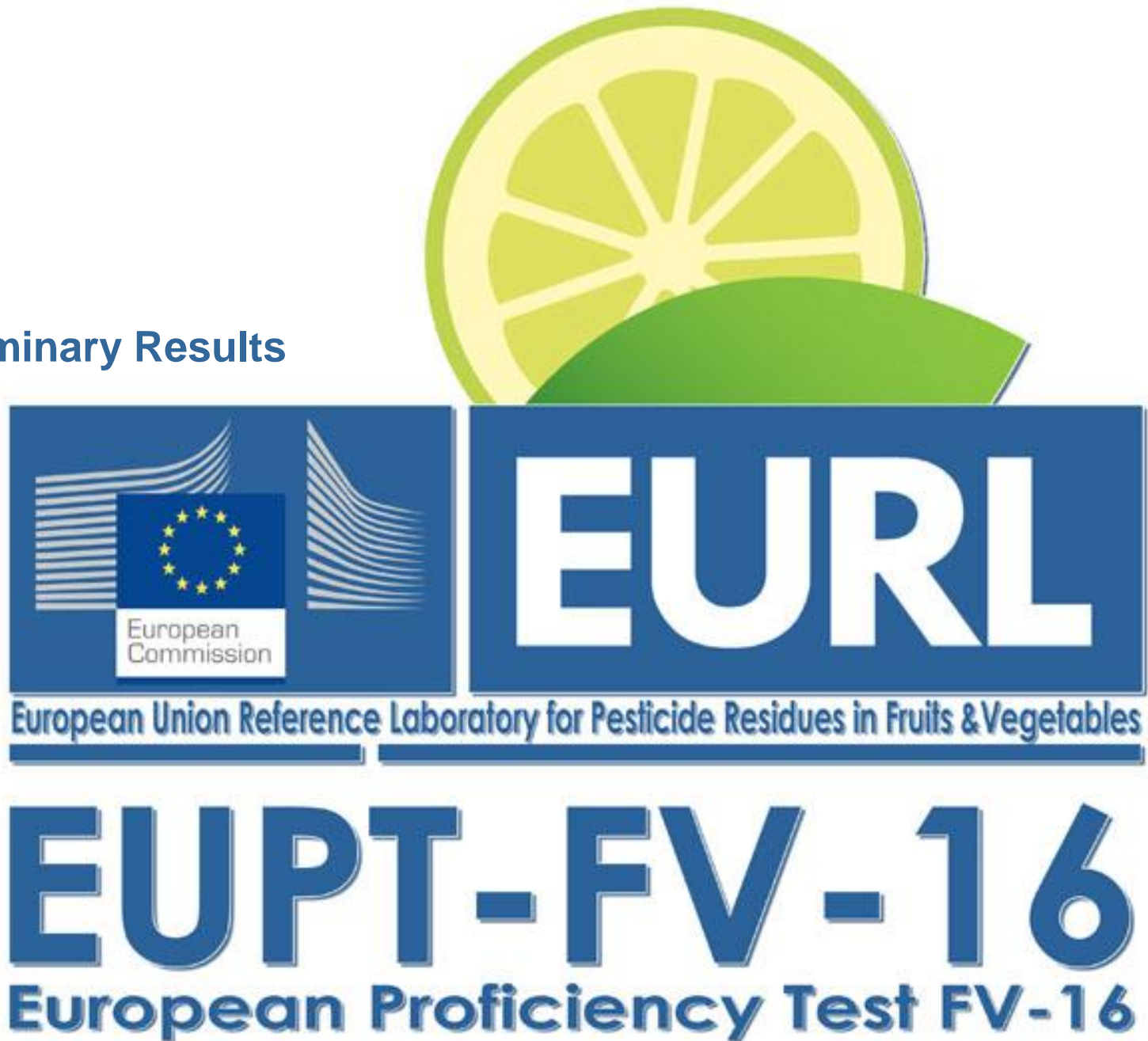


Preliminary Results



DG SANCO

Mrs. Almut Bitterhof

Mrs. Veerle Vanheusden

ORGANISING GROUP

Dr. Amadeo R. Fernández-Alba

Dr. Milagros Mezcua

Mr. Octavio Malato

Mrs. Carmen Ferrer

STATISTICAL GROUP

Dr. Carmelo Rodríguez

QUALITY CONTROL GROUP

Dr. Antonio Valverde

Mr. Stewart Reynolds

ADVISORY GROUP

Dr. Michelangelo Anastassiades

Mr. Richard Fussell

Dr. Miguel Gamón

Dr. Magnus Jezussek

Dr. André de Kok

Mr. Ralf Lippold

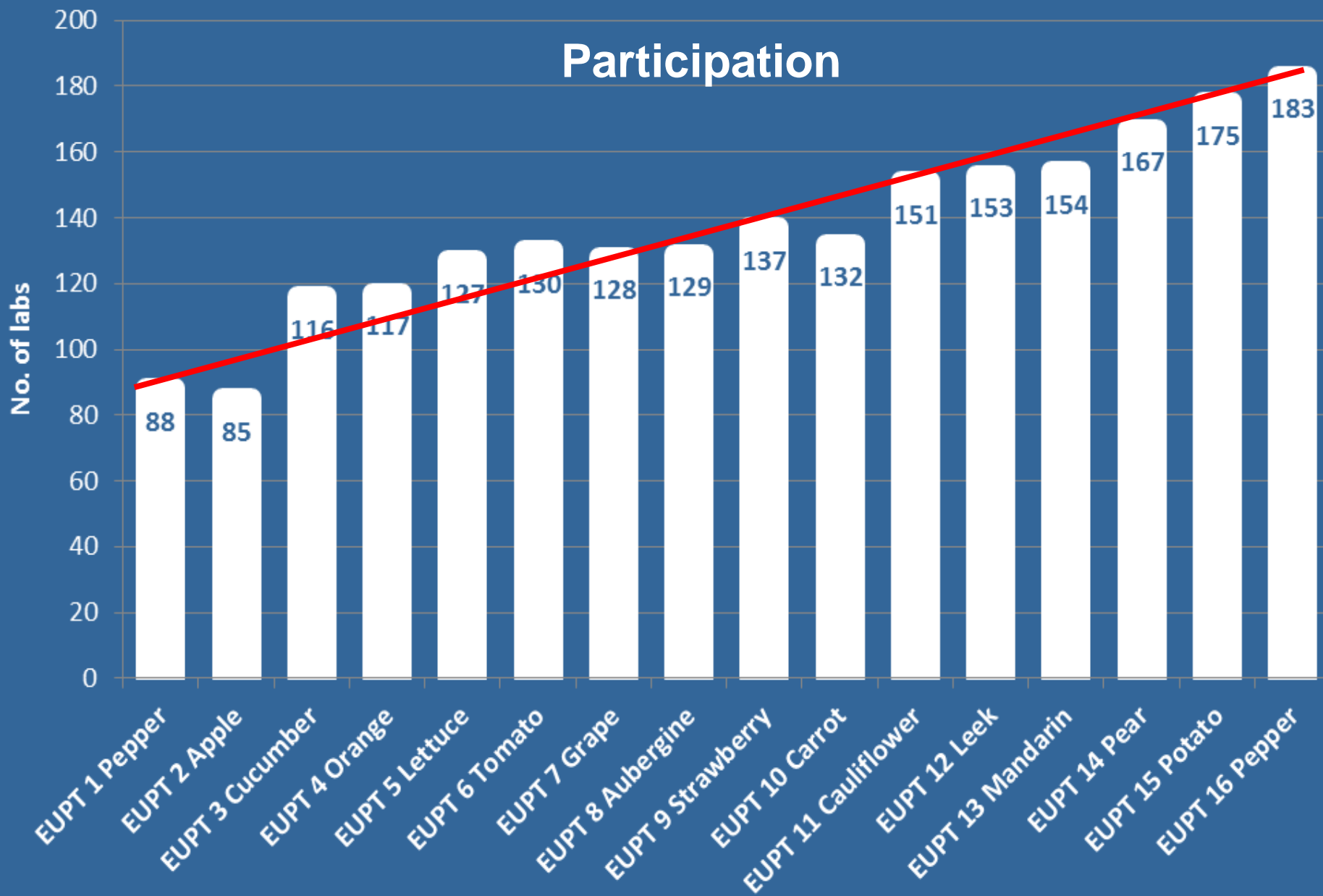
Dr. Sonja Masselter

Dr. Tuija Pihlström

Dr. Mette Erecius Poulsen

Dr. Darinka Stajnbaher

11th September 2014



Participation

Total No. of Labs = 183

EU/EFTA Labs = 169

Other countries Labs = 14

Total No. of Countries = 43

EU/EFTA countries = 31

Other countries = 12

(28 EU + 3 EFTA + 3 Candidate countries + 9 other countries)

11th September 2014

Country	No. of Labs	Country	No. of Labs	Country	No. of Labs
Austria	2	Hungary	4	Serbia	1
Belgium	3	Iceland	1	Singapore	1
Brazil	2	Ireland	1	Slovakia	2
Bulgaria	2	Israel	1	Slovenia	3
China	2	Italy	23	Spain	32
Croatia	7	Kenya	1	Sweden	2
Cyprus	1	Latvia	1	Switzerland	3
Czech Republic	3	Lithuania	1	Thailand	1
Denmark	2	Luxembourg	1	The Netherlands	4
Egypt	1	Norway	1	Turkey	1
Estonia	2	Peru	1	United Kingdom	4
Finland	2	Poland	10	Uruguay	1
France	8	Portugal	2	Total No. of Labs = 183 Total No. of Non-EU countries Labs = 14 Total No. of Countries = 43 EU countries, EFTA countries, 3 rd countries	
Germany	28	Romania	4		
Greece	10	Saudi Arabia	1		

11th September 2014

Pesticides Target List

175 pesticides

Clofentezine	Fenarimol	Lambda-Cyhalothrin	Phosmet oxon
3-hydroxy-carbofuran	Fenazaquin	Linuron	Phoxim
Acephate	Fenbuconazole	Lufenuron	Pirimicarb
Acetamiprid	Fenhexamid	Malaoxon	Pirimiphos-methyl
Acrinathrin	Fenitrothion	Malathion	Prochloraz
Aldicarb	Fenoxycarb	Mepanipyrim	Procymidone
Aldicarb Sulfone	Fenpropathrin	Metaflumizone*	Profenofos
Aldicarb Sulfoxide	Fenpropimorph	Metalaxyl and metalaxyl-M	Propargite
Amitraz	Fenthion	Metconazole	Propiconazole
Azinphos-methyl	Fenthion oxon	Methamidophos	Propyzamide
Azoxystrobin	Fenthion oxon sulfone	Methidathion	Prothioconazole
Benfuracarb	Fenthion oxon sulfoxide	Methiocarb	Prothiofos
Bifenthrin	Fenthion sulfone	Methiocarb sulfone	Pyraclostrobin
Bitertanol	Fenthion sulfoxide	Methiocarb sulfoxide	Pyridaben
Boscalid	Fipronil	Methomyl	Pyrimethanil
Bromopropylate	Fludioxonil	Methoxyfenozide	Pyriproxyfen
Bromuconazole	Flufenoxuron	Monocrotophos	Quinoxifen
Bupirimate	Fluopicolide*	Myclobutanil	Spinosad
Buprofezin	Fluquinconazole	Omethoate	Spirodiclofen
Cadusafos*	Flusilazole	Orthophenylphenol	Spiroxamine
Captan	Flutolanil*	Oxadixyl	Tau-Fluvalinate
Carbaryl	Flutriafol	Oxamyl	Tebuconazole
Carbendazim	Folpet	Oxydemeton-methyl	Tebufenozide
Carbofuran	Fosthiazate	Paclobutrazole	Tebufenpyrad
Carbosulfan	Hexaconazole	Paraoxon-methyl	Teflubenzuron
Chlorfenapyr	Hexythiazox	Parathion-ethyl	Tefluthrin
Chlorfenvinphos	Imazalil	Parathion-methyl	Tetraconazole
Chlorobenzilate*	Imidacloprid	Penconazole	Tetradifon
Chlorothalonil	Indoxacarb	Pencycuron	Thiabendazole
Chlorpropham	Iprodione	Pendimethalin	Thiacloprid
Chlorpyrifos	Iprovalicarb	Phenthoate	Thiamethoxam
Chlorpyrifos-methyl	Isofenphos-methyl	Phosalone	Thiodicarb
	Kresoxim-methyl	Phosmet	

***Not in the coordinated
multiannual control programme**

11th September 2014

Pesticides used for the treatment

Acetamiprid	Endosulfan beta
Acrinathrin	Fenamiphos
Buprofezin	Fenhexamid
Captan	Fludioxonil
Chlorothalonil	Lambda-Cyhalothrin
Chlorpyrifos	Methoxyfenozone
Cypermethrin	Pirimicarb
Cyprodinil	Pyridaben
Diazinon	Spinosad
Difenoconazole	Tetraconazole
Endosulfan alpha	Total: 21

ALL INCLUDED IN MACP

COMMISSION IMPLEMENTING REGULATION (EU)
No 788/2012 of 31 August 2012

concerning a coordinated multiannual control programme of the Union for 2013, 2014 and 2015 to ensure compliance with maximum residue levels of pesticides and to assess the consumer exposure to pesticide residues in and on food of plant and animal origin .

CALENDAR

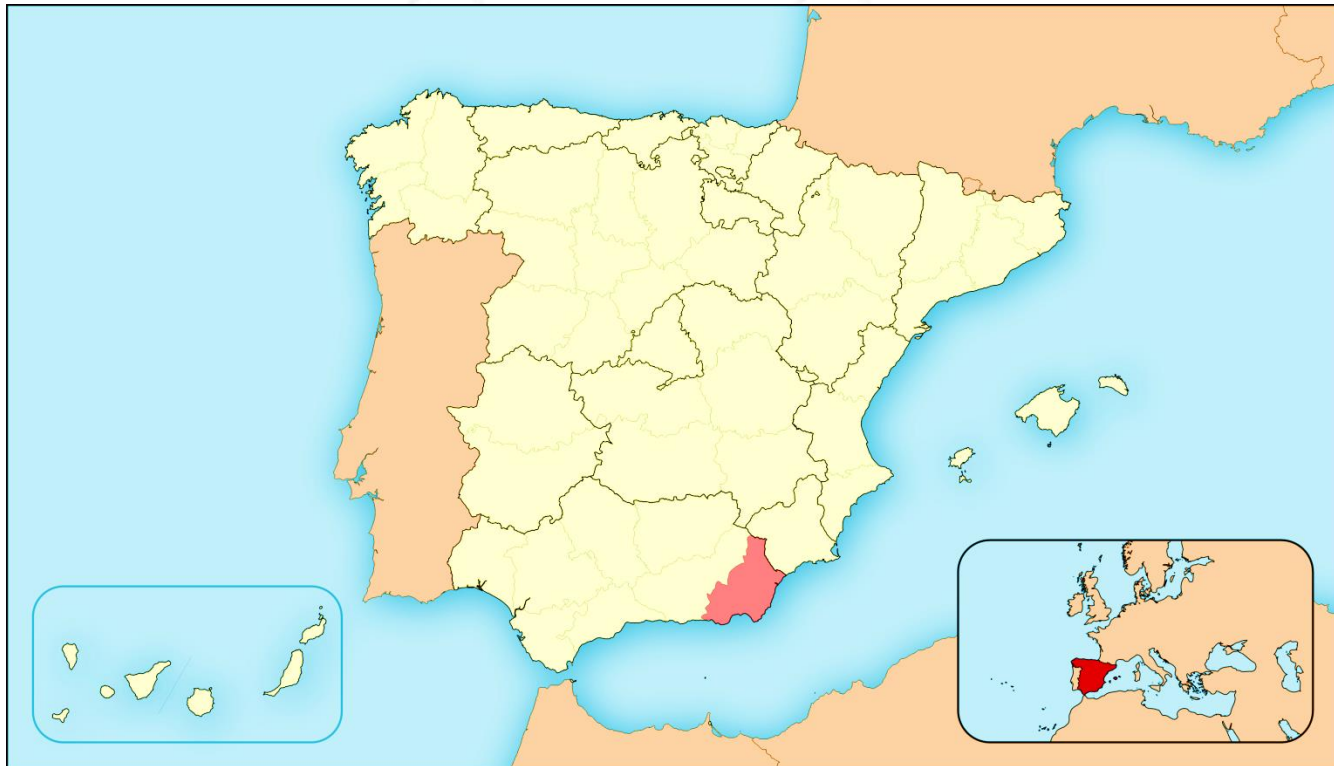
ACTIVITY	DATE
- Publishing the Target Pesticide List, Calendar and Matrix on the Web page.	15 th November 2013
- Receiving Application Form from invited laboratories.	23 rd Dec 2013 – 31 st Jan 2014
- Specific Protocol published on the Web site.	3 rd Feb 2014 at the latest
- Deadline for receiving Laboratory scope: Form 0	3 rd -14 th February 2014
- Sample distribution.	24 th February 2014
- Deadline for receiving sample acceptance: Form 1	28 th February 2014
- Deadline for receiving results: Forms 2, 3 and 4	17 th March 2014
- Filling in Form 5	24 th -28 th March 2014
- Preliminary Report: only results, no statistical treatment.	End of April 2014
- Final Report distributed to the Laboratories.	November 2014

EUPT-FV-16

European Proficiency Test FV-16



Organic peppers were grown in the EURL-FV greenhouse in Almería, Spain.





