746	0.0007	0.0	0.0962	0.0319	2.0
45	0.0866	0.0127	0.7	0.0764	0.0121	0.8
46	0.0719	-0.0020	-0.1	0.0571	-0.0072	-0.4
48	0.0665	-0.0074	-0.4	0.0594	-0.0049	-0.3
49	0.0550	-0.0189	-1.0	0.0630	-0.0013	-0.1
50	0.0640	-0.0099	-0.5	0.0560	-0.0083	-0.5
51	0.0610	-0.0129	-0.7	0.0510	-0.0133	-0.8

Results	Heptachlor		Hexachlorobenzene (HCB)	
Minimum required reporting level (MRRL) [mg/kg] powder provided	0.008		0.016	
Number of results	43		43	
Not analysed	0		0	
Not detected - false negatives	0		0	
Spike level [mg/kg] (x* / spike value) or from homogeneity	0.090	82.1%	0.080	80.4%
Minimum value [mg/kg]	0.0400		0.0210	
Median [mg/kg]	0.0750		0.0630	
Robust mean x* [mg/kg]	0.0739		0.0643	
Uncertainty of the robust mean [mg/kg] (coefficient of variation)	0.0022	2.9%	0.0024	3.8%
Average/mean [mg/kg]	0.0735		0.0640	
Maximum value [mg/kg]	0.1170		0.1020	
Standard deviation [mg/kg] (coefficient of variation)	0.0140	18.9%	0.0154	24.0%
Standard deviation (EUPT) [mg/kg] (coefficient of variation)	0.0185		0.0161	
Robust standard deviation s* [mg/kg] (coefficient of variation)	0.0114	15.4%	0.0128	20.0%
Acceptable results (percentage)	42	97.7%	41	95.3%
Questionable results (percentage)	1	2.3%	2	4.7%
Unacceptable results (percentage)	0	0%	0	0%

3.1.5 Nitrofen and Terbufos

Lab code	Nitrofen [mg/kg]			Terbufos [mg/kg]		
	Result	Deviation	z-Scores (EUPT)	Result	Deviation	z-Scores (EUPT)
2	0.0880	0.0046	0.2	0.1230	0.0019	0.1
3	0.0850	0.0016	0.1	0.0620	-0.0591	-2.0
5	0.1010	0.0176	0.8	0.1220	0.0009	0.0
6	0.0970	0.0136	0.7	0.1000	-0.0211	-0.7
7	0.0670	-0.0164	-0.8	0.1010	-0.0201	-0.7
8	0.0690	-0.0144	-0.7			
9	0.0530	-0.0304	-1.5	0.1150	-0.0061	-0.2
10	0.0985	0.0151	0.7	0.1320	0.0109	0.4
11	0.0810	-0.0024	-0.1	0.1180	-0.0031	-0.1
12	0.0948	0.0114	0.5	0.1310	0.0099	0.3
13	0.0648	-0.0186	-0.9	0.1360	0.0149	0.5
14	0.0617	-0.0217	-1.0	0.1180	-0.0031	-0.1
15	0.0721	-0.0113	-0.5	0.1040	-0.0171	-0.6
16	0.1030	0.0196	0.9	0.1290	0.0079	0.3
17	0.0833	-0.0001	0.0	0.1710	0.0499	1.6
19	0.0992	0.0158	0.8	0.1670	0.0459	1.5
20	0.0761	-0.0073	-0.4	0.1140	-0.0071	-0.2
21	0.0847	0.0013	0.1	nd	-0.1051	-3.5
22	0.0799	-0.0035	-0.2	0.0893	-0.0318	-1.1
23	0.0900	0.0066	0.3	0.1250	0.0039	0.1
24	0.0659	-0.0175	-0.8	0.1190	-0.0021	-0.1
25	0.0990	0.0156	0.7	0.1220	0.0009	0.0
26	0.0538	-0.0296	-1.4			
27	0.0898	0.0064	0.3	0.1120	-0.0091	-0.3
28	0.1000	0.0166	0.8	0.1100	-0.0111	-0.4
29	0.0720	-0.0114	-0.5	0.1190	-0.0021	-0.1
30	0.0971	0.0137	0.7	0.1740	0.0529	1.7
31	0.0920	0.0086	0.4			
32	nd	-0.0674	-3.5			
33	0.0555	-0.0279	-1.3	0.0858	-0.0353	-1.2
34	0.0753	-0.0081	-0.4	0.1390	0.0179	0.6
35	0.1040	0.0206	1.0	0.1410	0.0199	0.7
36	0.0960	0.0126	0.6	0.1320	0.0109	0.4
38	0.0657	-0.0177	-0.8	0.1490	0.0279	0.9
39	0.0791	-0.0043	-0.2	0.1240	0.0029	0.1
42	0.1230	0.0396	1.9	0.1150	-0.0061	-0.2
44	0.0978	0.0144	0.7	nd	-0.1051	-3.5
45	0.0994	0.0160	0.8	0.1450	0.0239	0.8
46	nd	-0.0674	-3.5	nd	-0.1051	-3.5
48	0.0807	-0.0027	-0.1	0.1120	-0.0091	-0.3
49	0.0740	-0.0094	-0.5	0.1210	-0.0001	0.0
50	0.0790	-0.0044	-0.2	0.1120	-0.0091	-0.3
51	0.0710	-0.0124	-0.6	0.0500	-0.0711	-2.3

Results	Nitrofen		Terbufos	
Minimum required reporting level (MRRL) [mg/kg] powder provided	0.016		0.016	
Number of results	43		39	
Not analysed	0		4	
Not detected - false negatives	2		3	
Spike level [mg/kg] (x* / spike value) or from homogeneity	0.110	75.8%	0.150	80.7%
Minimum value [mg/kg]	0.0530		0.0500	
Median [mg/kg]	0.0833		0.1200	
Robust mean x* [mg/kg]	0.0834		0.1211	
Uncertainty of the robust mean [mg/kg] (coefficient of variation)	0.0032	3.9%	0.0041	3.4%
Average/mean [mg/kg]	0.0834		0.1205	
Maximum value [mg/kg]	0.1230		0.1740	
Standard deviation [mg/kg] (coefficient of variation)	0.0159	19.1%	0.0254	21.0%
Standard deviation (EUPT) [mg/kg] (coefficient of variation)	0.0209		0.0303	
Robust standard deviation s* [mg/kg] (coefficient of variation)	0.0165	19.8%	0.0196	16.1%
Acceptable results (percentage)	41	95.3%	35	89.7%
Questionable results (percentage)	0	0%	1	2.6%
Unacceptable results (percentage)	2	4.7%	3	7.7%

3.2 Voluntary analytes

3.2.1 BAC-C12 and BAC-C14

Lab code	BAC-C12 [mg/kg]			BAC-C14 [mg/kg]		
	Result	Deviation	z-Scores (EUPT)	Result	Deviation	z-Scores (EUPT)
2	0.1680	-0.0330	-0.7	0.1440	-0.0273	-0.6
3						
5						
6	0.2600	0.0590	1.2	0.2100	0.0387	0.9
7						
8						
9	0.1620	-0.0390	-0.8	0.1160	-0.0553	-1.3
10						
11						
12	0.2330	0.0320	0.6	0.1920	0.0207	0.5
13						
14	0.2100	0.0090	0.2	0.1970	0.0257	0.6
15						
16	0.2480	0.0470	0.9	0.2010	0.0297	0.7
17						
19	0.2200	0.0190	0.4	0.1400	-0.0313	-0.7
20						
21	0.2290	0.0280	0.6	0.1920	0.0207	0.5
22						
23	0.1720	-0.0290	-0.6	0.1480	-0.0233	-0.5
24						
25						
26	0.2050	0.0040	0.1	0.1620	-0.0093	-0.2
27	0.1840	-0.0170	-0.3	0.1560	-0.0153	-0.4
28	0.2900	0.0890	1.8	0.2100	0.0387	0.9
29	0.1970	-0.0040	-0.1	0.1720	0.0007	0.0
30						
31						
32						
33	0.1820	-0.0190	-0.4	0.2140	0.0427	1.0
34	0.1740	-0.0270	-0.5	0.1470	-0.0243	-0.6
35						
36	0.2000	-0.0010	0.0	0.1900	0.0187	0.4
38						
39	0.1920	-0.0090	-0.2	0.1540	-0.0173	-0.4
42	nd	-0.1610	-3.5	nd	-0.1313	-3.5
44	nd	-0.1610	-3.5	nd	-0.1313	-3.5
45						
46	nd	-0.1610	-3.5	nd	-0.1313	-3.5
48	0.1670	-0.0340	-0.7	0.1400	-0.0313	-0.7
49	nd	-0.1610	-3.5	nd	-0.1313	-3.5
50	nd	-0.1610	-3.5	nd	-0.1313	-3.5
51	0.1700	-0.0310	-0.6	0.1630	-0.0083	-0.2

Results	BAC-C12		BAC-C14	
Minimum required reporting level (MRRL) [mg/kg] powder provided	0.04		0.04	
Number of results	24		24	
Not analysed	19		19	
Not detected - false negatives	5		5	
Spike level [mg/kg] (x* / spike value) or from homogeneity	0.260	77.3%	0.220	77.9%
Minimum value [mg/kg]	0.1620		0.1160	
Median [mg/kg]	0.1970		0.1630	
Robust mean x* [mg/kg]	0.2010		0.1713	
Uncertainty of the robust mean [mg/kg] (coefficient of variation)	0.0100	5.0%	0.0092	5.4%
Average/mean [mg/kg]	0.2033		0.1709	
Maximum value [mg/kg]	0.2900		0.2140	
Standard deviation [mg/kg] (coefficient of variation)	0.0356	17.7%	0.0290	16.9%
Standard deviation (EUPT) [mg/kg] (coefficient of variation)	0.0503		0.0428	
Robust standard deviation s* [mg/kg] (coefficient of variation)	0.0348	17.3%	0.0321	18.7%
Acceptable results (percentage)	19	79.2%	19	79.2%
Questionable results (percentage)	0	0%	0	0%
Unacceptable results (percentage)	5	20.8%	5	20.8%

3.2.2 Chlorfenvinphos and Fluquinconazole

Lab code	Chlorfenvinphos [mg/kg]			Fluquinconazole [mg/kg]		
	Result	Deviation	z-Scores (EUPT)	Result	Deviation	z-Scores (EUPT)
2	0.1650	0.0097	0.2	0.1290	0.0056	0.2
3	0.0770	-0.0783	-2.0			
5	0.1660	0.0107	0.3	0.1200	-0.0034	-0.1
6	0.1700	0.0147	0.4	0.1200	-0.0034	-0.1
7	0.1080	-0.0473	-1.2	0.1070	-0.0164	-0.5
8	0.1450	-0.0103	-0.3	0.0486	-0.0748	-2.4
9	0.1460	-0.0093	-0.2	0.1200	-0.0034	-0.1
10	0.1710	0.0157	0.4	0.1264	0.0030	0.1
11	0.1530	-0.0023	-0.1	0.1120	-0.0114	-0.4
12	0.1710	0.0157	0.4	0.1540	0.0306	1.0
13	0.1650	0.0097	0.2	0.1210	-0.0024	-0.1
14	0.1570	0.0017	0.0	0.1200	-0.0034	-0.1
15	0.1500	-0.0053	-0.1	0.1160	-0.0074	-0.2
16	0.1920	0.0367	0.9	0.1570	0.0336	1.1
17	0.1460	-0.0093	-0.2	0.1350	0.0116	0.4
19	0.1860	0.0307	0.8			
20	0.1330	-0.0223	-0.6	0.1260	0.0026	0.1
21	0.1430	-0.0123	-0.3	0.1270	0.0036	0.1
22	0.1760	0.0207	0.5	0.1150	-0.0084	-0.3
23	0.1670	0.0117	0.3	0.1090	-0.0144	-0.5
24	0.1130	-0.0423	-1.1	0.1060	-0.0174	-0.6
25	0.1930	0.0377	1.0			
26	0.1260	-0.0293	-0.8	0.0677	-0.0557	-1.8
27	0.1640	0.0087	0.2	0.1150	-0.0084	-0.3
28	0.1800	0.0247	0.6	0.1400	0.0166	0.5
29	0.1730	0.0177	0.5	0.1330	0.0096	0.3
30	0.1850	0.0297	0.8	0.1260	0.0026	0.1
31						
32						
33	0.0879	-0.0674	-1.7	0.0643	-0.0591	-1.9
34	0.1550	-0.0003	0.0	0.1240	0.0006	0.0
35	0.1650	0.0097	0.2	0.1550	0.0316	1.0
36	0.1440	-0.0113	-0.3	0.1350	0.0116	0.4
38	0.1670	0.0117	0.3	0.1340	0.0106	0.3
39	0.1340	-0.0213	-0.5	0.1240	0.0006	0.0
42	0.1340	-0.0213	-0.5	0.1060	-0.0174	-0.6
44	0.1810	0.0257	0.7	0.1440	0.0206	0.7
45	0.1700	0.0147	0.4			
46	nd	-0.1153	-3.5	nd	-0.0834	-3.7
48	0.1630	0.0077	0.2	0.1360	0.0126	0.4
49	nd	-0.1153	-3.5	nd	-0.0834	-3.7
50	0.1320	-0.0233	-0.6	0.1500	0.0266	0.9
51	0.1190	-0.0363	-0.9	0.1020	-0.0214	-0.7

Results	Chlorfenvinphos		Fluquinconazole	
Minimum required reporting level (MRRL) [mg/kg] powder provided	0.04		0.04	
Number of results	41		37	
Not analysed	2		6	
Not detected - false negatives	2		2	
Spike level [mg/kg] (x* / spike value) or from homogeneity	0.180	86.3%	0.140	88.1%
Minimum value [mg/kg]	0.0770		0.0486	
Median [mg/kg]	0.1630		0.1240	
Robust mean x* [mg/kg]	0.1553		0.1234	
Uncertainty of the robust mean [mg/kg] (coefficient of variation)	0.0050	3.2%	0.0037	3.0%
Average/mean [mg/kg]	0.1532		0.1207	
Maximum value [mg/kg]	0.1930		0.1570	
Standard deviation [mg/kg] (coefficient of variation)	0.0269	17.3%	0.0237	19.2%
Standard deviation (EUPT) [mg/kg] (coefficient of variation)	0.0388		0.0309	
Robust standard deviation s* [mg/kg] (coefficient of variation)	0.0252	16.2%	0.0176	14.3%
Acceptable results (percentage)	39	79.2%	19	79.2%
Questionable results (percentage)	0	0%	0	0%
Unacceptable results (percentage)	2	20.8%	5	20.8%

