EUPT AO 18 (HONEY)

EURL/NRL Joint Workshop
18-20 October 2023, Stuttgart (Germany)

European Commission

EURL-AO

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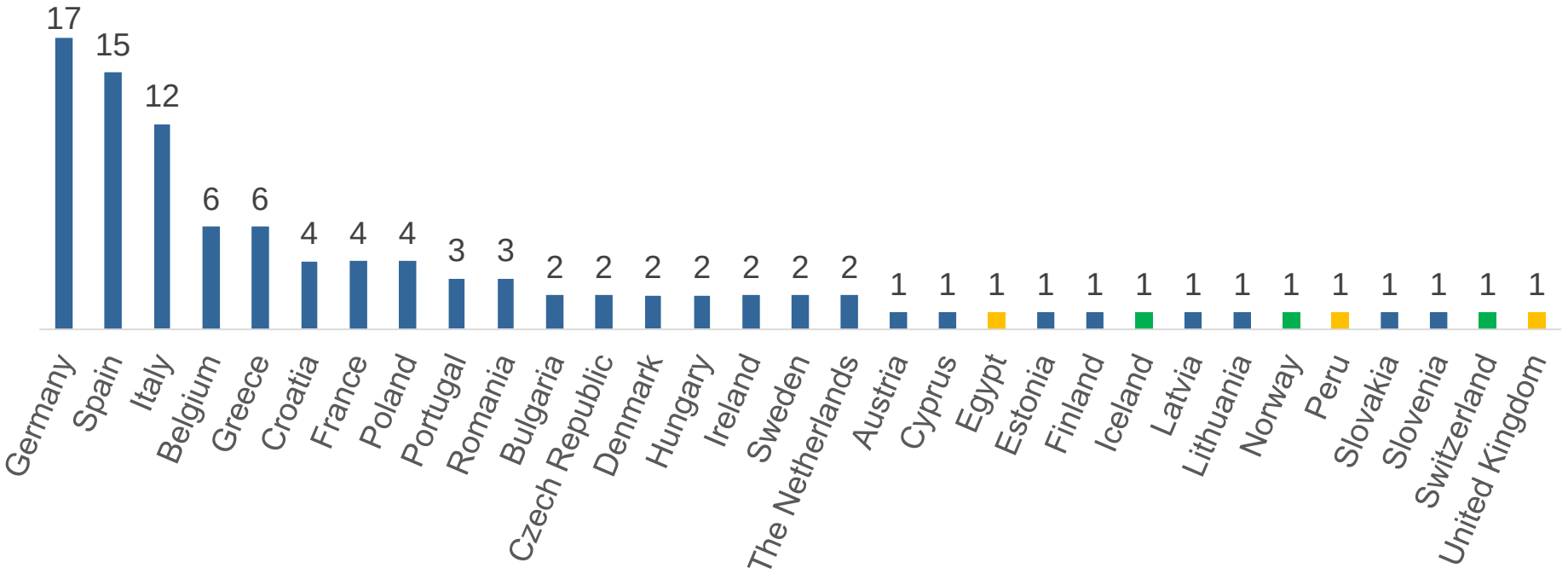
General information

Time schedule of EUPT AO 18

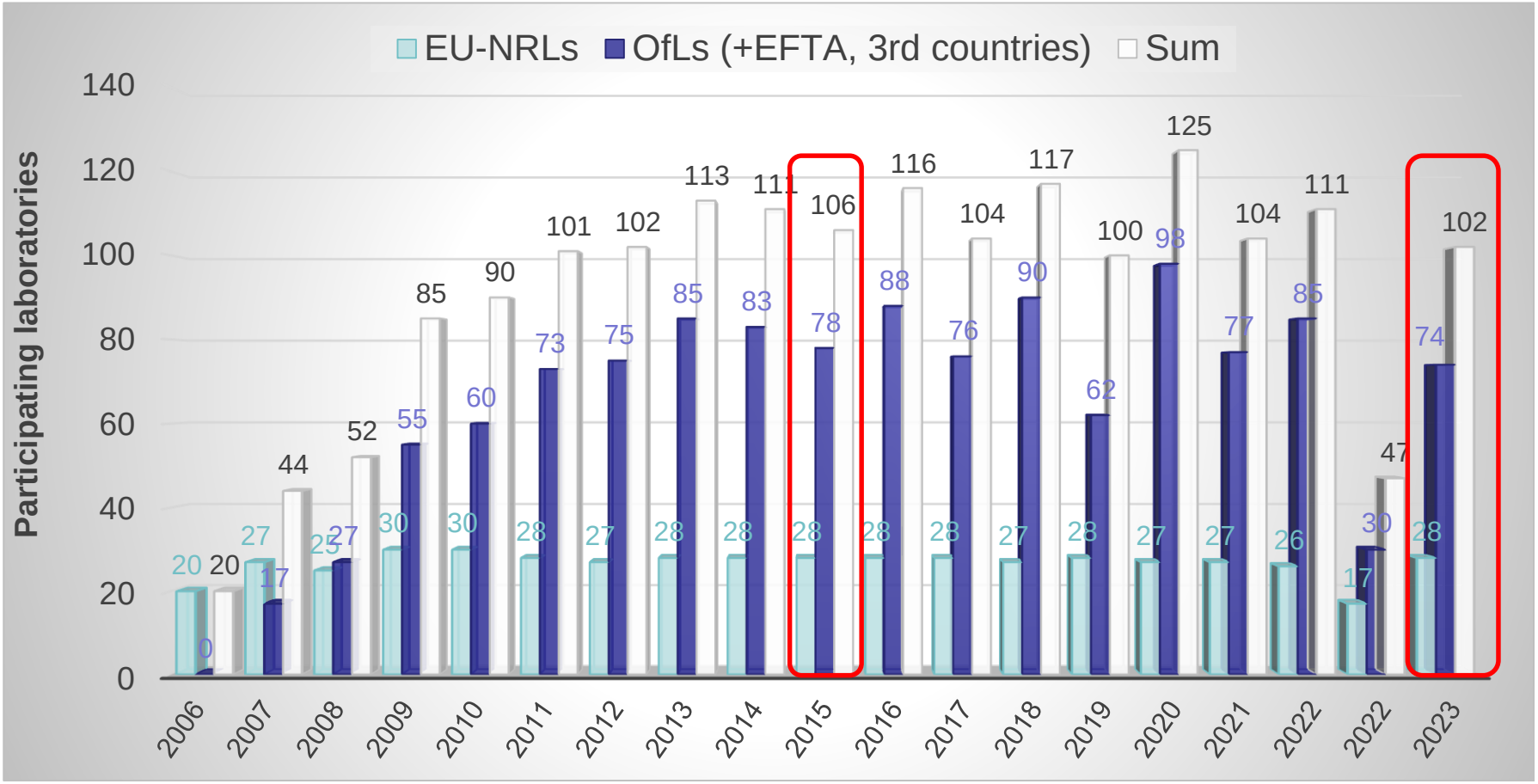
Actor	Activity	Date
EURL	Preliminary announcement matrix honey at EURL-NRL workshop	27 October 2022
EURL	First information supplied to laboratories and call for participation	Mid of January 2023
Participant	**Registration** via EUPT website	**23 January – 10 March 2023**
Participant	**Scope selection** via EUPT webtool	**27 March – 14 April 2023**
Participant	Proof of shipment address in EURL-Datapool	Ending 06 April 2023
EURL	**Dispatch of test material**	**17 April 2023**
Participant	Confirmation of test material receipt	18 – 24 April 2023
Participant	**Deadline for reporting of test results**	**22 May 2023**
Participant	Deadline for reporting of additional method information (no changes of reported results possible)	31 May 2023
EURL	Deadline for preliminary report	22 July 2023
EURL	Dispatch of the final report as pdf-file	Approx. end of 2023

Participants

- 102 labs registered
  - 31 different countries
  - 99 EU/EFTA labs
  - 3 EFTA and 3 third countries
  - all labs have submitted results for at least one compound



Participants (2006 – 2023)



matrix	vegetable oil	vegetable oil	liquid whole egg	butter fat	swine meat	poultry meat	liquid cream	raw poultry meat	liquid whole egg	honey	pork fat	liquid whole egg	milk powder	bovine liver	rape seed oil	liquid whole egg	native rape seed oil	infant formula	**honey**
EUPT AO	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	BF1	**18**

Target list of pesticides

- 115 **mandatory** analytes
  - 17 were spiked into the test item
- 14 voluntary analytes
  - 2 were spiked into the test item
- analytes were chosen based on:
  - multiannual control programme (MACP)
  - working document SANCO/12745/2013
  - cover full residue definition
  - suggestions/voting by EUPT committee

Target list of mandatory pesticides

Analyte	MRRL (mg/kg)	Analyte	MRRL (mg/kg)
2,4-Dimethylphenylformamid (amitraz metabolite)	0.010	Hexachlorcylcohexane (HCH), alpha-isomer	0.010
4,4´-Methoxychlor	0.010	Hexachlorcylcohexane (HCH), beta-isomer	0.010
Acetamiprid	0.010	Hexachlorcylcohexane (HCH), gamma-isomer (Lindane)	0.010
Aldrin	0.010	Hexachlorobenzene (HCB)	0.010
Azinphos-methyl	0.010	Hexythiazox	0.010
Azoxystrobin	0.010	Imazalil	0.010
Bifenthrin	0.010	Imidacloprid	0.010
Bixafen (parent only)	0.010	Indoxacarb (sum of isomers)	0.010
Boscalid (parent only)	0.010	Iprodione	0.010
Bromopropylate	0.010	Malaoxon	0.010
BTS 44595 (M201-04) (prochloraz metabolite)	0.010	Malathion	0.010
BTS 44596 (M201-03) (prochloraz metabolite)	0.010	Metaflumizone (sum of isomers)	0.010
Buprofezin	0.010	Methidathion	0.010
Carbendazim (carbendazim only)	0.010	Methiocarb	0.010
Chlordane, Cis-	0.010	Methiocarb-sulfone	0.010
Chlordane, Trans-	0.010	Methiocarb-sulfoxide	0.010
Chlorfenvinphos	0.010	Myclobutanil (parent only)	0.010
Chlorobenzilate	0.010	N-2,4-Dimethylphenyl-N-methylformamidin (amitraz metabolite)	0.010
Chlorpropham (parent only)	0.010	o,p´-DDT	0.010
Chlorpyrifos	0.010	Oxychlordane	0.010
Chlorpyrifos-methyl	0.010	p,p´-DDD	0.010
Clothianidin	0.010	p,p´-DDE	0.010
Coumaphos	0.010	p,p´-DDT	0.010
Cyfluthrin (sum of isomers)	0.010	Paraoxon-methyl	0.010
Cyhalothrin, Lambda- (sum of isomers)	0.010	Parathion	0.010
Cypermethrin (sum of isomers)	0.010	Parathion-methyl (parent only)	0.010
Cyproconazole	0.010	Pendimethalin	0.010
Cyprodinil	0.010	Permethrin (sum of isomers)	0.010
Deltamethrin	0.010	Phosalone	0.010

Analyte	MRRL (mg/kg)	Analyte	MRRL (mg/kg)
Diazinon	0.010	Pirimicarb	0.010
Dieldrin	0.010	Pirimicarb, Desmethyl-	0.010
Diethyl-m-toluamid, N,N-, (DEET)	0.010	Pirimiphos-ethyl	0.010
Difenoconazole	0.010	Pirimiphos-methyl	0.010
Dimethoate	0.010	Prochloraz	0.010
Dimethomorph	0.010	Profenofos	0.010
Dimoxystrobin	0.010	Propargite	0.010
Endosulfan, Alpha-	0.010	Prothioconazole-desthio (sum of isomers)	0.010
Endosulfan, Beta-	0.010	Pyraclostrobin	0.010
Endosulfan-sulfate	0.010	Pyrazophos	0.010
Endrin	0.010	Pyrimethanil	0.010
Epoxiconazole	0.010	Resmethrin (sum of isomers)	0.010
Ethoprophos	0.010	Spinosad[1]	0.010
Etofenprox	0.010	Spinosyn A[2]	0.010
Famoxadone	0.010	Spinosyn D[2]	0.010
Fenhexamid	0.010	Spiroxamine	0.010
Fenitrothion	0.010	tau-Fluvalinate	0.010
Fenpropidin (parent only)	0.010	Tebuconazole	0.010
Fenpropimorph (sum of isomers) (parent only)	0.010	Tebufenozide	0.010
Fenvalerate/Esfenvalerate (sum of RR, SS, RS and SR isomers)	0.010	Terbuthylazine	0.010
Fipronil	0.005	Tetraconazole	0.010
Fipronil sulfone (MB46136)	0.005	Tetramethrin	0.010
Fluquinconazole	0.010	Thiacloprid	0.010
Flusilazole (parent only)	0.010	Thiamethoxam	0.010
Flutriafol	0.010	Thiophanate-methyl	0.010
Heptachlor	0.010	Triazophos	0.010
Heptachlorepoxid, Cis-	0.010	Trichlorfon	0.010
Heptachlorepoxid, Trans-	0.010	Trifloxystrobin (parent only)	0.010
Heptenophos	0.010	Vinclozolin	0.010

[1] The residue definition for amitraz is: amitraz including the metabolites containing the 2,4-dimethylaniline moiety expressed as amitraz. If an amitraz metabolite was detected, please report additionally the value for amitraz according to the residue definition and the equation used for calculation of amitraz in the comments field.

[2] Results for Spinosad should be reported either if individual standards for Spinosyn A and D or a mixture of Spinosyn A and D are used for quantification.

[3] Results for Spinosyn A or D should be reported, if the individual standards were used for quantification.

Target list of voluntary pesticides

Analyte	MRRL (mg/kg)
Ametoctradin (parent only)	0.01
Benzovindiflupyr	0.01
Fenpyrazamine (parent only)	0.01
Fenthion	0.01
Fenthionoxon	0.01
Fenthionoxonsulfone	0.01
Fenthionoxonsulfoxide	0.01
Fenthionsulfone	0.01
Fenthionsulfoxide	0.01
Flonicamid (parent only)	0.01
Orthophenylphenol (2-phenylphenol)	0.01
Picoxystrobin	0.01
Spirotetramat	0.01
Spirotetramat-enol	0.01

Pesticides spiked

Mandatory analytes
2,4-Dimethylphenylformamid (amitraz metabolite)
Acetamiprid
Azoxystrobin
Boscalid
Chlorpyrifos
Deltamethrin
Diazinon
Etofenprox
Famoxadone
Hexachlorocylcohexane (HCH), gamma-isomer (Lindane)

Mandatory analytes
Imidacloprid
N-2,4-Dimethylphenyl-N-methylformamidin (amitraz metabolite)
Parathion-methyl
Permethrin (sum of isomers)
Phosalone
Pyraclostrobin
Thiacloprid
Voluntary analytes
Fenpyrazamine
Orthophenylphenol (2-phenylphenol)

Preparation of EUPT AO 18

Preparation

15 kg honey were obtained from a commercial trader (FoodQS GmbH, Germany)

1 certified spike solution

- custom made by HPC
- solvent: acetone
- checked against EURL solutions

Preparation

spiking
15 kg honey was spiked with 30 mL of certified spike solution

homogenisation
homogeni-sation by stirring for 30 min

bottling
minimum of 50 g for each test item (no blank material provided)

storage conditions
test items were stored at 4°C until shipment

shipment
test items were shipped uncooled

Homogeneity and stability testing

Homogeneity and stability testing

Homogeneity test

- 11 randomly selected test items
  - (including the first and the last unit portioned)
  - analysed in duplicate

Stability test

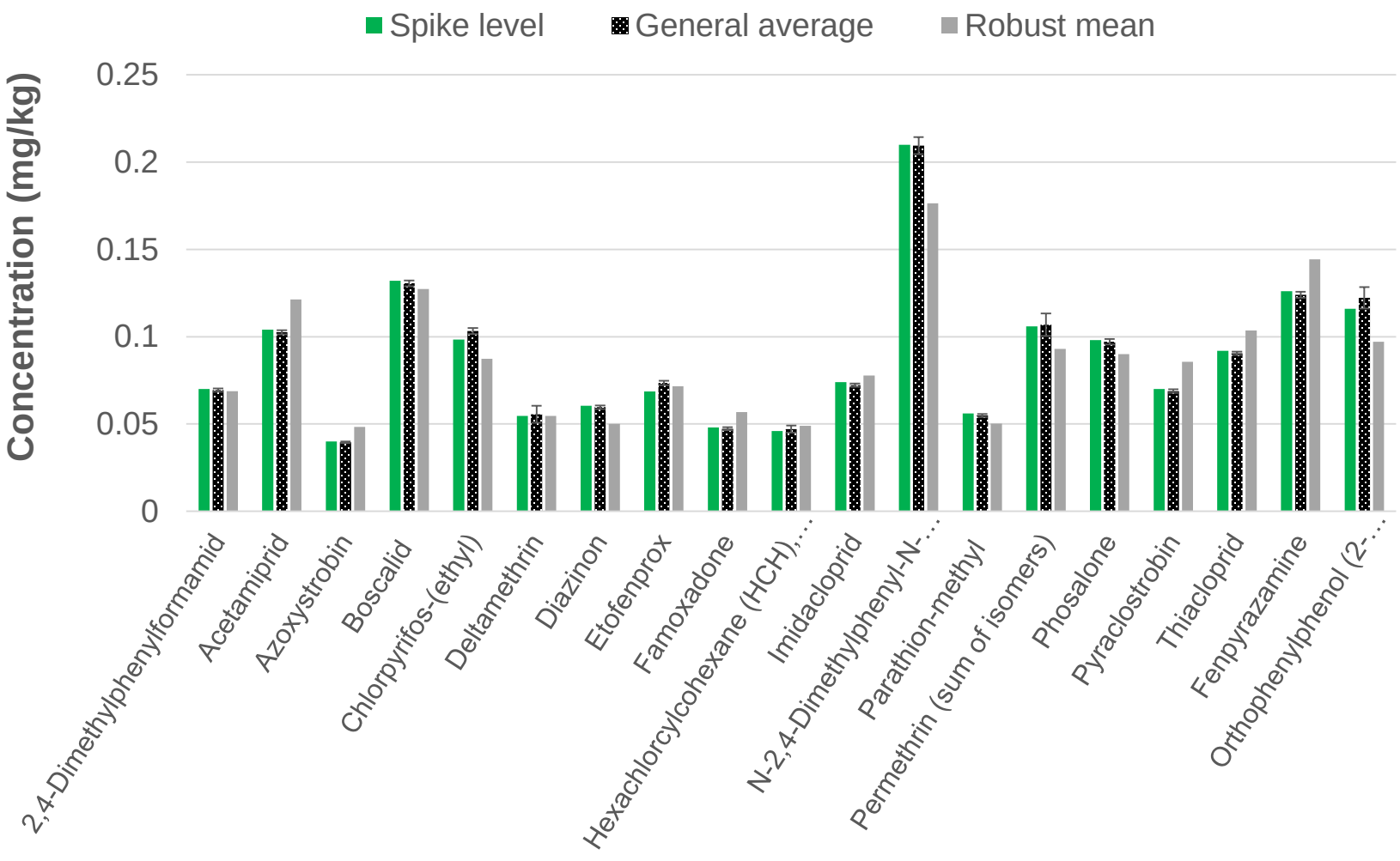
- 3 randomly selected test items were analysed for stability testing
  - analysed in duplicate
  - analysis I: before dispatch of test item (11.04.2023)
  - analysis II: after deadline of result submission (22.05.2023 / 26.05.2023)
  - test items were stored at 4°C between analyses