Before harvest, the peppers were treated with commercial formulations



Pesticide	Type	Commercial Product
Acetamiprid	Commercial Formulation	Epik 20SG (20%)
Acrinathrin	Commercial Formulation	Jokari (7.5%)
Buprofezin	Commercial Formulation	Geiser (25%)
Captan	Commercial Formulation	Merpan 50 (50%)
Chlorothalonil	Commercial Formulation	Daconil 50 SC (50%)
Chlorpyrifos	Commercial Formulation	Clorifos 48 EC (48%)
Cypermethrin	Commercial Formulation	Citron (10%)
Cyprodinil + Fludioxonil	Commercial Formulation	Switch (37.5%+25%)
Diazinon	Commercial Formulation	Fegazinogole (60%)
Difenoconazole	Commercial Formulation	Core (25%)
Endosulfan a y β	Commercial Formulation	Sulfanex 35 EC (35%)
Fenamiphos	Commercial Formulation	Nemacur 40LE (40%)
Fenhexamid	Commercial Formulation	Teldor (50%)
Lambda Cyhalothrin	Commercial Formulation	Karate Zeon (1.5%)
Methoxyfenozide	Commercial Formulation	Runner (24%)
Pirimicarb	Commercial Formulation	Aphox (50%)
Pyridaben	Commercial Formulation	Sanmmite 20% (20%)
Spinosad	Commercial Formulation	Spintor 480 SC (48%)
Tetraconazole	Commercial Formulation	Eminent (10%)



Homogeneity

- The homogeneity in the treated sample was studied using the 2006 Harmonised Protocol.



-The sampling standard deviation of all the pesticides must be lower than the critical value.

$$S_{sample}^2 < c$$

All the pesticides passed the homogeneity test

Stability

1st Analysis - prior to the sample shipment

2nd Analysis - after the deadline for reporting results

FEBRUARY 2014						
SUN	MON	TUE	WED	THU	FRI	SAT
						1
2	3	4	5	6	7	8
9	10	11	12	13	14	15
16	17	18	19	20	21	22
23	24	25	26	27	28	



MARCH 2014						
SUN	MON	TUE	WED	THU	FRI	SAT
30	31					1
2	3	4	5	6	7	8
9	10	11	12	13	14	15
16	17	18	19	20	21	22
23	24	25	26	27	28	29

All the pesticides passed the stability test

Stability

Additionally:

3rd Analysis - reproducing the delivery conditions that the samples experienced during 48 hours

FEBRUARY 2014						
SUN	MON	TUE	WED	THU	FRI	SAT
						1
2	3	4	5	6	7	8
9	10	11	12	13	14	15
16		18		20	21	22
23	24	25	26	27	28	

Preliminary Results



Acetamiprid **Endosulfan beta** Acrinathrin **Fenamiphos**
 Buprofezin Fenhexamid Captan Fludioxonil
 Chlorothalonil Lambda-Cyhalothrin Chlorpyrifos
 Methoxyfenozide Cypermethrin **Pirimicarb** Cyprodinil
 Pyridaben Diazinon Spinosad Difenoconazole
 Tetraconazole **Endosulfan alpha**



- **Endosulfan sulfate**
- **Fenamiphos sulfone and Fenamiphos sulfoxide**
- **Pirimicarb-desmethyl**
- **Propiconazole**
- **Tebuconazole**



Pesticides	Robust mean (mg/kg)
Endosulfan sulfate	0,011
Propiconazole	0,011
Tebuconazole	0,020
Desmethyl-pirimicarb	0,027

Pesticides	Robust mean (mg/kg)
Lambda-Cyhalothrin	0,078
Diazinon	0,084
Spinosad	0,098
Tetraconazole	0,105
Fenamiphos sulfone	0,109
Captan	0,132
Pyridaben	0,149
Methoxyfenozide	0,170
Fludioxonil	0,245
Acrinathrin	0,263
Buprofezin	0,469
Cypermethrin	0,528
Cyprodinil	0,588
Acetamiprid	0,636
Pirimicarb	0,730
Fenhexamid	0,855
Difenoconazole	0,945
Endosulfan beta	0,964
Endosulfan alpha	1,149
Chlorothalonil	2,179
Chlorpyrifos	3,667
Fenamiphos sulfoxide	6,625
Fenamiphos	7,297

Pesticides	Robust mean (mg/kg)
Endosulfan sulfate	0,011
Propiconazole	0,011
Tebuconazole	0,020
Desmethyl-pirimicarb	0,027

0.011-0.027 mg/kg



Pesticides	Robust mean (mg/kg)
Lambda-Cyhalothrin	0,078
Diazinon	0,084
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Fenamiphos	7,297

0.078-0.100 mg/kg

Pesticides	Robust mean (mg/kg)
Lambda-Cyhalothrin	0,078
Diazinon	0,084
Spinosad	0,098
Tetraconazole	0,105
Fenamiphos sulfone	0,109
Captan	0,132
Pyridaben	0,149
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Fenamiphos	7,297

0.078-0.100 mg/kg

0.100-0.500 mg/kg

Pesticides	Robust mean (mg/kg)
Lambda-Cyhalothrin	0,078
Diazinon	0,084
Spinosad	0,098
Tetraconazole	0,105
Fenamiphos sulfone	0,109
Captan	0,132
Pyridaben	0,149
Methoxyfenozide	0,170
Fludioxonil	0,245
Acrinathrin	0,263
Buprofezin	0,469
Cypermethrin	0,528
Cyprodinil	0,588
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Pirimicarb	0,730
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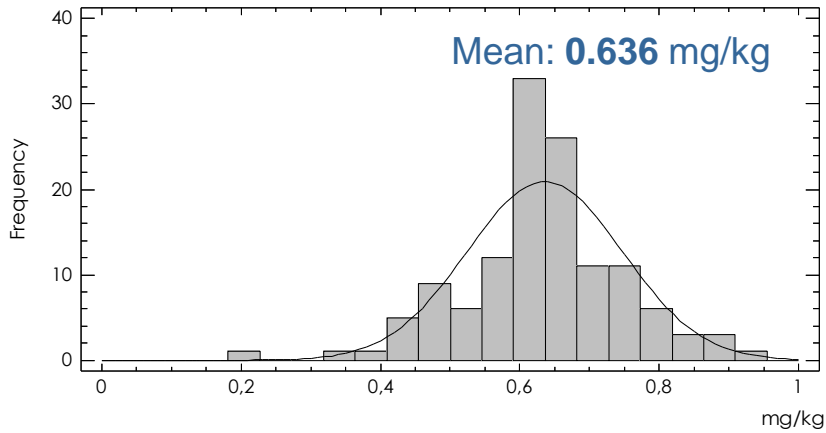
0.078-0.100 mg/kg