3.2.3 Chlorate (evaluation only for information!)

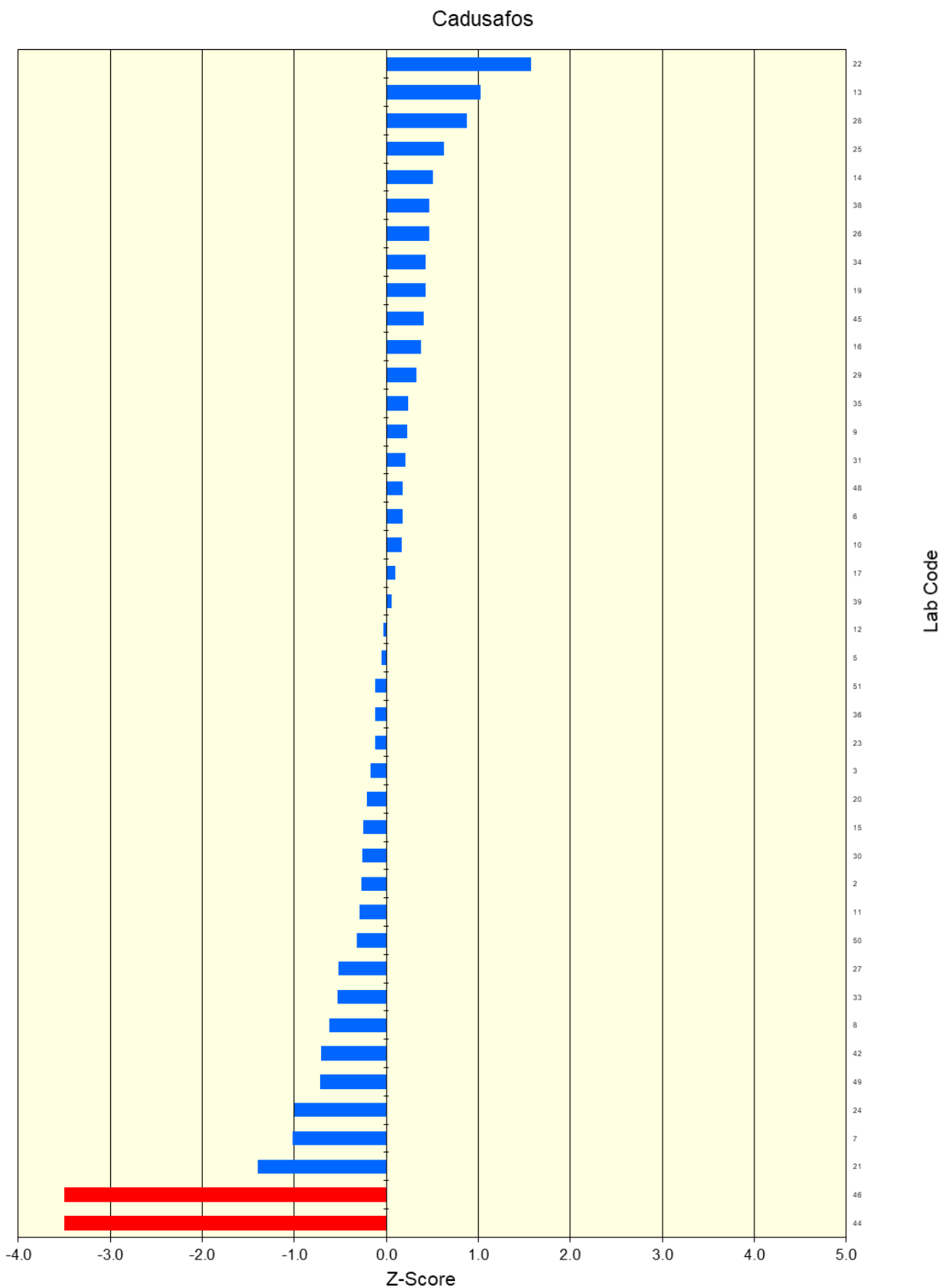
Lab code	Chlorate [mg/kg]		
	Result	Deviation	z-Scores (EUPT)
2			
3			
5	0.1420	-0.0040	-0.1
6			
7			
8			
9	0.1420	-0.0040	-0.1
10			
11			
12	0.1500	0.0040	0.1
13			
14	0.1340	-0.0120	-0.3
15			
16			
17			
19	0.0470	-0.0990	-2.7
20			
21			
22			
23	0.1490	0.0030	0.1
24			
25	nd	-0.1060	-3.7
26			
27			
28	0.2000	0.0540	1.5
29	0.1500	0.0040	0.1
30			
31			
32			
33			
34	0.3450	0.1990	5.0
35			
36	0.1600	0.0140	0.4
38			
39			
42	nd	-0.1060	-3.7
44	nd	-0.1060	-3.7
45			
46	nd	-0.1060	-3.7
48	0.1090	-0.0370	-1.0
49	nd	-0.1060	-3.7
50	nd	-0.1060	-3.7
51	0.1250	-0.0210	-0.6

Results	Chlorate	
Minimum required reporting level (MRRL) [mg/kg] powder provided	0.04	
Number of results	18	
Not analysed	25	
Not detected - false negatives	6	
Spike level [mg/kg] (x* / spike value) or from homogeneity	0.190	76.8%
Minimum value [mg/kg]	0.0470	
Median [mg/kg]	0.1455	
Robust mean x* [mg/kg]	0.1460	
Uncertainty of the robust mean [mg/kg] (coefficient of variation)	0.0127	8.67%
Average/mean [mg/kg]	0.1544	
Maximum value [mg/kg]	0.3450	
Standard deviation [mg/kg] (coefficient of variation)	0.0699	47.9%
Standard deviation (EUPT) [mg/kg] (coefficient of variation)	0.0365	
Robust standard deviation s* [mg/kg] (coefficient of variation)	0.0351	24.0%
Acceptable results (percentage)	10	55.6%
Questionable results (percentage)	1	5.6%
Unacceptable results (percentage)	7	38.9%