Statistical evaluation according to ISO 13528:2022

Homogeneity test results

Analyte	General average [mg/kg]	between-sample STD, s_s	Check value, $\sqrt{c}$	Homogeneity $s_s < \sqrt{c}$
2,4-Dimethylphenylformamid	0.0694	0.0000	0.00710	passed
Acetamiprid	0.1026	0.0008	0.01044	passed
Azoxystrobin	0.0396	0.0000	0.00404	passed
Boscalid	0.1306	0.0000	0.01336	passed
Chlorpyrifos-(ethyl)	0.1033	0.0000	0.01063	passed
Deltamethrin	0.0555	0.0000	0.00769	passed
Diazinon	0.0596	0.0000	0.00614	passed
Etofenprox	0.0735	0.0001	0.00755	passed
Famoxadone	0.0473	0.0000	0.00488	passed
Hexachlorocylcohexane (HCH), gamma-isomer (Lindane)	0.0471	0.0000	0.00516	passed
Imidacloprid	0.0722	0.0000	0.00740	passed
N-2,4-Dimethylphenyl-N-methylformamidin	0.2095	0.0000	0.02191	passed
Parathion-methyl	0.0549	0.0000	0.00564	passed
Permethrin (sum of isomers)	0.1069	0.0000	0.01338	passed
Phosalone	0.0971	0.0000	0.01000	passed
Pyraclostrobin	0.0688	0.0005	0.00703	passed
Thiacloprid	0.0906	0.0004	0.00922	passed
Fenpyrazamine (parent only)	0.1240	0.0007	0.01267	passed
Orthophenylphenol (2-phenylphenol)	0.1223	0.0000	0.01426	passed

Homogeneity test results



Stability test results

	Stability test I (11.04.2023)			Stability test II (22.05.2023)			Result		
	Average [mg/kg], $\overline{y}_1$	**RSD**	**% Recovery** (vs. homogeneity test)	**Average** [mg/kg], $\overline{y}_2$	**RSD**	**% Recovery** (vs. stability test I)	**Diff Averages** $\lvert\overline{y}_1 - \overline{y}_2\rvert$	Stability **check value**, $\lvert\overline{y}1 - \overline{y}2\rvert / \sigma_{pt}$	**Result** of stability test, (passed = $\lvert\overline{y}1 - \overline{y}2\rvert / \sigma_{pt} \leq 0.3$)
2,4-Dimethylphenylform-amid	0.069	4.6%	99%	0.068	0.4%	99%	0.001	0.0	**passed**
Acetamiprid	0.096	0.9%	94%	0.099	0.2%	103%	0.003	0.1	**passed**
Azoxystrobin	0.037	0.7%	93%	0.038	0.5%	104%	0.001	0.1	**passed**
Boscalid	0.125	1.4%	96%	0.125	0.2%	100%	0.000	0.0	**passed**
Chlorpyrifos-(ethyl)	0.098	2.3%	95%	0.097	0.7%	100%	0.000	0.0	**passed**
Deltamethrin	0.053	3.0%	96%	0.056	0.2%	105%	0.003	0.2	**passed**
Diazinon	0.055	1.6%	93%	0.056	0.6%	100%	0.000	0.0	**passed**
Etofenprox	0.073	2.4%	100%	0.070	0.3%	96%	0.003	0.2	**passed**
Famoxadone	0.046	3.0%	97%	0.045	0.3%	99%	0.000	0.0	**passed**
Hexachlorcylcohexane (HCH), gamma-isomer (Lindane)	0.044	1.0%	93%	0.046	1.7%	105%	0.002	0.2	**passed**

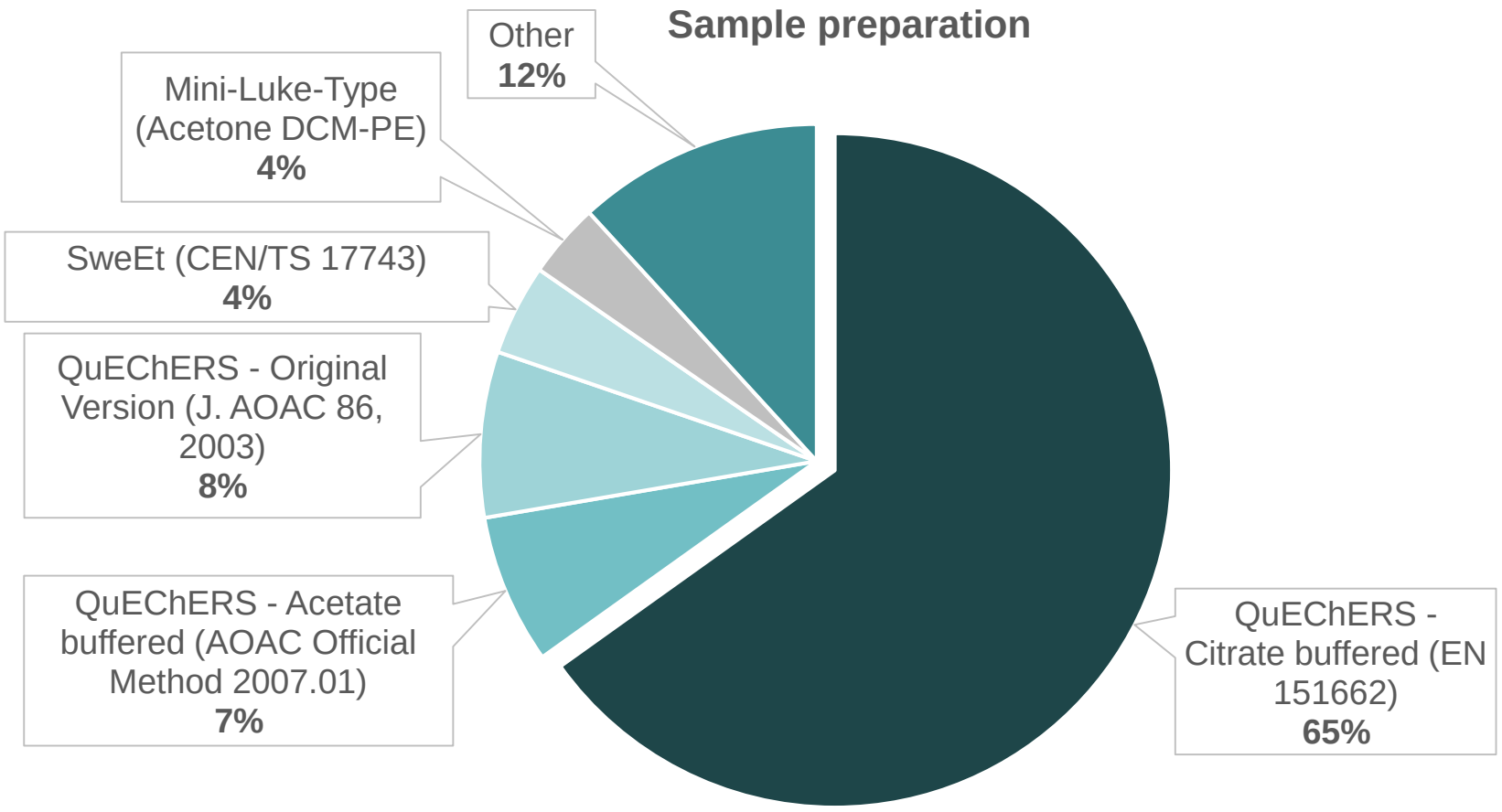
Stability test results

	Stability test I (11.04.2023)			Stability test II (22.05.2023)			Result		
	Average [mg/kg], $\bar{y}_1$	**RSD**	**% Recovery** (vs. homogeneity test)	**Average** [mg/kg], $\bar{y}_2$	**RSD**	**% Recovery** (vs. stability test I)	**Diff Averages** $\lvert\bar{y}_1 - \bar{y}_2\rvert$	Stability **check value**, $\lvert\bar{y}1 - \bar{y}2\rvert / \sigma_{pt}$	**Result** of stability test, (passed = $\lvert\bar{y}1 - \bar{y}2\rvert / \sigma_{pt} \leq 0.3$)
Imidacloprid	0.066	3.8%	91%	0.071	0.7%	108%	0.005	0.3	passed
N-2,4-Dimethylphenyl-N-methylformamidin	0.200	0.6%	96%	0.196	1.5%	98%	0.005	0.1	passed
Parathion-methyl	0.049	4.1%	90%	0.048	0.7%	98%	0.001	0.1	passed
Permethrin (sum of isomers)	0.103	0.9%	96%	0.107	1.8%	104%	0.004	0.2	passed
Phosalone	0.093	1.0%	96%	0.094	0.5%	101%	0.001	0.1	passed
Pyraclostrobin	0.065	1.9%	95%	0.067	0.6%	102%	0.001	0.1	passed
Thiacloprid	0.085	1.5%	94%	0.088	0.7%	104%	0.003	0.1	passed
Fenpyrazamine (parent only)	0.116	1.2%	94%	0.120	0.5%	103%	0.004	0.1	passed
Orthophenylphenol	0.116	2.3%	95%	0.125	0.0%	108%	0.010	0.3	passed

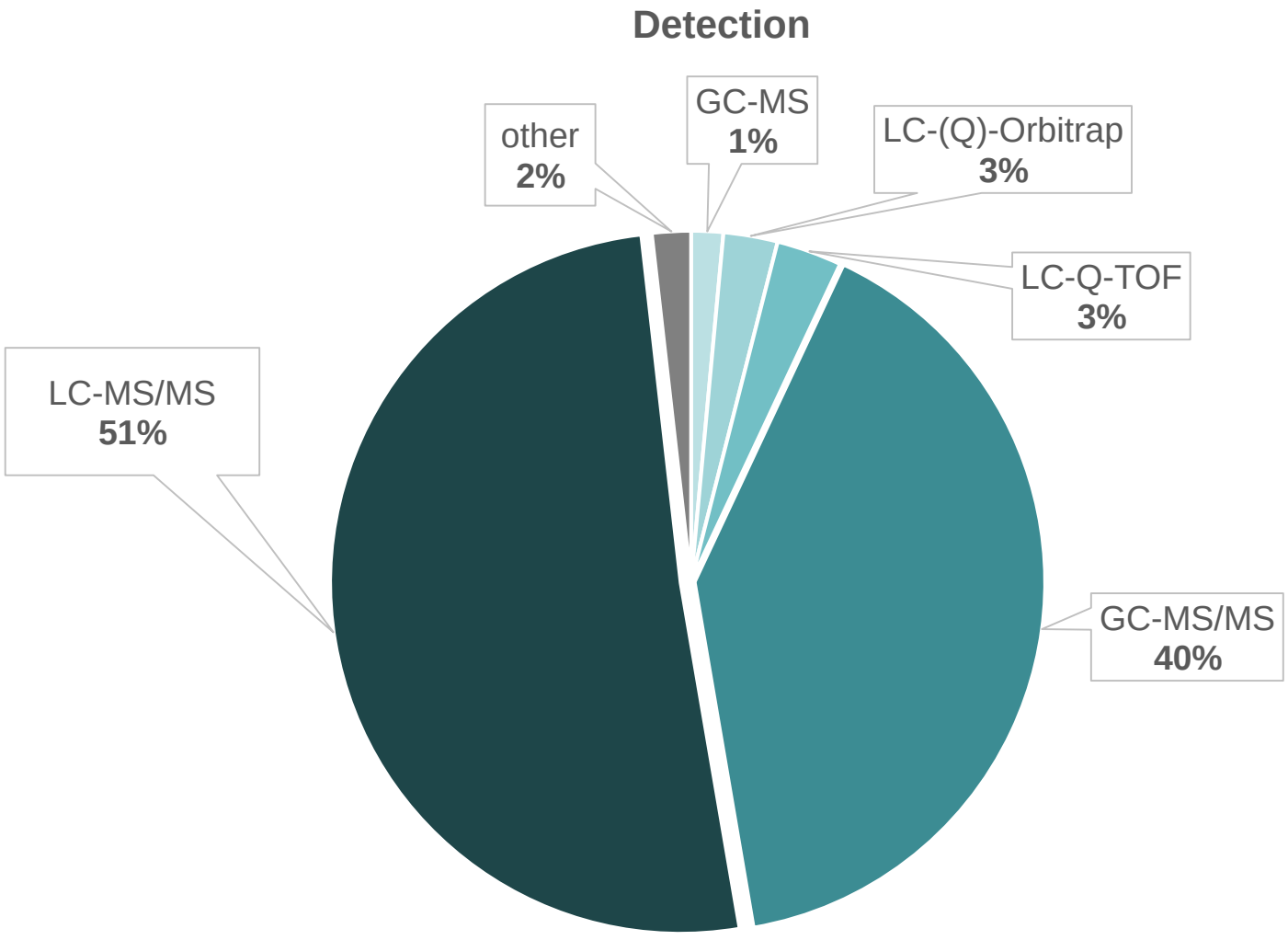
→ All analytes of the proficiency test are considered to be adequately stable during the result submission period

Analytical methods

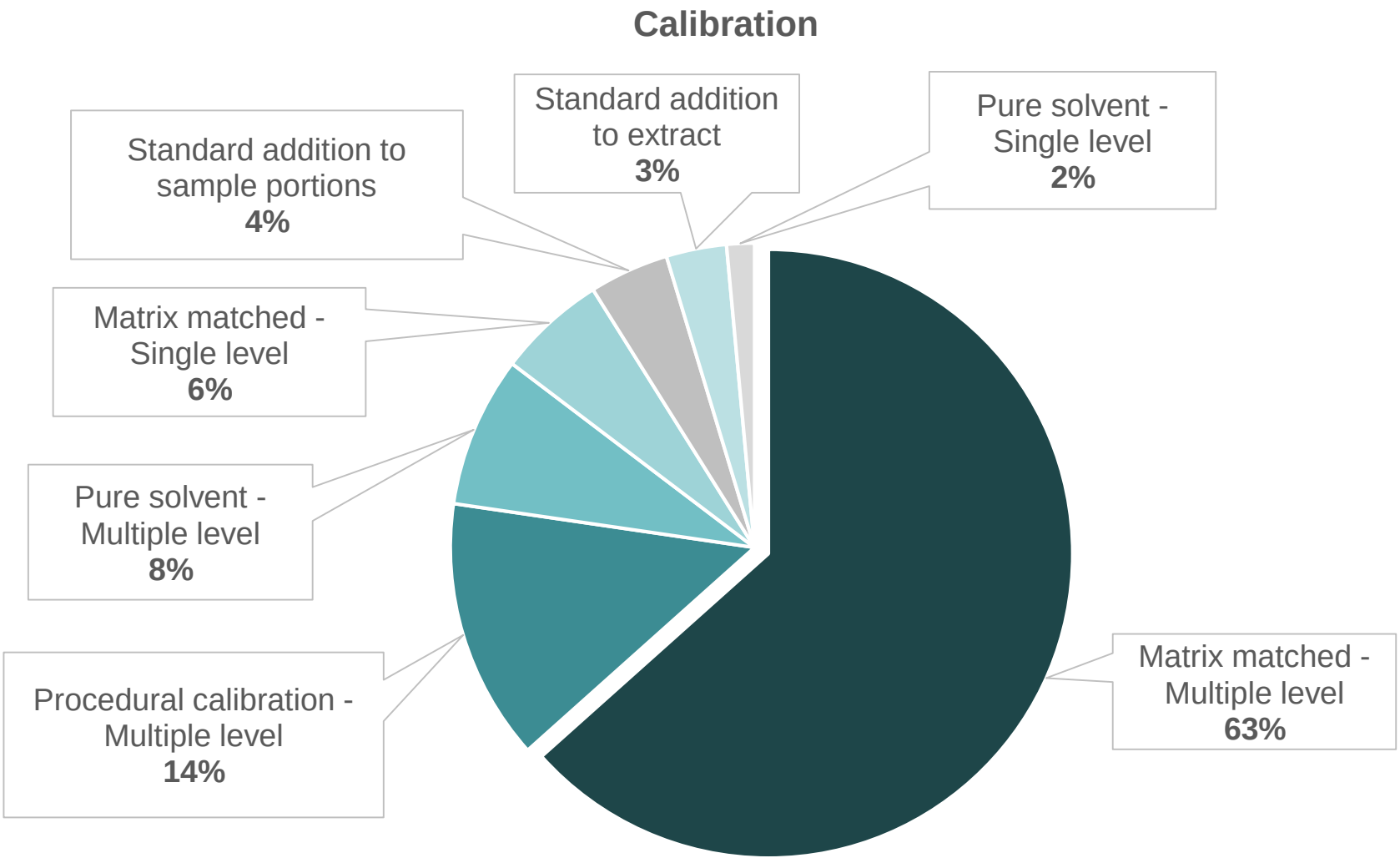
Methods used by participants



Methods used by participants



Methods used by participants



Results

Statistical Parameters

Evaluation according to general protocol (10th Edition):