0.100-0.500 mg/kg

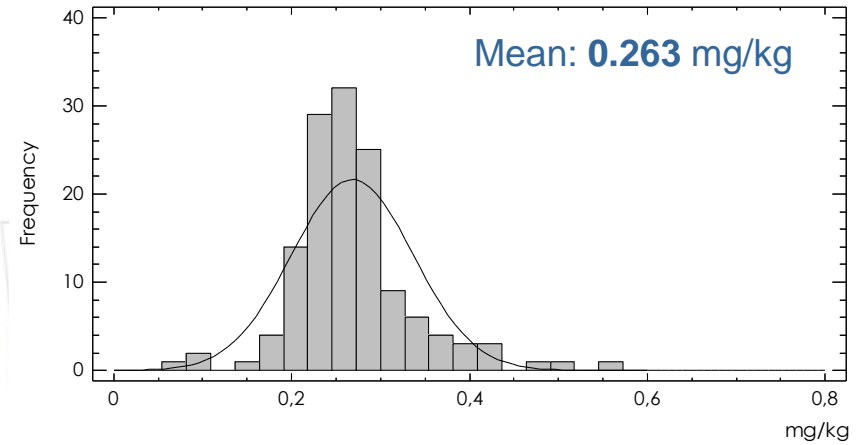
> 0.500 mg/kg

Distribution of data-Histograms

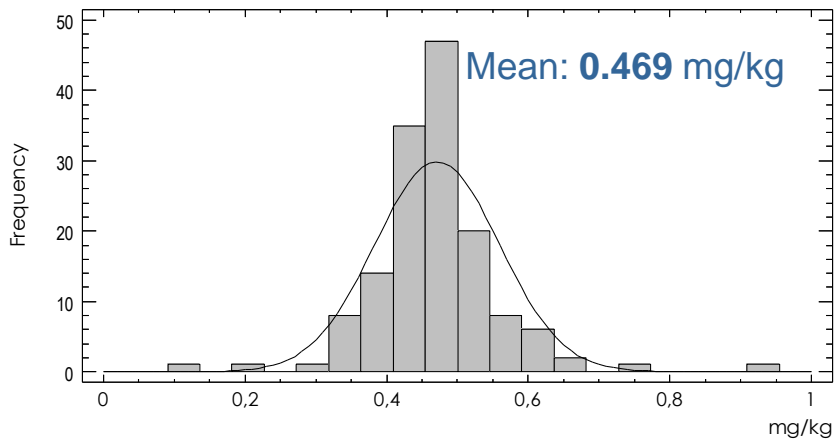
Acetamiprid



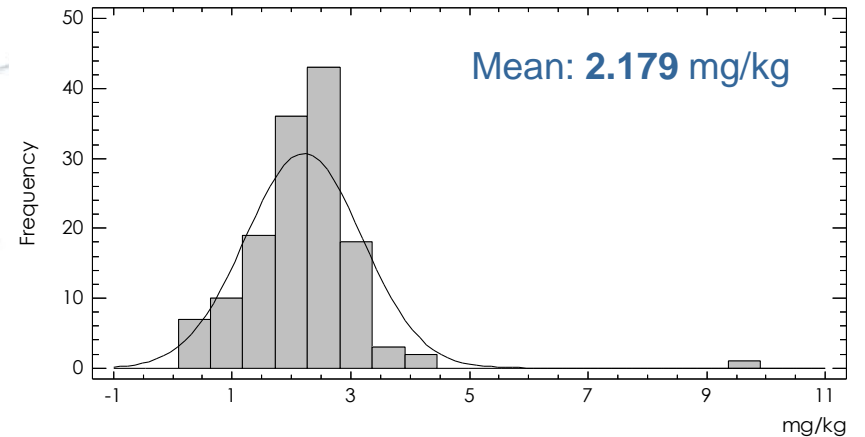
Acrinathrin



Buprofezin

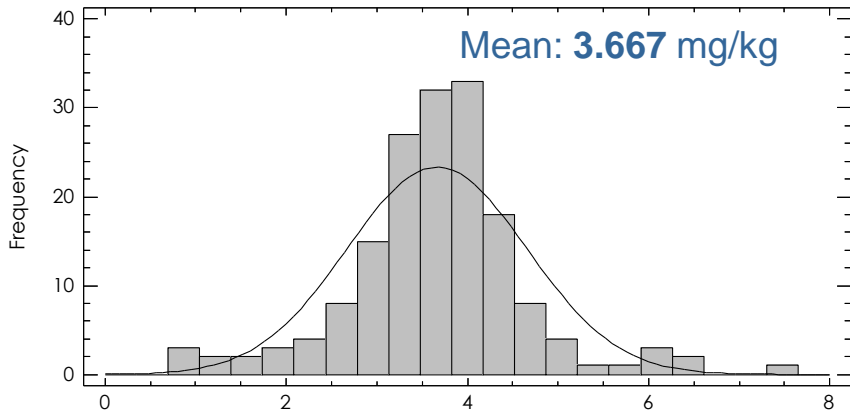


Chlorothalonil

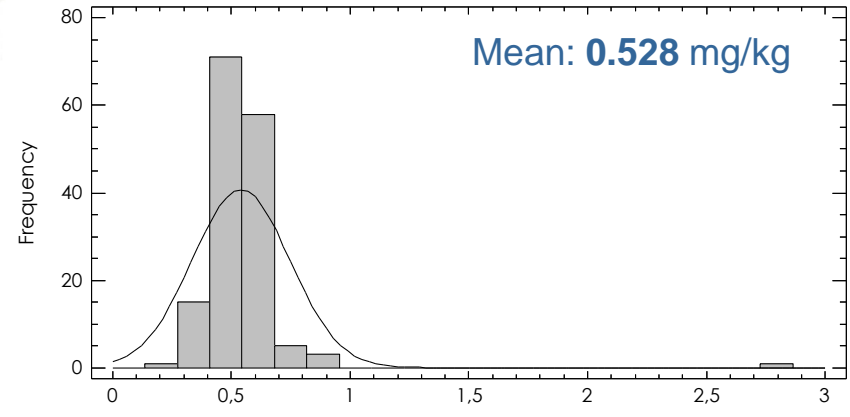


Distribution of data-Histograms

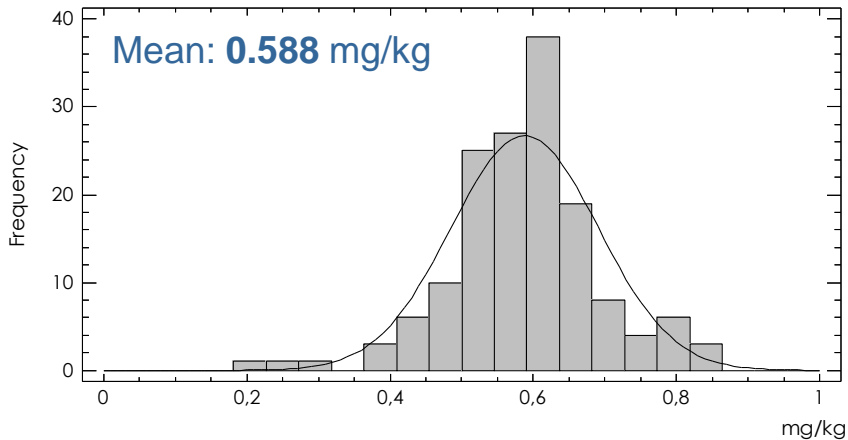
Chlorpyrifos



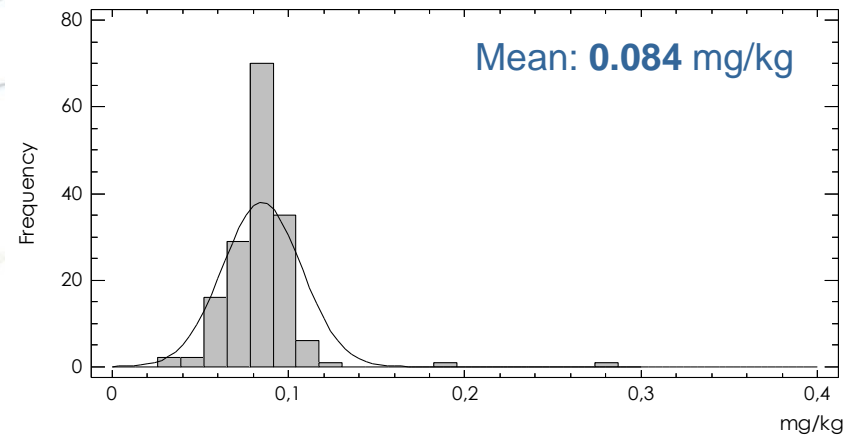
Cypermethrin



Cyprodinil

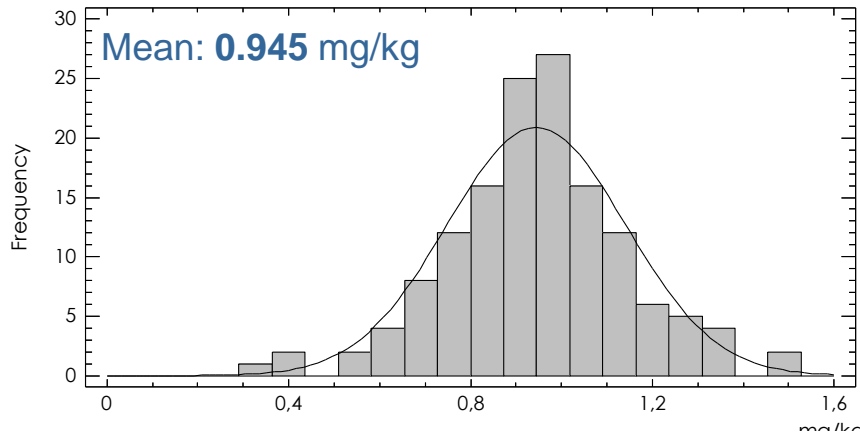


Diazinon

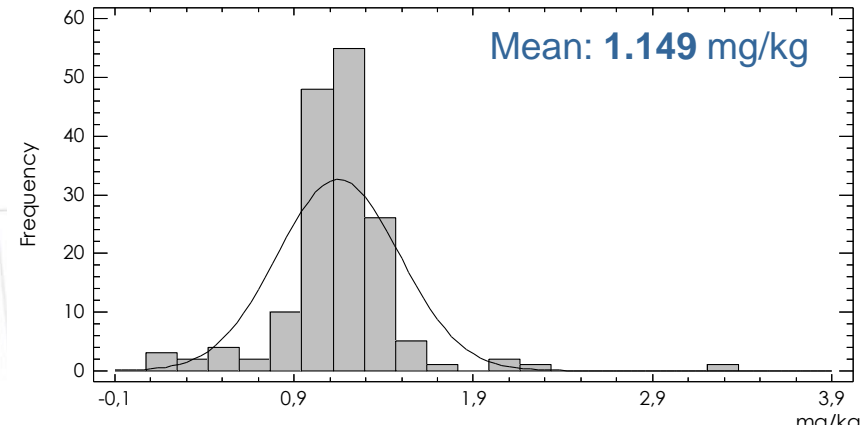


Distribution of data-Histograms

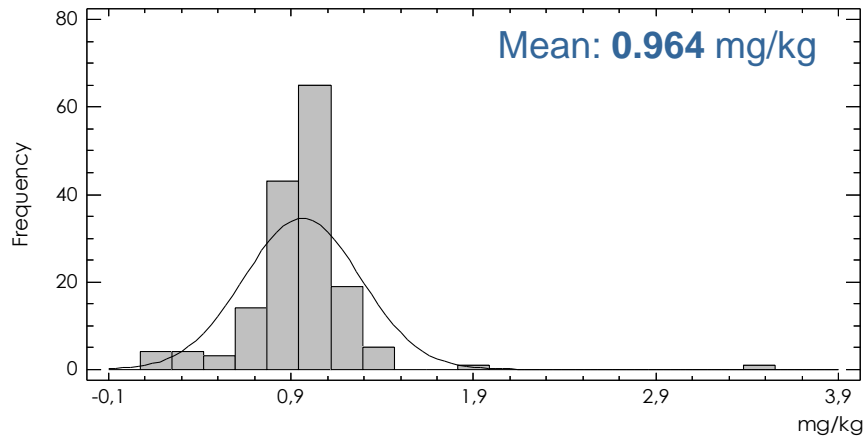
Difenoconazole



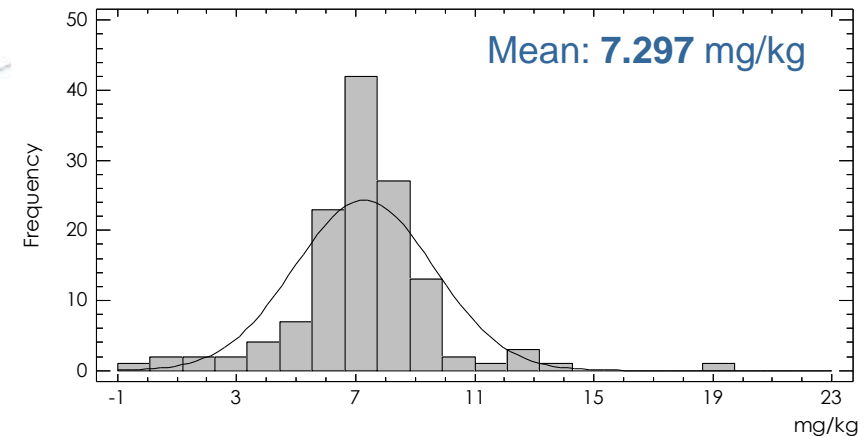
Endosulfan alpha



Endosulfan beta

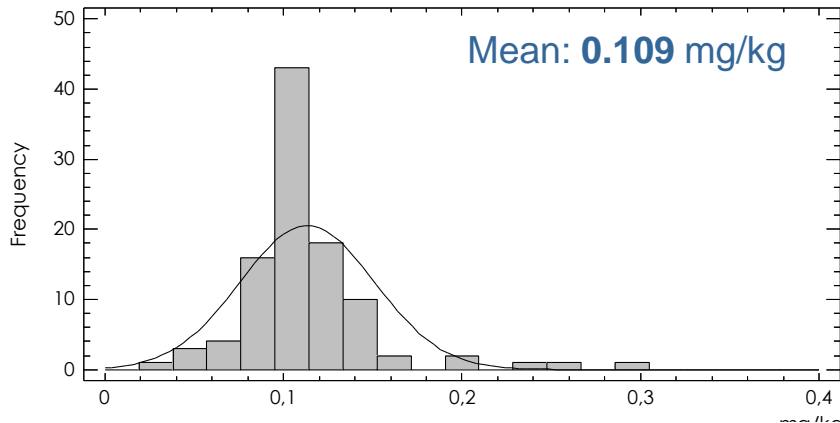


Fenamiphos

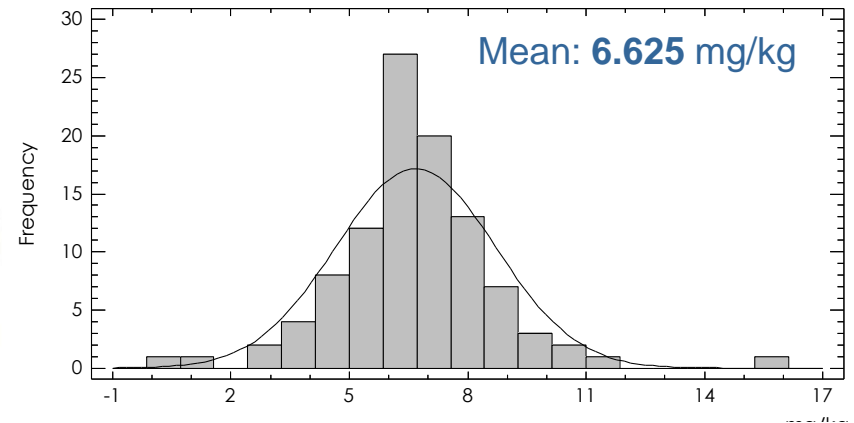


Distribution of data-Histograms

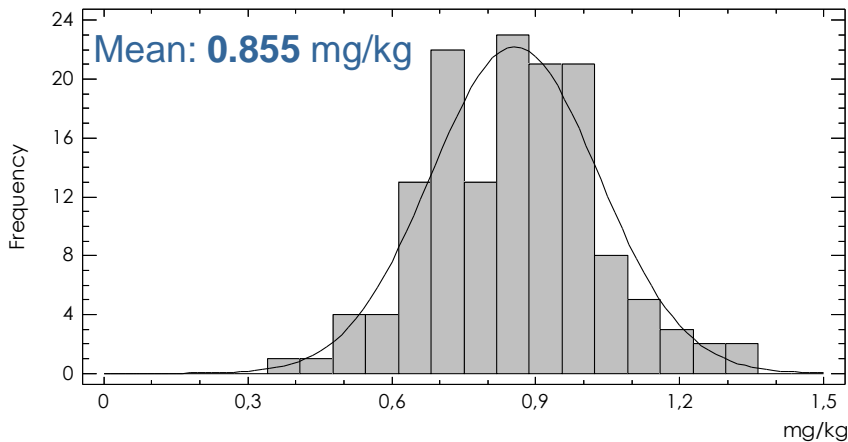
Fenamiphos sulfone



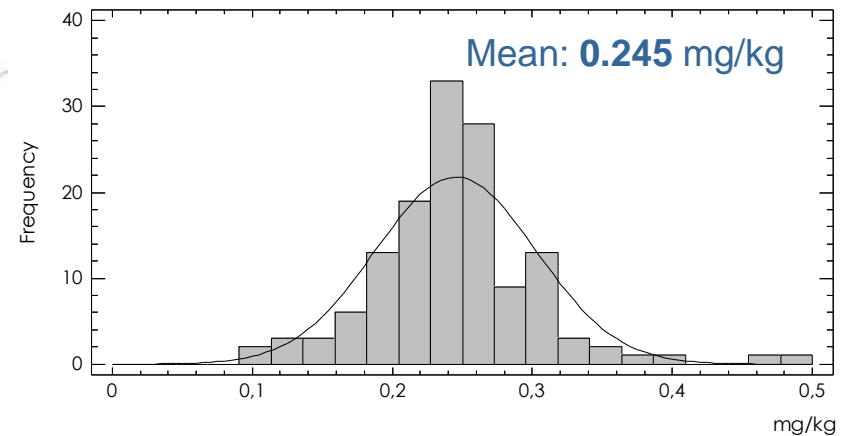
Fenamiphos sulfoxide



Fenhexamid

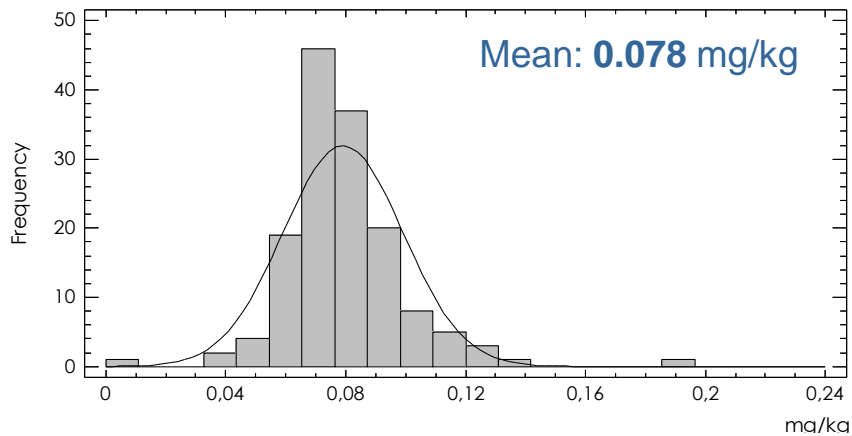


Fludioxinil

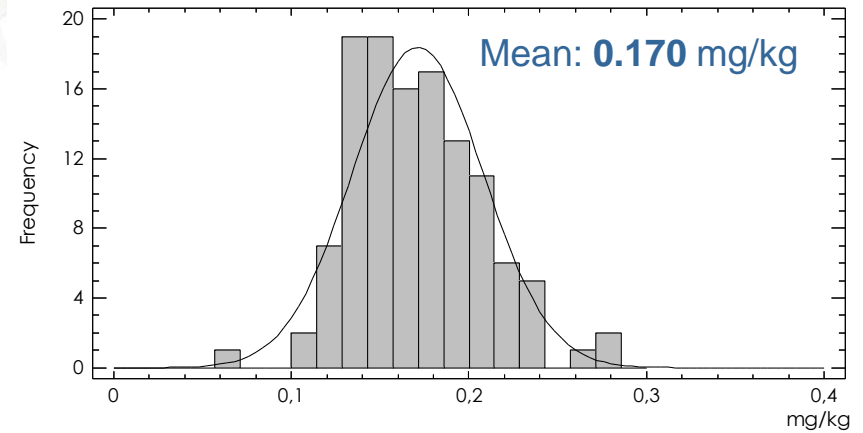


Distribution of data-Histograms

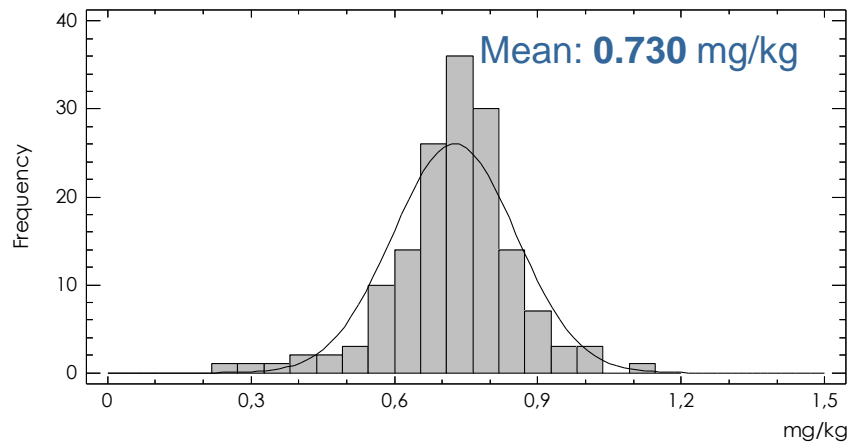
Lambda-cyhalothrin



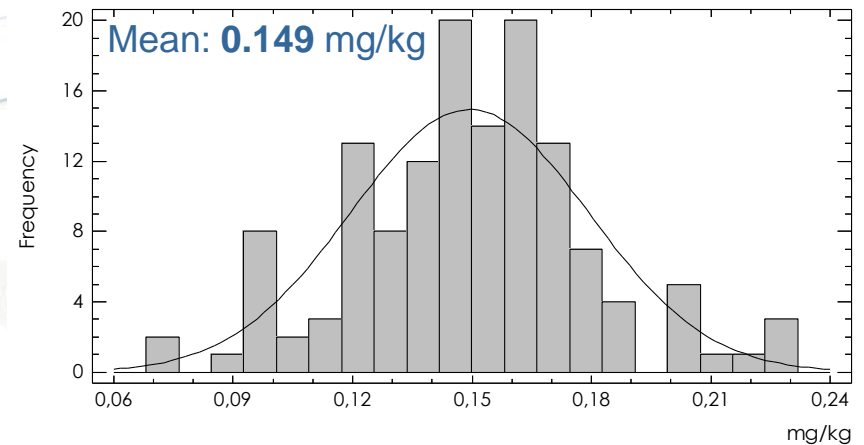
Methoxyfenozide



Pirimicarb

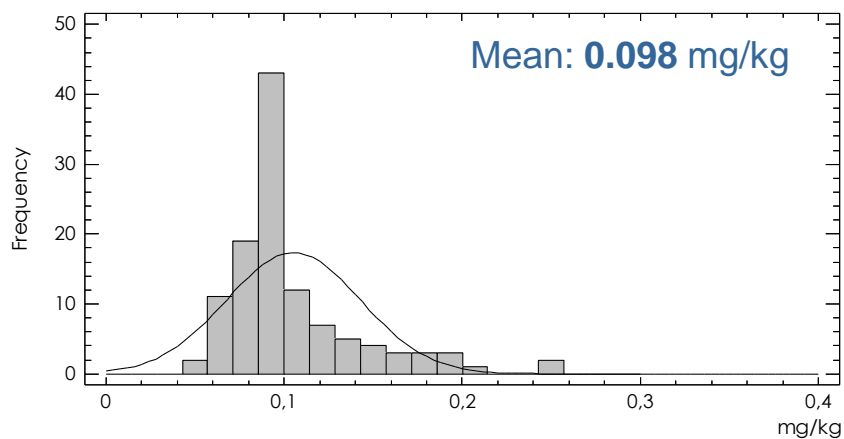


Pyridaben

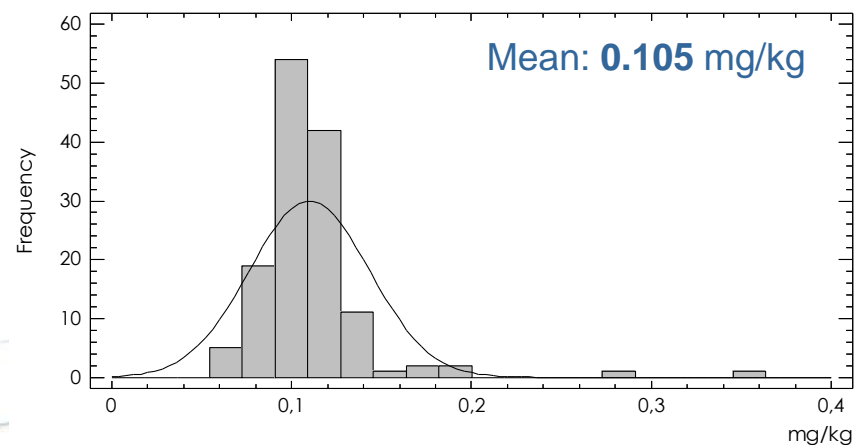


Distribution of data-Histograms

Spinosad (sum of spinosyn A and spinosyn D, expr. as spinosad)