4 Graphs

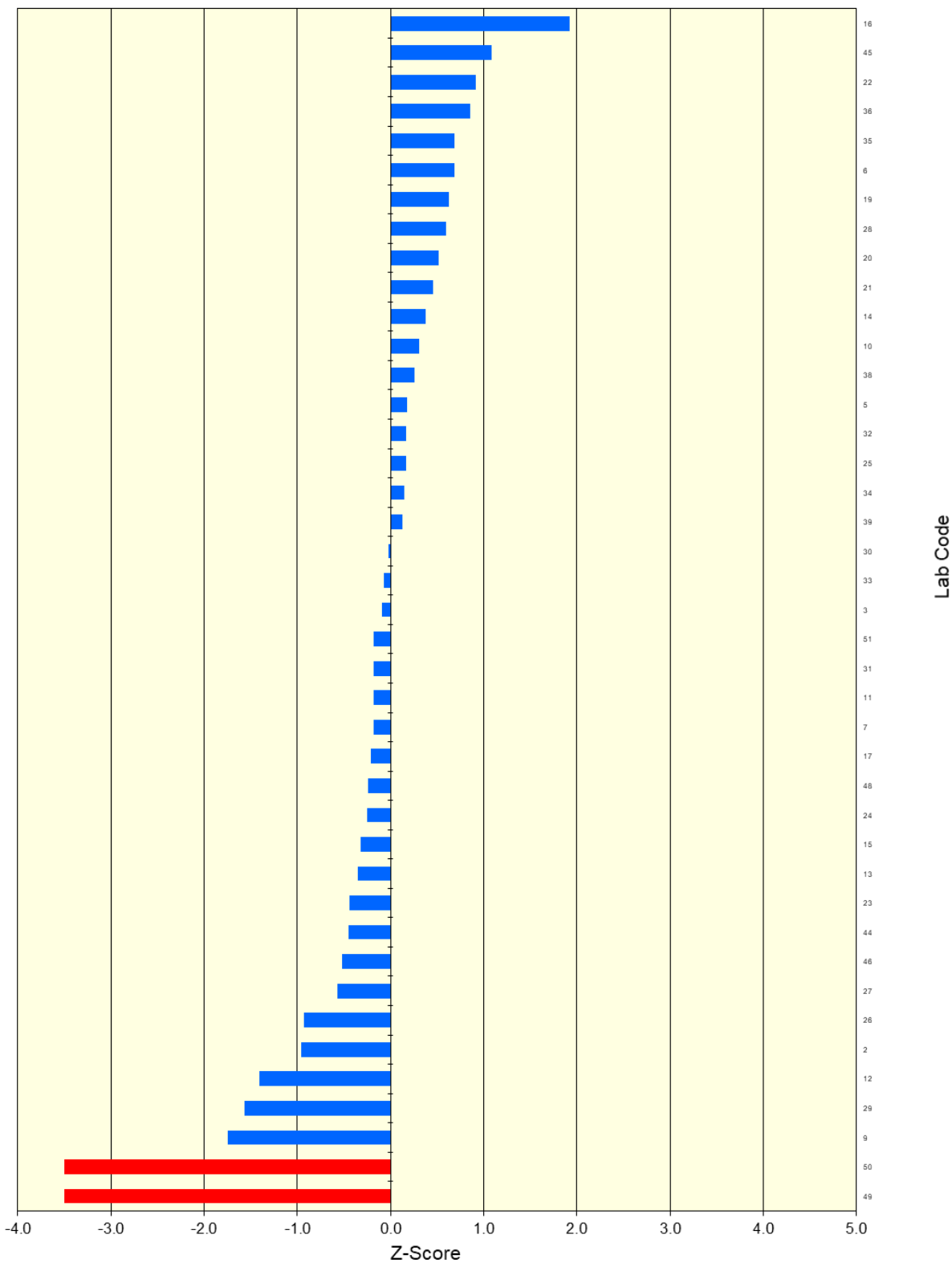
4.1 Individual z-scores

4.1.1 Cadusafos

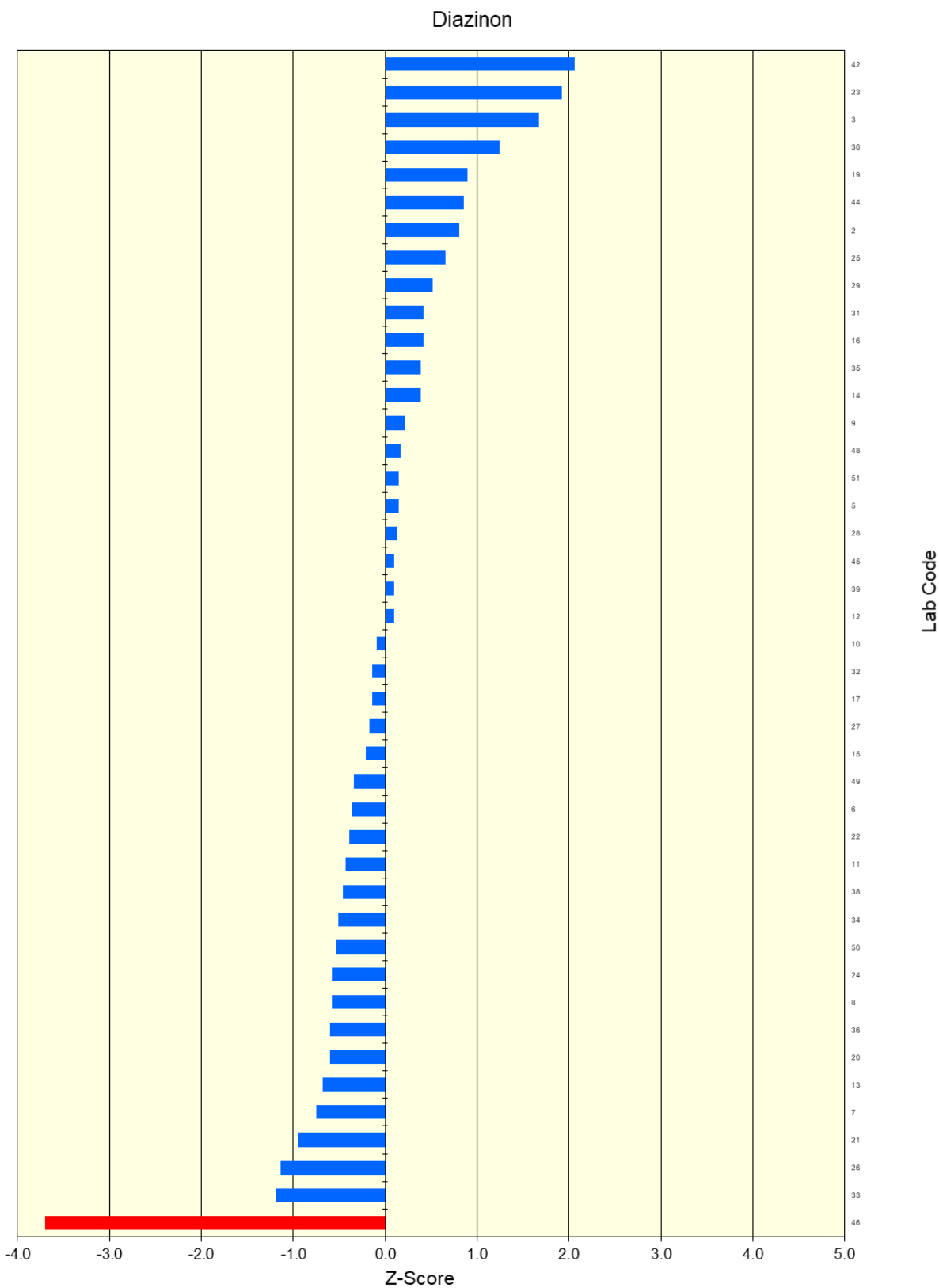


4.1.2 trans-Chlordane

Chlordane-trans

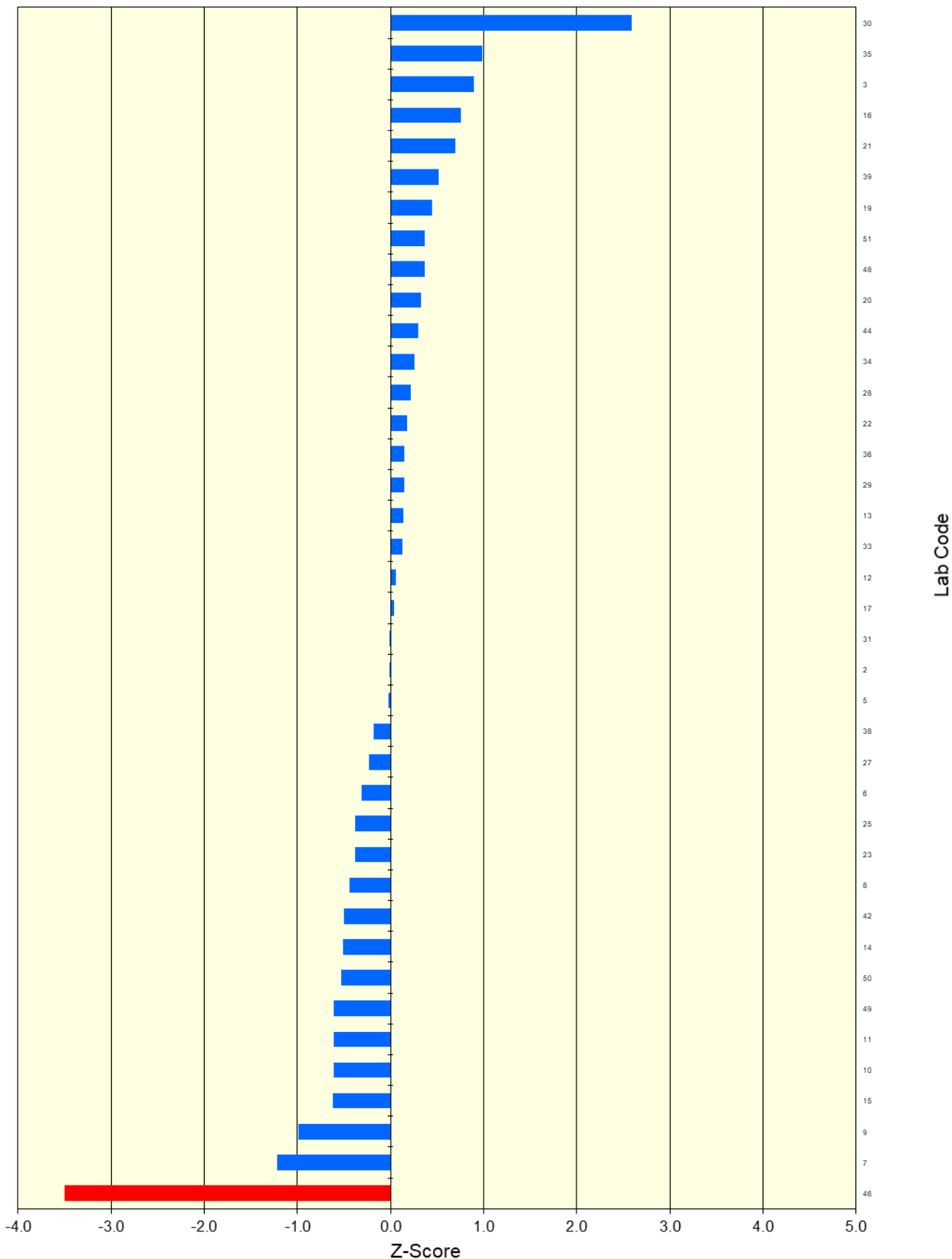


4.1.3 Diazinon

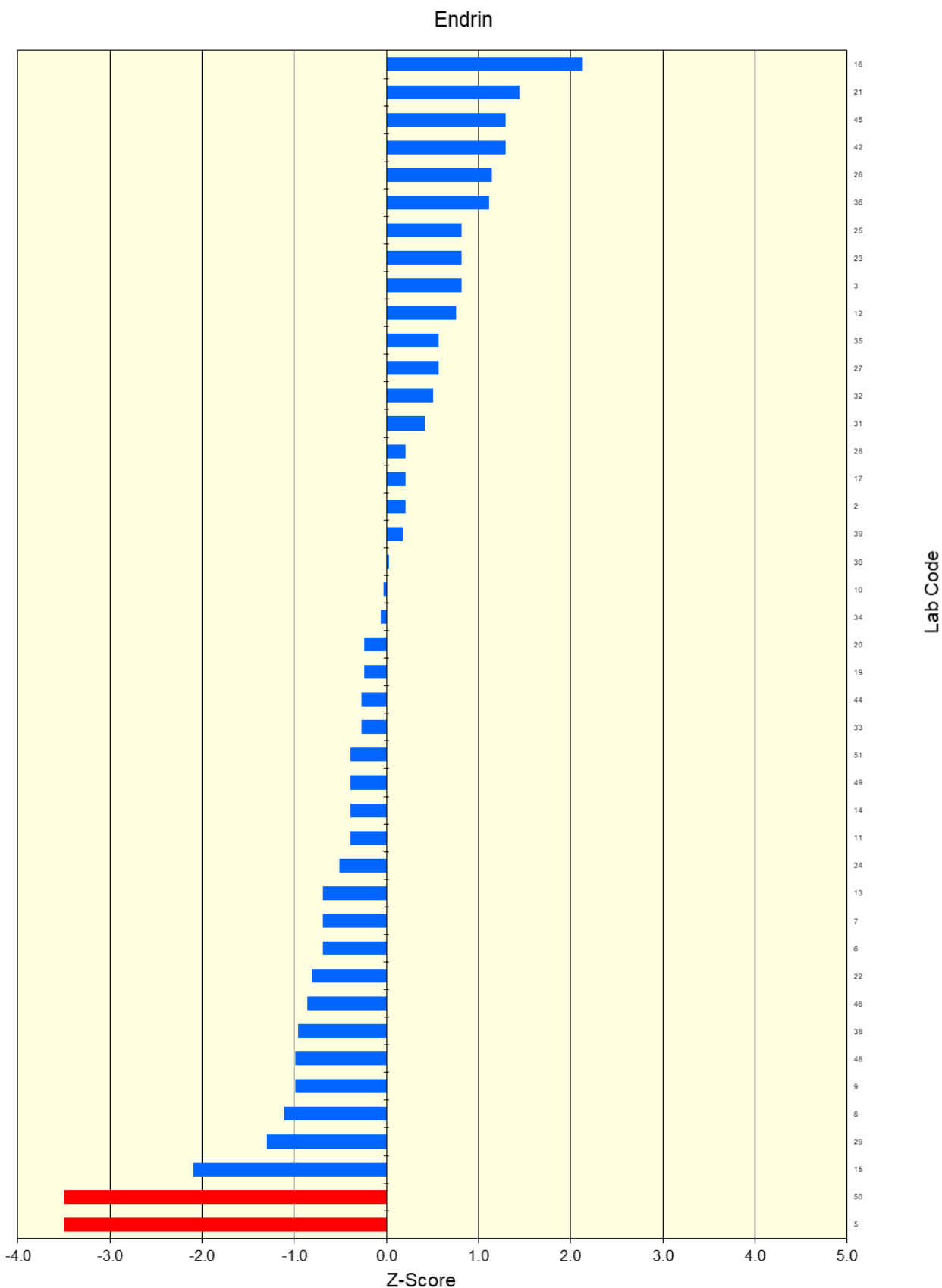


4.1.4 Disulfoton-sulfoxide

Disulfoton-sulfoxide

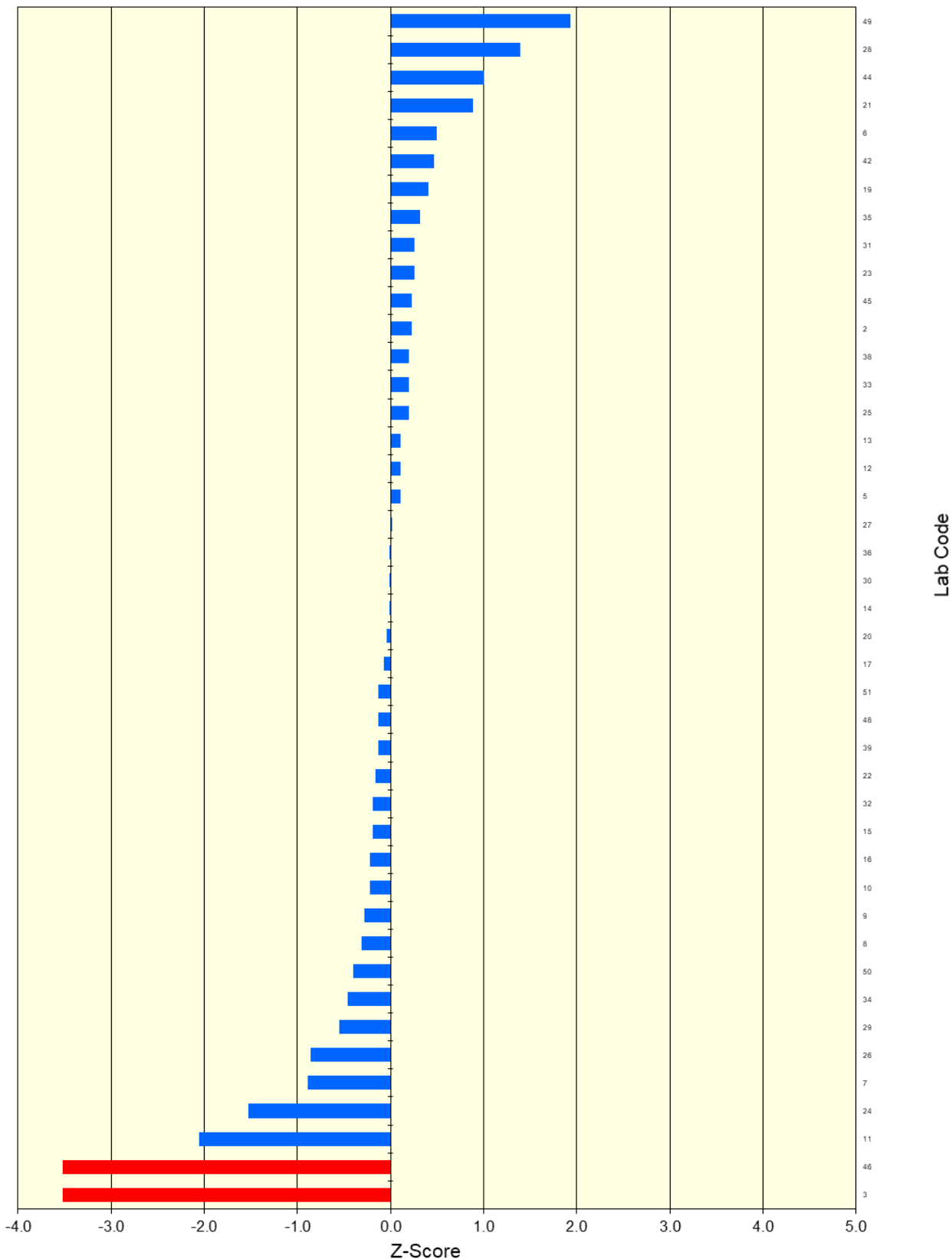


4.1.5 Endrin

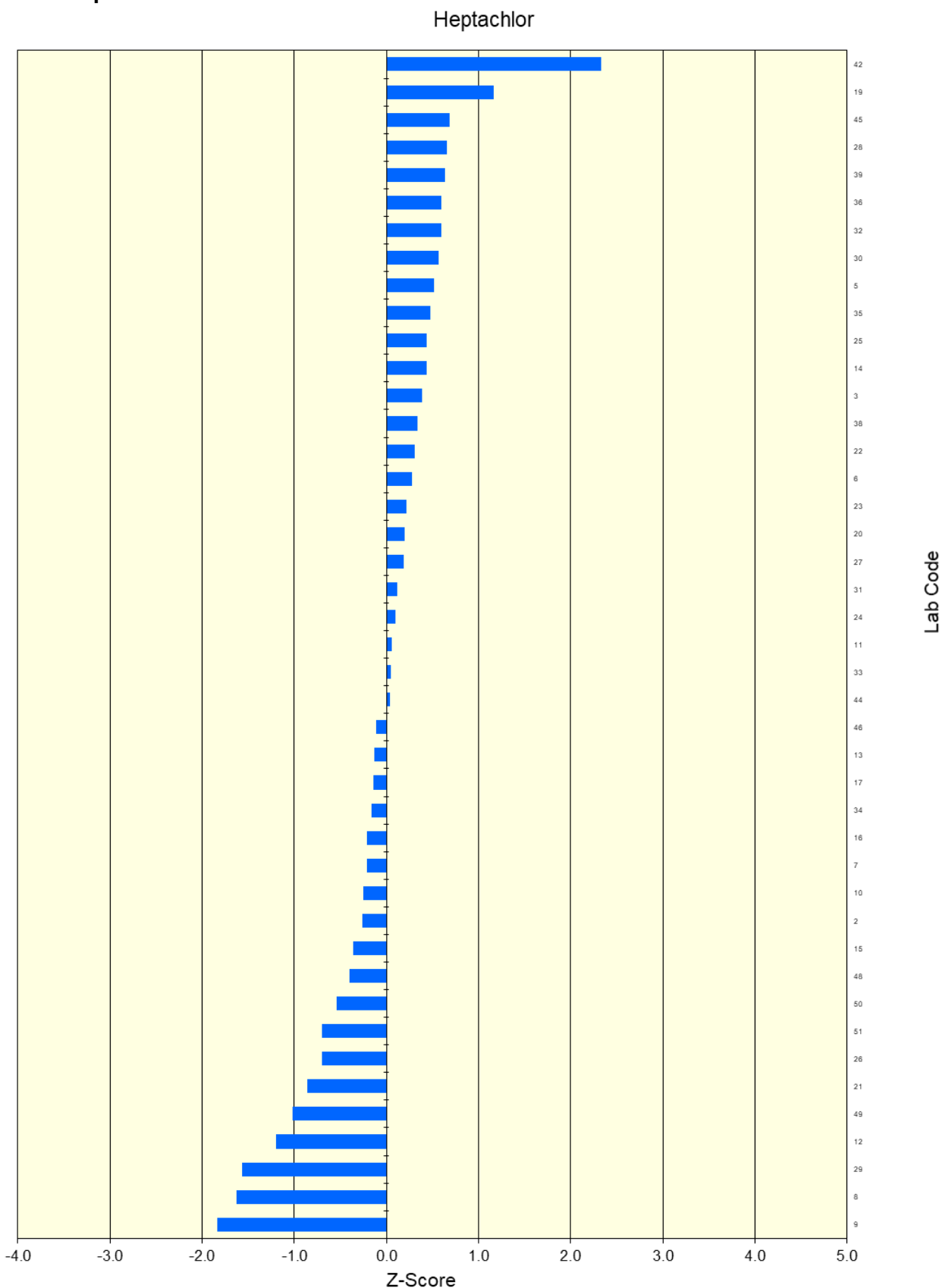


4.1.6 Fipronil-sulfone

Fipronil-sulfone

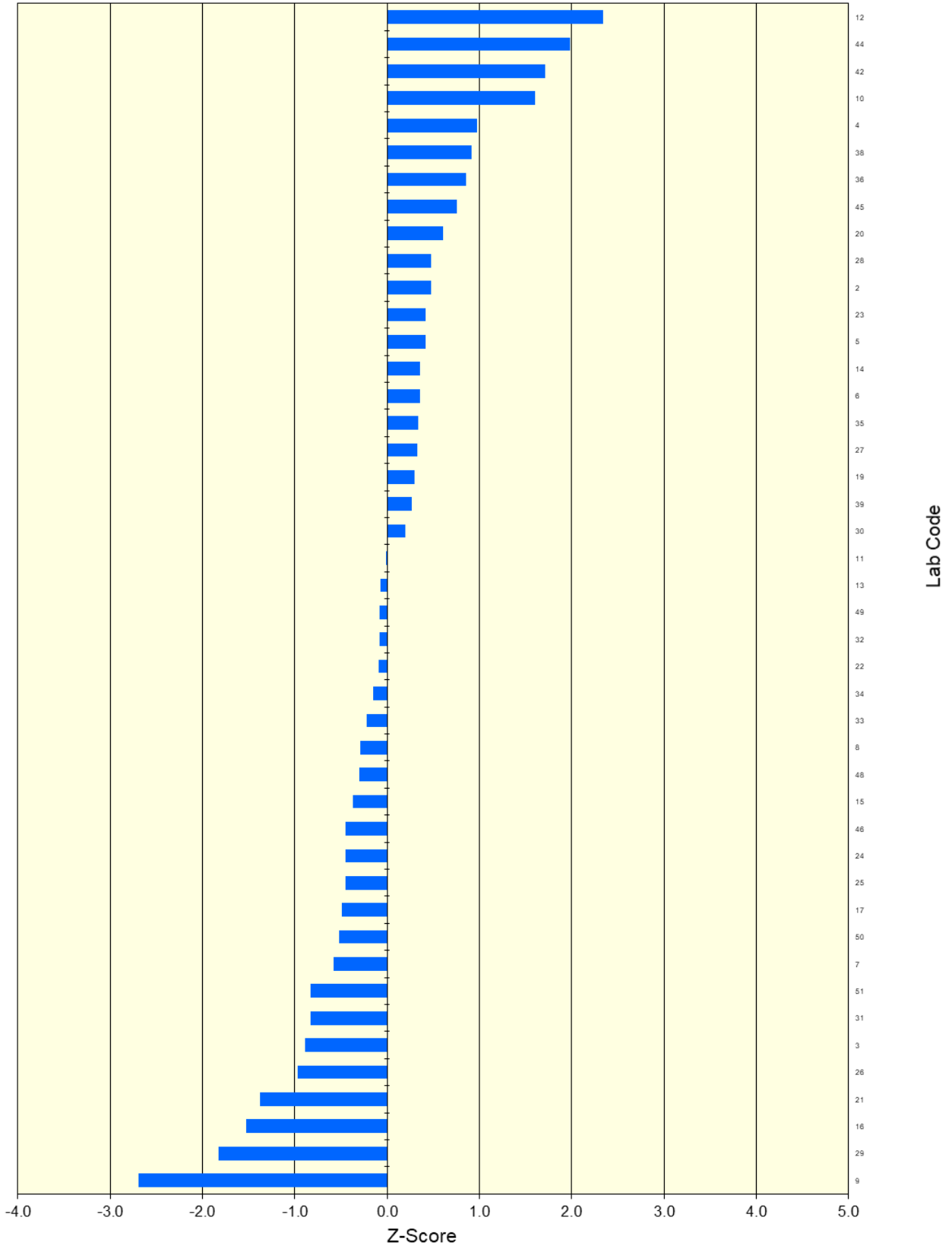


4.1.7 Heptachlor

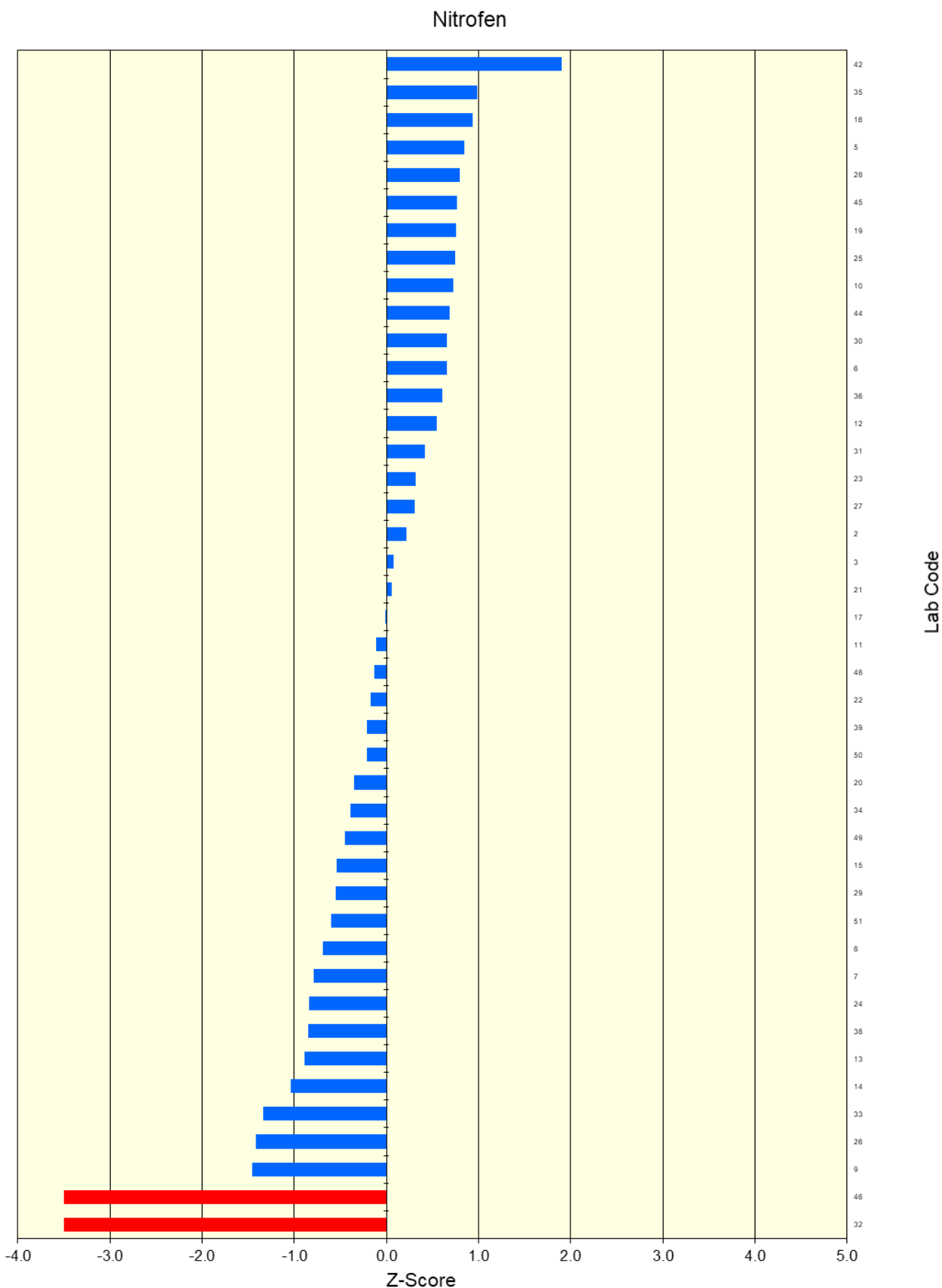


4.1.8 Hexachlorobenzene (HCB)

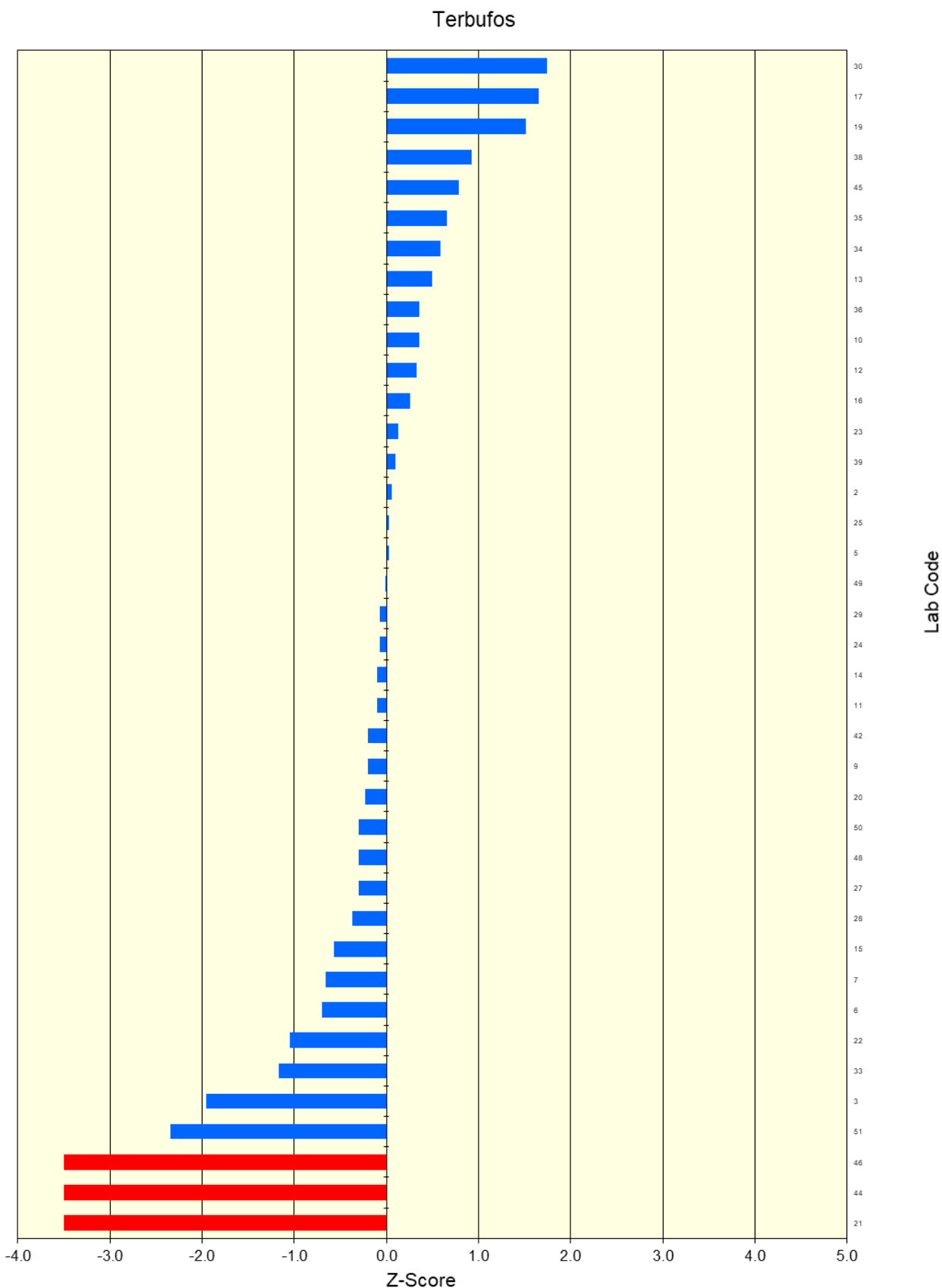
Hexachlorobenzene



4.1.9 Nitrofen

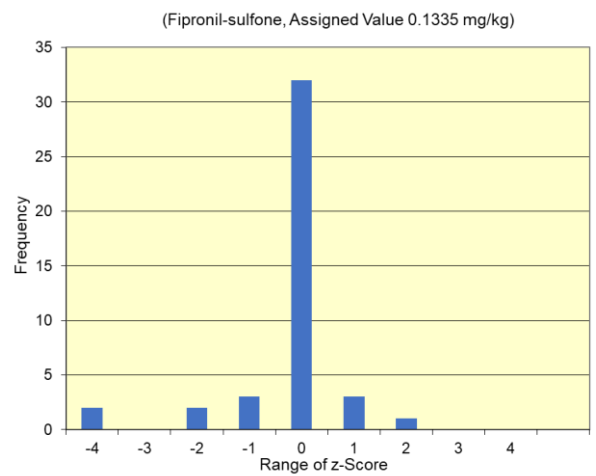
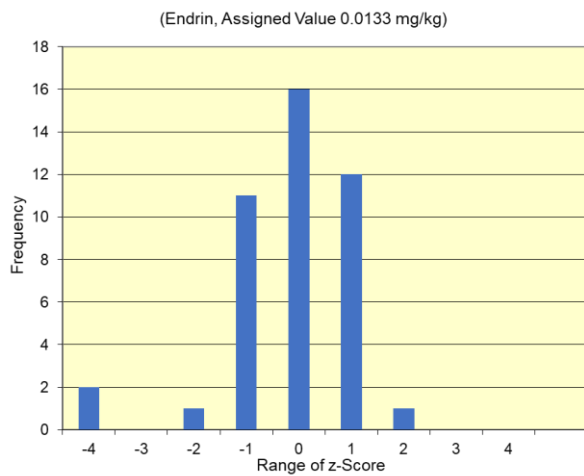
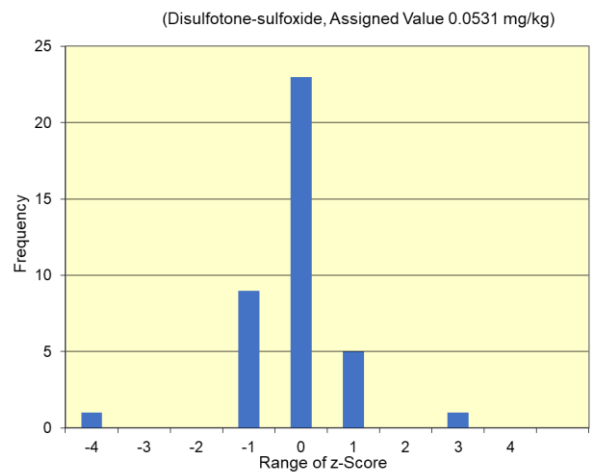
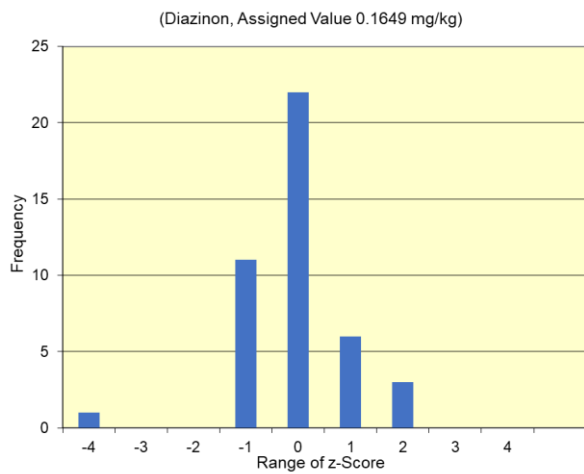
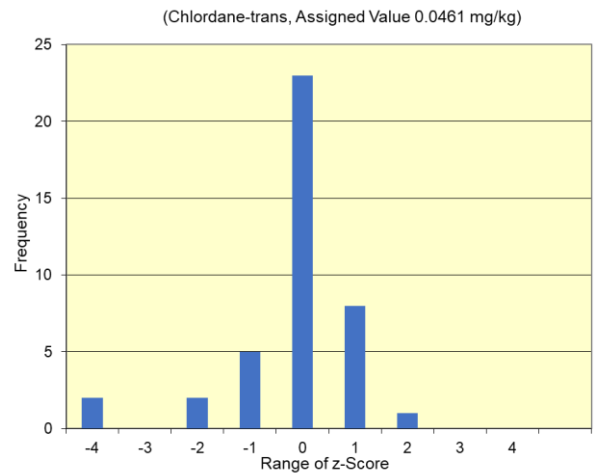
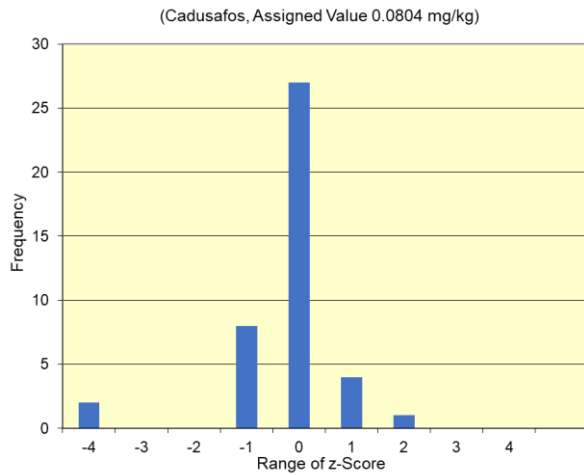


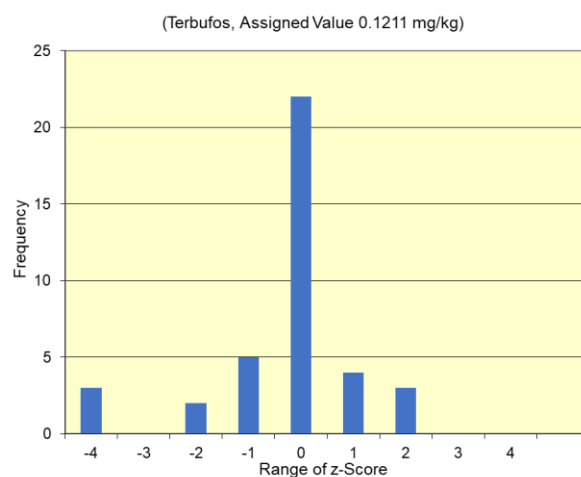
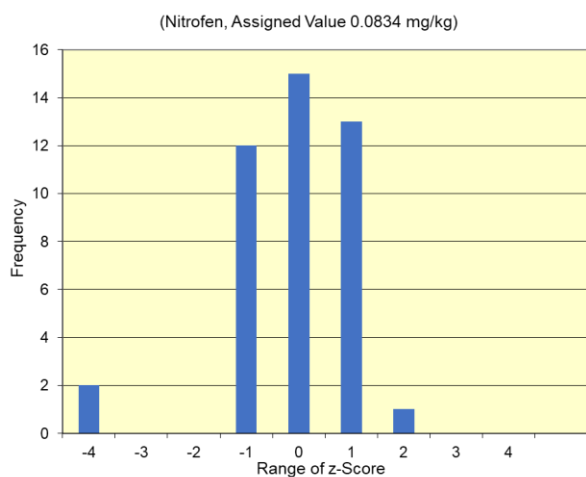
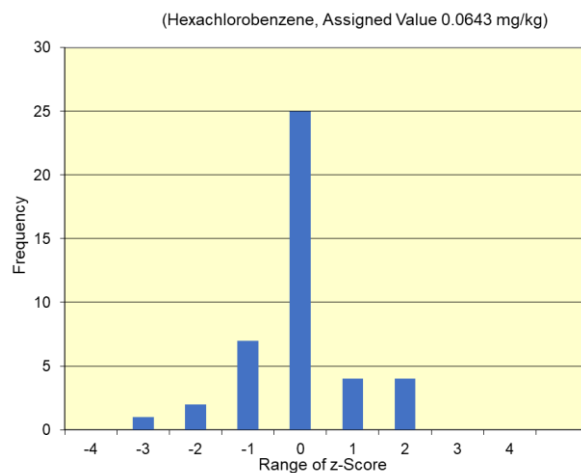
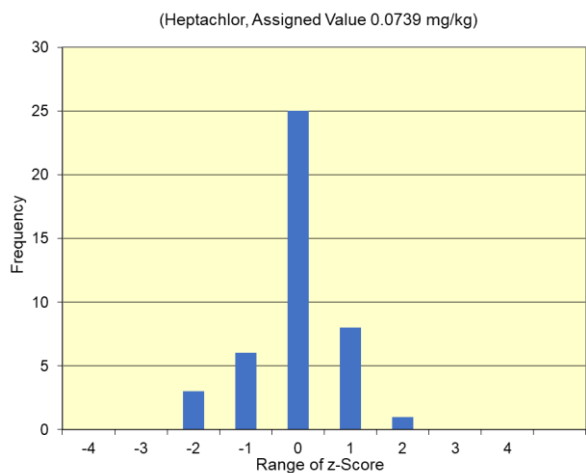
4.1.10 Terbufos



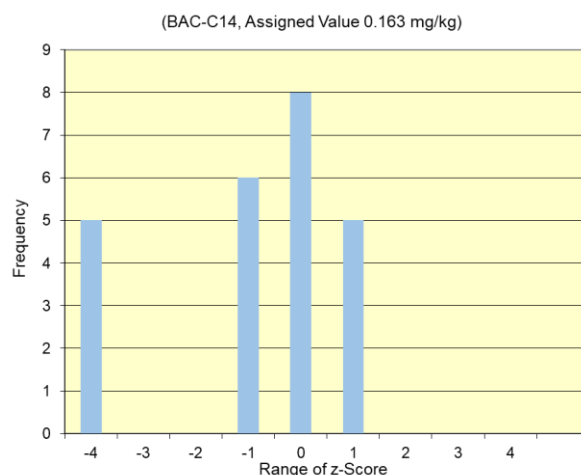
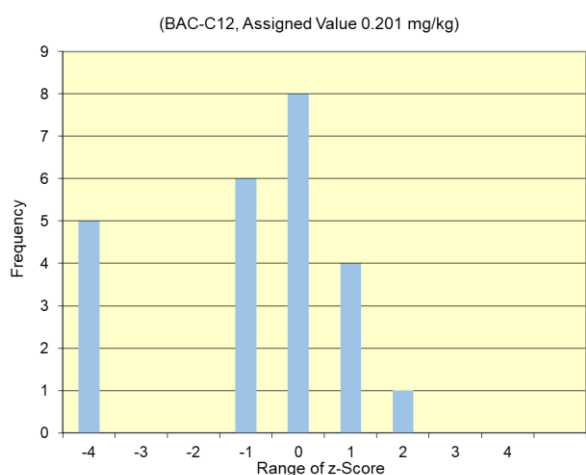