- Assigned value:

  robust mean

  calculated according Algorithm A (ISO 13528:2020-09)

- Only EU and EFTA countries laboratories

Statistical Parameters

Results excluded from the population used for robust statistics:

→ **3 labs from non EU/EFTA countries**

→ **Results of 99 EU/EFTA labs were used for robust statistics**

Results of robust statistic

Mandatory

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	Acceptable	Questionable	Unacceptable
2,4-Dimethylphenylformamid (am)	0.0688	14.1%	67	58	1	8	8	35	0.075	92%	86.6%	1.5%	11.9%
Acetamiprid	0.1214	12.6%	89	84	2	3	2	13	0.125	97%	94.4%	2.2%	3.4%
Azoxystrobin	0.0483	13.8%	90	86	1	3	2	12	0.050	97%	95.6%	1.1%	3.3%
Boscalid	0.1273	11.9%	89	87	0	2	0	13	0.135	94%	97.8%	0.0%	2.2%
Chlorpyrifos	0.0873	16.1%	96	89	2	5	2	6	0.095	92%	92.7%	2.1%	5.2%
Deltamethrin	0.0546	16.8%	96	88	3	5	2	6	0.060	91%	91.7%	3.1%	5.2%
Diazinon	0.0501	16.5%	94	90	1	3	1	8	0.055	91%	95.7%	1.1%	3.2%
Etofenprox	0.0717	14.8%	87	81	1	5	4	15	0.075	96%	93.1%	1.1%	5.7%
Famoxadone	0.0568	15.2%	84	76	3	5	5	18	0.060	95%	90.5%	3.6%	6.0%
Lindane	0.0490	15.2%	94	85	1	8	5	8	0.054	91%	90.4%	1.1%	8.5%
Imidacloprid	0.0778	13.6%	89	85	0	4	1	13	0.080	97%	95.5%	0.0%	4.5%
N-2,4-Dimethylphenyl-N-methylform	0.1764	27.4%	68	53	0	15	7	34	0.185	95%	77.9%	0.0%	22.1%
Parathion-methyl	0.0502	22.8%	89	77	3	9	5	13	0.065	77%	86.5%	3.4%	10.1%
Permethrin (sum of isomers)	0.0930	23.0%	95	87	6	2	1	7	0.100	93%	91.6%	6.3%	2.1%
Phosalone	0.0900	15.2%	86	83	0	3	2	16	0.100	90%	96.5%	0.0%	3.5%
Pyraclostrobin	0.0857	12.1%	83	78	0	5	3	19	0.090	95%	94.0%	0.0%	6.0%
Thiacloprid	0.1036	11.5%	88	83	2	3	1	14	0.105	99%	94.3%	2.3%	3.4%
Fenpyrazamine	0.1443	12.3%	67	65	0	2	1	35	0.150	96%	97.0%	0.0%	3.0%
Orthophenylphenol (2-phenylphenol)	0.0971	16.2%	58	53	0	5	4	44	0.110	88%	91.4%	0.0%	8.6%

Results of robust statistic

Mandatory

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	Acceptable	Questionable	Unacceptable
2,4-Dimethylphenylformamid (am)	0.0688	14.1%	67	58	1	8	8	35	0.075	92%	86.6%	1.5%	11.9%
Acetamiprid	0.1214	12.6%	89	84	2	3	2	13	0.125	97%	94.4%	2.2%	3.4%
Azoxystrobin	0.0483	13.8%	90	86	1	3	2	12	0.050	97%	95.6%	1.1%	3.3%
Boscalid	0.1273	11.9%	89	87	0	2	0	13	0.135	94%	97.8%	0.0%	2.2%
Chlorpyrifos	0.0873	16.1%	96	89	2	5	2	6	0.095	92%	92.7%	2.1%	5.2%
Deltamethrin	0.0546	16.8%	96	88	3	5	2	6	0.060	91%	91.7%	3.1%	5.2%
Diazinon	0.0501	16.5%	94	90	1	3	1	8	0.055	91%	95.7%	1.1%	3.2%
Etofenprox	0.0717	14.8%	87	81	1	5	4	15	0.075	96%	93.1%	1.1%	5.7%
Famoxadone	0.0568	15.2%	84	76	3	5	5	18	0.060	95%	90.5%	3.6%	6.0%
Lindane	0.0490	15.2%	94	85	1	8	5	8	0.054	91%	90.4%	1.1%	8.5%
Imidacloprid	0.0778	13.6%	89	85	0	4	1	13	0.080	97%	95.5%	0.0%	4.5%
N-2,4-Dimethylphenyl-N-methylform	0.1764	27.4%	68	53	0	15	7	34	0.185	95%	77.9%	0.0%	22.1%
Parathion-methyl	0.0502	22.8%	89	77	3	9	5	13	0.065	77%	86.5%	3.4%	10.1%
Permethrin (sum of isomers)	0.0930	23.0%	95	87	6	2	1	7	0.100	93%	91.6%	6.3%	2.1%
Phosalone	0.0900	15.2%	86	83	0	3	2	16	0.100	90%	96.5%	0.0%	3.5%
Pyraclostrobin	0.0857	12.1%	83	78	0	5	3	19	0.090	95%	94.0%	0.0%	6.0%
Thiacloprid	0.1036	11.5%	88	83	2	3	1	14	0.105	99%	94.3%	2.3%	3.4%
							51						
Fenpyrazamine	0.1443	12.3%	67	65	0	2	1	35	0.150	96%	97.0%	0.0%	3.0%
Orthophenylphenol (2-phenylphenol)	0.0971	16.2%	58	53	0	5	4	44	0.110	88%	91.4%	0.0%	8.6%

False positive results

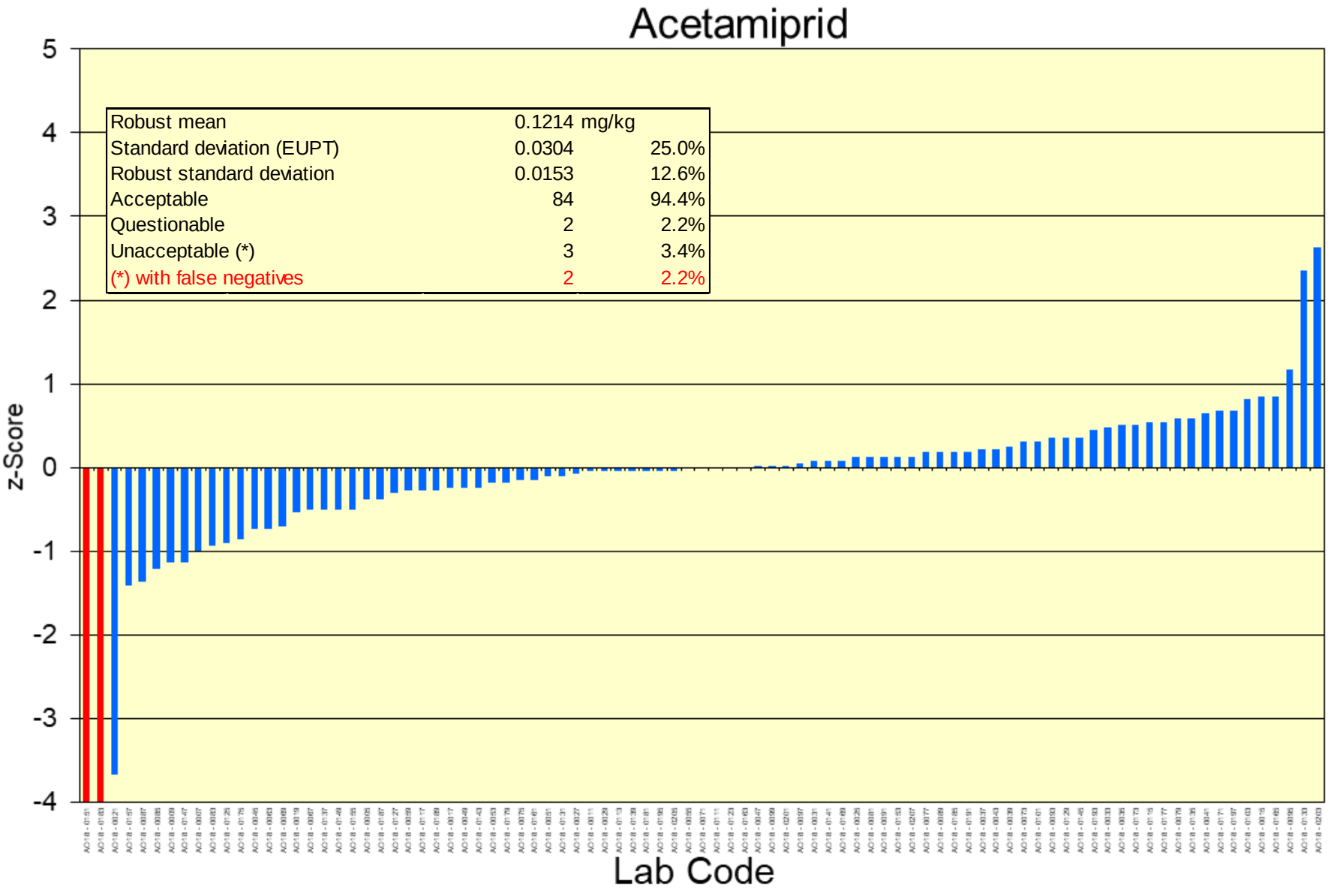
5 laboratories reported 6 tentative false positive results for 4 pesticides

Pesticide detected	Number	Reported concentrations above the MRRL value [mg/kg] (lab code)
Chlorpyrifos-methyl	1	0.082 (61)
Tetramethrin	1	0.006 (103)
HCH-alpha	1	0.022 (179)
Dimoxystrobin	1	0.240 (117)
HCH-beta	3	0.029 (117);0.06 (69); 0.052 (149)

Lab 103 no fp as <RL (0.01 mg/kg), Lab 117 possible mismatch with lindane

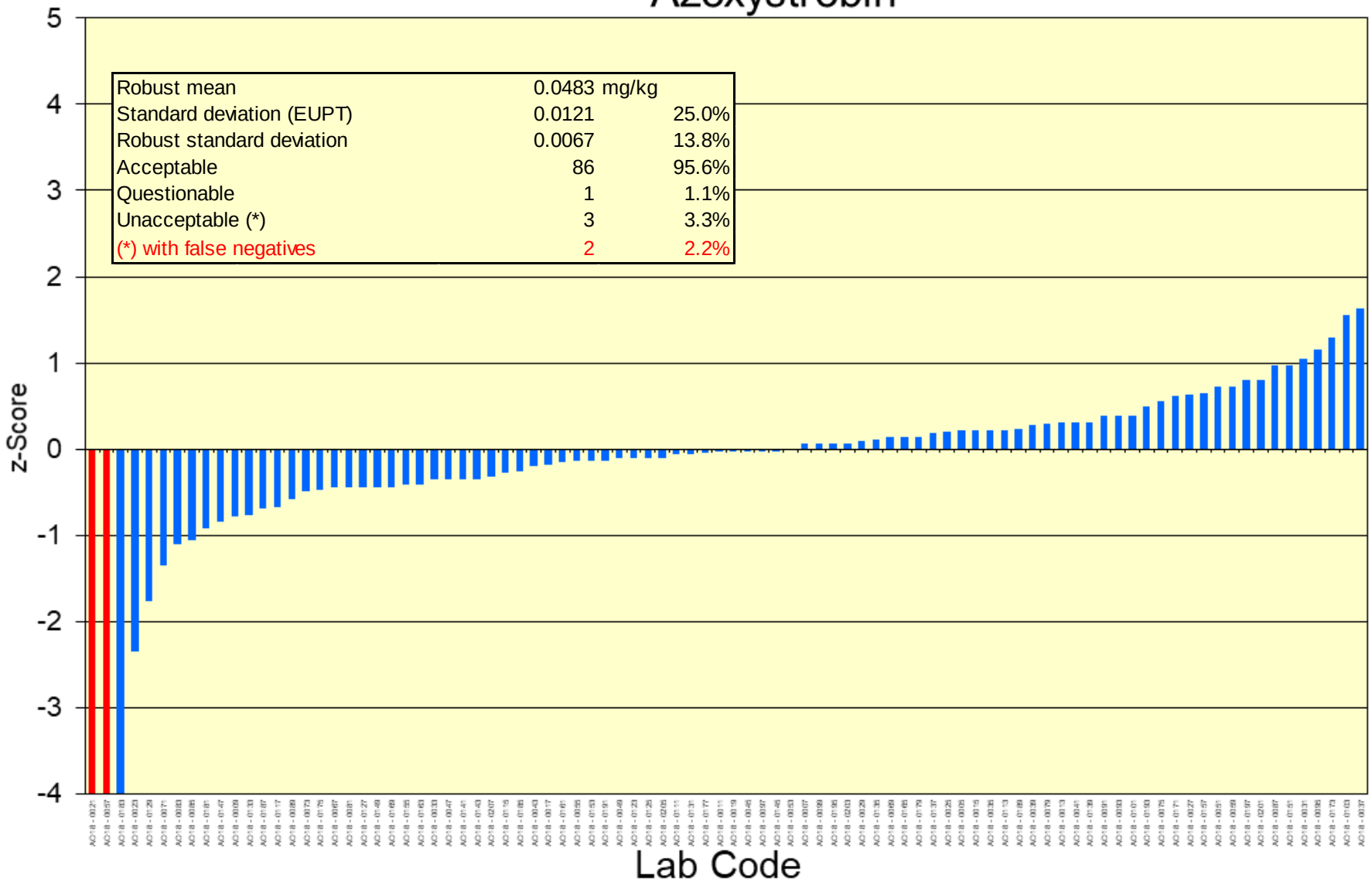
z-scores

mandatory pesticides


Acetamiprid
Robust mean 0.1214 mg/kg
Standard deviation (EUPT) 0.0304 25.0%
Robust standard deviation 0.0153 12.6%
Acceptable 84 94.4%
Questionable 2 2.2%
Unacceptable (*) 3 3.4%
(*) with false negatives 2 2.2%
z-Score
5
4
3
2
1
0
-1
-2
-3
-4
Lab Code

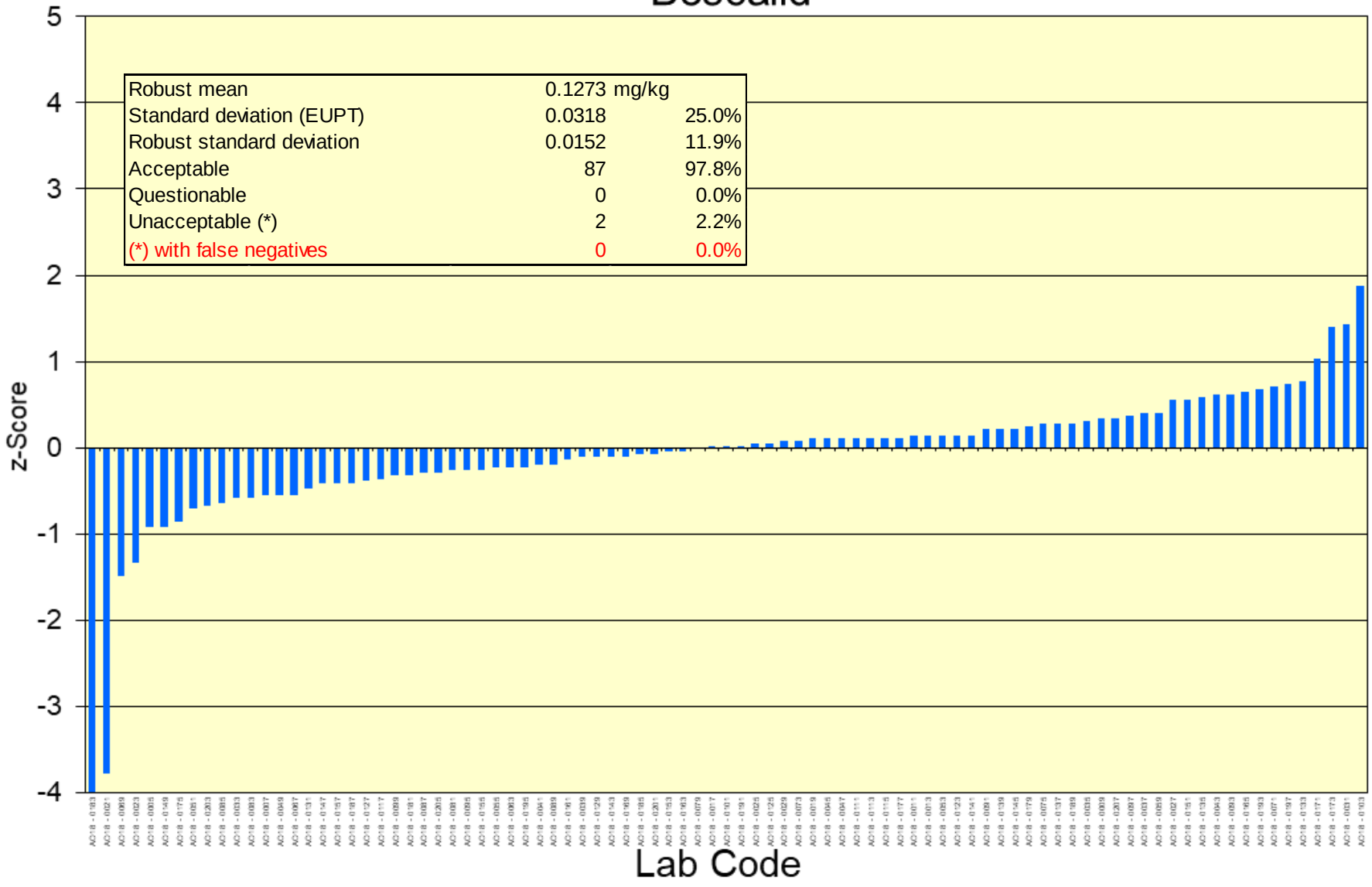
Azoxystrobin

Robust mean	0.0483 mg/kg	
Standard deviation (EUPT)	0.0121	25.0%
Robust standard deviation	0.0067	13.8%
Acceptable	86	95.6%
Questionable	1	1.1%
Unacceptable (*)	3	3.3%
(*) with false negatives	2	2.2%