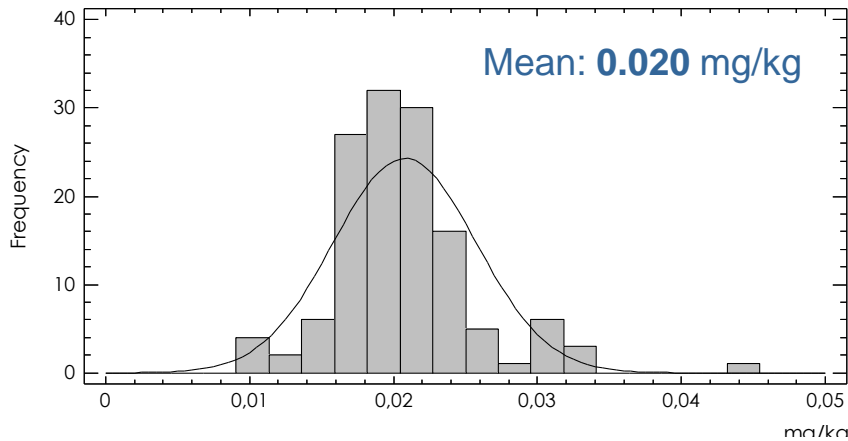


Tetraconazol

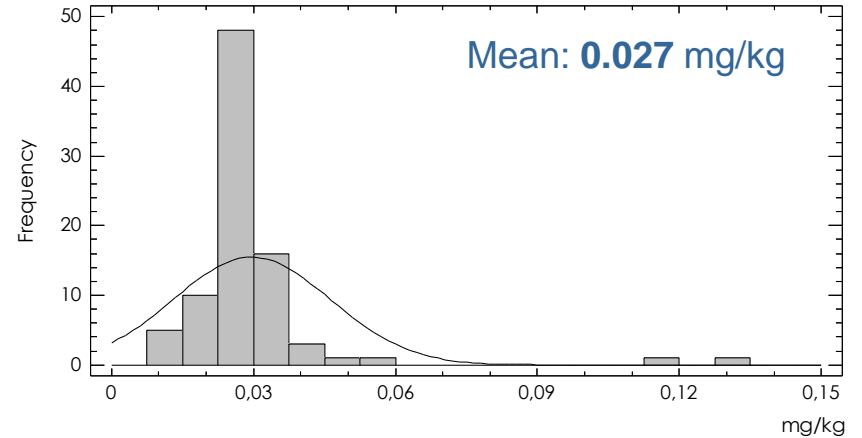


Distribution of data-Histograms (special cases)

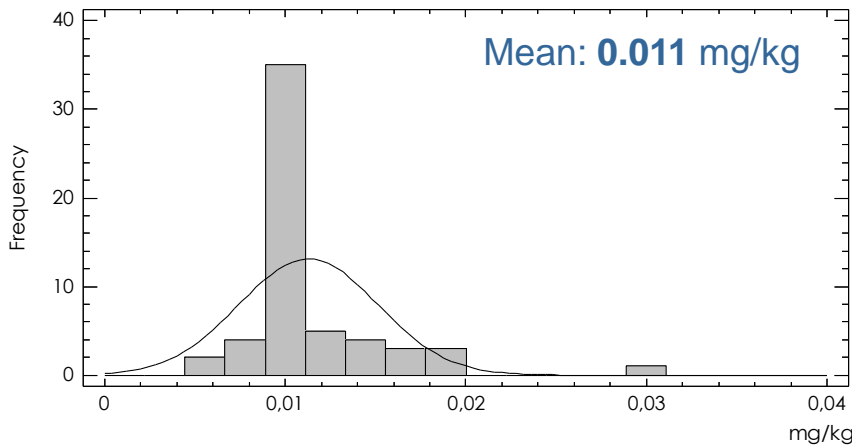
Tebuconazole



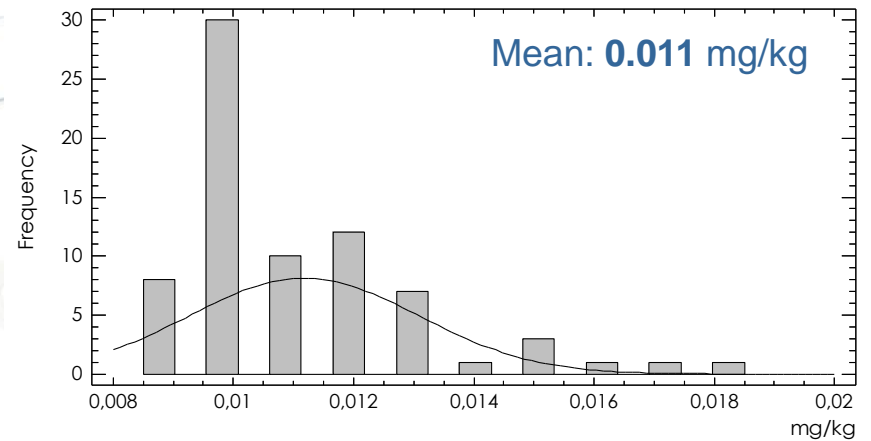
Desmethyl-pirimicarb



Endosulfan sulfate

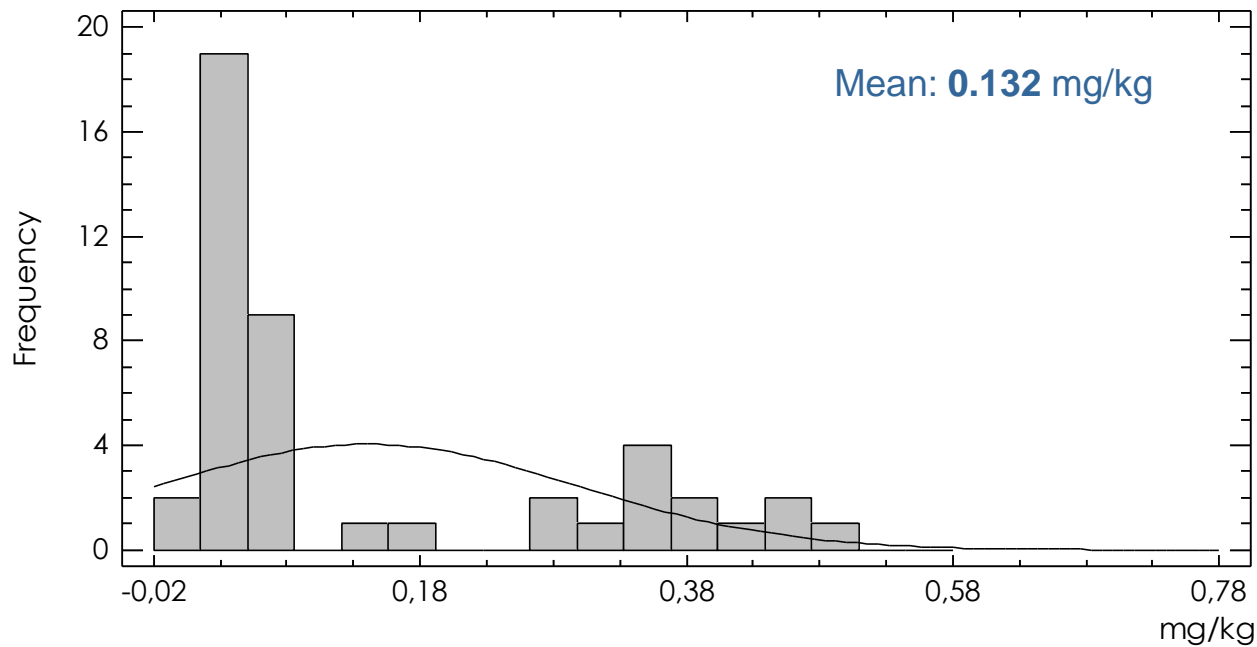


Propiconazole



Distribution of data-Histograms (special cases)

Captan



Captan

129 laboratories analysed captan

54 detected captan

75 did not detect captan

30 % GC-ECD

40% GC-ECD

70 % GC-MS or GC-MS/MS

60% GC-MS or GC-MS/MS

?



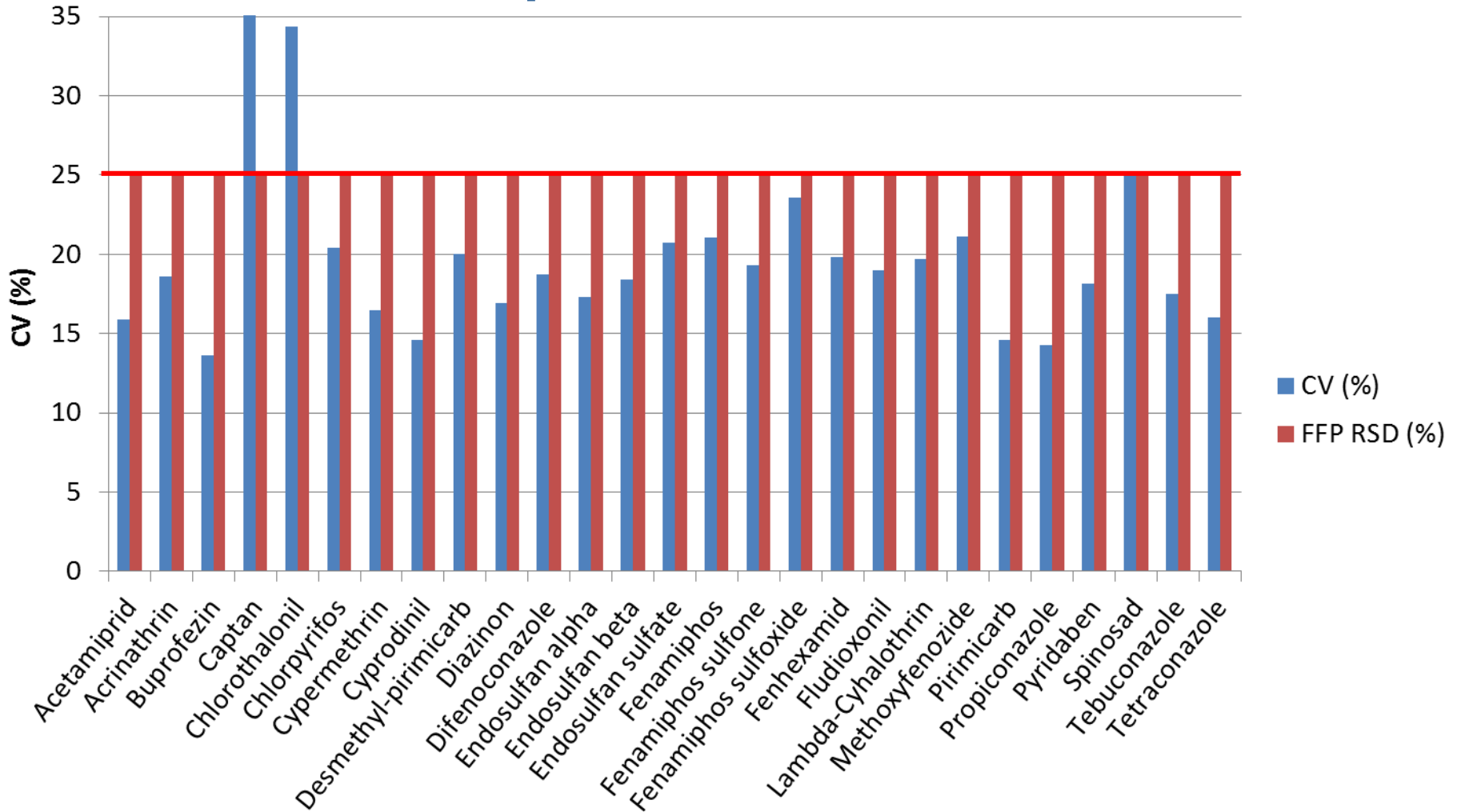
Captan expressed as captan

Captan + THPI expressed as captan

THPI expressed as captan

Pesticides	MRRL (mg/kg)	Robust mean (mg/kg)	CV (%)
Acetamiprid	0.01	0,636	15,89
Acrinathrin	0.01	0,263	18,62
Buprofezin	0.01	0,469	13,61
Captan	0.01	0,132	120,09
Chlorothalonil	0.01	2,179	34,39
Chlorpyrifos	0.01	3,667	20,40
Cypermethrin	0.01	0,528	16,45
Cyprodinil	0.01	0,588	14,63
Desmethyl-pirimicarb	0.01	0,027	20,02
Diazinon	0.01	0,084	16,92
Difenoconazole	0.01	0,945	18,71
Endosulfan alpha	0.01	1,149	17,30
Endosulfan beta	0.01	0,964	18,42
Endosulfan sulfate	0.01	0,011	20,71
Fenamiphos	0.01	7,297	21,03
Fenamiphos sulfone	0.01	0,109	19,33
Fenamiphos sulfoxide	0.01	6,625	23,57
Fenhexamid	0.01	0,855	19,84
Fludioxonil	0.01	0,245	18,96
Lambda-Cyhalothrin	0.01	0,078	19,72
Methoxyfenozide	0.01	0,170	21,15
Pirimicarb	0.01	0,730	14,57
Propiconazole	0.01	0,011	14,27
Pyridaben	0.01	0,149	18,17
Spinosad	0.01	0,098	25,18
Tebuconazole	0.01	0,020	17,50
Tetraconazole	0.01	0,105	16,02