4.2 Histograms of z-scores (including false negative results)

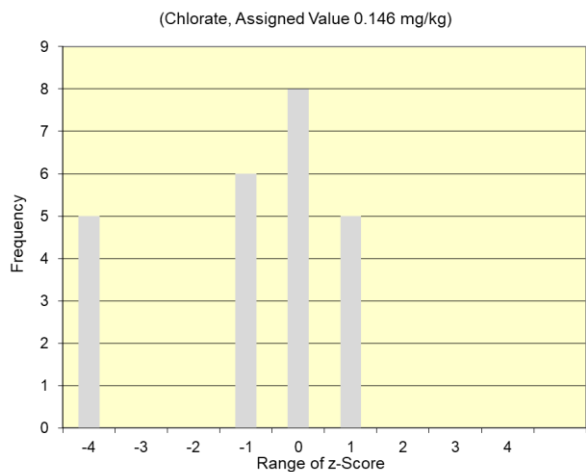
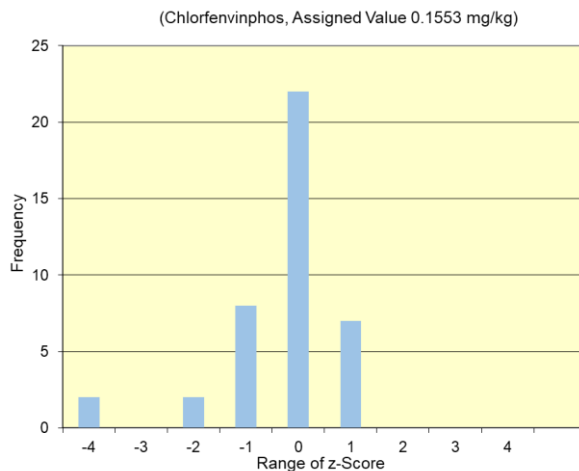
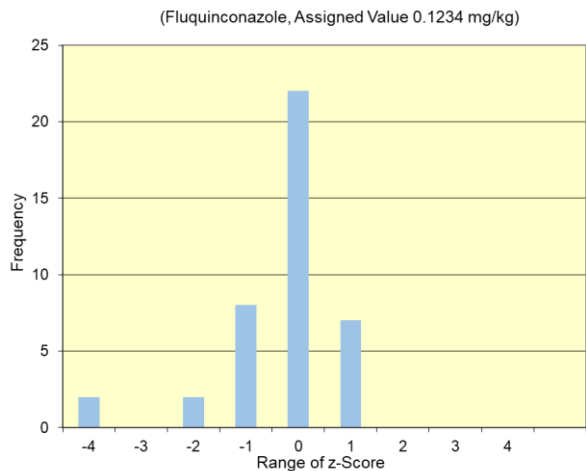
4.2.1 Mandatory pesticides





4.2.2 Voluntary pesticides





5 False positive results

Laboratories reported quantitative results for pesticides that were not present in the test item. Quantitative results for the compounds that exceeded the MRRL were considered as false positives. Results below the corresponding MRRL concentrations were ignored. In addition to this rule, the results of the blank material were checked. One laboratory reported in total one false positive result for cis-chlordane (**Table 13**). Terbufos-sulfoxide was not spiked in the test item. Anyways three laboratories reported values for this analyte near or above the MRRL value. The EURL AO checked the test items and the spike solution and detected terbufos-sulfoxide at 0.009 mg/kg in the test item and 75 ng/mL in the spike solution (which is 0.1% of spiked terbufos).

Table 13: Summary of false positive results.

Analyte detected	Reported concentrations above the MRRL [mg/kg] (lab code)
Chlordane- <i>cis</i>	0.034 (49)
Terbufos-sulfoxide ^(x)	0.003 (25); 0.0041 (39); 0.0029 (48)

^(x) Terbufos sulfoxide was found to be present in the spike solution. Therefore the results given by the three labs is not considered as false positive result as the result was above the RL (0.005 mg/kg) of the lab, but lower than the MRRL value (0.01 mg/kg) of the EUPT (according to chapter 1.3.7)

Laboratories reporting false positive results are kindly asked to provide feedback regarding any actions undertaken to find out the reasons for false positive results, if they have not done so already. In case of OfLs, please inform your corresponding NRL, too.

6 Information on investigative activities of participants

Laboratories are expected to undertake follow-up activities to trace back the sources of erroneous or strongly deviating results (typically those with $|z| > 2.0$) including all false positives. The following feedback on investigative activities for i.e. false positives, false negatives, for results with $|z| > 3.0$ or non submission of data were reported (**Table 14**).

Table 14: Comments reported as follow-up activities by laboratories with deviating results.