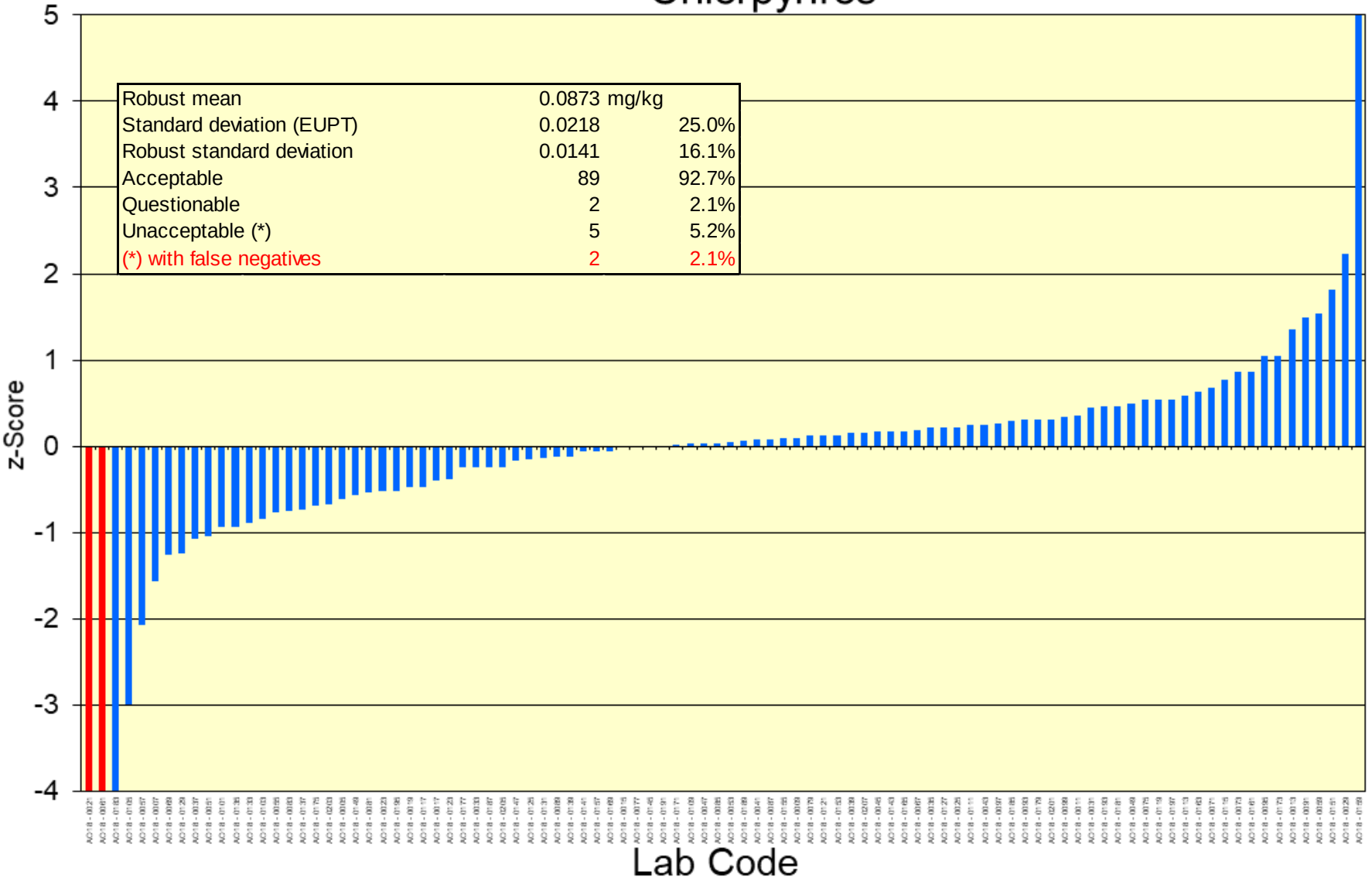
Boscalid

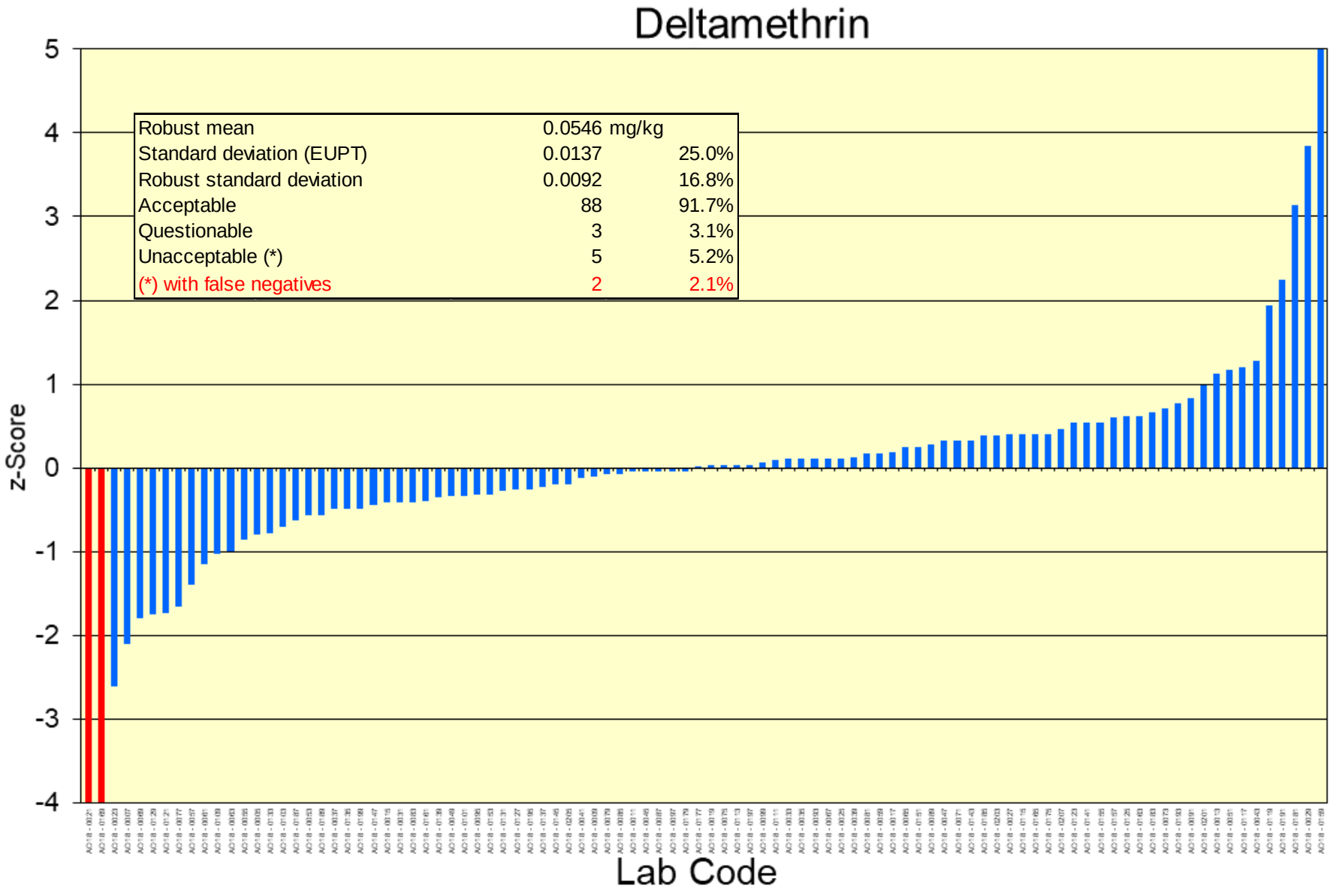
Robust mean	0.1273 mg/kg	
Standard deviation (EUPT)	0.0318	25.0%
Robust standard deviation	0.0152	11.9%
Acceptable	87	97.8%
Questionable	0	0.0%
Unacceptable (*)	2	2.2%
(*) with false negatives	0	0.0%



Chlorpyrifos

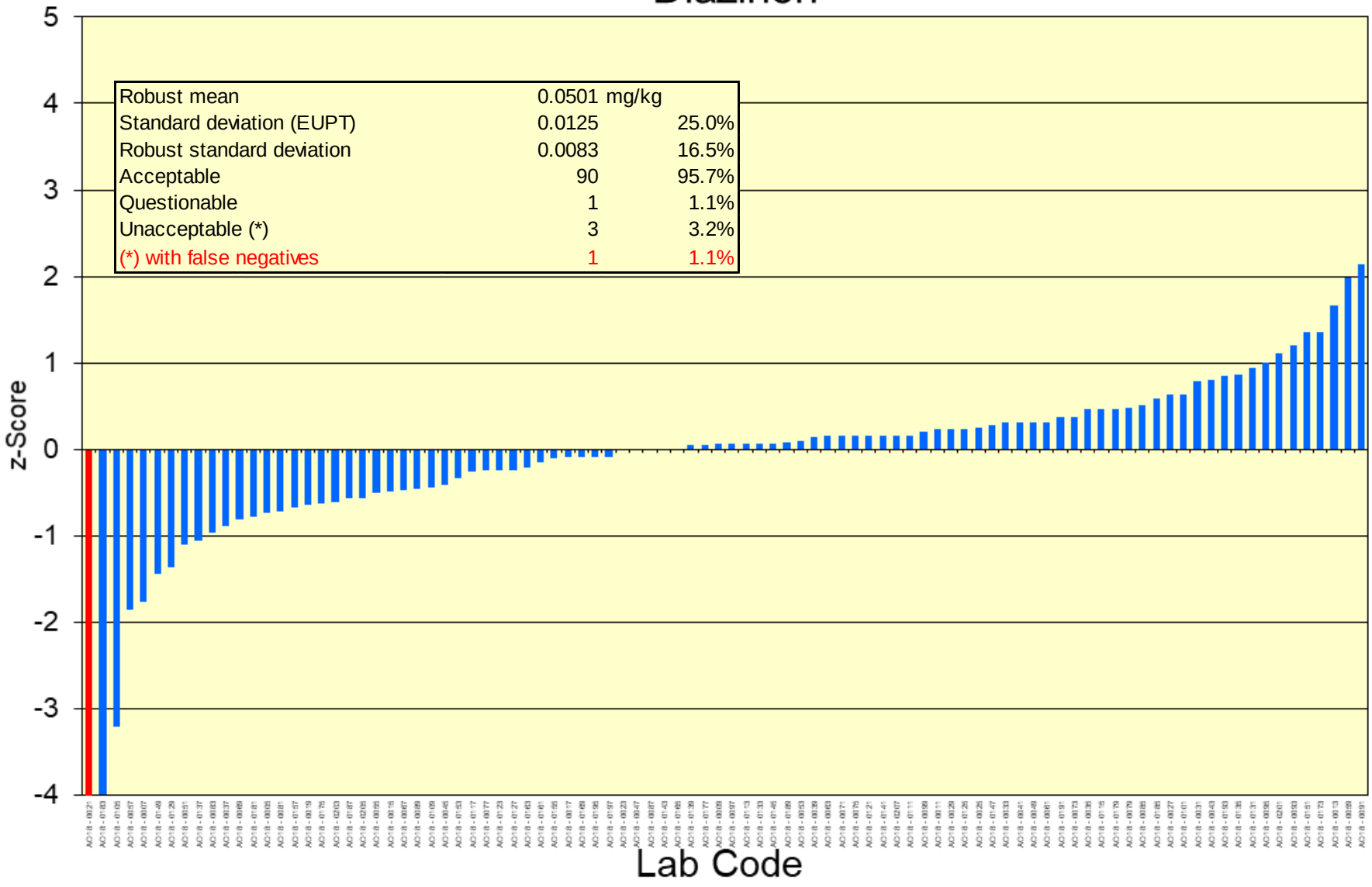
Robust mean	0.0873 mg/kg	
Standard deviation (EUPT)	0.0218	25.0%
Robust standard deviation	0.0141	16.1%
Acceptable	89	92.7%
Questionable	2	2.1%
Unacceptable (*)	5	5.2%
(*) with false negatives	2	2.1%




Deltamethrin
Robust mean 0.0546 mg/kg
Standard deviation (EUPT) 0.0137 25.0%
Robust standard deviation 0.0092 16.8%
Acceptable 88 91.7%
Questionable 3 3.1%
Unacceptable (*) 5 5.2%
(*) with false negatives 2 2.1%
z-Score
5
4
3
2
1
0
-1
-2
-3
-4
Lab Code

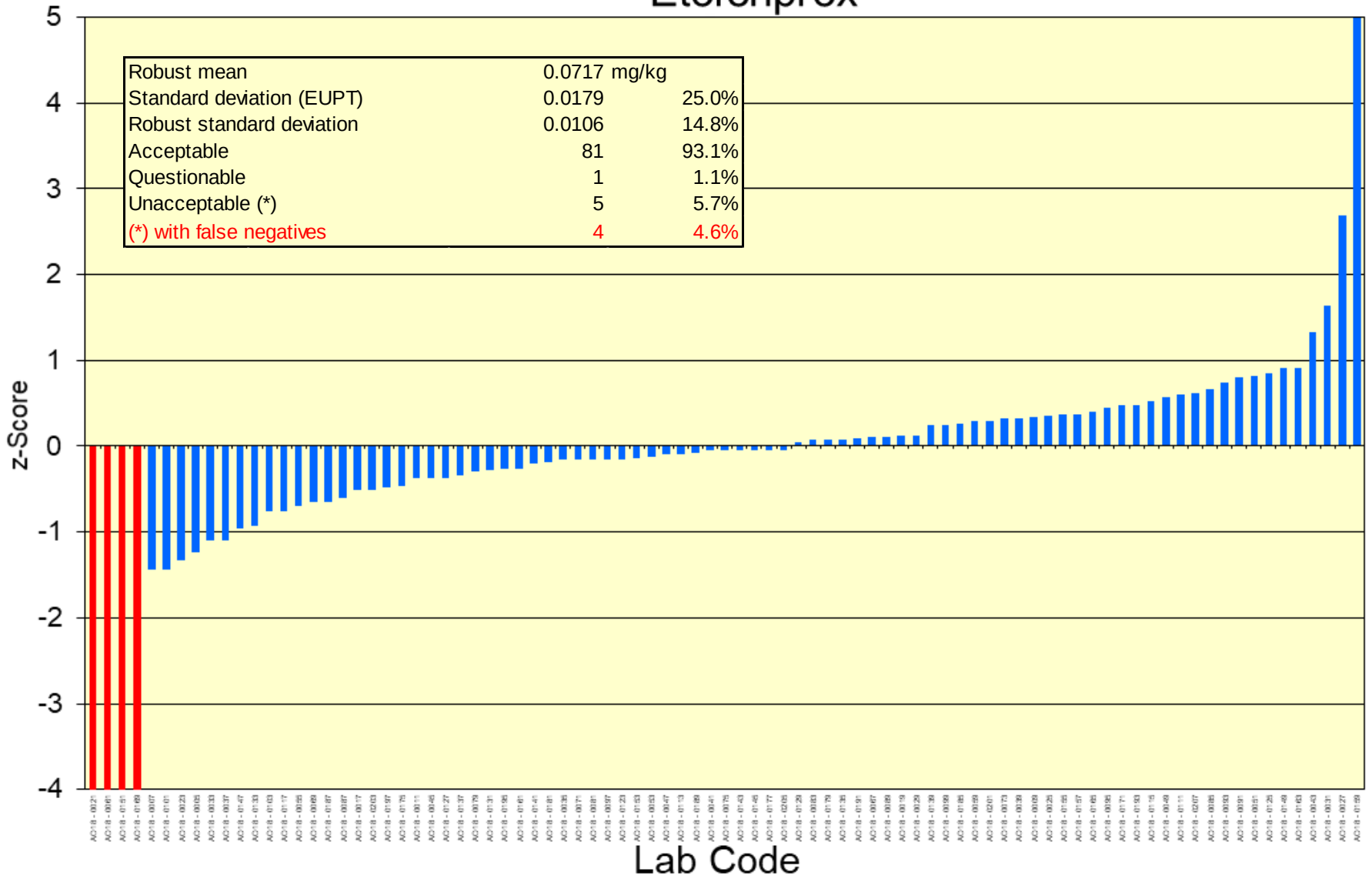
Diazinon

Robust mean	0.0501 mg/kg	
Standard deviation (EUPT)	0.0125	25.0%
Robust standard deviation	0.0083	16.5%
Acceptable	90	95.7%
Questionable	1	1.1%
Unacceptable (*)	3	3.2%
(*) with false negatives	1	1.1%