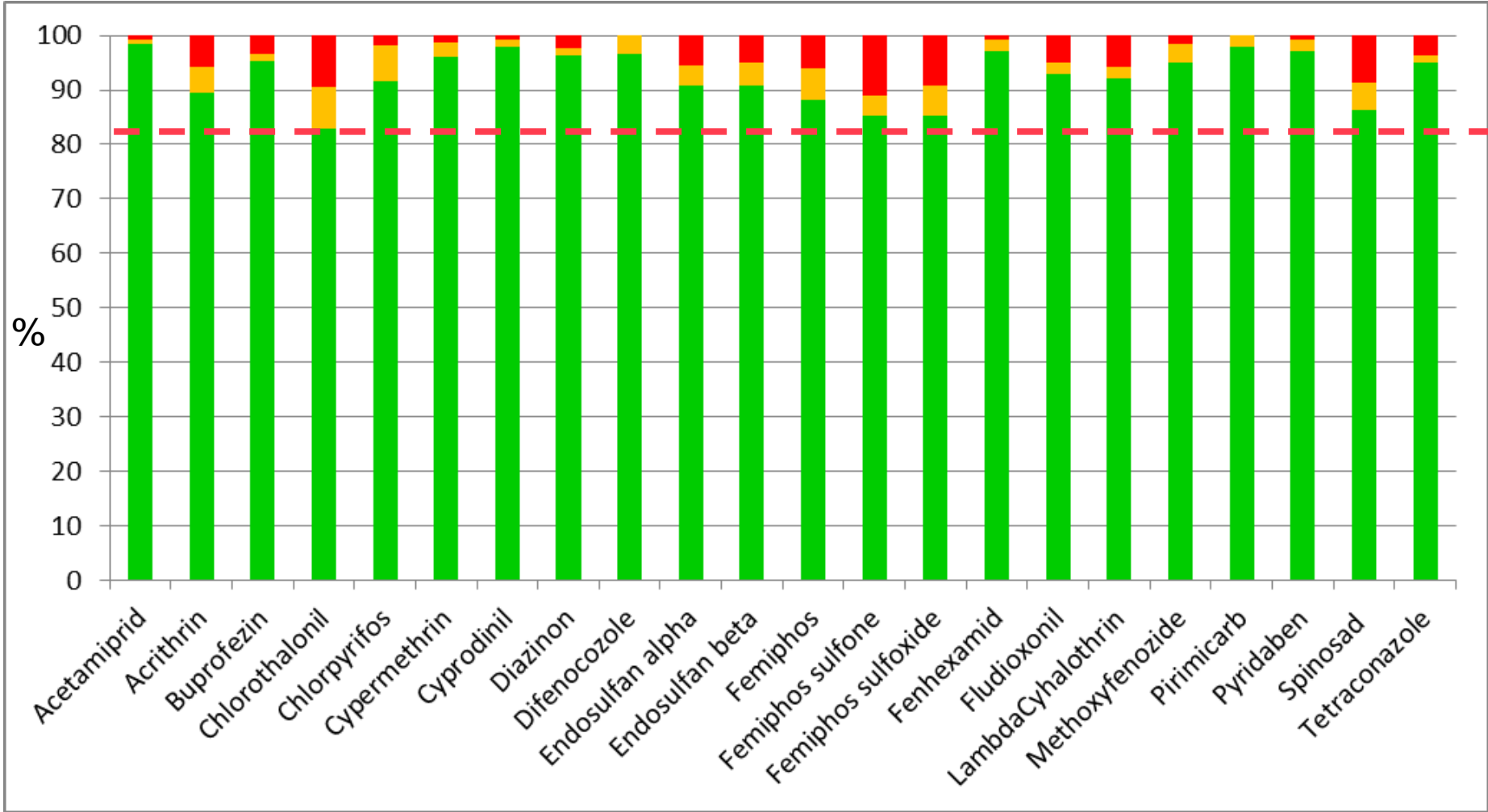
Dispersion of Results



Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results (out of 169)
Acetamiprid	129	1	39	76
Acrinathrin	136	5	28	80
Buprofezin	145	3	21	86
Chlorothalonil	139	8	22	82
Chlorpyrifos	167	0	2	99
Cypermethrin	154	1	14	91
Cyprodinil	152	1	16	90
Diazinon	164	1	4	97
Difenoconazole	142	0	27	84
Endosulfan alpha	160	2	7	95
Endosulfan beta	159	2	8	94
Fenamiphos	131	3	35	78
Fenamiphos sulfone	102	7	60	60
Fenamiphos sulfoxide	102	7	60	60
Fenhexamid	143	1	25	85
Fludioxonil	138	5	26	82
Lambda-Cyhalothrin	147	6	16	87
Methoxyfenozide	119	2	48	70
Pirimicarb	154	0	15	91
Pyridaben	138	1	30	82
Spinosad	115	1	53	68
Tetraconazole	139	1	29	82

z-Scores

z-Scores classification



Acceptable

Questionable

Unacceptable

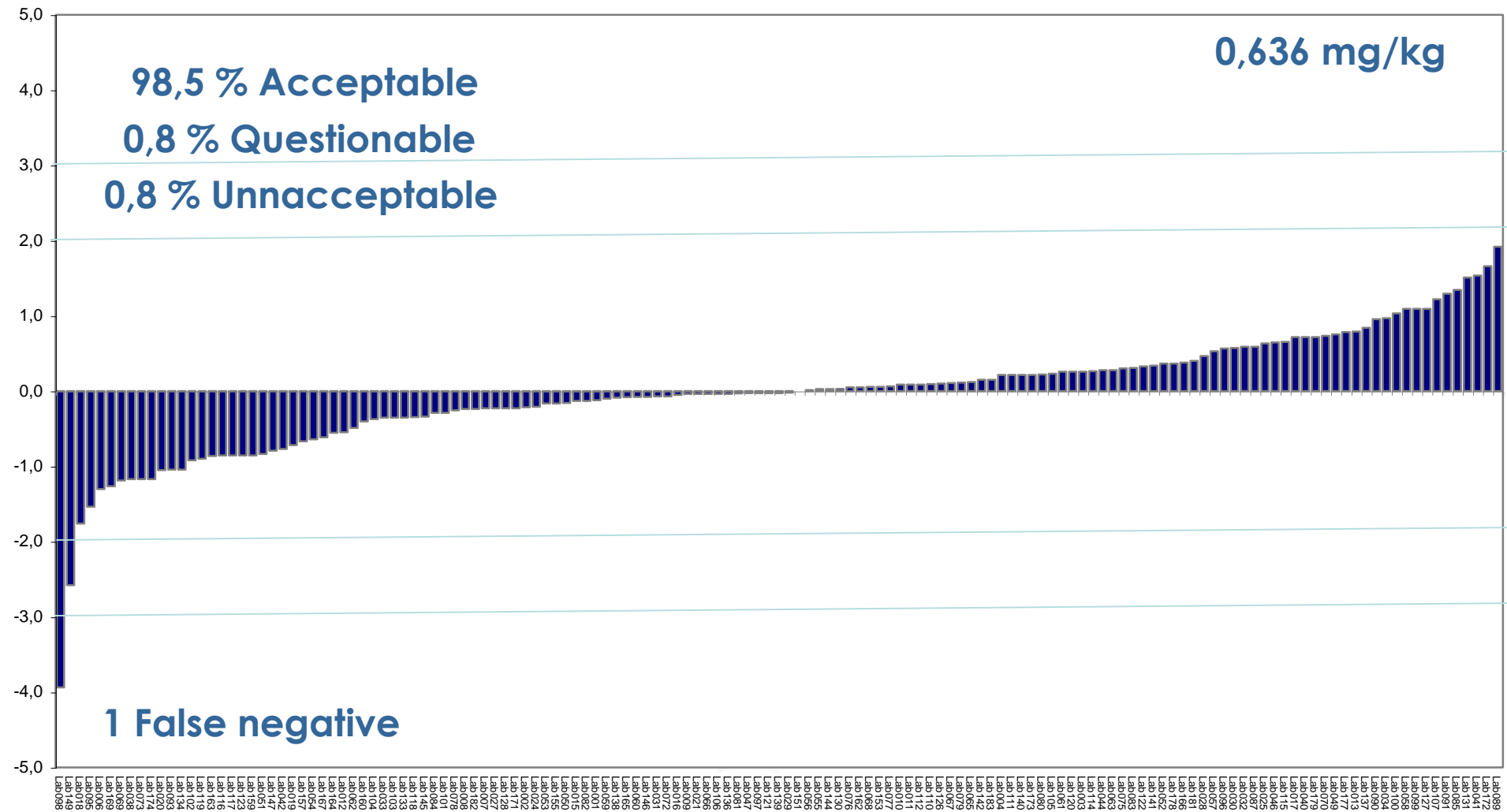
Pesticides	Robust Mean (mg/kg)	% Acceptable z-scores	% Questionable z-scores	% Unacceptable z-scores
Acetamiprid	0,636	98,5	0,8	0,8
Acrinathrin	0,263	89,4	5,0	5,7
Buprofezin	0,469	95,3	1,4	3,4
Chlorothalonil	2,179	83,0	7,5	9,5
Chlorpyrifos	3,667	91,6	6,6	1,8
Cypermethrin	0,528	96,1	2,6	1,3
Cyprodinil	0,588	98,0	1,3	0,7
Diazinon	0,084	96,4	1,2	2,4
Difenoconazole	0,945	96,5	3,5	0,0
Endosulfan alpha	1,149	90,7	3,7	5,6
Endosulfan beta	0,964	90,7	4,3	5,0
Fenamiphos	7,297	88,1	6,0	6,0
Fenamiphos sulfone	0,109	85,3	3,7	11,0
Fenamiphos sulfoxide	6,625	85,3	5,5	9,2
Fenhexamid	0,855	97,2	2,1	0,7
Fludioxonil	0,245	93,0	2,1	4,9
Lambda-Cyhalothrin	0,078	92,2	2,0	5,9
Methoxyfenozide	0,170	95,0	3,3	1,7
Pirimicarb	0,730	98,1	1,9	0,0
Pyridaben	0,149	97,1	2,2	0,7
Spinosad	0,098	86,2	5,2	8,6
Tetraconazole	0,105	95,0	1,4	3,6