Lab code	Submitted comments
37	<p>The test results were sent by email on 26/09/2022, because the official page for sending the results was not functional and it was not possible to connect to it. When recalculating the measured values, we used a calculation factor of 8, as indicated in red in the target list. By comparing our results with the results in the Preliminary Report dated 25/11/2022, we found a match in the Z-score interval $<-0.8 \ 0.9>$.</p> <p>We do not agree with the disqualification of our analysis results in the ready-to-eat form, considering that the instruction stated in the Specific protocol says: All results <u>should be</u> reported on the product basis (provided powder). The expression „should be“ that was used indicates a preferred form, not a mandatory one. The ready-to-eat form is legally required according to regulation COMMISSION DELEGATED REGULATION (EU) 2016/127, Article 4, paragraph 5. This form of result expression is also required by EFSA, with which we communicate annually and send information about the measured parameters. We have provided the results in mg/kg and added a clear note: "ready to eat".</p> <p>In the target list, the formulation of the text gives the impression that the ready-to-eat results are desired.</p> <ol style="list-style-type: none"> The MRRL values in the table are in mg/kg ready-to-eat without any warning that the results must be reported on powder basis. The note below the table says: The calculation factor from powder to ready-to-eat product is 8 (MRRL ready to eat value x8), evoking the calculation direction of the results from powder to ready-to-eat. <p>The electronic system for entering results was out of order on the required date. In the event that any notification regarding the preferred results form had been posted on the Result Submission Website, we were unable to see it. It is still not possible to log into the system. We took part in the EUPT-AO-BF1 baby nutrition test for the first time, and since we deal with this issue as a priority, it is important for us that our submitted results are officially evaluated, possibly including a comment, whether to provide the results in powder, or ready-to-eat form.</p> <p>Producing the test results requires high material costs, special technical equipment, time and hard work, therefore, we ask for your understanding and the re-evaluation of our results.</p>
42	<p>Please note that there is an error in the preliminary report. Our lab (042) did not analyze BAC-C14. However, we have a result for that in the report with a quite nice z-score. Also the voluntary pesticides (BAC-C12 and chlorate) were not analyzed and were not detected for that reason.</p> <p>[EURL comment: The error was corrected and a new version 1.1 was published]</p>
48	<p>There is a mistake in the pdf-report by the Laboratory 48. BAC C 14 are reported but not mentioned in the pdf report. Please check this and correct this.</p> <p>[EURL comment: The error was corrected and a new version 1.1 was published]</p>
51	<p>We reported a value for BAC C14 which was not considered. We have now a false positive. Please check the preliminary report.</p> <p>[EURL comment: The error was corrected and a new version 1.1 was published]</p>

Lab code	Submitted comments
5	<p>For the BF1 PT we had to fill in our LOQs for all pesticides in our scope before we received the PT material.</p> <p>Since infant formula is not a common matrix in our lab, it was difficult to provide accurate LOQ values beforehand.</p> <p>For some of the pesticides, our reported LOQ was higher than the MRRL provided (e.g., endrin). However, after analyzing the product we were able to determine endrin at a lower concentration than our reported LOQ, but above the MRRL. In this situation, we did not know if we were supposed to report the result or not. We decided not to report it since it was lower than our reported LOQ.</p> <p>However, it would be good to know what to do in these situations for future PTs, and I hoped you would be able to enlighten me about what is preferred.</p> <p>(It should be noted that the LOQs reported was for “ready to eat” -product, while the results reported were for the powder. This was accepted by e-mail before the results were reported.)</p>
39	<p>We are one of the three labs that reported a result for terbufos-sulfoxide. This was evaluated as false positive. We have checked all performance criteria according to SANTE No 11312/2021 (e.g. ion ratio, retention time) and found that these are fulfilled. We therefore believe that terbufos-sulfoxide is present in the test item.</p> <p>In addition, it is confusing that the MRRLs are given on ready-to-eat in the target list and the results should be reported on powder. It would be nice to have consistency to avoid misunderstandings.</p> <p>[EURL comment: it can be confirmed that terbufos-sulfoxide was present in the test items. The result is not considered as false positives anymore]</p>

7 Test for homogeneity

11 bottles of treated test material were randomly chosen and analysed in duplicate. The identification and quantification of spiked analytes was performed by applying two methods as described briefly in chapters 7.1 (QuEChERS-AO method) and in chapter 7.2 (QuPPE method for chlorate).

7.1 QuEChERS-AO method for LC and GC-amenable pesticides

Preparation	Extraction procedure	Quantification
<ul style="list-style-type: none"> • transfer 2 g of the homogenous sample into a 50 ml centrifuge tube • add 13 mL water • add the ISTD-solution • shake for a few seconds and wait for at least 10 min 	<ul style="list-style-type: none"> • add 10 ml of acetonitrile, close the tube and shake for 10 min • shake for 10 min • add extraction salts (4 g magnesium sulfate, 1 g sodium chloride, 1 g trisodium citrate x 2 H₂O, 0.5 g disodium citrate x 6 H₂O) • shake for 10 min and centrifuge 5 min • Transfer 8 mL supernatant in a 15 mL tube and freeze out (-20°C) acetonitrile phase over night • Filtrate solution to separate frozen out fat • Transfer an aliquote of 6 mL of supernatant in a tube and add 150 mg PSA and 900 mg MgSO₄ • Shake for 10 min and centrifuge 5 min • Transfer an aliquot of the extract into a vial and analyse by LC or GC-MS/MS 	<ul style="list-style-type: none"> • LC-MS/MS system • GC-MS/MS system • procedural calibration with at least 3 calibration levels

7.2 QuPPE method for chlorate

Preparation	Extraction procedure	Quantification
<ul style="list-style-type: none"> • transfer 2 g of the homogenous sample into a 50 ml centrifuge tube • add 8 mL water • add the ISTD-solution • shake for a few seconds and wait for at least 10 min 	<ul style="list-style-type: none"> • add 10 mL acidified methanol • shake for 15 min and centrifuge for 5 min • Transfer an aliquote of 2 mL of supernatant in a 15 mL which already contains 2 mL acetonitrile and 100 mg C18 sorbent • shake vigorously for 1 min and centrifuge for 5 min • Transfer an aliquot of the extract into a vial and analyse by LC -MS/MS 	<ul style="list-style-type: none"> • LC-MS/MS System • procedural calibration with at least 3 calibration levels, internal standards

7.3 Statistical evaluation

The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC⁴. The individual data from the homogeneity test are given in the following tables. The acceptance criterion for the test material to be sufficiently homogenous for the proficiency test is that the following criterion is fulfilled:

$$\frac{\text{between-sample standard deviation (s}_s\text{)}/\text{standard deviation for proficiency assessment(St}_{\text{arget}})}{\text{assessment(St}_{\text{arget}})} \leq 0.3$$

7.3.1 Results from homogeneity analysis

Individual results of the analysis of 11 randomly chosen EUPT AO BF1 test items (including the first and the last test item) analysed in duplicate are listed in **Table 15**. Cochran's test

⁴ Thompson M., Ellison S. L. R. and Wood R., The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories. Pure & Appl Chem **78**, 145-196 (2006.)

showed a statistically significant difference between replicates in test item 60 for chlorate and test item 17 for BAC-C12 and -C14. As a consequence, the outlying group was removed from statistical evaluation (**Table 15**). Finally, all analytes passed the homogeneity test and the test item was considered to be sufficiently homogenous and suitable for EUPT BF1.

Table 15: Homogeneity results of the analysis of 11 EUPT AO BF1 test items in duplicate. Results are given in µg/kg. Values in red were excluded from statistical evaluation.

Sample	Replicate	Chlordane, gamma (trans)-	Diazinon	Endrin	Heptachlor	Hexachlorobenzene (HCB)	Nitrofen	Cadusafos	Disulfoton-sulfoxide	Fipronil-sulfone	Terbufos	BAC-C12	BAC-C14	Chlorate	Chlorfenvinphos	Fluquinconazole
1	1	54.2	196	15.6	89.2	80.1	104	86.4	53.1	125	139	154	130	143	166	115
	2	53.7	195	15.0	90.4	80.0	111	96.9	53.0	140	153	159	140	165	164	128
125	1	54.1	194	15.1	90.8	79.7	100	88.3	53.5	129	137	159	131	149	168	118
	2	54.4	195	14.9	90.5	80.0	111	96.9	53.0	141	136	153	129	146	166	126
47	1	54.2	194	15.0	90.4	80.9	102	88.8	55.6	139	144	208	174	151	176	123
	2	54.2	196	14.8	90.4	80.0	111	95.9	52.1	140	140	176	147	150	167	128
106	1	53.3	195	15.3	90.7	79.3	105	89.5	53.9	131	143	163	134	136	173	116
	2	53.8	194	15.0	90.0	80.0	110	94.7	51.7	137	134	157	131	141	163	130
68	1	54.0	196	15.2	89.2	80.6	100	89.3	53.9	131	145	176	145	156	170	116
	2	54.4	195	14.9	89.0	77.7	111	104	58.7	161	149	175	146	160	180	135
99	1	54.0	196	15.4	89.3	79.4	105	88.0	54.1	130	144	168	141	160	170	115
	2	54.6	195	15.1	89.5	81.4	110	98.2	53.0	140	136	218	179	141	168	129
114	1	54.3	194	15.3	90.4	78.6	106	88.2	53.9	135	147	160	130	142	172	124
	2	53.5	195	15.0	89.5	80.9	109	95.7	53.0	137	139	161	136	174	168	123
146	1	53.1	196	14.6	89.1	79.5	103	87.5	54.3	138	149	164	135	143	174	123
	2	54.3	194	14.9	89.5	80.3	111	97.6	53.8	144	145	184	150	174	168	127
60	1	54.3	196	15.4	89.0	80.6	103	88.2	54.2	133	142	167	137	1697	172	120
	2	54.1	194	14.9	89.6	80.2	109	97.6	52.0	140	146	171	143	145	178	126
17	1	66.3	238	19.6	115	88.0	128	104	72.2	170	187	505	409	214	221	162
	2	53.5	196	14.6	90.3	79.0	109	103	55.6	153	153	164	142	147	178	132
190	1	53.2	196	15.1	90.9	79.5	106	101	55.1	132	146	166	139	155	173	122
	2	54.0	195	14.7	89.6	80.4	110	101	55.4	148	152	159	134	155	175	130
General average		54.5	197	15.2	91.0	80.3	108	94.5	54.8	140	146	170	142	155	173	126
Standard deviation		2.66	9.21	1.01	5.42	1.91	5.79	5.91	4.17	10.6	10.9	16.8	13.5	17.5	11.7	10.0
Coefficient of variation (Cv)		4.9%	4.7%	6.6%	5.9%	2.4%	5.4%	6.3%	7.6%	7.6%	7.4%	9.9%	9.5%	11%	6.8%	7.9%

7.3.2 Between-test-portion ranges

Table 16: Difference between the results of 11 EUPT AO BF1 test items analysed in duplicate in µg/kg.

Sample	Chlordane, gamma (trans)-	Diazinon	Endrin	Heptachlor	Hexachlorobenzene (HCB)	Nitrofen	Cadusafos	Disulfoton-sulfoxide	Fipronil-sulfone	Terbufos	BAC-C12	BAC-C14	Chlorate	Chlorfenvinphos	Fluquinconazole
1	0.50	1.10	0.60	1.20	0.10	6.60	10.5	0.14	15.0	13.7	4.60	10.3	21.9	2.02	13.0
125	0.30	0.90	0.20	0.30	0.30	10.7	8.52	0.49	12.0	0.92	5.90	2.37	2.83	2.16	8.06
47	0.00	1.60	0.20	0.00	0.90	8.80	7.12	3.49	0.68	4.02	31.6	26.9	1.28	9.11	4.88
106	0.50	0.90	0.30	0.70	0.70	4.20	5.18	2.25	6.83	9.20	6.28	3.55	5.22	9.92	13.7
69	0.40	0.70	0.30	0.20	2.90	10.1	14.7	4.72	30.6	4.04	1.66	1.17	4.08	9.62	18.9
99	0.60	0.30	0.30	0.20	2.00	5.90	10.3	1.18	9.96	7.77	49.7	37.6	19.1	1.85	13.3
114	0.80	1.00	0.30	0.90	2.30	3.40	7.54	0.91	2.07	8.11	1.31	5.28	31.8	3.70	0.81
146	1.20	1.60	0.30	0.40	0.80	7.70	10.1	0.46	5.74	3.19	20.7	15.1	30.4	5.97	3.80
60	0.20	1.30	0.50	0.60	0.40	6.50	9.45	2.19	7.37	3.89	4.22	6.12	-	6.39	6.09
17	12.8	42.4	5.00	24.8	9.00	18.7	0.37	16.6	17.1	34.5	-	-	66.9	43.3	30.1
190	0.80	1.20	0.40	1.30	0.90	4.70	0.00	0.36	16.3	6.15	7.34	5.72	0.28	1.51	8.33
S _w	2.76	9.07	1.09	5.31	2.15	6.31	6.15	3.82	9.77	8.75	14.3	11.4	19.1	10.1	9.56
C _{v(w)}	5.1%	4.6%	7.2%	5.8%	2.7%	5.9%	6.5%	7.0%	7.0%	6.0%	100%	100%	101%	5.8%	7.6%

Variable	Description
C _{v(w)}	Coefficient of variation (between samples)
S _w	Within-sample standard deviation

7.3.3 Sample averages and results of the test for homogeneity

The homogeneity test showed standard deviations of the sample averages (s_x) in the range of 0.637 – 13.59 $\mu\text{g}/\text{kg}$ (3.0-8.0%) (Table 17). Homogeneity is given if the ratio of s_s/s_{target} is below or equal 0.3 for all spiked analytes.

Table 17: Mean of 11 EUPT AO BF1 test items analysed in duplicate in $\mu\text{g}/\text{kg}$.