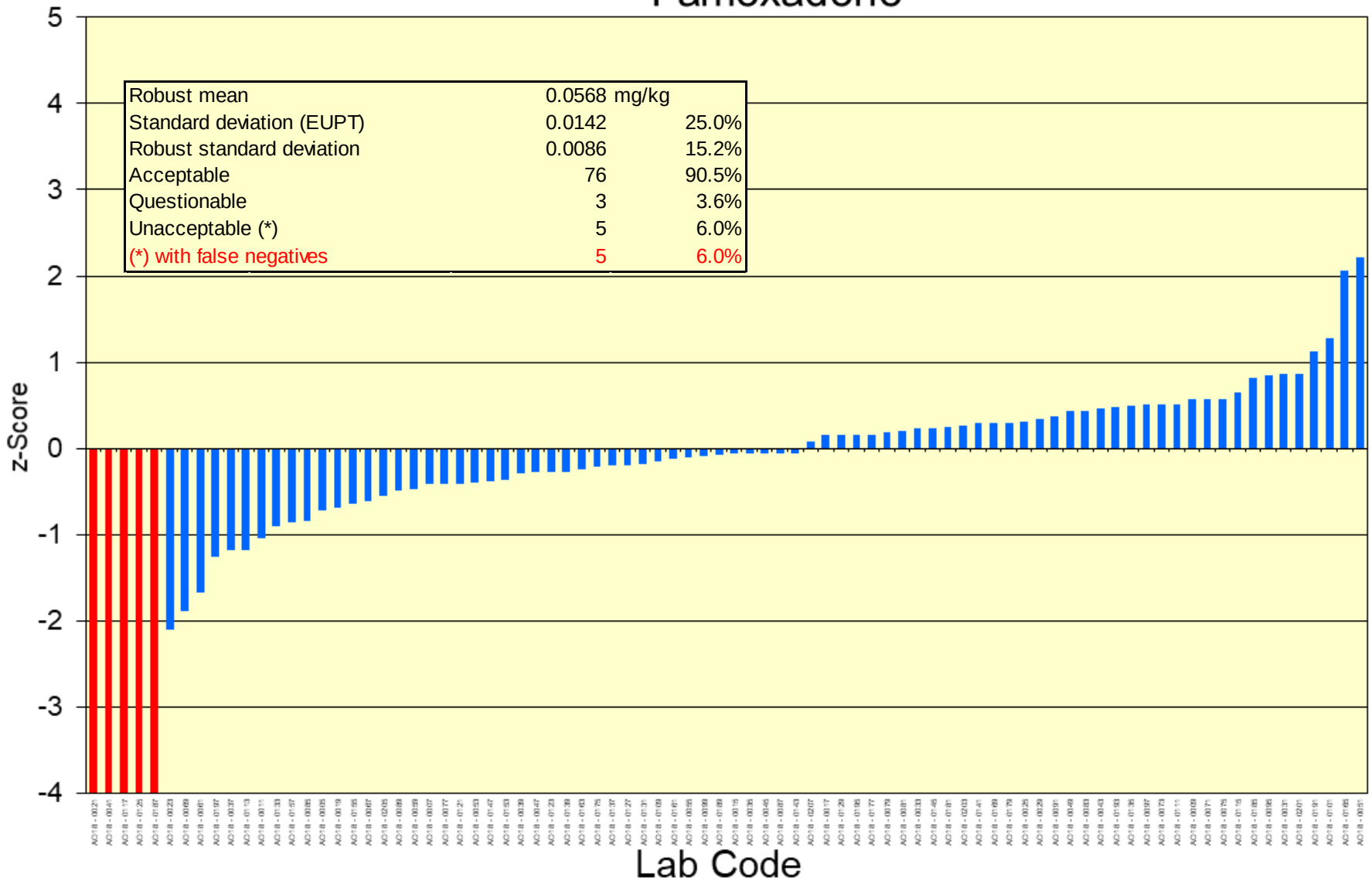
Etofenprox

Robust mean	0.0717 mg/kg	
Standard deviation (EUPT)	0.0179	25.0%
Robust standard deviation	0.0106	14.8%
Acceptable	81	93.1%
Questionable	1	1.1%
Unacceptable (*)	5	5.7%
(*) with false negatives	4	4.6%

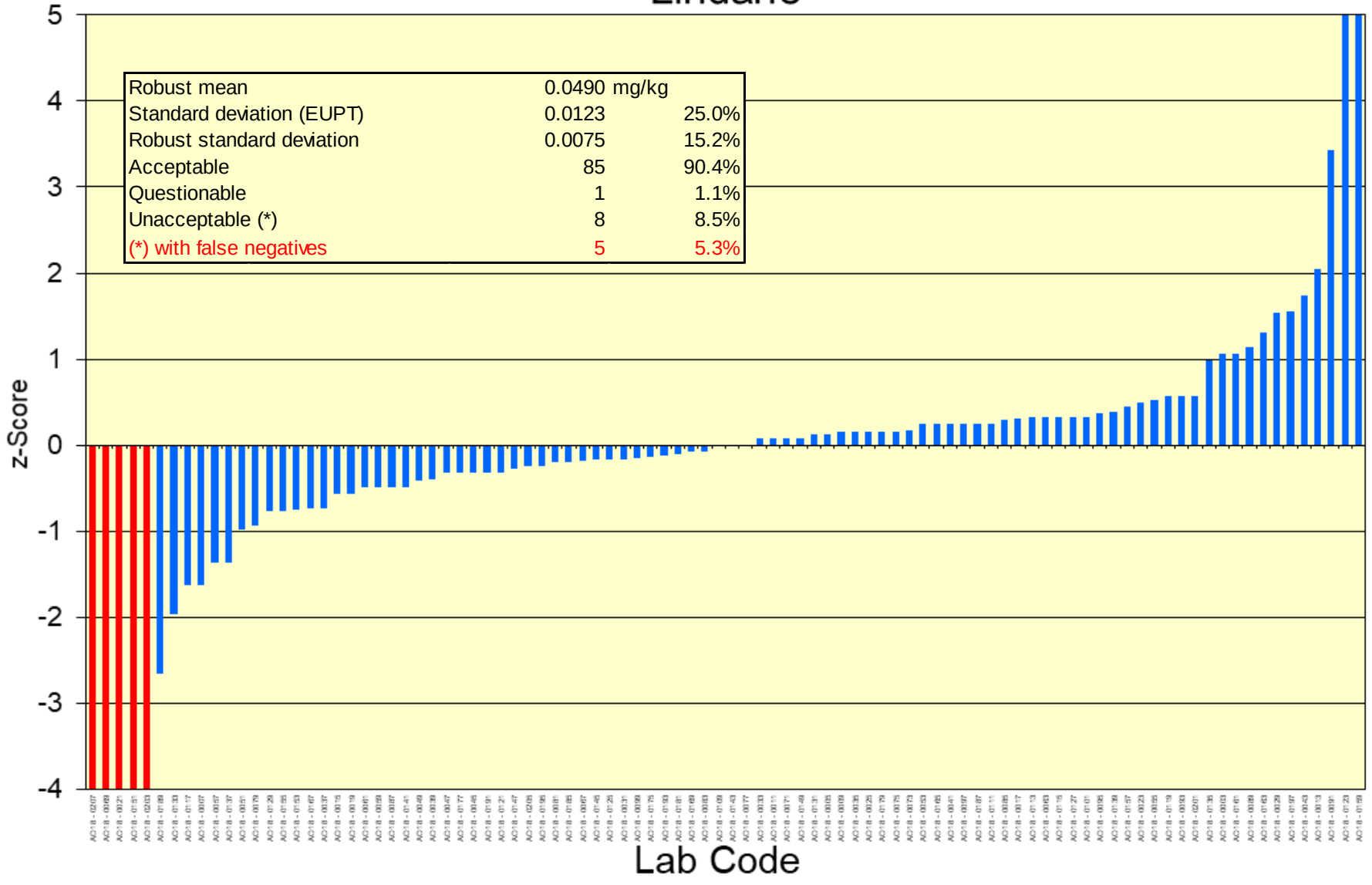


Famoxadone

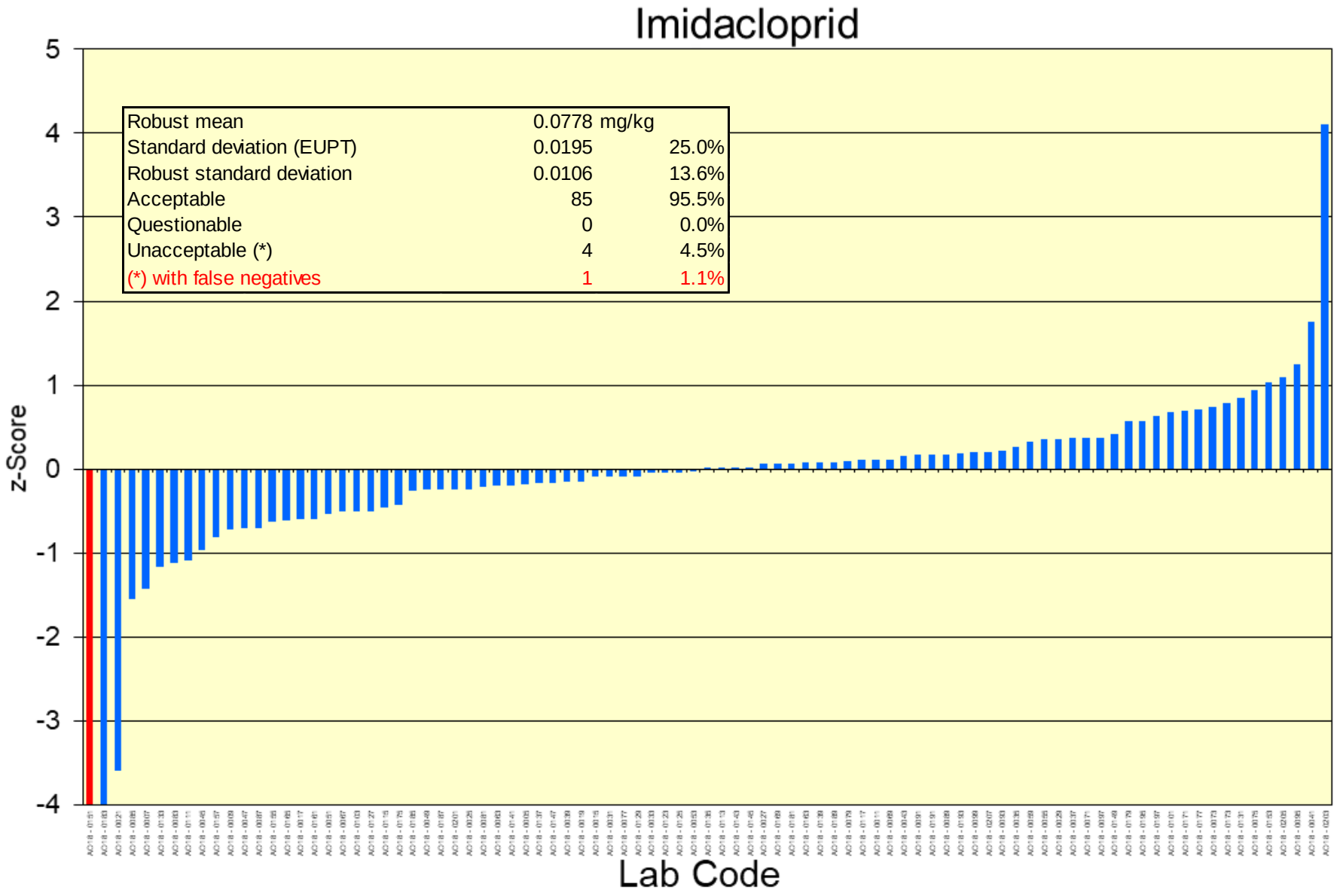
Robust mean	0.0568 mg/kg	
Standard deviation (EUPT)	0.0142	25.0%
Robust standard deviation	0.0086	15.2%
Acceptable	76	90.5%
Questionable	3	3.6%
Unacceptable (*)	5	6.0%
(*) with false negatives	5	6.0%