Acetamiprid

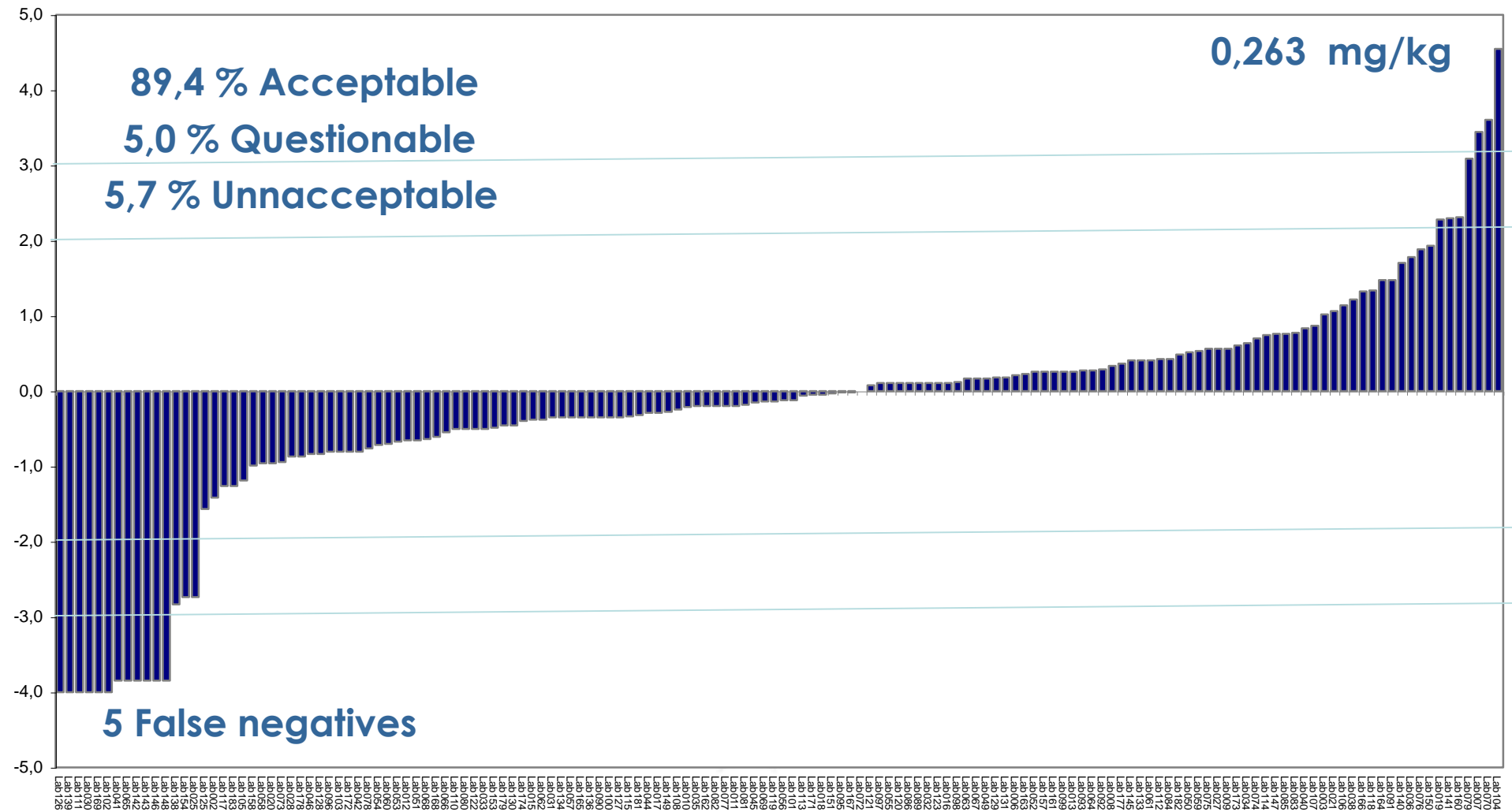
0,636 mg/kg

98,5 % Acceptable
0,8 % Questionable
0,8 % Unacceptable

1 False negative



Acrinathrin



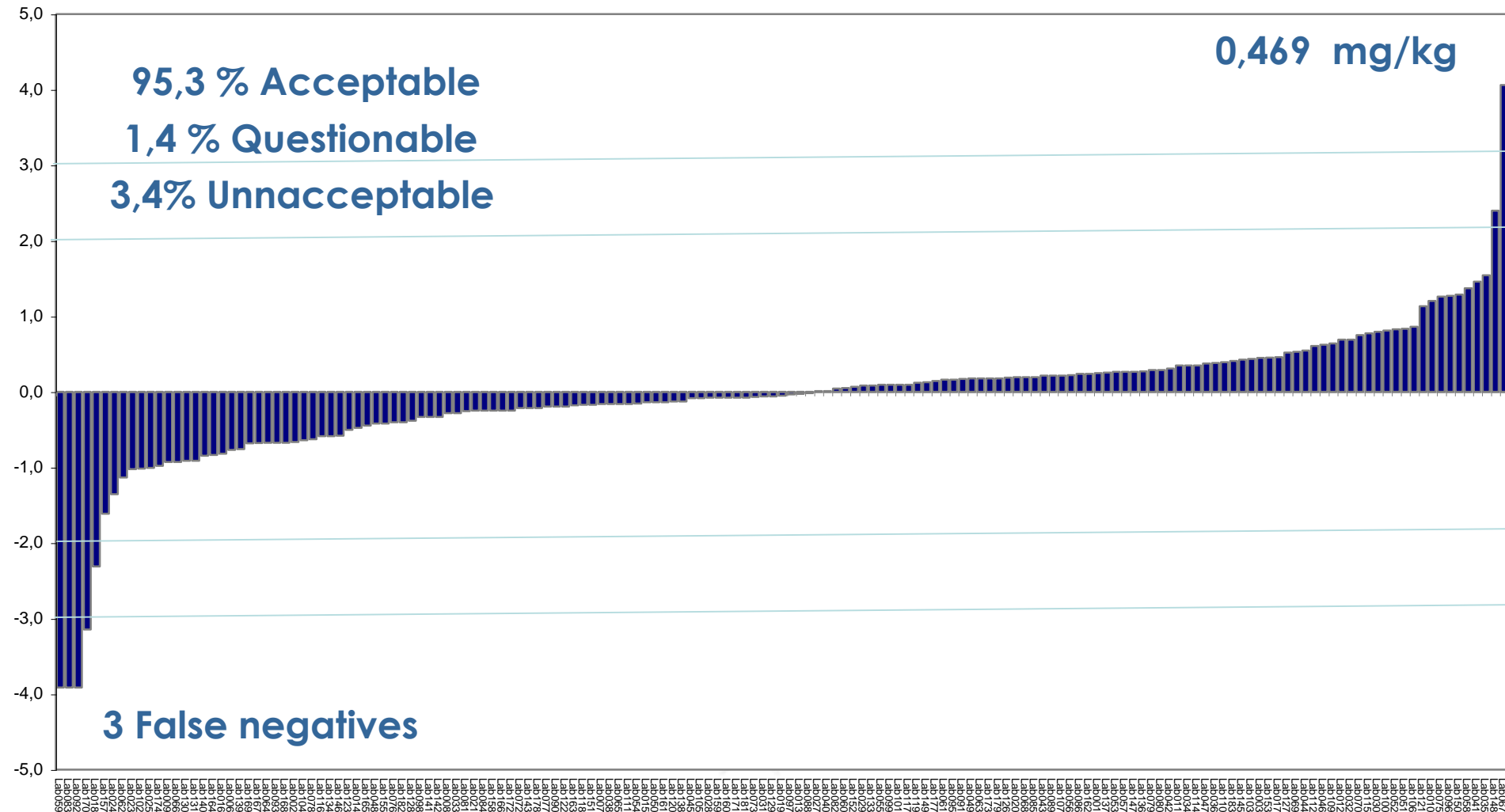


Buprofezin

0,469 mg/kg

95,3 % Acceptable
1,4 % Questionable
3,4% Unacceptable

3 False negatives



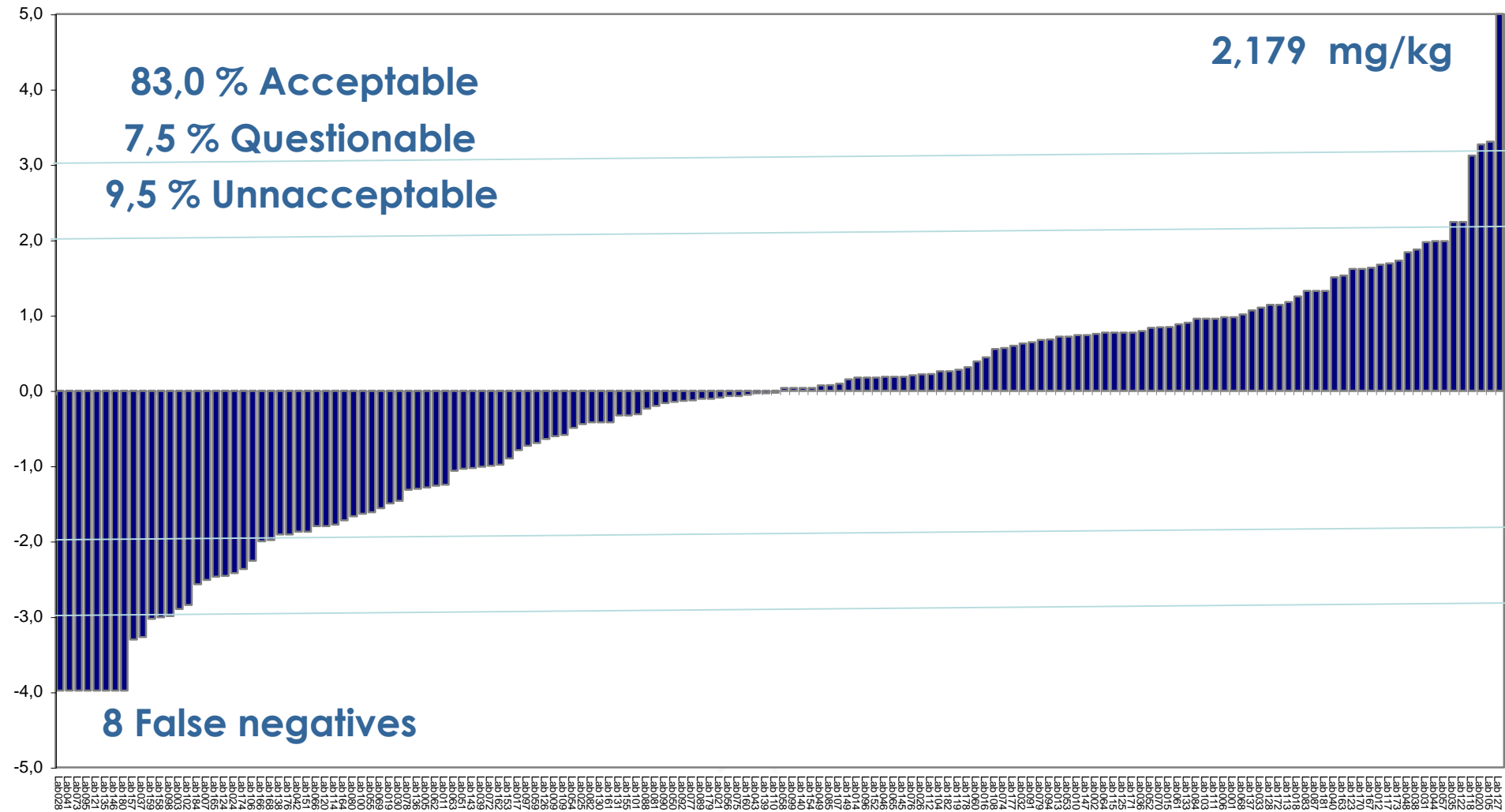


Chlorothalonil

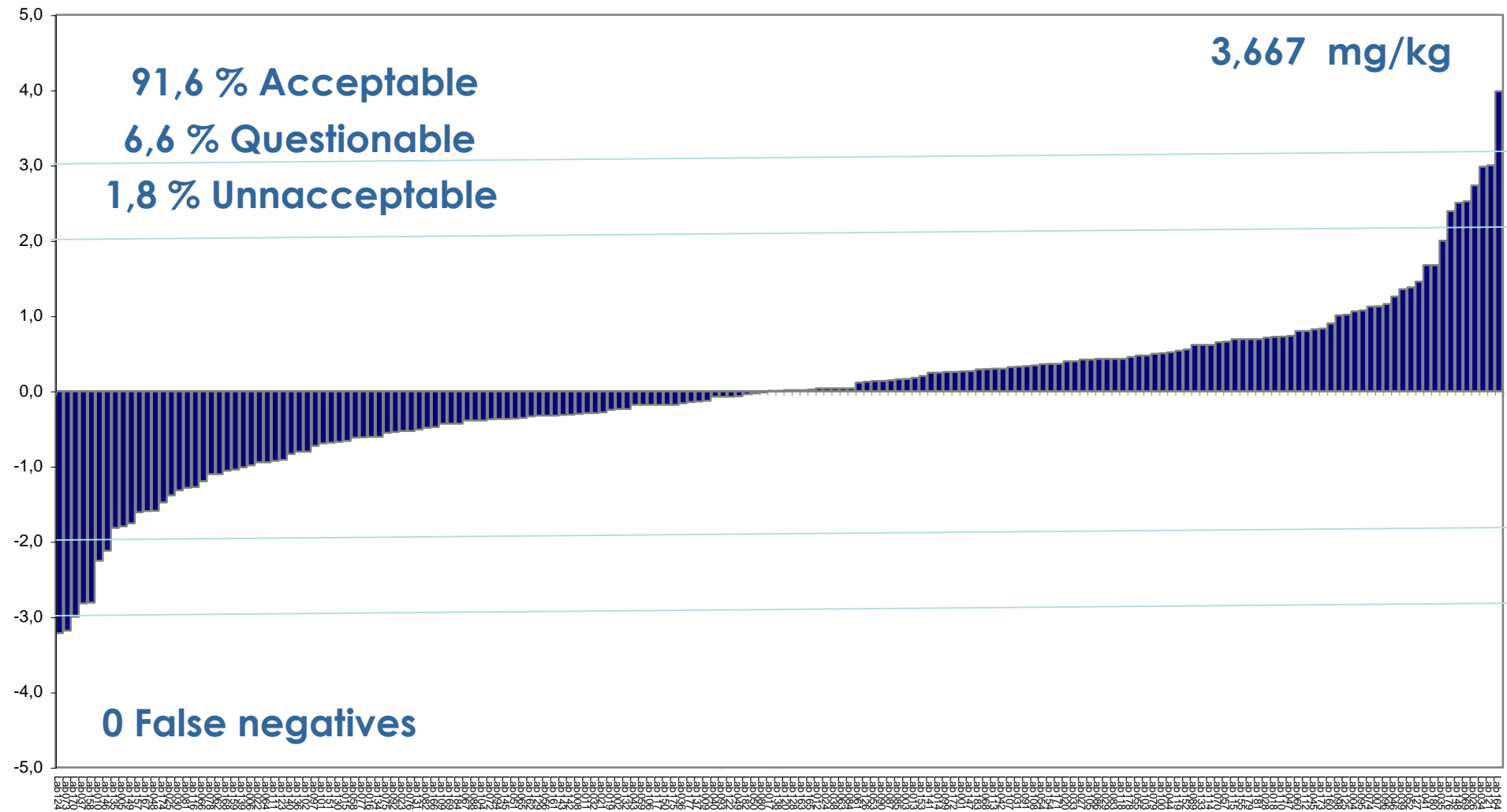
2,179 mg/kg

83,0 % Acceptable
7,5 % Questionable
9,5 % Unacceptable

8 False negatives



Chlorpyrifos



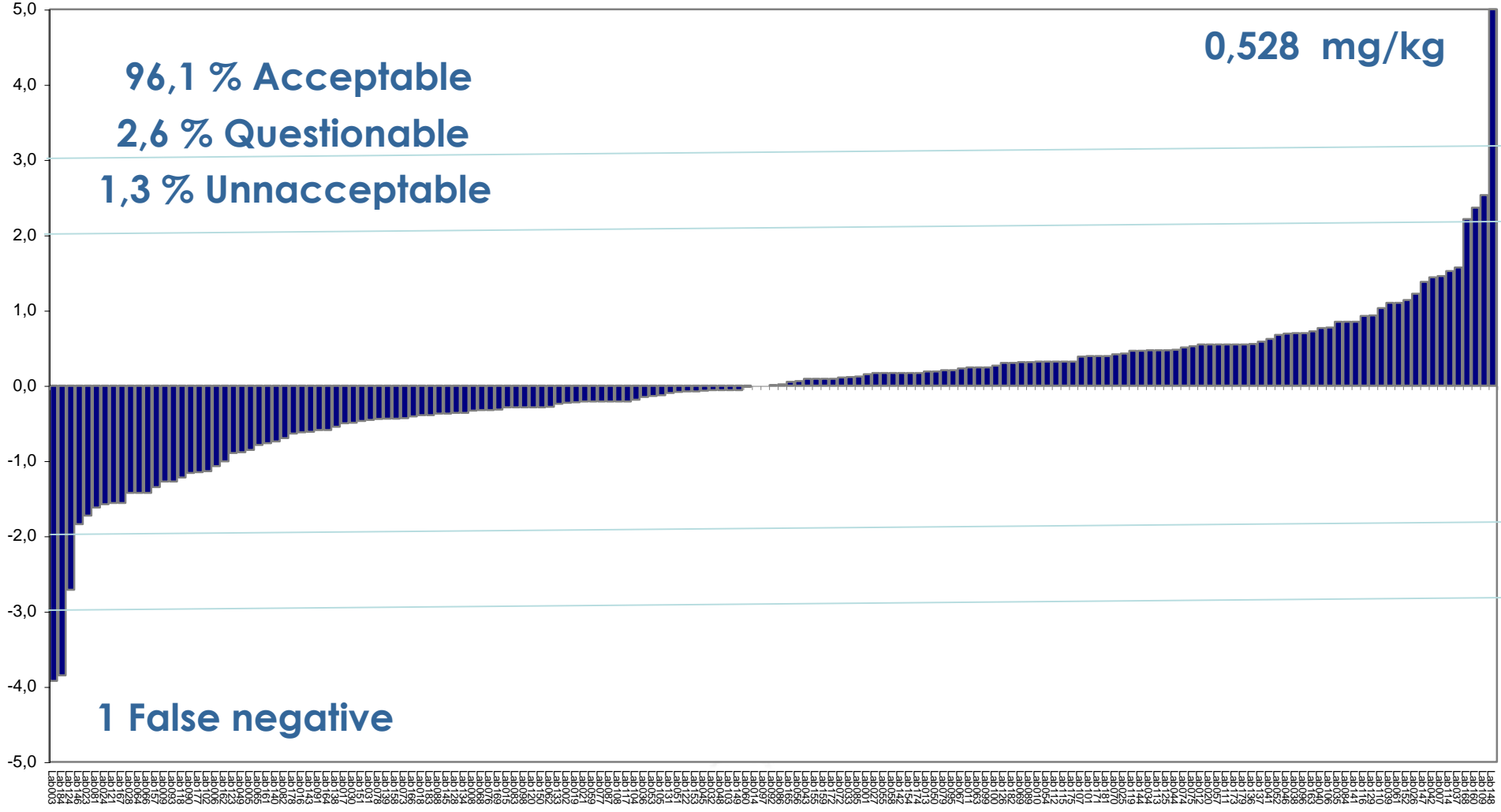


Cypermethrin

0,528 mg/kg

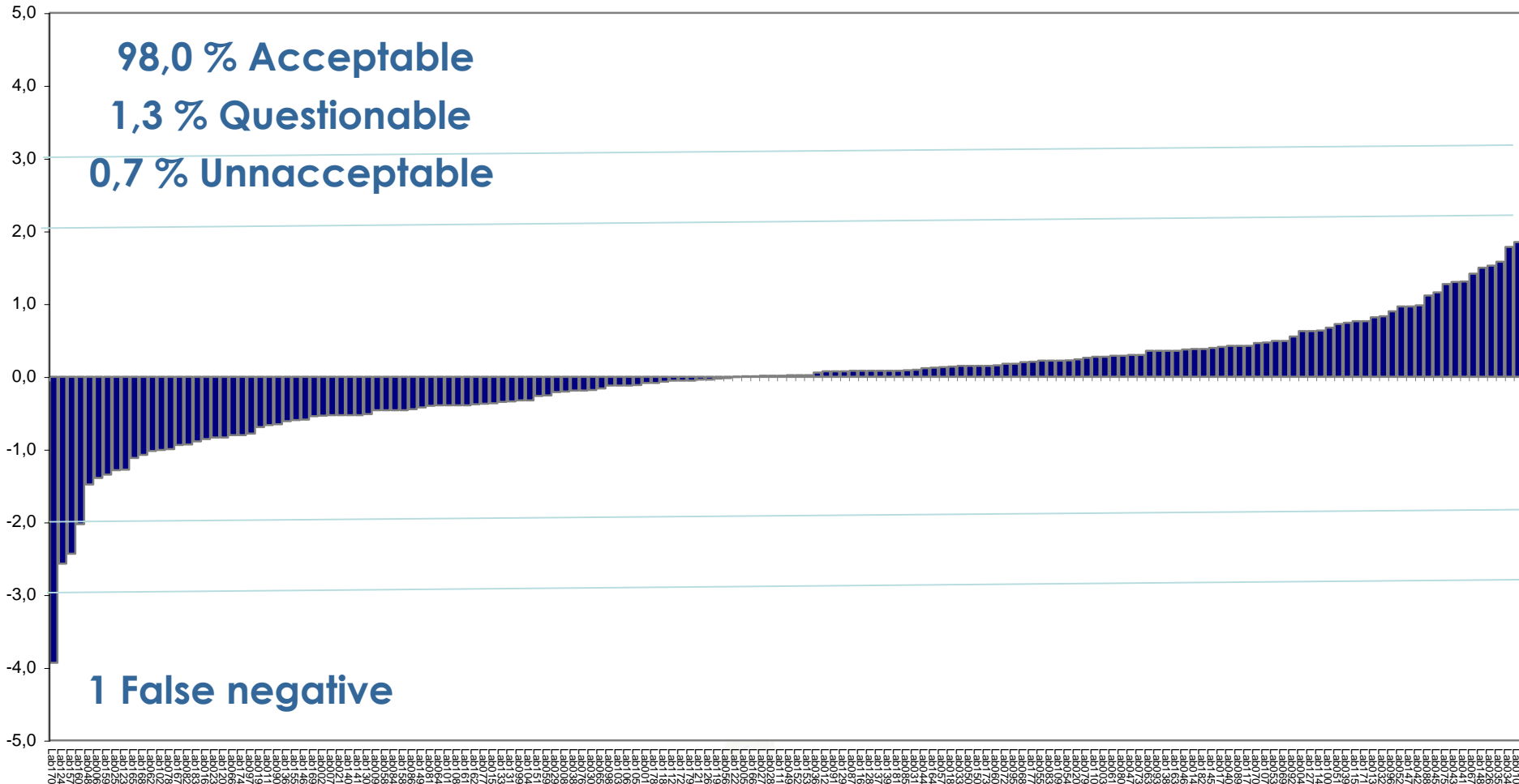
96,1 % Acceptable
2,6 % Questionable
1,3 % Unacceptable