Sample	Chlordane, gamma (trans)-	Diazinon	Endrin	Heptachlor	Hexachlorobenzene (HCB)	Nitrofen	Cadusafos	Disulfoton-sulfoxide	Fipronil-sulfone	Terbufos	BAC-C12	BAC-C14	Chlorate	Chlorfenvinphos	Fluquinconazole
1	54.0	195	15.3	89.8	80.1	107	91.7	53.1	133	146	157	135	154	165	121
125	54.3	194	15.0	90.7	79.9	105	92.6	53.3	135	136	156	130	147	167	122
47	54.2	195	14.9	90.4	80.5	106	92.4	53.9	140	142	192	160	150	172	126
106	53.6	195	15.2	90.4	79.7	108	92.1	52.8	134	138	160	133	139	168	123
69	54.2	195	15.1	89.1	79.2	105	96.6	56.3	146	147	176	146	158	175	125
99	54.3	196	15.3	89.4	80.4	107	93.1	53.6	135	140	193	160	150	169	122
114	53.9	195	15.2	90.0	79.8	107	92.0	53.5	136	143	161	133	158	170	123
146	53.7	195	14.8	89.3	79.9	107	92.5	54.0	141	147	174	142	159	171	125
60	54.2	195	15.2	89.3	80.4	106	92.9	53.1	137	144	169	140	-	175	123
17	59.9	217	17.1	103	83.5	118	103	63.9	161	170	-	-	181	200	147
190	53.6	196	14.9	90.3	80.0	108	101	55.2	140	149	163	137	155	174	126
Average	54.5	197	15.2	91.0	80.3	108	94.5	54.8	140	146	170	142	155	173	126
Standard deviation s_x	1.80	6.62	0.64	3.91	1.14	3.64	3.99	3.20	8.07	9.03	13.6	10.9	10.9	9.38	7.37
$C_v(x)$	3.3%	3.4%	4.2%	4.3%	1.4%	3.4%	4.2%	5.8%	5.8%	6.2%	8.0%	7.7%	7.0%	5.4%	5.9%
$C_v(\text{target})$	25%														
S_s	0	1.63	0	1.09	0	0	0	1.71	4.16	6.57	9.12	7.41	0	6.07	2.93
S_s/s_{target}	0.00	0.03	0.00	0.05	0.00	0.00	0.00	0.13	0.12	0.18	0.21	0.21	0.00	0.14	0.09
Homogeneity	o.k.	o.k.	o.k.	o.k.	o.k.	o.k.	o.k.	o.k.	o.k.	o.k.	o.k.	o.k.	o.k.	o.k.	o.k.

Variable	Description
s_x	Standard deviation of the sample averages
$C_v(x)$	Coefficient of variation of the sample averages
$C_v(\text{target})$	Fit-for-purpose coefficient of variation (25%, as used in all EUPTs)
S_s	Estimation of the between sample standard deviation
s_{target}	Estimated fit-for-purpose standard deviation (25% of the average)

8 Test for stability

The analytical methods outlined in chapter 7 were also used for stability testing. The stability test was performed according to ISO 13528, Annex B⁵. Tests were performed using three test items (chosen randomly). The samples were analysed before the start (stab test I, 18.08.2022) and after the deadline of the proficiency test (stab test II, 27.09.2022), respectively. Between the measurements the test material was stored at -18°C. According to ISO 13528 the analyte is considered to be adequately stable if $|\bar{y}_1 - \bar{y}_2| \leq 0.3\sigma$, with \bar{y}_1 being the average of the first stability test, \bar{y}_2 the average of the second stability test and σ standard deviation used for proficiency assessment (25% of the assigned value). As a consequence stability is given if the ratio of \bar{y}_2/\bar{y}_1 is in the range of 92.5 – 107.5%. The result of the stability test is given in **Table 18**. All pesticides passed the stability test.

Table 18: Statistical evaluation of the stability test.

		Stab test I	Stab test II	\bar{y}_2/\bar{y}_1	Stability
Analyte		Average (\bar{y}_1) ($\mu\text{g}/\text{kg}$)	Average (\bar{y}_2) ($\mu\text{g}/\text{kg}$)	(%)	
mandatory	Chlordane, gamma (trans)-	54.6	54.8	100	passed
	Diazinon	195	196	100	passed
	Endrin	15.0	14.7	98	passed
	Heptachlor	90.0	90.7	101	passed
	Hexachlorobenzene (HCB)	80.0	80.1	100	passed
	Nitrofen	109	110	100	passed
	Cadusafos	80.7	78.7	97.5	passed
	Disulfoton-sulfoxide	52.6	50.2	95.5	passed
	Fipronil-sulfone	136	131	96.6	passed
	Terbufos	128	123	95.9	passed
voluntary	BAC-C12	160	159	99.7	passed
	BAC-C14	134	134	99.9	passed
	Chlorate	173	162	93.6	passed
	Chlorfenvinphos	166	159	95.7	passed
	Fluquinconazole	122	119	97.4	passed

⁵ ISO 13528:2015 – Statistical methods for use in proficiency testing by interlaboratory comparison

9 List of participating laboratories

Lab Name	City	Country	NRL
AGES - Innsbruck	Innsbruck	Austria	NRL
Croatian Veterinary Institute - Zagreb	Zagreb	Croatia	NRL
SGL - Pesticide Lab (Nicosia)	Nicosia	Cyprus	NRL
CAFIA - Pesticide Lab (Praha)	Praha	Czech Republic	
SVI Prague - Pesticide Lab	Praha 6	Czech Republic	NRL
VSCHT / UCT Prague - Food Analysis (323)	Praha	Czech Republic	
Laboratoriet Ringsted - Pesticide Lab	Ringsted	Denmark	
DTU, National Food Institute	Lyngby	Denmark	NRL
Health and Safety Laboratory	Tallinn	Estonia	
Finnish Customs Laboratory	Espoo	Finland	
Finnish Food Authority	Helsinki	Finland	NRL
ANSES - LSAI (Unité PBM)	Maisons-Alfort Cedex	France	NRL
LUA Sachsen - Pesticide Lab, Dresden	Dresden	Germany	
Landeslabor Schleswig-Holstein, Neumünster	Neumünster	Germany	
CVUA RRW - Pesticide Lab (Krefeld)	Krefeld	Germany	
CVUA-MEL - Pesticide Lab (Münster)	Münster	Germany	
Eurofins - Germany, Hamburg	Hamburg	Germany	
BVL Unit 504 NRL for Pesticide Residues	Berlin	Germany	NRL
LGL Erlangen - Pesticide Lab	Erlangen	Germany	
LUA Rheinland-Pfalz, Institut für LM-Chemie Speyer	Speyer	Germany	
LHL - Pesticide Lab (Wiesbaden)	Wiesbaden	Germany	
LALLF - Pesticide Lab (Rostock)	Rostock	Germany	
LAVES - Pesticide Lab (Oldenburg)	Oldenburg	Germany	
Landeslabor Berlin-Brandenburg, Berlin	Berlin (Mitte)	Germany	
GCSL - Pesticide Lab (Athens)	Athens	Greece	NRL
Benaki Phytopathological Institute, Kifissia	Kifissia	Greece	
NFCSSO - Pesticide Lab (Velence)	Velence	Hungary	NRL
FCSCN Ltd Pesticide Res. Anal. Lab. Miskolc	Miskolc	Hungary	
The Food Chemistry Laboratories - DAFM	Co. Kildare	Ireland	NRL
IZSLER - Pesticide Lab	Brescia	Italy	
IZSUM - Italy, Perugia	Perugia	Italy	
BIOR (Riga) - Pesticide Lab	Riga	Latvia	NRL
NIBIO - Department of Pesticide Chemistry	ÅS	Norway*	NRL
Nat. Vet. Research Institute - Poland, Puławy	Puławy	Poland	NRL

Lab Name	City	Country	NRL
Pesticide Lab (Funchal - Madeira Island)	Funchal - Madeira Island	Portugal	
State Veterinary and Food Institute (Bratislava)	Bratislava	Slovakia	NRL
Pesticide Lab of PHA SR - Bratislava	Bratislava	Slovakia	
Pesticide Lab - Maribor	Maribor	Slovenia	NRL
Laboratorio Agroambiental de Zaragoza	Zaragoza	Spain	
Laboratori Agència Salut Pública Barcelona	Barcelona	Spain	
Analytica Alimentaria GmbH - Almeria, Spain	Almeria	Spain	
Laboratorio Regional de la CCAA de La Rioja	Logroño	Spain	
Laboratorio Químico Microbiológico (San Gines)	San Ginés (Murcia)	Spain	
Eurofins Food & Feed - Pesticide Lab (Lidköping)	Lidköping	Sweden	
Groen Agro Control - Netherlands	Delfgauw	The Netherlands	
Wageningen Food Safety Research (WFSR)	Wageningen	The Netherlands	NRL

* EFTA

10 Methods used to determine the individual pesticides of EUPT AO BF1

Due to the huge amount of data, methods used by participating labs are summarised in a separate excel-file and can be downloaded from CIRCABC:

[EUPT BF1_Information on methods.xlsx](#)

11 Summary of the proficiency test

52 laboratories from 24 different countries registered for participation in EUPT AO BF1. 46 laboratories reported results, from which 43 were taken into account.

The matrix of the test items was infant formula with incurred and spiked pesticides. The distribution of the test items to the participants was randomly. The test items contained 10 pesticides from the list of mandatory analytes and 5 voluntary pesticide.

For each pesticide and the relevant compounds included in the residue definitions, a MRRL was set that the laboratories were expected to achieve. The MRRL were established by the organiser and confirmed by the EURL Scientific Committee. The MRRL value was set on ready to eat basis. The laboratories had to use a conversion factor 8 to calculate on the powder provided.

In total, 419 (97.4%) out of 430 quantitative results for 10 mandatory pesticides were reported. 395 (94.3%) results gave z-scores within the range of ± 2 and 9 (2.1%) results were in the range between z-scores $|2|$ and $|3|$. In total 15 (3.6%) results were outside the range of ± 3 whereas 15 (100.0%) of these results were false negatives. The percentage of analysed mandatory pesticides was higher than in other EUPT AO at a high level of 97%. Usually it is ~87%. The rate of false negative results for the mandatory pesticides is at 3.6% in EUPT AO BF1.

126 (73.3%) out of 172 quantitative results for 4 voluntary pesticides were reported. 111 results (88.1%) gave z-scores that were within the range of $|2|$ and one result (0.8%) was in the range between $|2|$ to $|3|$. In total 14 (11.1%) of the results were outside the range of $|3|$, and all of these results were false negatives.

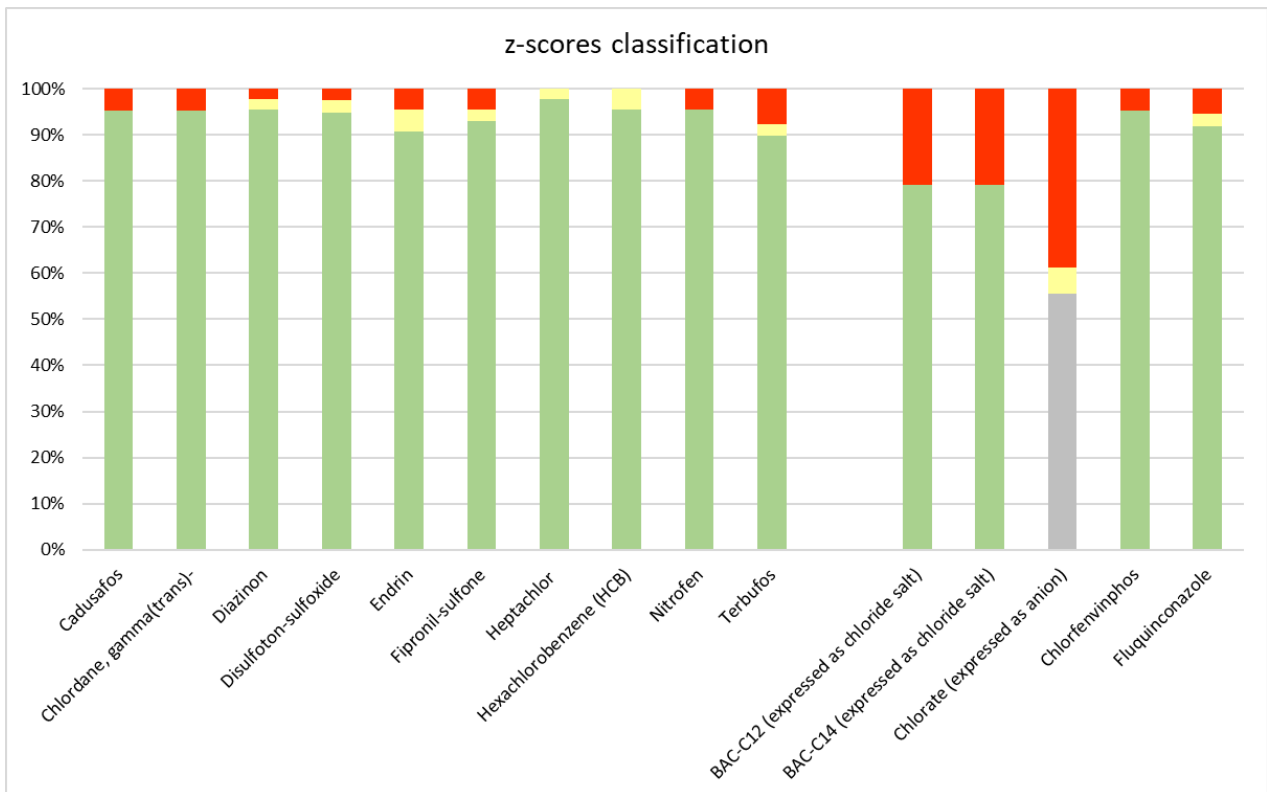


Figure 2: Percentage of labs with z-scores classified as acceptable (green), questionable (yellow) and unacceptable (red) for all spiked analytes. **Chlorate is evaluated for information only!**

Only one laboratory reported one false positive result for one analyte. Three laboratories reported results for terbufos-sulfoxide that was expected not to be present in the test item. The EURL AO has checked the presence of terbufos-sulfoxide in the test items as well as in the spike solution. As traces of terbufos-sulfoxide were found, the results of the three labs were not considered as false positives.

Since 2010 the criterion “Average Squared z-Scores” is used to evaluate the overall performance of the laboratories. In 2016 the precondition “scope” was introduced as an additional criterion: laboratories had to analyse at least 90% of the pesticides from the mandatory target list. 33 of 43 laboratories (77%) reported quantitative results for 9 or 10 pesticides (at least 90% of the pesticides present in the sample) and analysed 39 to 44 pesticides (at least 90% of the mandatory pesticides), with no false positive results. Therefore these laboratories were considered to have sufficient scope to be classified in Category A. 1 of 10 laboratories belongs to Category B caused by reporting of a false positive result. 2 laboratories were categorized in Category B just because of reporting false negative results.