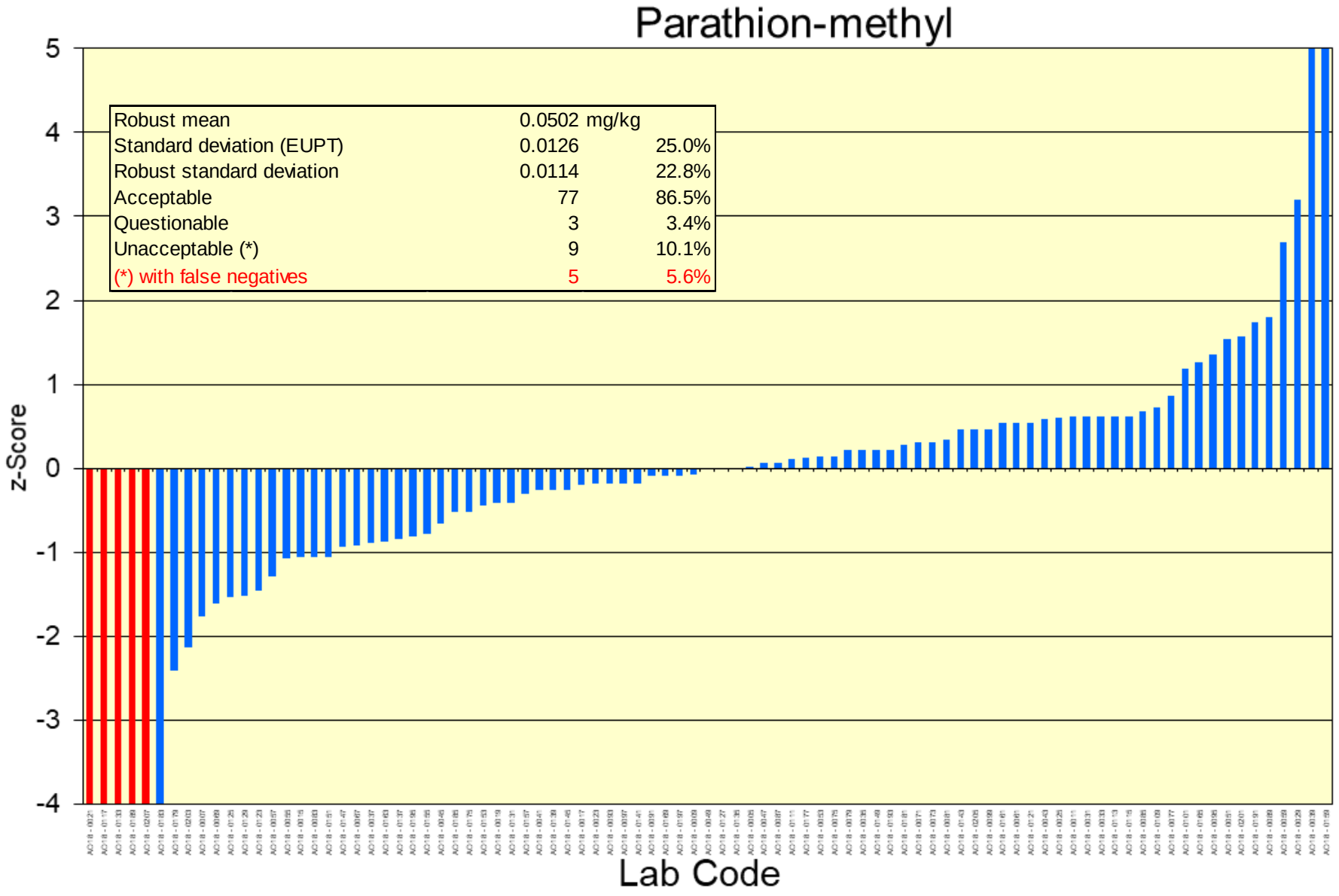


Lindane



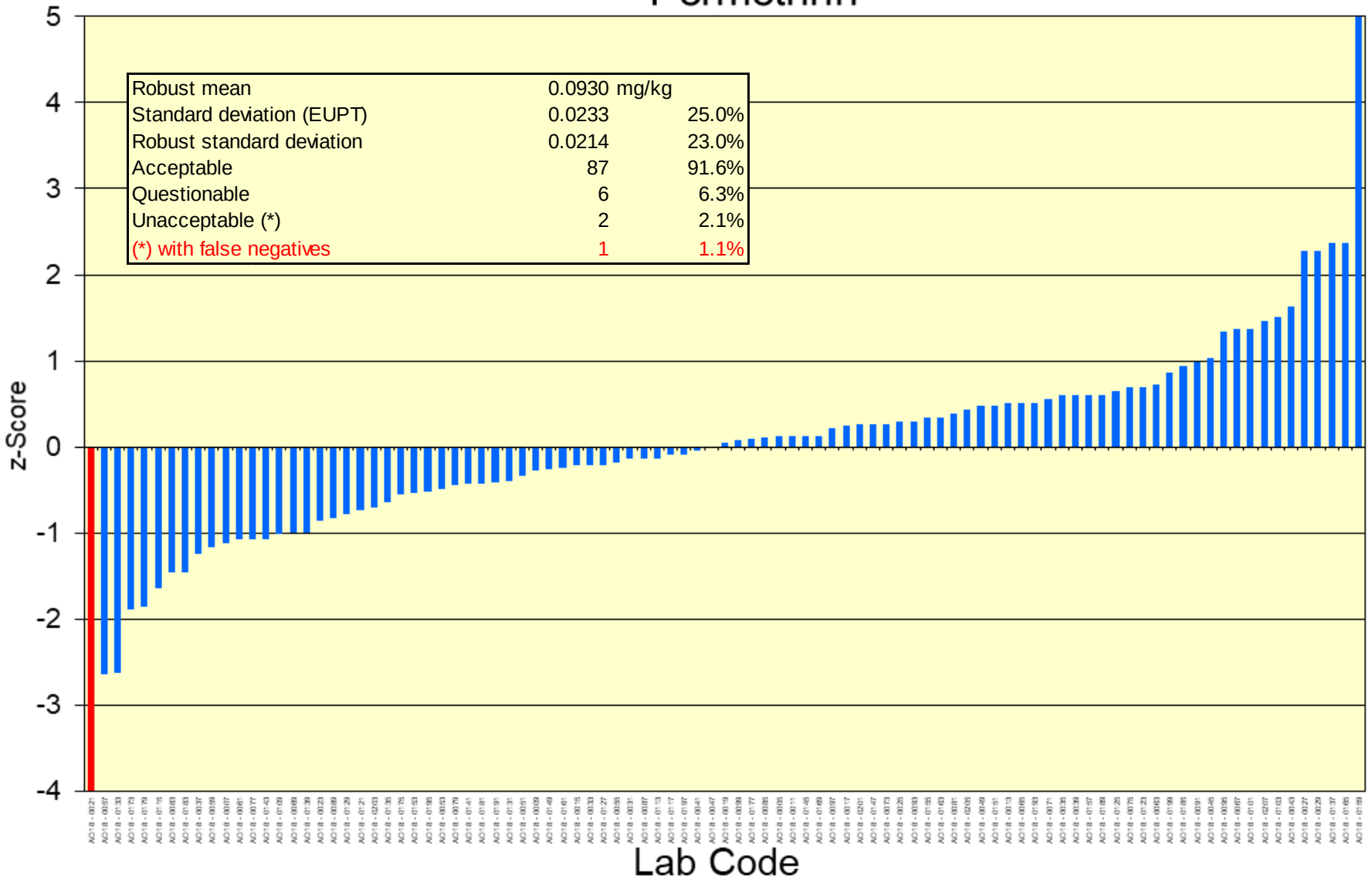
Robust mean	0.0490 mg/kg	
Standard deviation (EUPT)	0.0123	25.0%
Robust standard deviation	0.0075	15.2%
Acceptable	85	90.4%
Questionable	1	1.1%
Unacceptable (*)	8	8.5%
(*) with false negatives	5	5.3%

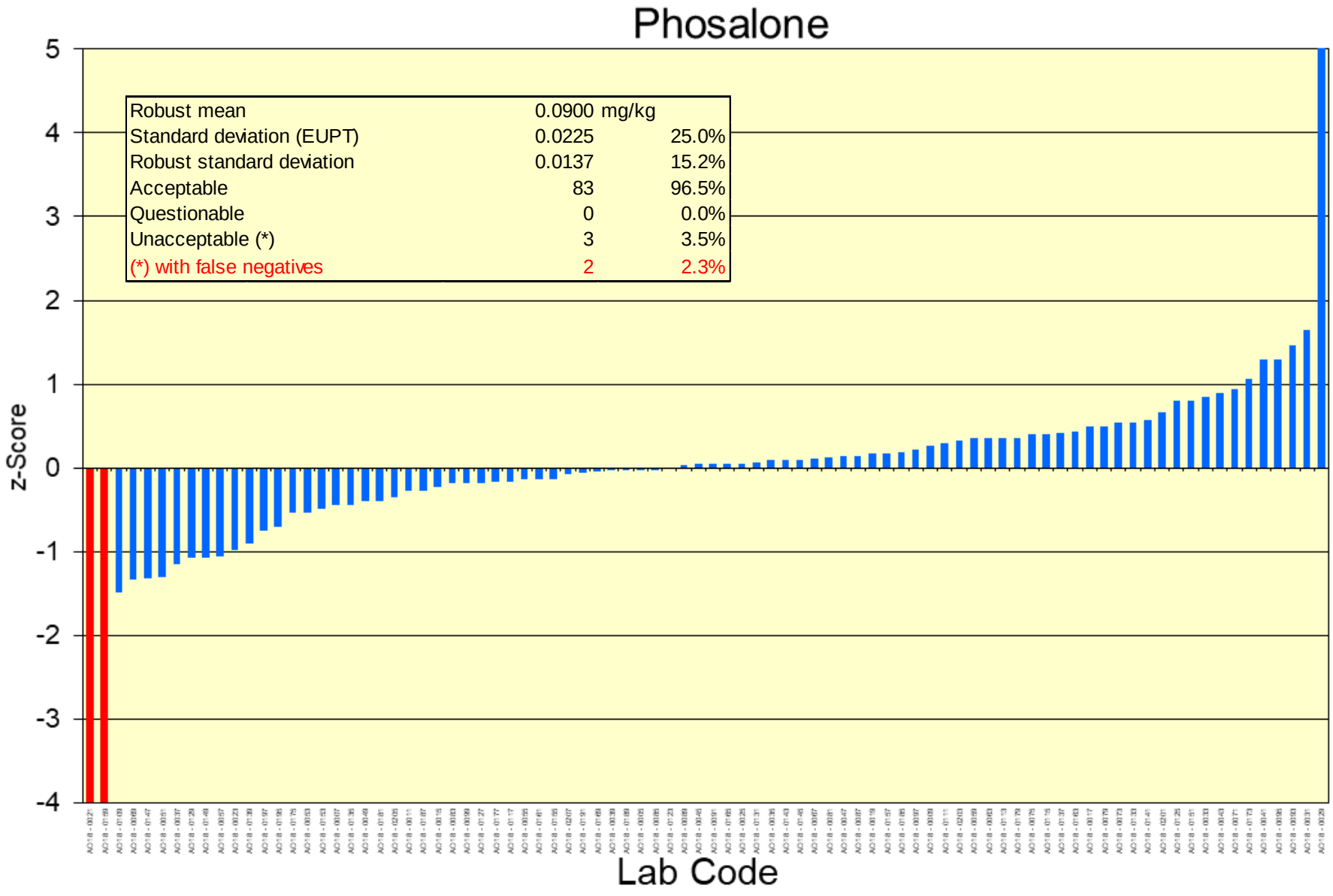

Imidacloprid
Robust mean 0.0778 mg/kg
Standard deviation (EUPT) 0.0195 25.0%
Robust standard deviation 0.0106 13.6%
Acceptable 85 95.5%
Questionable 0 0.0%
Unacceptable (*) 4 4.5%
(*) with false negatives 1 1.1%
z-Score
5
4
3
2
1
0
-1
-2
-3
-4
Lab Code

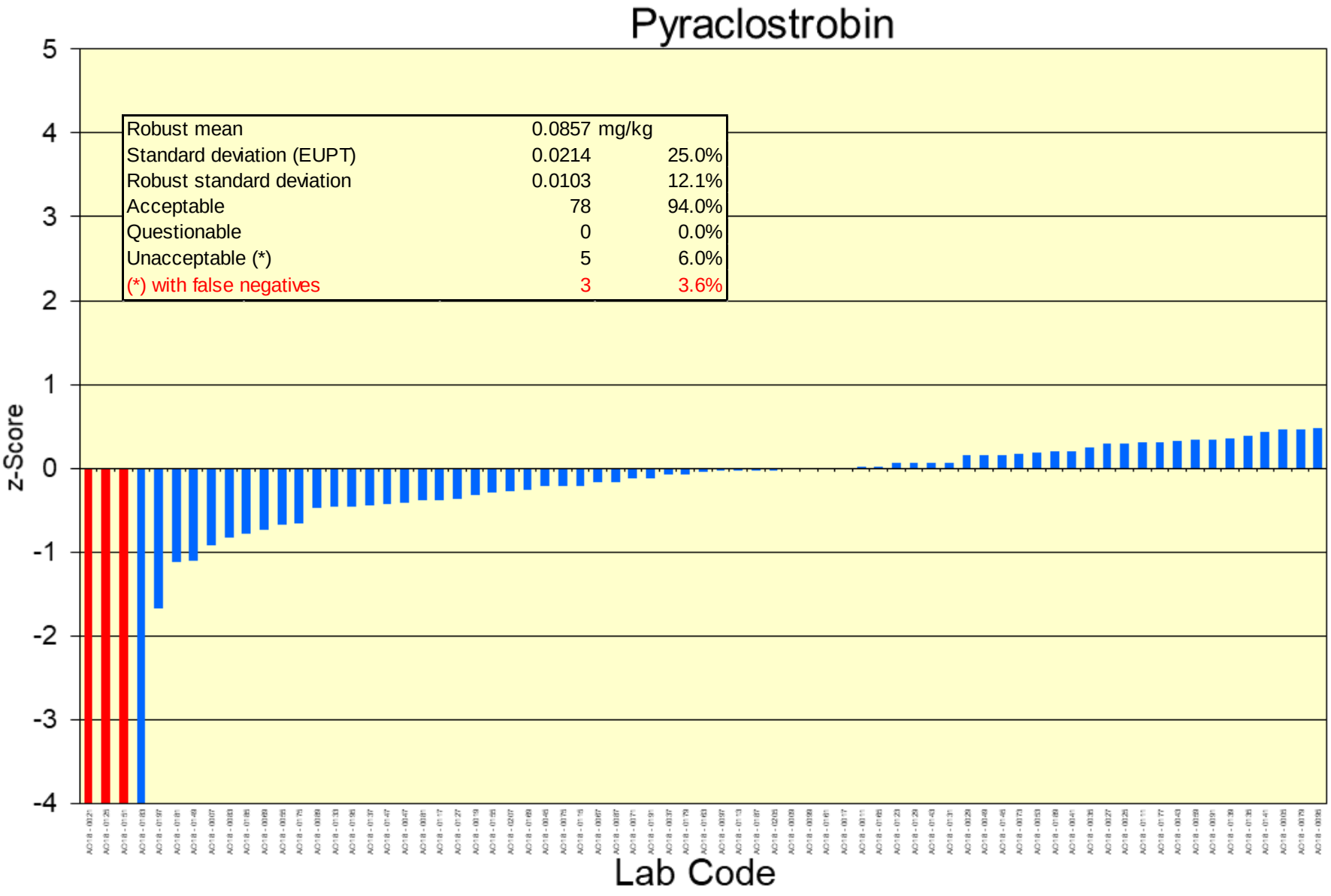

Parathion-methyl
z-Score
Lab Code
5
4
3
2
1
0
-1
-2
-3
-4
Robust mean 0.0502 mg/kg
Standard deviation (EUPT) 0.0126 25.0%
Robust standard deviation 0.0114 22.8%
Acceptable 77 86.5%
Questionable 3 3.4%
Unacceptable (*) 9 10.1%
(*) with false negatives 5 5.6%

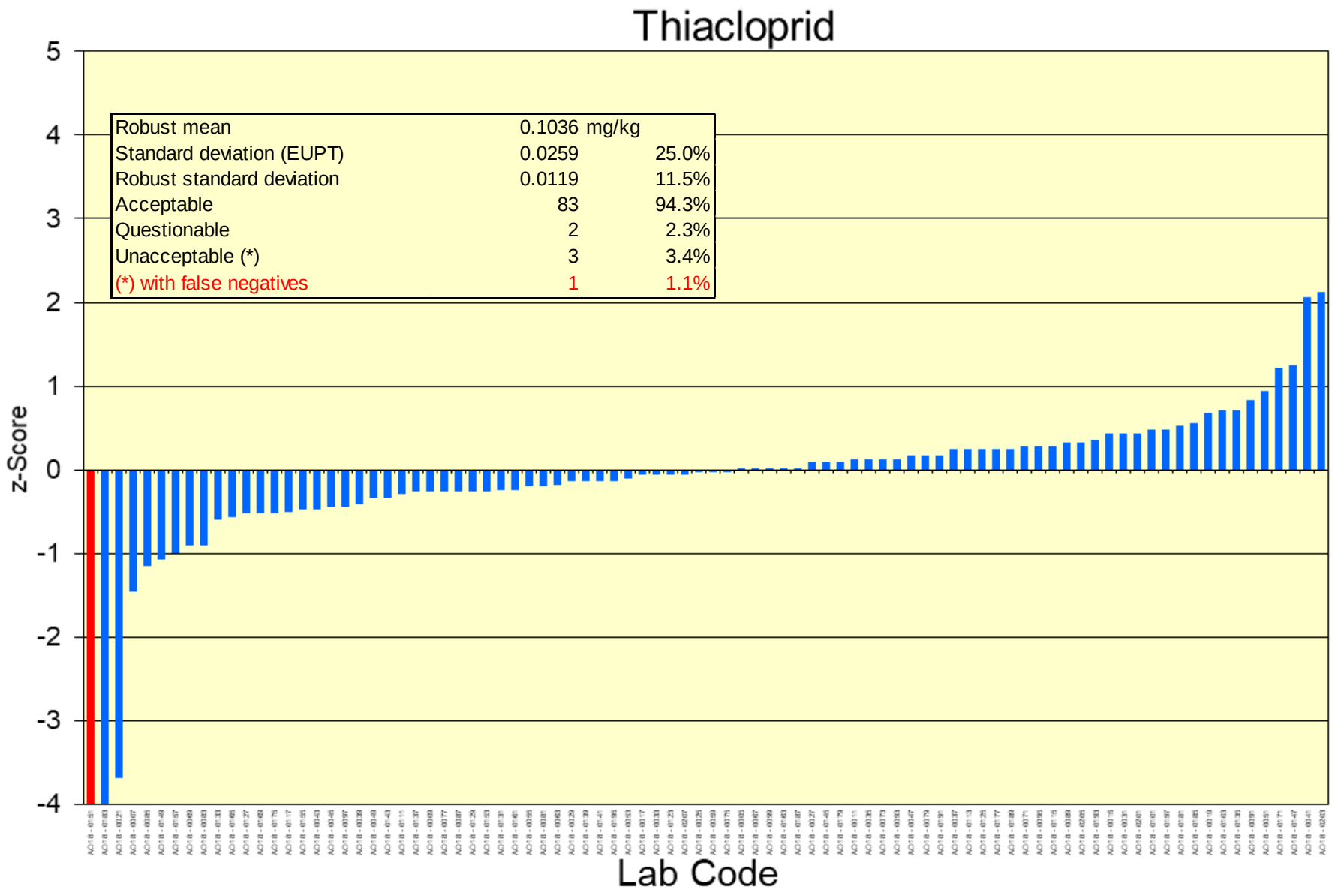
Permethrin

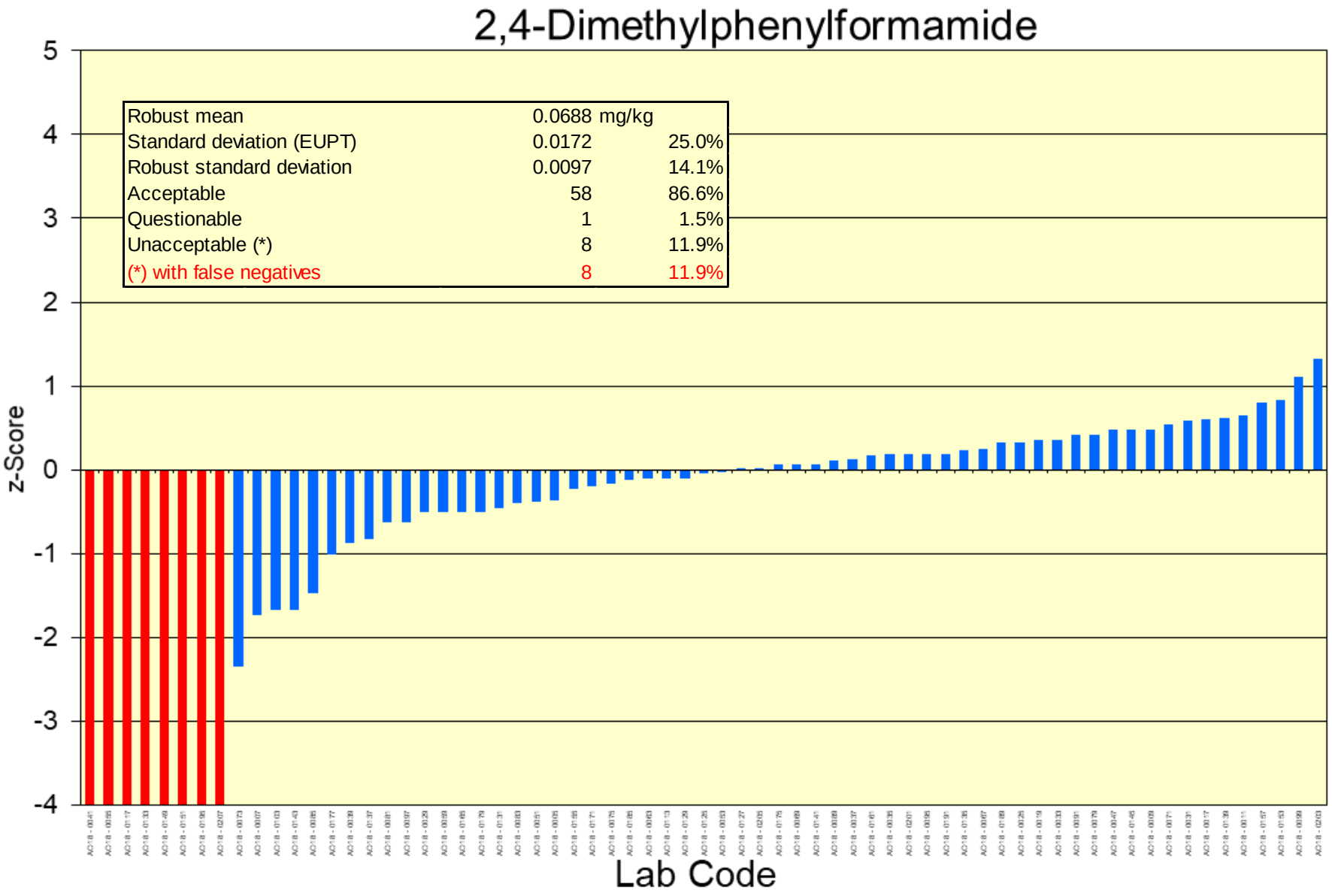
Robust mean	0.0930 mg/kg	
Standard deviation (EUPT)	0.0233	25.0%
Robust standard deviation	0.0214	23.0%
Acceptable	87	91.6%
Questionable	6	6.3%
Unacceptable (*)	2	2.1%
(*) with false negatives	1	1.1%