1 False negative



Cyprodinil

0,588 mg/kg

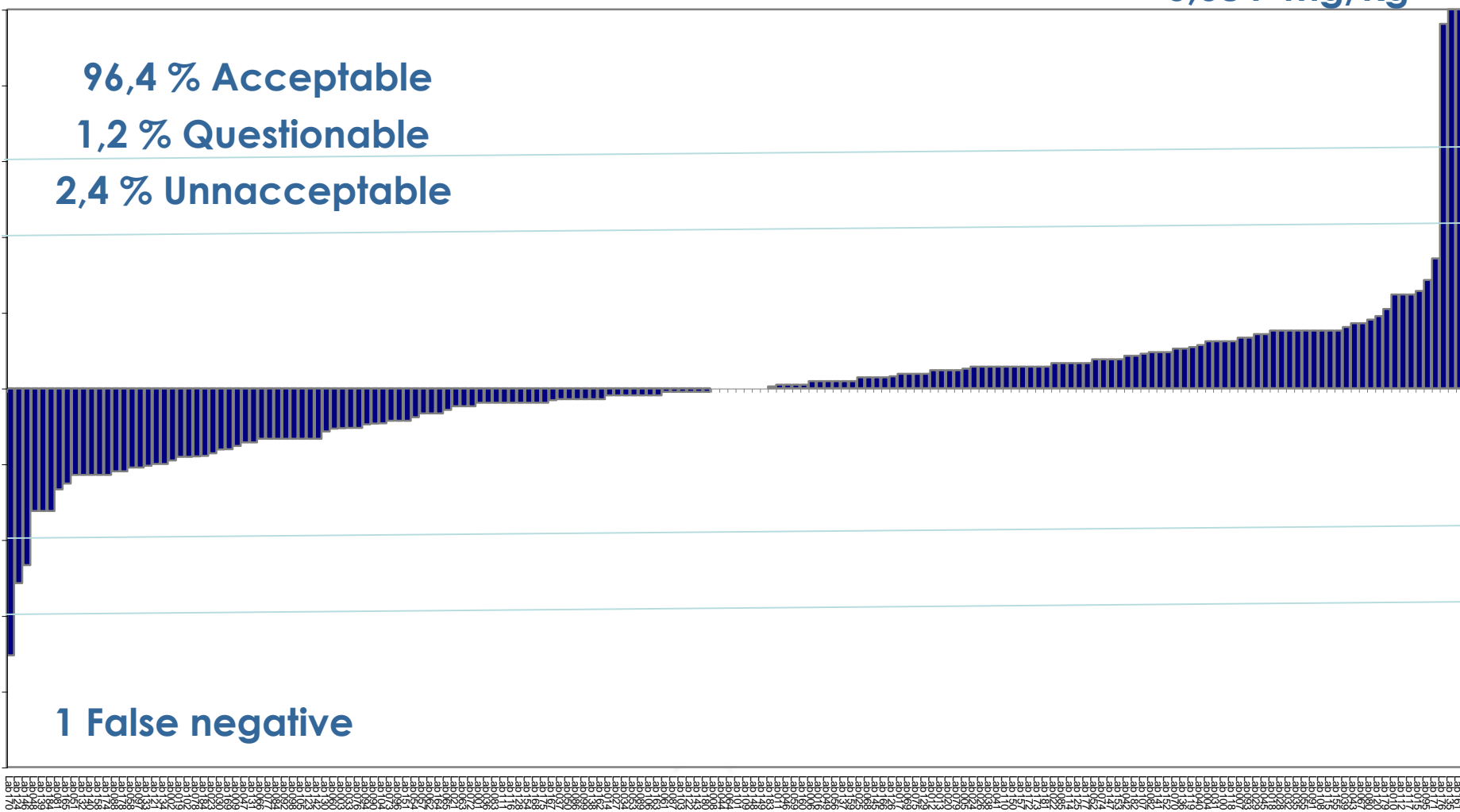


Diazinon

0,084 mg/kg

96,4 % Acceptable
1,2 % Questionable
2,4 % Unacceptable

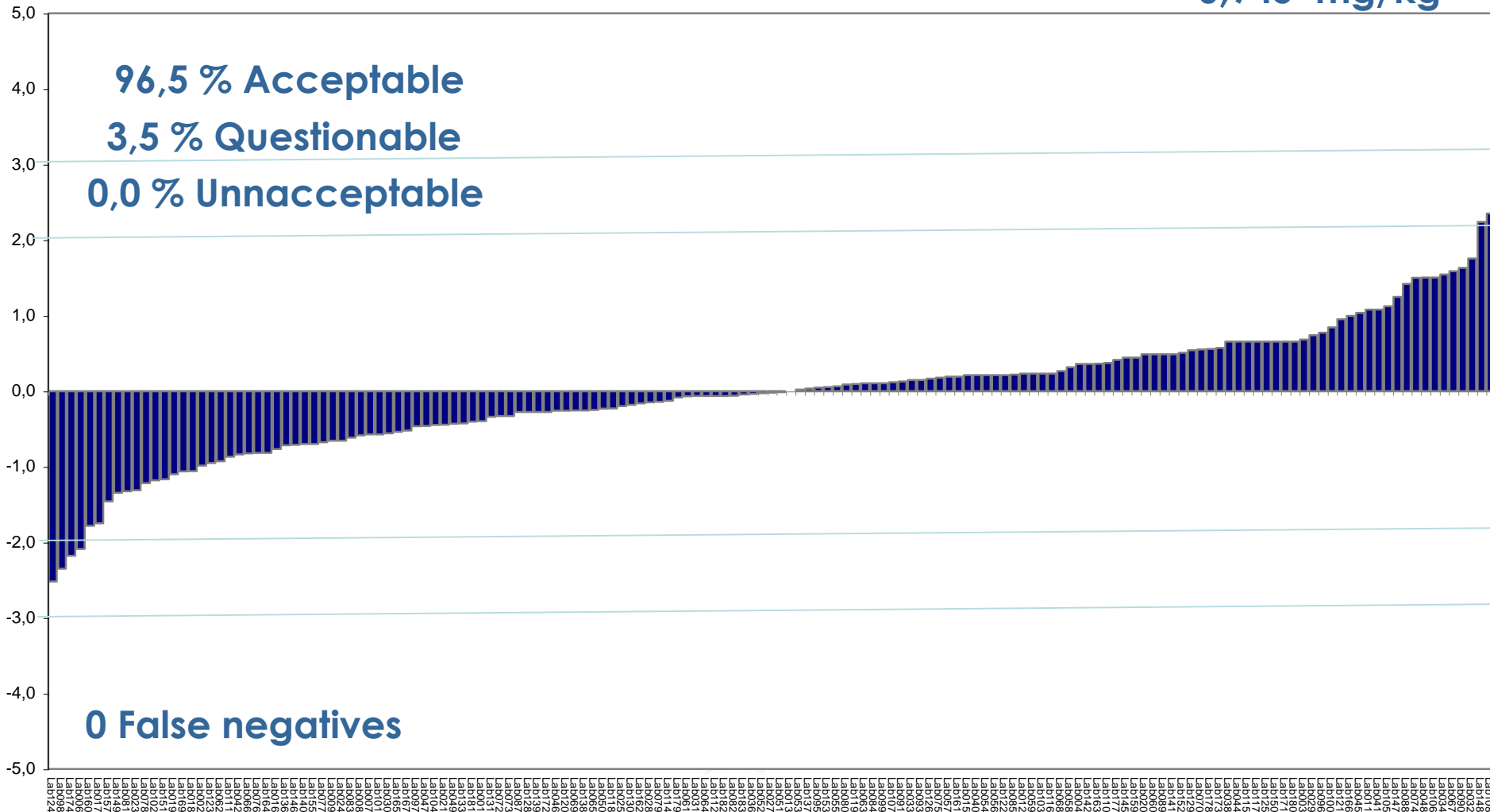
1 False negative





Difenoconazole

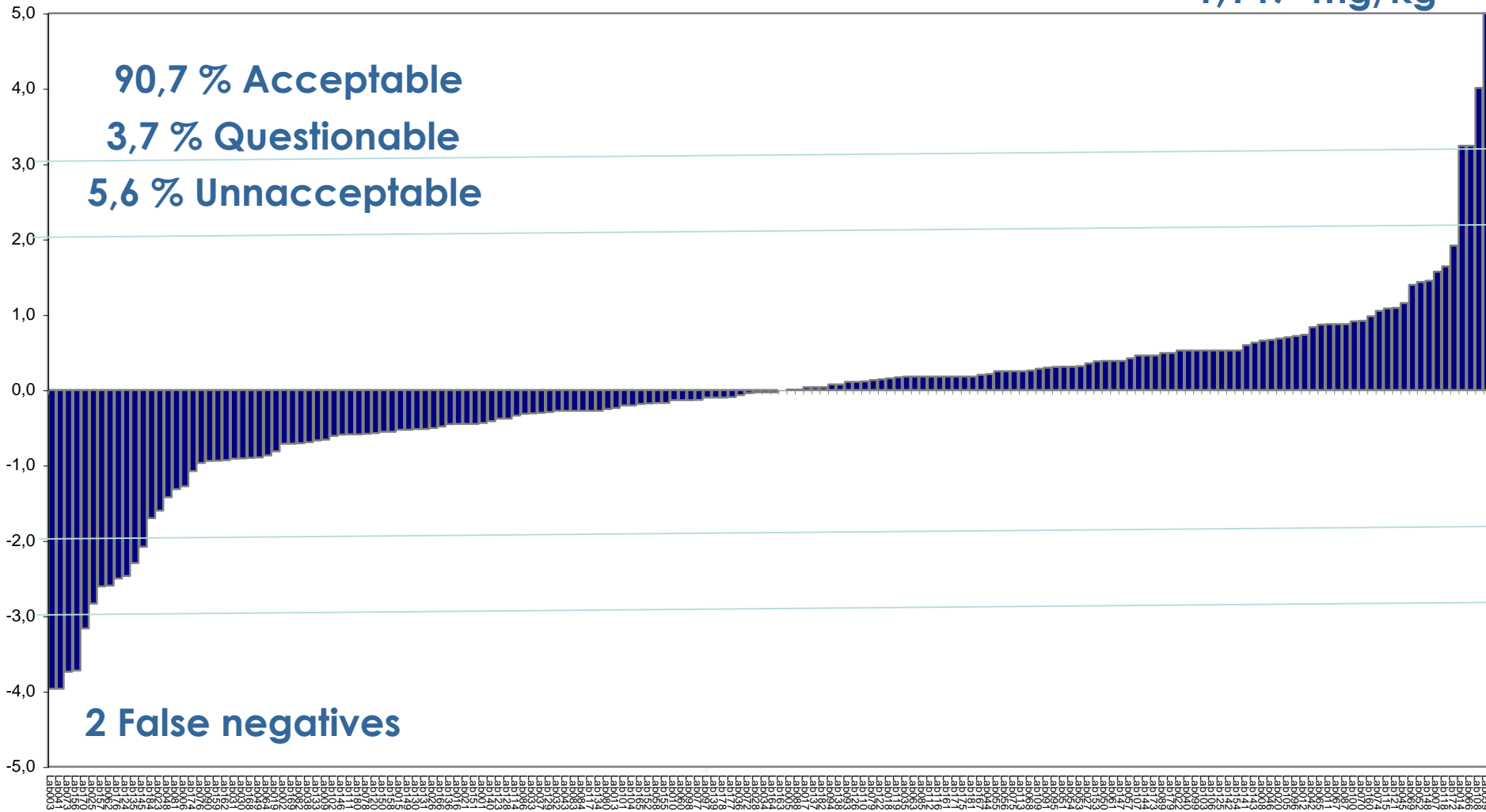
0,945 mg/kg





Endosulfan alpha

1,149 mg/kg

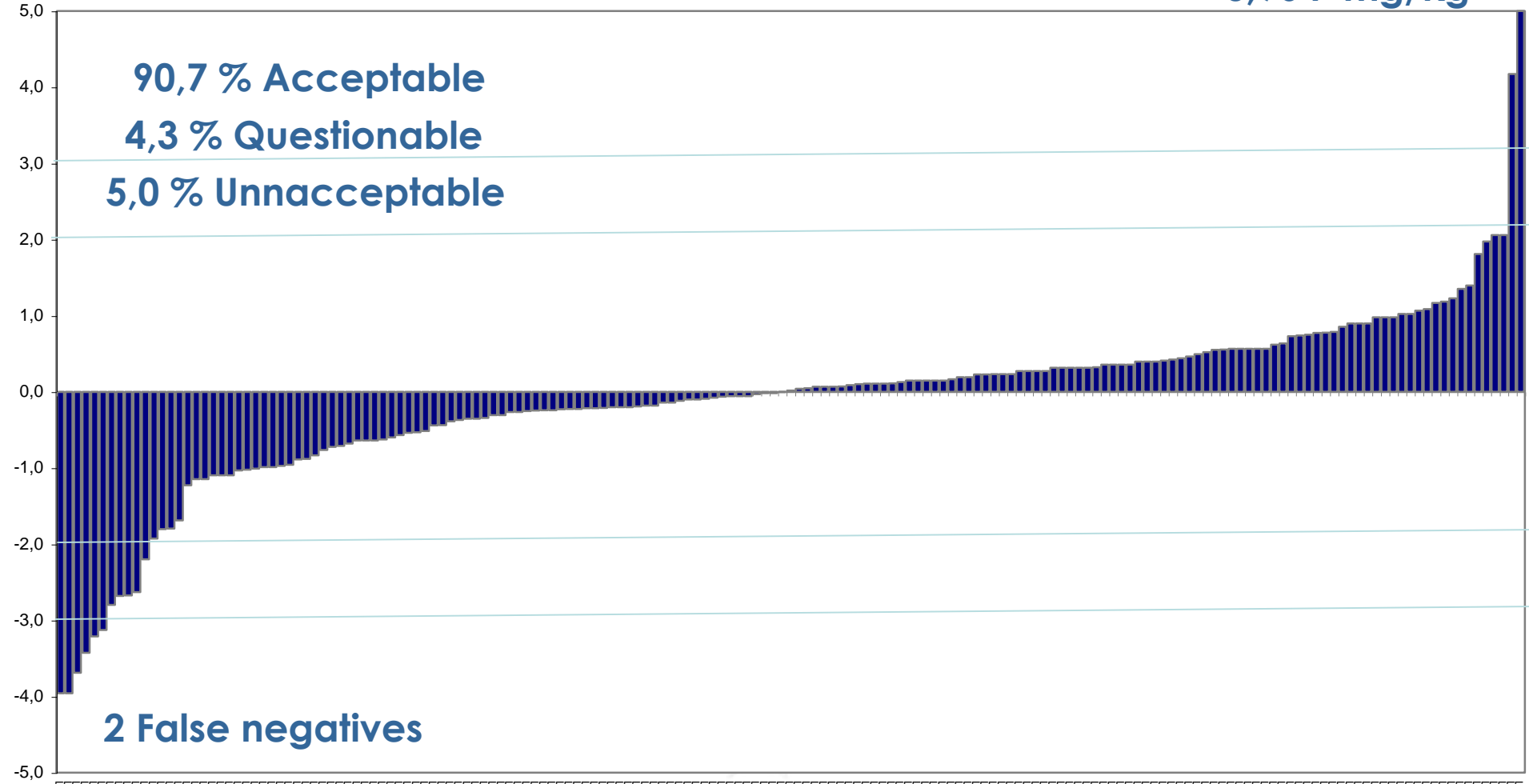


Endosulfan Beta

0,964 mg/kg