Laboratories in Category A were sub-classified into ‘good’, ‘satisfactory’ and ‘unsatisfactory’. An overview of the results of the calculation of the average of squared z-scores is given in **Figure 3**.

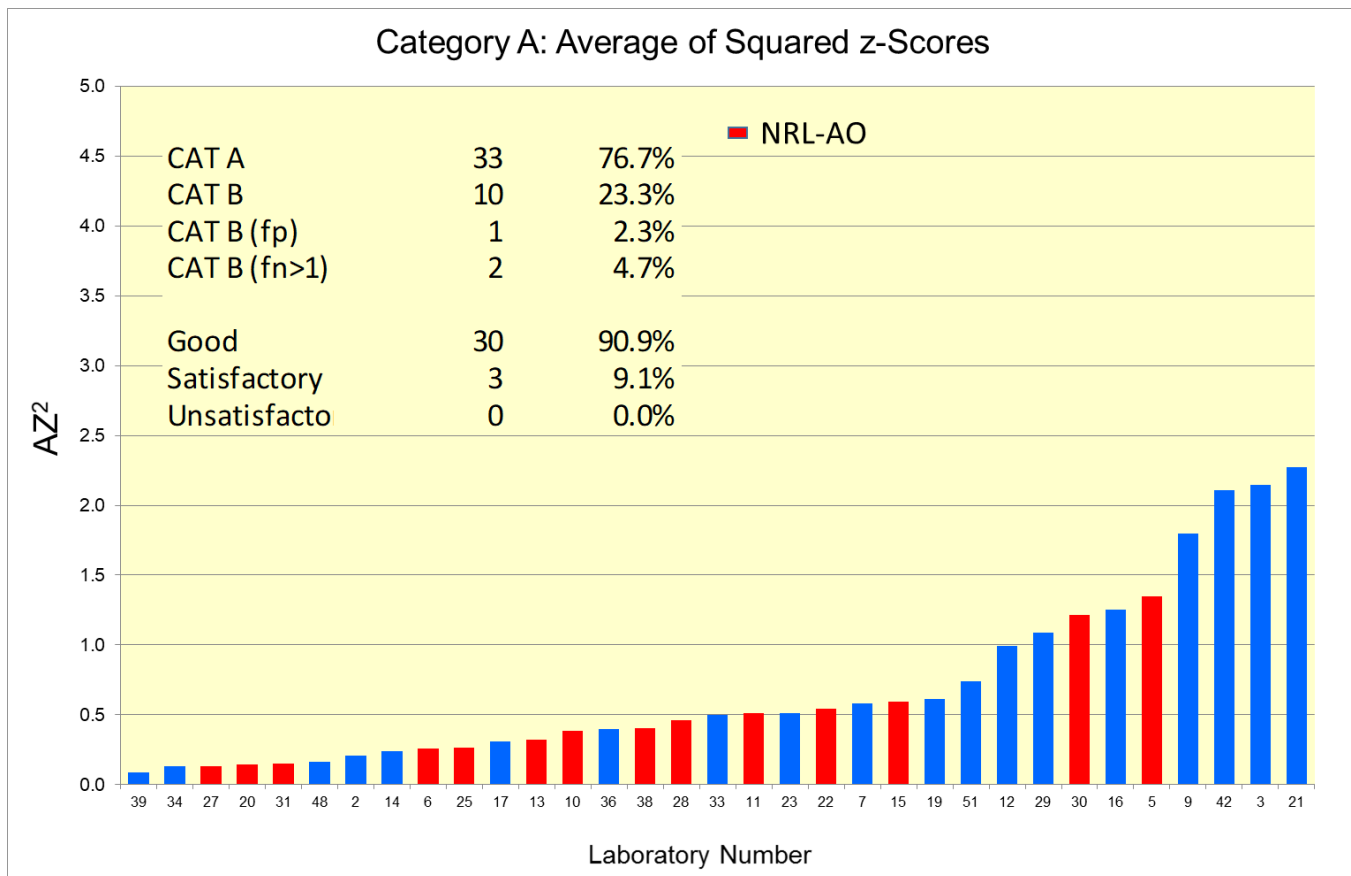


Figure 3: Results of the calculation of the average of squared z-scores.

12 Evaluation of the results of the questionnaire about EUPT AO BF1

The evaluation was opened from 16 November to 16 December 2022. Participants were asked to answer a few questions about their satisfaction on EUPT AO BF1 and give comments for improvement in the future. The following questions had to be answered:

- 1) How satisfied are you with EUPT AO BF1 in general (information, shipment, contact, reporting)?
- 2) How satisfied were you with the selected matrix?
- 3) How satisfied were you with the EUPT result submission webpage (availability, information, handling, etc.)?
- 4) How satisfied were you with the levels of pesticide concentrations?
- 5) How satisfied were you with the evaluation of results and information given in the preliminary report?

In addition, the participants had the possibility to comment on special wishes, matrices and improvements for future EUPTs.

The participants had to select between 1 point and 10 points whereas:

- 10 points means very satisfied
- 1 point means not satisfied
- If selecting 1 – 4 points the laboratories should give a reason for their selection.

16 laboratories (among them 7 NRLs and 9 OFLs) answered the survey. Overall, the feedback as regards organisation of the PT, matrix and level of pesticides, information on EUPT webpage and evaluation of results was moderate (**Figure 4-8**). For some laboratories the reporting form of the results was unclear (ready to eat vs. powder). However, it was stated in the specific protocol that “All results should be reported on the **product basis (provided powder)**”.

How satisfied are you with EUPT-AO BF 1 in general (information, shipment, contact, reporting)? 10 = very satisfied; 1 = not satisfied

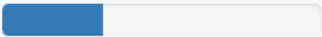
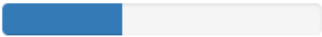
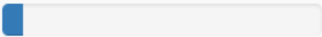
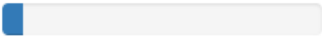
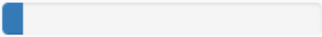
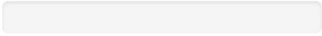
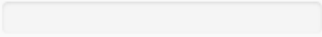
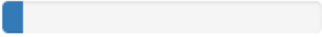
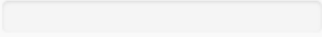
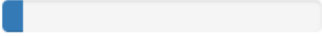
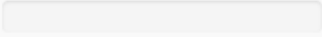
		Answers	Ratio
10		5	31.25 %
9		6	37.50 %
8		1	6.25 %
7		1	6.25 %
6		1	6.25 %
5		0	0.00 %
4		0	0.00 %
3		1	6.25 %
2		0	0.00 %
1		1	6.25 %
No Answer		0	0.00 %

Figure 4: Outcome of feedback from participants as regards satisfaction with EUPT AO BF1.

Comments by participants:

- There was no information on the form of the results in the Target list
- Support documents weren't clear and objective

How satisfied were you with the selected matrix? 10 = very satisfied; 1 = not satisfied


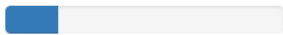
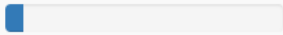
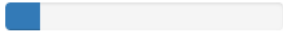
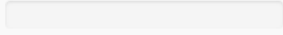
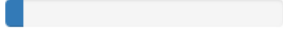
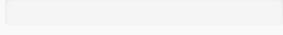
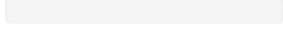
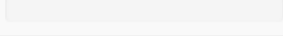
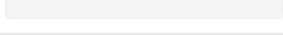
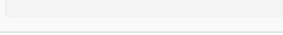
		Answers	Ratio
10		9	56.25 %
9		3	18.75 %
8		1	6.25 %
7		2	12.50 %
6		0	0.00 %
5		1	6.25 %
4		0	0.00 %
3		0	0.00 %
2		0	0.00 %
1		0	0.00 %
No Answer		0	0.00 %

Figure 5: Outcome of feedback from participants as regards satisfaction with selected matrix.

How satisfied were you with the levels of pesticide concentrations? 10 = very satisfied; 1 = not satisfied

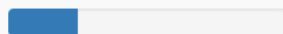

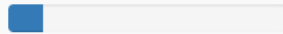
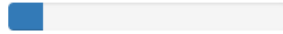
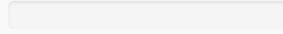
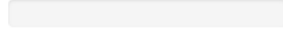
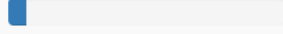
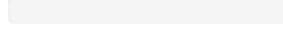
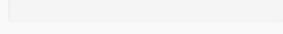
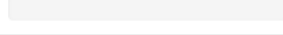
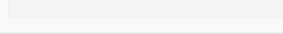
		Answers	Ratio
10		4	25.00 %
9		7	43.75 %
8		2	12.50 %
7		2	12.50 %
6		0	0.00 %
5		0	0.00 %
4		1	6.25 %
3		0	0.00 %
2		0	0.00 %
1		0	0.00 %
No Answer		0	0.00 %

Figure 6: Outcome of feedback from participants as regards satisfaction with levels of pesticides.

Comments by participants:

- Too high concentrations. Not consistent with real samples.

How satisfied were you with the EUPT result submission webpage (availability, information, handling...)? 10 = very satisfied; 1 = not satisfied

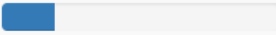
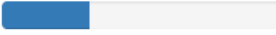
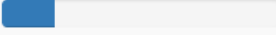
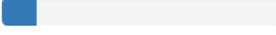
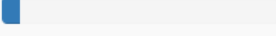
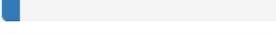
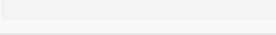
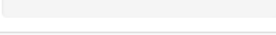
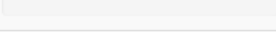
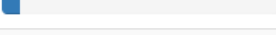
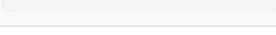
		Answers	Ratio
10		3	18.75 %
9		5	31.25 %
8		3	18.75 %
7		2	12.50 %
6		1	6.25 %
5		1	6.25 %
4		0	0.00 %
3		0	0.00 %
2		0	0.00 %
1		1	6.25 %
No Answer		0	0.00 %

Figure 7: Outcome of feedback from participants as regards satisfaction with results submission webpage.

Comments by participants:

- The electronic system for entering results was out of order on the required date.

How satisfied are you with the evaluation of results and information given in the preliminary report?
10 = very satisfied; 1 = not satisfied

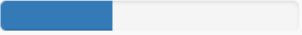
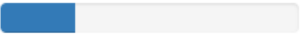
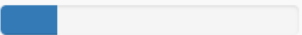
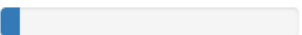
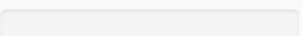
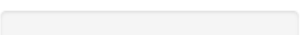
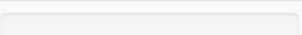
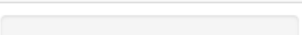
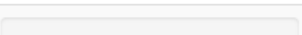
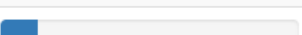
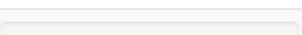
		Answers	Ratio
10		6	37.50 %
9		4	25.00 %
8		3	18.75 %
7		1	6.25 %
6		0	0.00 %
5		0	0.00 %
4		0	0.00 %
3		0	0.00 %
2		0	0.00 %
1		2	12.50 %
No Answer		0	0.00 %

Figure 8: Outcome of feedback from participants as regards satisfaction with preliminary report.

Comments by participants:

- Results expressed in ready to eat form should be evaluated too.
- Support documents weren't clear and objective

Additional comments are listed below:

- more sample material would be desirable
- Unclear information (to begin with) about the MRRL and whether it was for ready to eat product or the powder.
- 1. According to law, results for baby food are reported on ready to eat basis and, in this situation, labs need to use the conversion factor
2. Being the results of EUPT AO-BF1 expressed on the "powder" there's no need any information about conversion factor's.
3. MRRL presented on Specific Protocol are wrong. The real MRRL, for the powder, should is 8 times higher.
4. Due to the fact that documentation was confusing, we think EURL should accept results provided by the labs that didn't multiply their results by 8.
- In the specific protocol it was a little bit unclear whether the results should be reported on the formula basis or the final product basis.

Annex A: Robust data analysis using algorithm A

Algorithm A yields robust values of the average and standard deviation of the data to which it is applied. Algorithm A is reproduced from ISO 5725-5. Robustness is a property of the estimation algorithm, not of the estimates it produces, so it is not strictly correct to call the averages and standard deviations calculated by such an algorithm as robust. However, to avoid the use of excessively cumbersome terminology, the terms “robust average” and “robust standard deviation” should be understood in this protocol to mean estimates of the population mean or of the population standard deviation calculated using a robust algorithm.

- Denote the n items of data, sorted into increasing order, by: $x_1, x_2, \dots, x_i, \dots, x_n$.
- Denote the robust average and robust standard deviation of these data by x^* and s^* .

- Calculate initial values for x^* and s^* as:

$$x^* = \text{median of } x_i \quad (i = 1, 2, \dots, n)$$

$$s^* = 1.483 \text{ median of } |x_i - x^*| \quad (i = 1, 2, \dots, n)$$

- Update the values of x^* and s^* as follows. Calculate:

$$\delta = 1.5 * s^*$$

For each x_i ($i=1, 2, \dots, n$), calculate:

$$x_i^* = x^* - \delta \quad \text{if } x_i < x^* - \delta$$

$$x_i^* = x^* + \delta \quad \text{if } x_i > x^* + \delta$$

$$x_i^* = x^* \quad \text{otherwise}$$

- Calculate the new values of x^* and s^* from:

$$x^* = \sum x_i^* / n$$

$$s^* = 1.134 * (\sum (x_i^* - x^*)^2 / (n - 1))^{1/2}$$

where the summation is over i .

The robust estimates x^* and s^* are derived using an iterative calculation by updating the values of x^* and s^* several times using the modified data, until the process converges. Convergence is assumed when there is no change from one iteration to

the next in the third significant figure of the robust standard deviation and of the equivalent figure in the robust average.