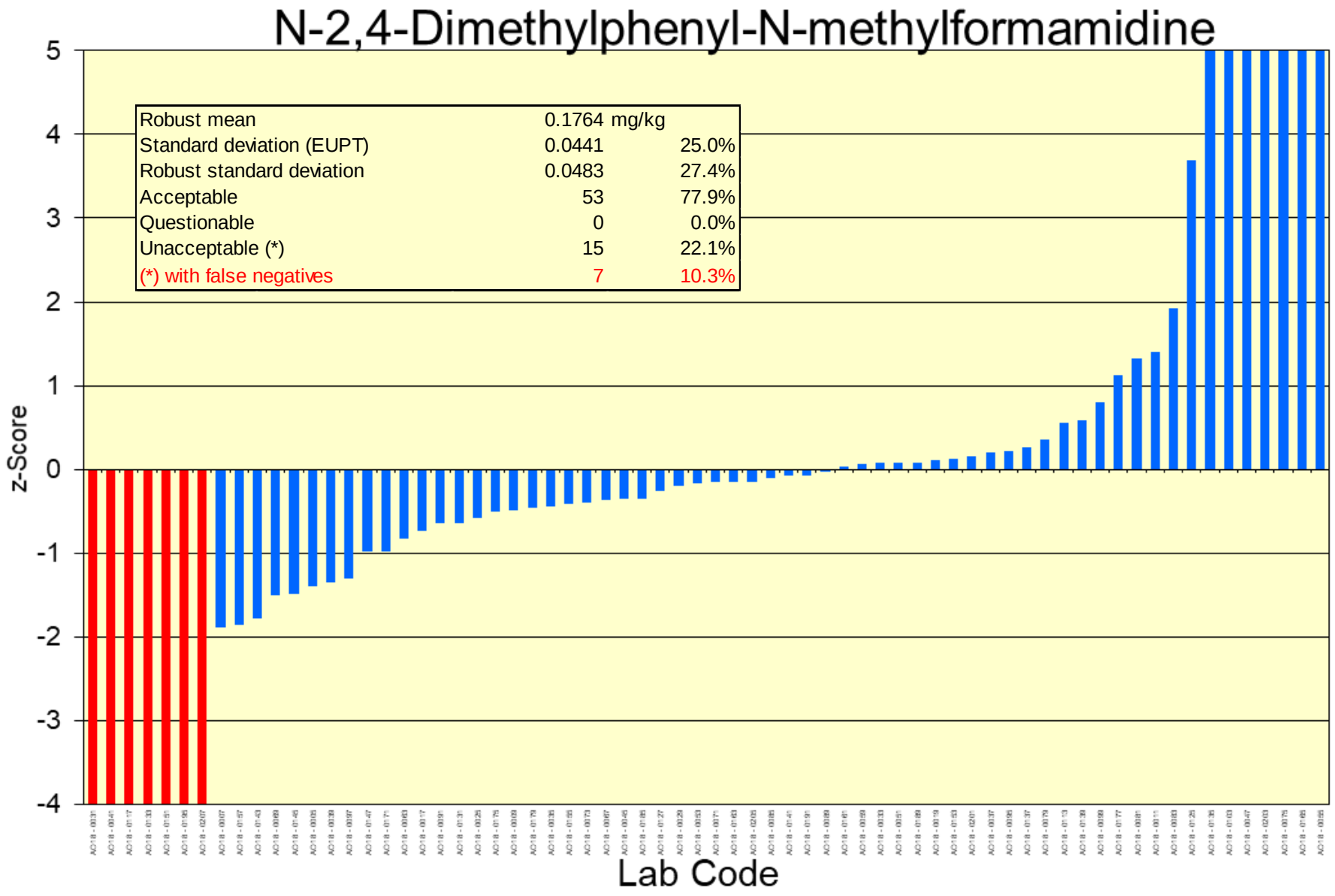



Phosalone
Robust mean 0.0900 mg/kg
Standard deviation (EUPT) 0.0225 25.0%
Robust standard deviation 0.0137 15.2%
Acceptable 83 96.5%
Questionable 0 0.0%
Unacceptable (*) 3 3.5%
(*) with false negatives 2 2.3%
z-Score
5
4
3
2
1
0
-1
-2
-3
-4
Lab Code


Pyraclostrobin
Robust mean 0.0857 mg/kg
Standard deviation (EUPT) 0.0214 25.0%
Robust standard deviation 0.0103 12.1%
Acceptable 78 94.0%
Questionable 0 0.0%
Unacceptable (*) 5 6.0%
(*) with false negatives 3 3.6%
z-Score
5
4
3
2
1
0
-1
-2
-3
-4
Lab Code


Thiacloprid
Robust mean 0.1036 mg/kg
Standard deviation (EUPT) 0.0259 25.0%
Robust standard deviation 0.0119 11.5%
Acceptable 83 94.3%
Questionable 2 2.3%
Unacceptable (*) 3 3.4%
(*) with false negatives 1 1.1%
z-Score
5
4
3
2
1
0
-1
-2
-3
-4
Lab Code

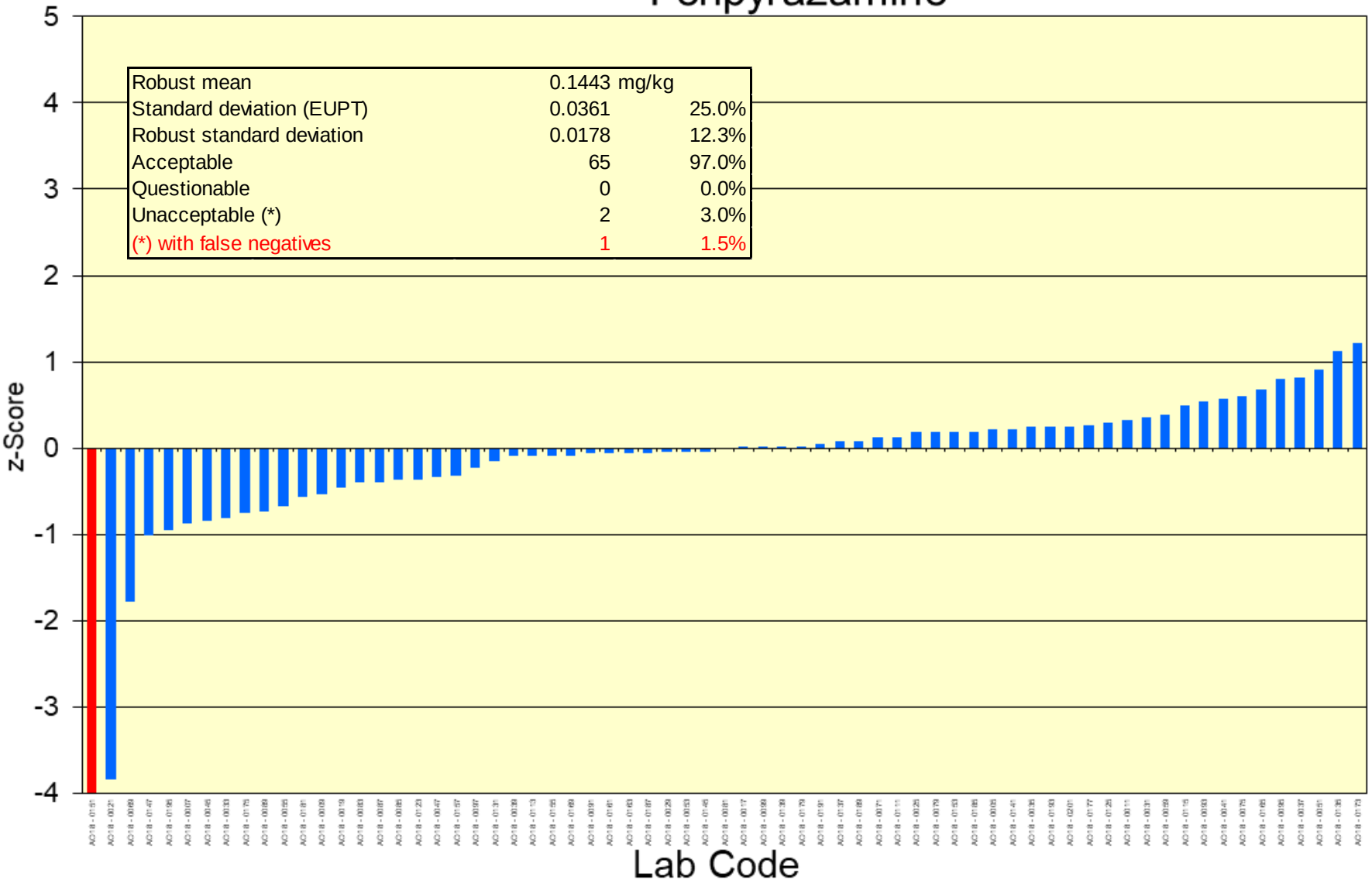

2,4-Dimethylphenylformamide
Robust mean 0.0688 mg/kg
Standard deviation (EUPT) 0.0172 25.0%
Robust standard deviation 0.0097 14.1%
Acceptable 58 86.6%
Questionable 1 1.5%
Unacceptable (*) 8 11.9%
(*) with false negatives 8 11.9%
z-Score
5
4
3
2
1
0
-1
-2
-3
-4
Lab Code

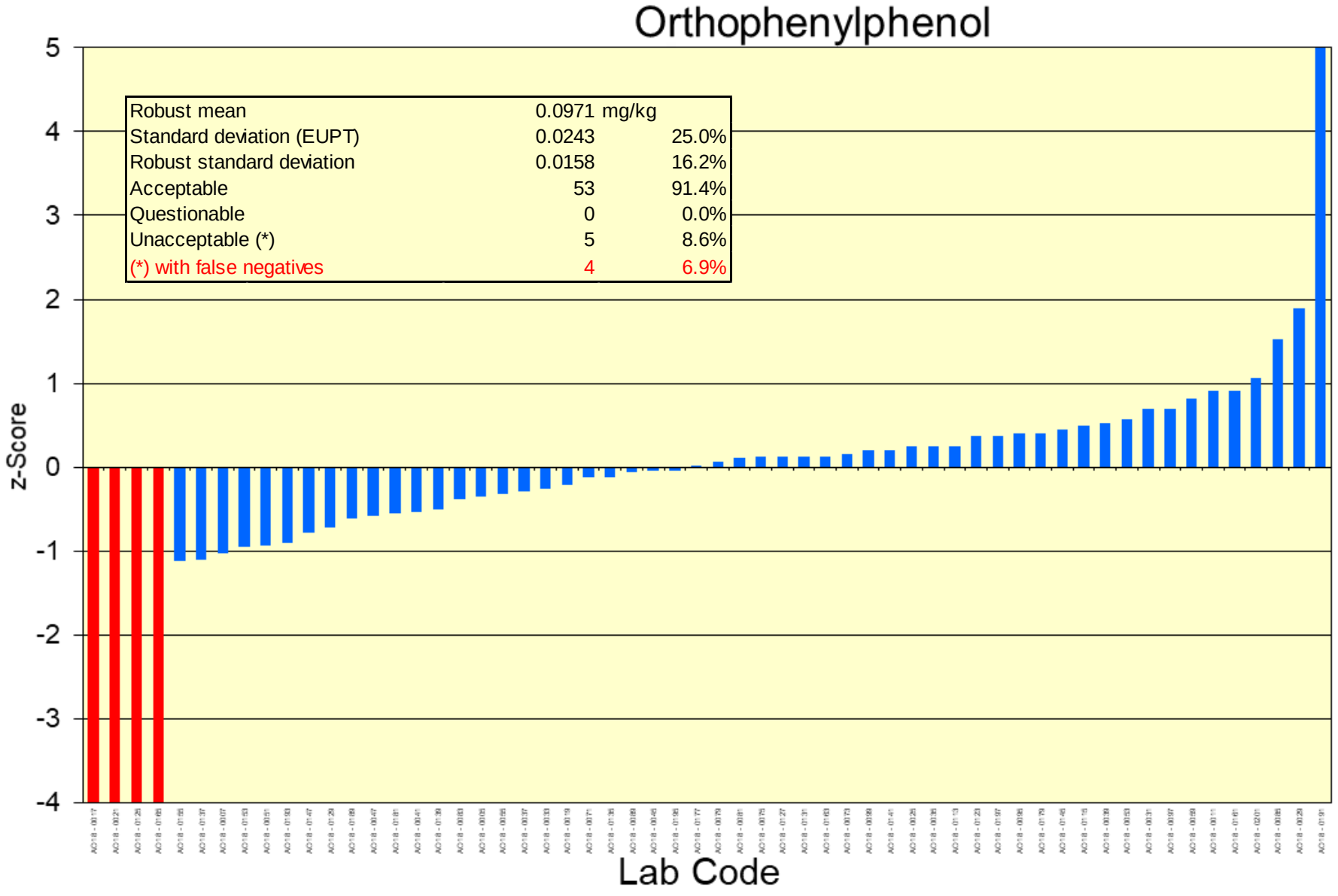

N-2,4-Dimethylphenyl-N-methylformamidine
Robust mean 0.1764 mg/kg
Standard deviation (EUPT) 0.0441 25.0%
Robust standard deviation 0.0483 27.4%
Acceptable 53 77.9%
Questionable 0 0.0%
Unacceptable (*) 15 22.1%
(*) with false negatives 7 10.3%
z-Score
5
4
3
2
1
0
-1
-2
-3
-4
Lab Code

voluntary pesticides

Fenpyrazamine

Robust mean	0.1443 mg/kg	
Standard deviation (EUPT)	0.0361	25.0%
Robust standard deviation	0.0178	12.3%
Acceptable	65	97.0%
Questionable	0	0.0%
Unacceptable (*)	2	3.0%
(*) with false negatives	1	1.5%

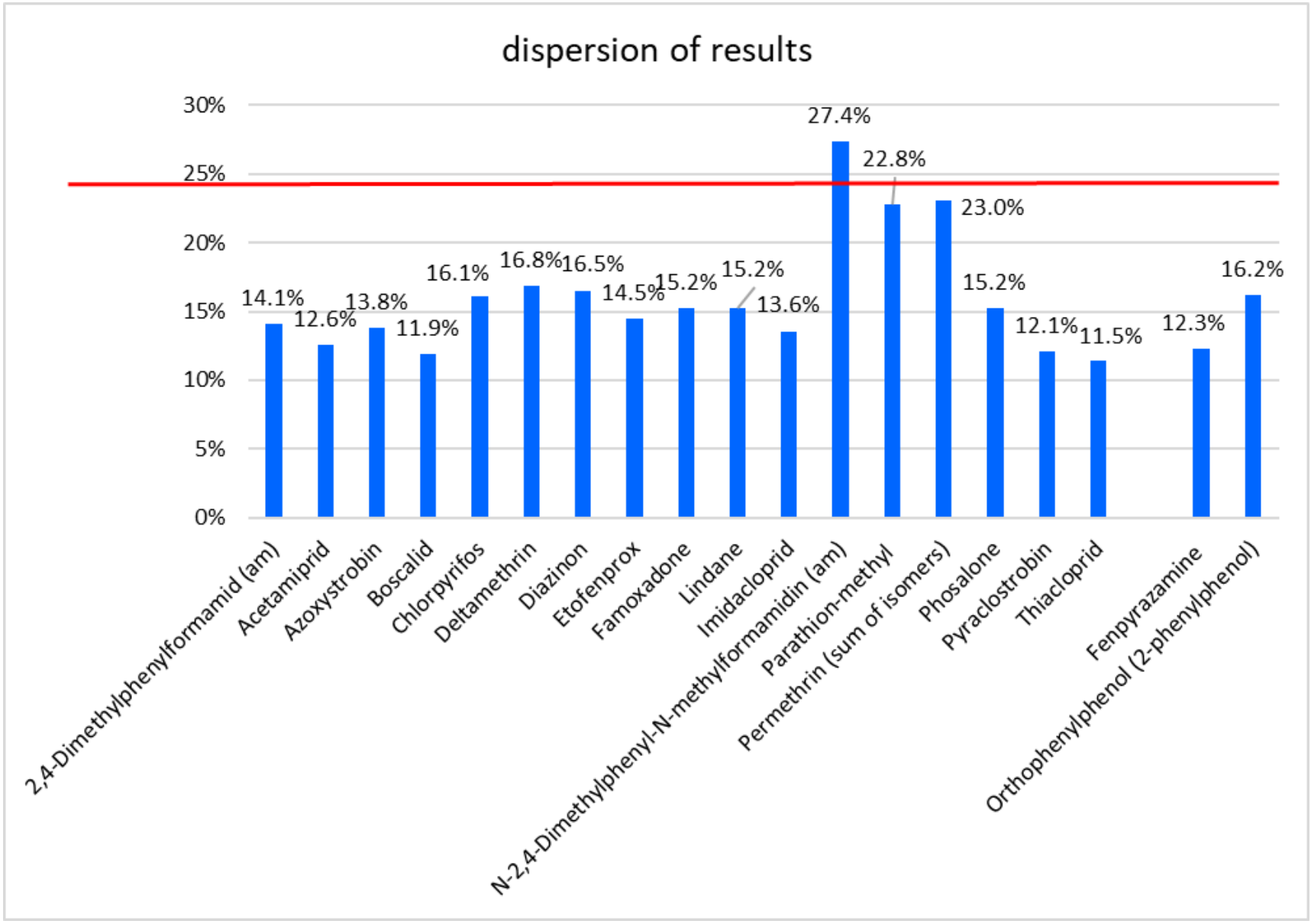


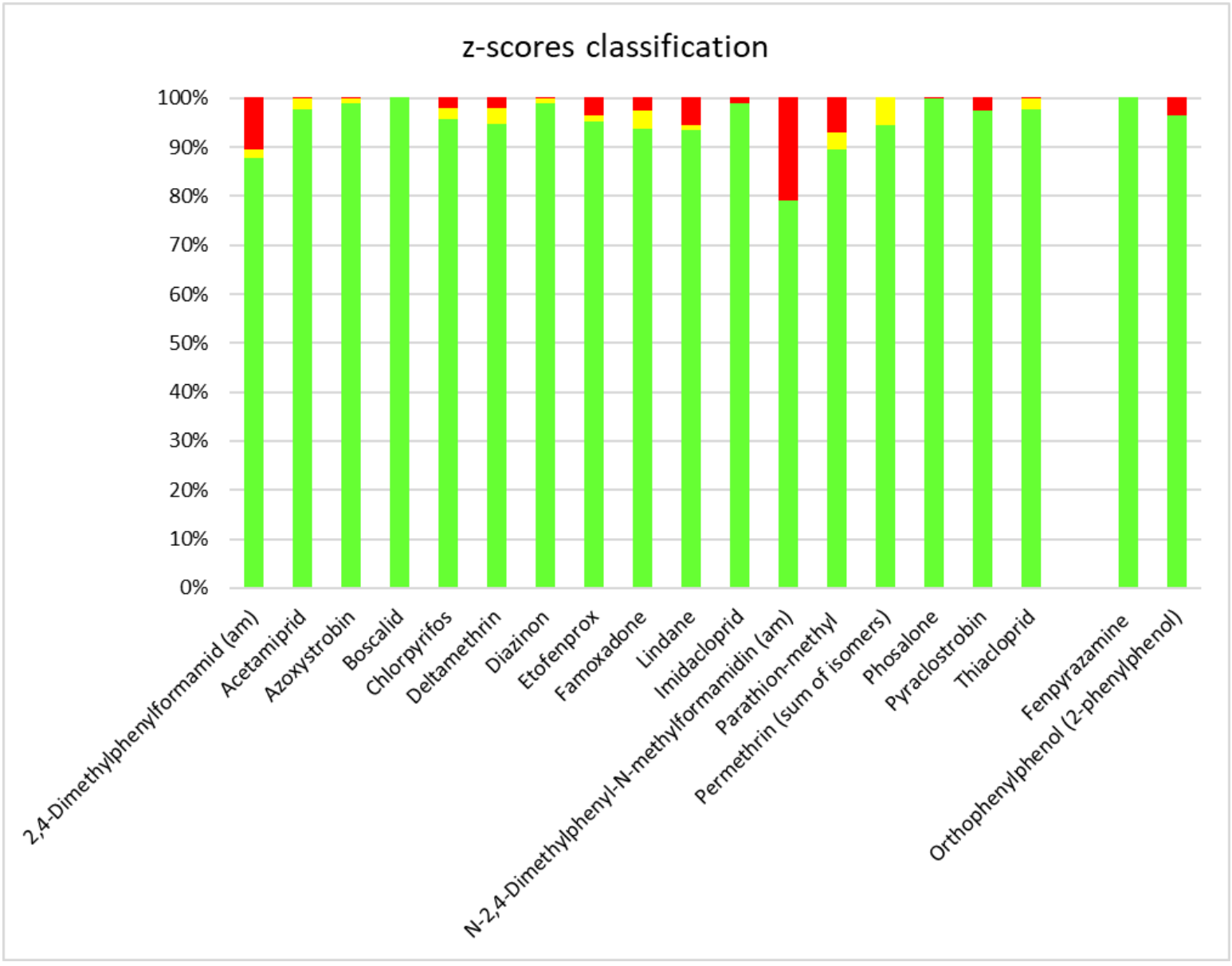

Orthophenylphenol
Robust mean 0.0971 mg/kg
Standard deviation (EUPT) 0.0243 25.0%
Robust standard deviation 0.0158 16.2%
Acceptable 53 91.4%
Questionable 0 0.0%
Unacceptable (*) 5 8.6%
(*) with false negatives 4 6.9%
z-Score
5
4
3
2
1
0
-1
-2
-3
-4
Lab Code

Results of robust statistic

Mandatory

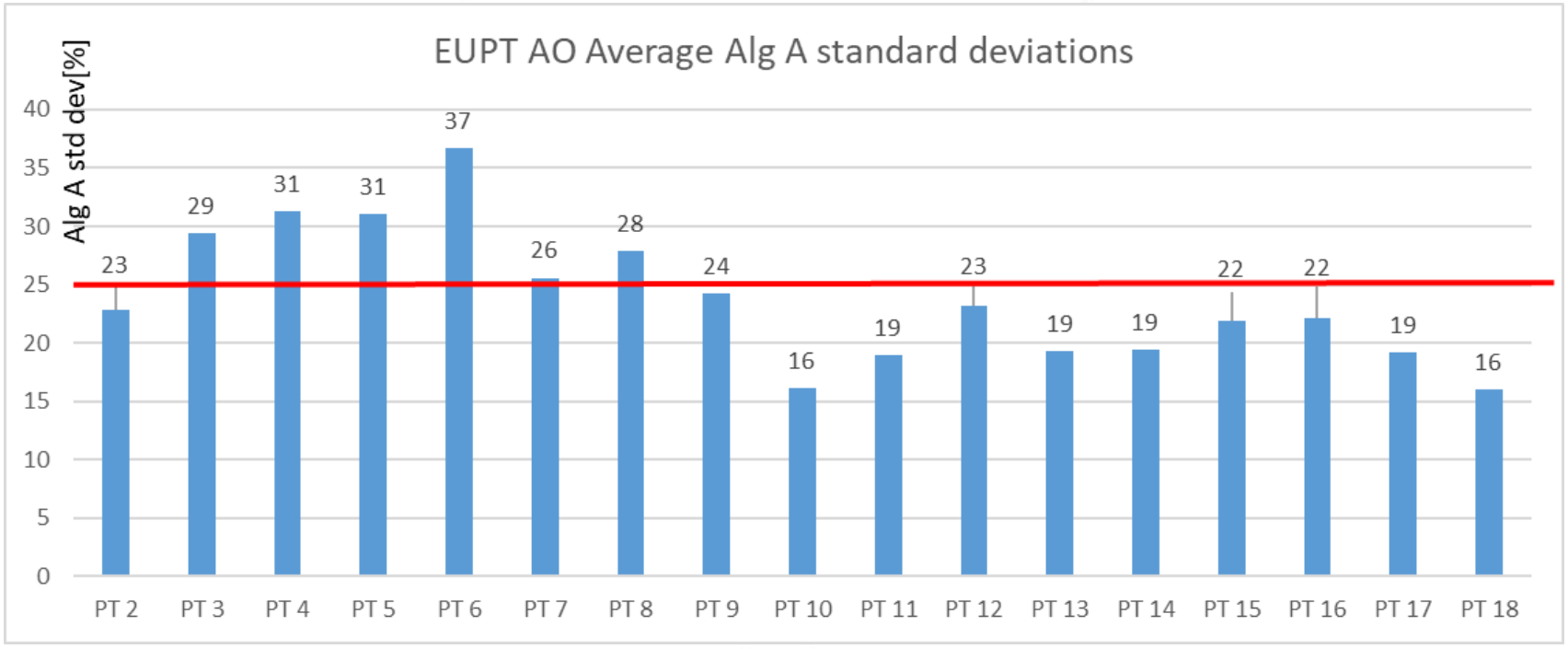
Analyte	Robust mean X* [mg/kg]	robust RSD (FFP-σ_{pt})	number of results	Acceptable	Questionable	Unacceptable	False Negatives	Not analysed	Spike value [mg/kg]	Ratio X* / spike	Acceptable	Questionable	Unacceptable
2,4-Dimethylphenylformamid (am)	0.0688	14.1%	67	58	1	8	8	35	0.075	92%	86.6%	1.5%	11.9%
Acetamiprid	0.1214	12.6%	89	84	2	3	2	13	0.125	97%	94.4%	2.2%	3.4%
Azoxystrobin	0.0483	13.8%	90	86	1	3	2	12	0.050	97%	95.6%	1.1%	3.3%
Boscalid	0.1273	11.9%	89	87	0	2	0	13	0.135	94%	97.8%	0.0%	2.2%
Chlorpyrifos	0.0873	16.1%	96	89	2	5	2	6	0.095	92%	92.7%	2.1%	5.2%
Deltamethrin	0.0546	16.8%	96	88	3	5	2	6	0.060	91%	91.7%	3.1%	5.2%
Diazinon	0.0501	16.5%	94	90	1	3	1	8	0.055	91%	95.7%	1.1%	3.2%
Etofenprox	0.0717	14.8%	87	81	1	5	4	15	0.075	96%	93.1%	1.1%	5.7%
Famoxadone	0.0568	15.2%	84	76	3	5	5	18	0.060	95%	90.5%	3.6%	6.0%
Lindane	0.0490	15.2%	94	85	1	8	5	8	0.054	91%	90.4%	1.1%	8.5%
Imidacloprid	0.0778	13.6%	89	85	0	4	1	13	0.080	97%	95.5%	0.0%	4.5%
N-2,4-Dimethylphenyl-N-methylform	0.1764	27.4%	68	53	0	15	7	34	0.185	95%	77.9%	0.0%	22.1%
Parathion-methyl	0.0502	22.8%	89	77	3	9	5	13	0.065	77%	86.5%	3.4%	10.1%
Permethrin (sum of isomers)	0.0930	23.0%	95	87	6	2	1	7	0.100	93%	91.6%	6.3%	2.1%
Phosalone	0.0900	15.2%	86	83	0	3	2	16	0.100	90%	96.5%	0.0%	3.5%
Pyraclostrobin	0.0857	12.1%	83	78	0	5	3	19	0.090	95%	94.0%	0.0%	6.0%
Thiacloprid	0.1036	11.5%	88	83	2	3	1	14	0.105	99%	94.3%	2.3%	3.4%
Fenpyrazamine	0.1443	12.3%	67	65	0	2	1	35	0.150	96%	97.0%	0.0%	3.0%
Orthophenylphenol (2-phenylphenol)	0.0971	16.2%	58	53	0	5	4	44	0.110	88%	91.4%	0.0%	8.6%


dispersion of results
30%
25%
20%
15%
10%
5%
0%
14.1%
12.6%
13.8%
11.9%
16.1%
16.8%
16.5%
14.5%
15.2%
15.2%
13.6%
27.4%
22.8%
23.0%
15.2%
12.1%
11.5%
12.3%
16.2%
2,4-Dimethylphenylformamid (am)
Acetamiprid
Azoxystrobin
Boscalid
Chlorpyrifos
Deltamethrin
Diazinon
Etofenprox
Famoxadone
Lindane
Imidacloprid
N-2,4-Dimethylpheny-N-methylformamidin (am)
Parathion-methyl
Permethrin (sum of isomers)
Phosalone
Pyraclostrobin
Thiacloprid
Fenpyrazamine
Orthophenylphenol (2-phenylphenol)


z-scores classification
100%
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
2,4-Dimethylphenylformamid (am)
Acetamiprid
Azoxystrobin
Boscalid
Chlorpyrifos
Deltamethrin
Diazinon
Etofenprox
Famoxadone
Lindane
Imidacloprid
N-2,4-Dimethylphenyl-N-methylformamidin (am)
Parathion-methyl
Permethrin (sum of isomers)
Phosalone
Pyraclostrobin
Thiacloprid
Fenpyrazamine
Orthophenylphenol (2-phenylphenol)

|z| (0 - 2)	2 < |z| < 3	3 ≤ |z|

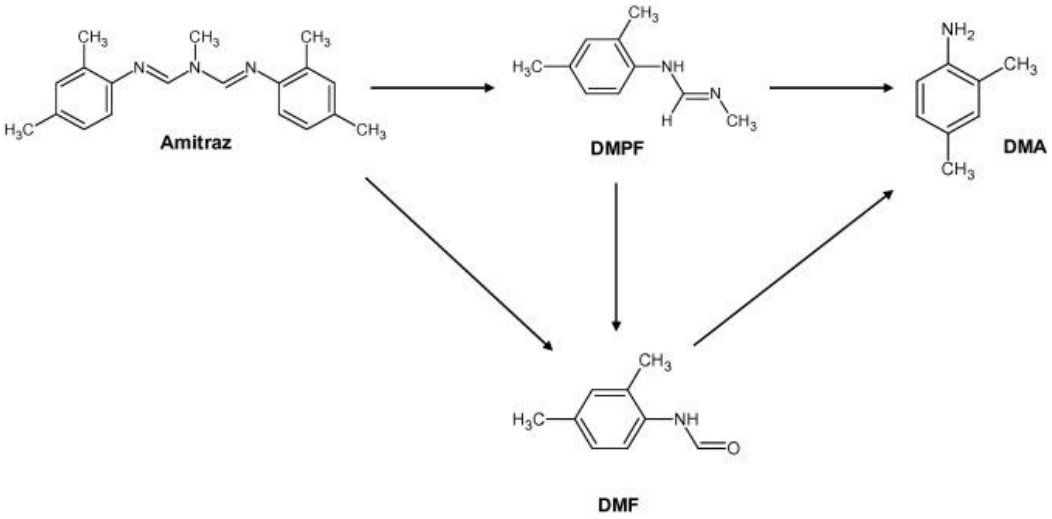
Alg A std deviation development



Question regarding the calculation of Amitraz

Residue Definition according to Reg. (EC) No. 396/2005:

- Amitraz (amitraz including the metabolites containing the 2,4 - dimethylaniline moiety expressed as amitraz)



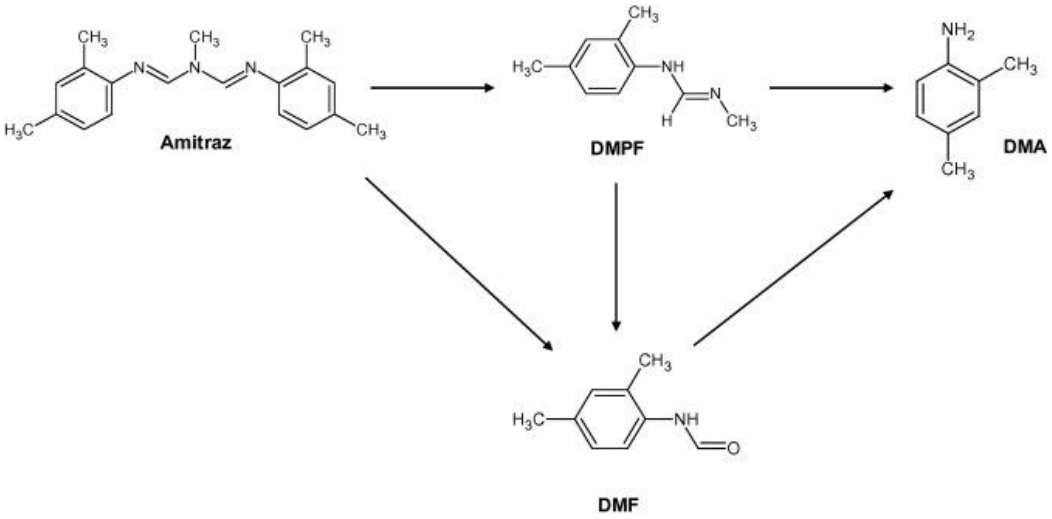
- ***Calculation of Amitraz:***

Hint: The formula below was used by the majority of the participants. It is not to be seen as a recommendation of the EURLs.

- ***c(Amitraz) = c(Amitraz) + c(DMPF*1.809) + c(DMF*1.967) (+ c(DMA))***
- ***c(Amitraz)= 0.319 + 0.135 = 0.454 mg/kg (using robust mean of PT)***

Residue Definition according to Reg. (EC) No. 396/2005:

- Amitraz (amitraz including the metabolites containing the 2,4 - dimethylaniline moiety expressed as amitraz)



- ***Calculation of Amitraz***
  (31 calculations provided, mainly using formula on page before)
- ***Results of participants between 0.21 – 1.35 mg/kg***
- ***0.394 mg/kg robust mean for calculated Amitraz results***
- ***with SD 0.116 mg/kg***

Poor performance feedback

Poor performance

- 63 cases of poor performance (individual z-score >3 or false positive/negative results)
  - 21% from EU/EFTA NRLs
  - 79 % from EU/EFTA OFLs
  - 40 false negatives (some might be due to scope selection (2 labs responsible for ~50%)
  - 6 false positives
- for 43 cases feedback was given
- most frequent reasons for underperformance
  - inappropriate / erroneous calibration approach → 11
  - Erroneous analytical standard → 5
  - Measurement problems → 3
  - Transcription error → 3

Thank You
for Your Attention

EURL EUROPEAN UNION REFERENCE LABORATORY