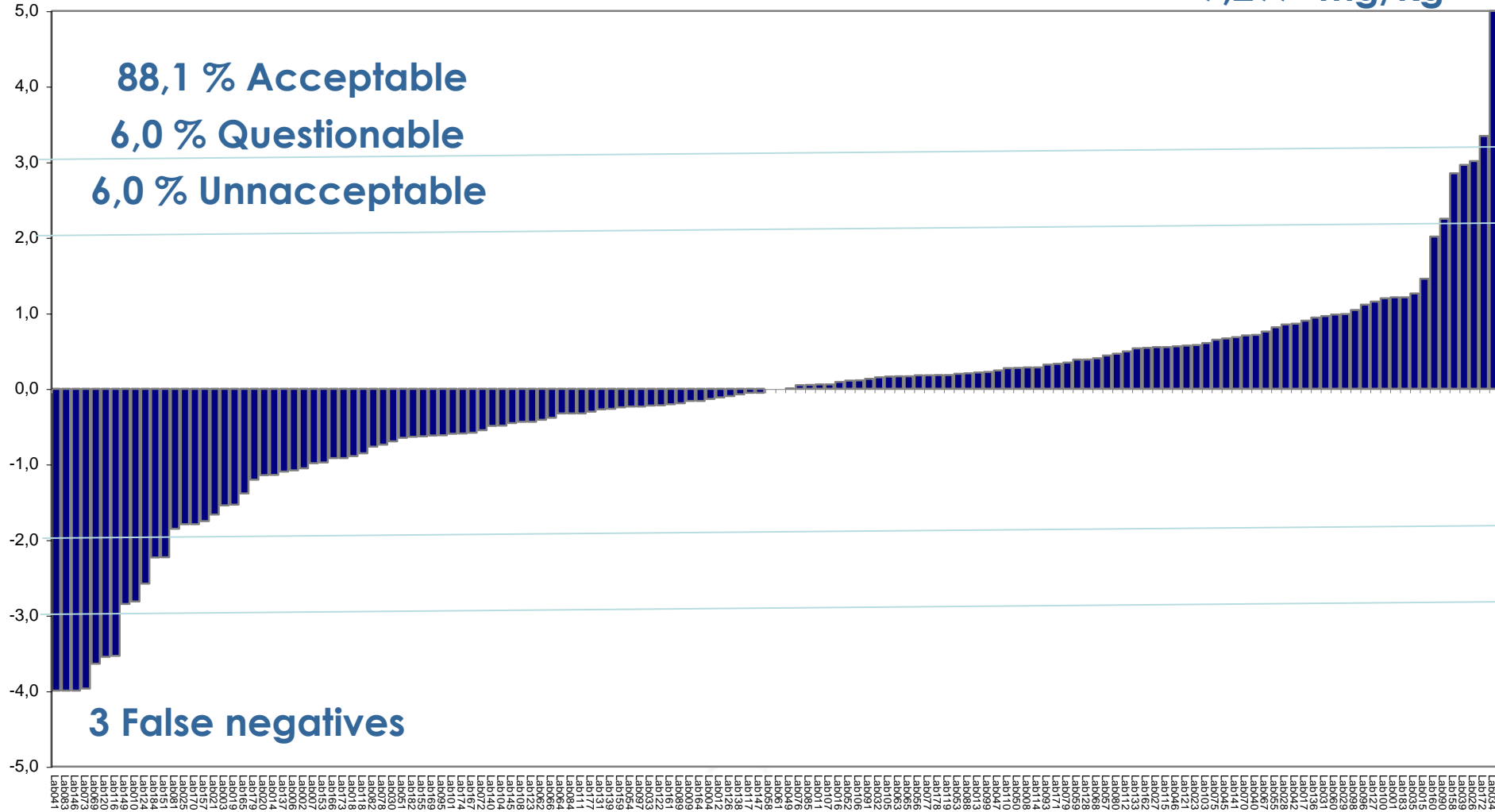
90,7 % Acceptable
4,3 % Questionable
5,0 % Unacceptable

2 False negatives



Fenamiphos

7,297 mg/kg



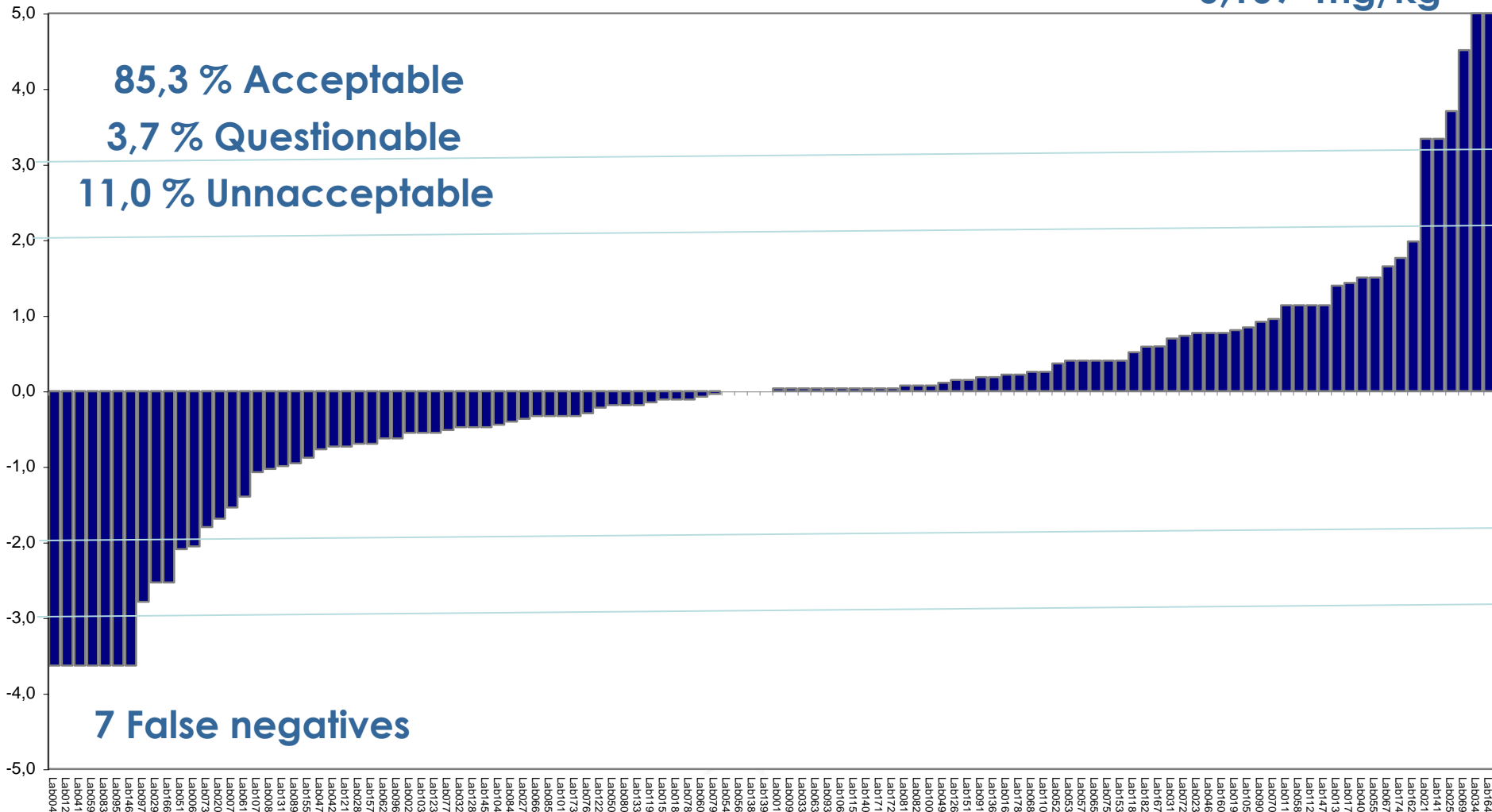


Fenamiphos sulfone

0,109 mg/kg

85,3 % Acceptable
3,7 % Questionable
11,0 % Unacceptable

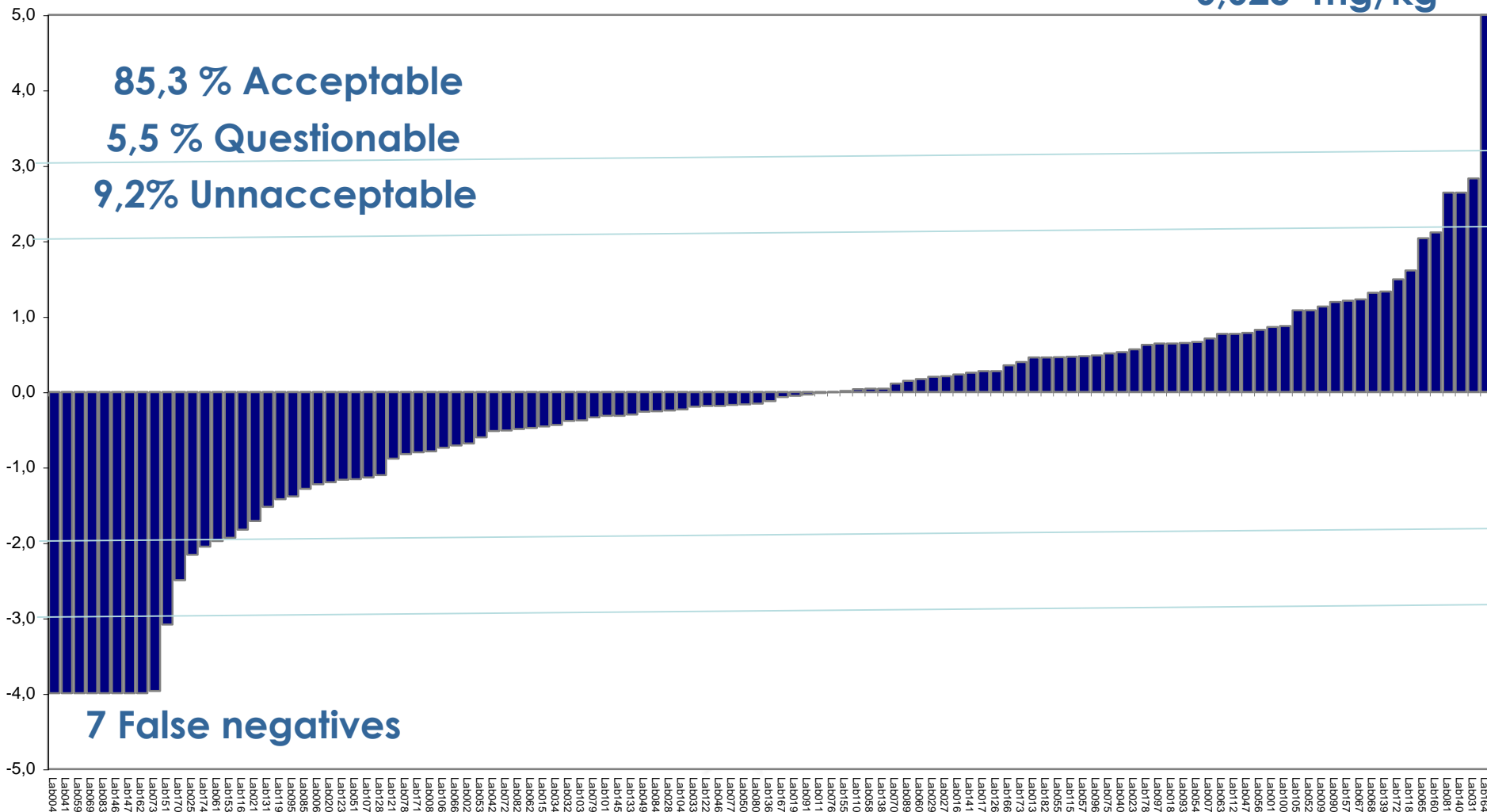
7 False negatives





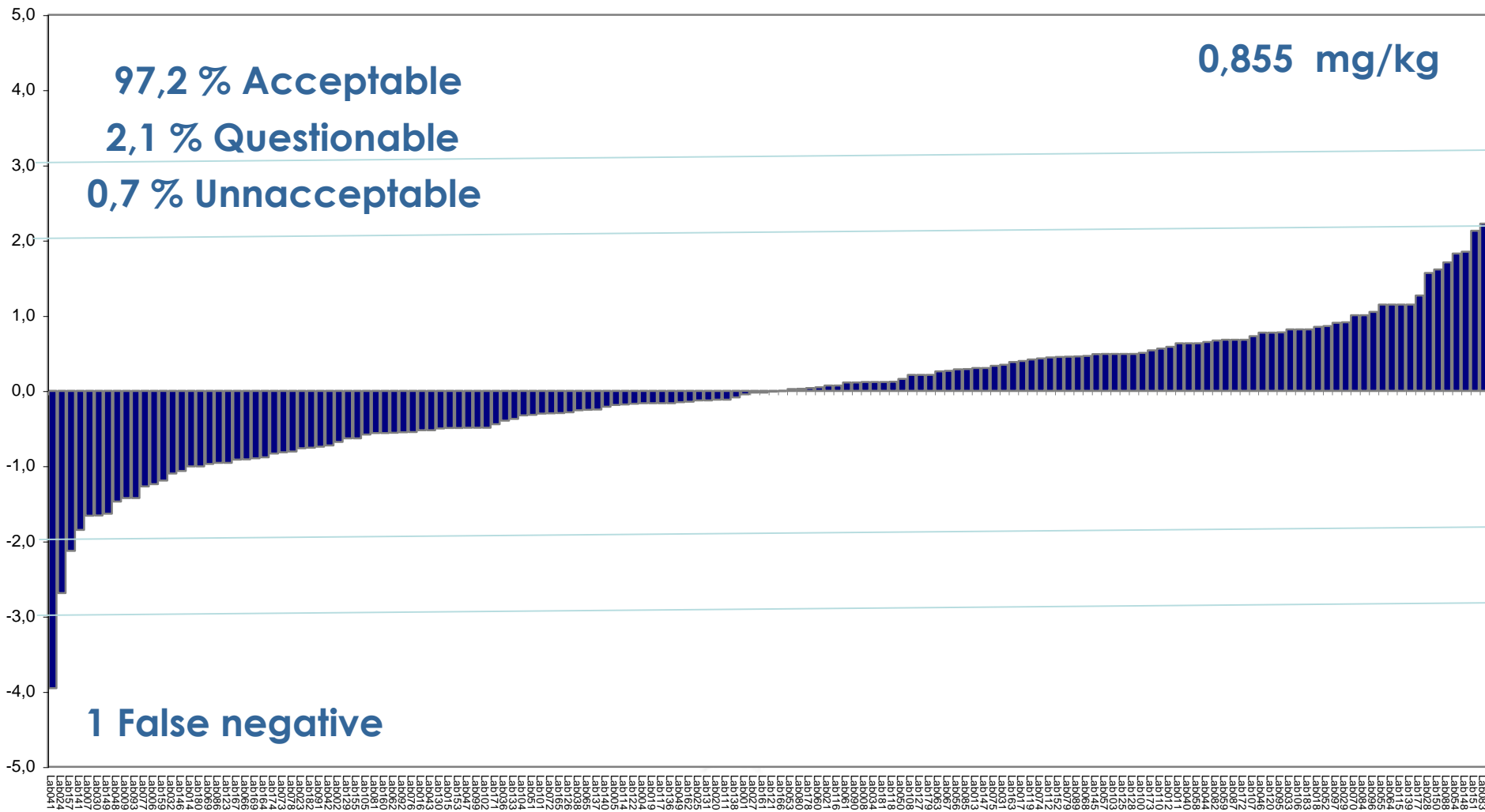
Fenamiphos sulfoxide

6,625 mg/kg



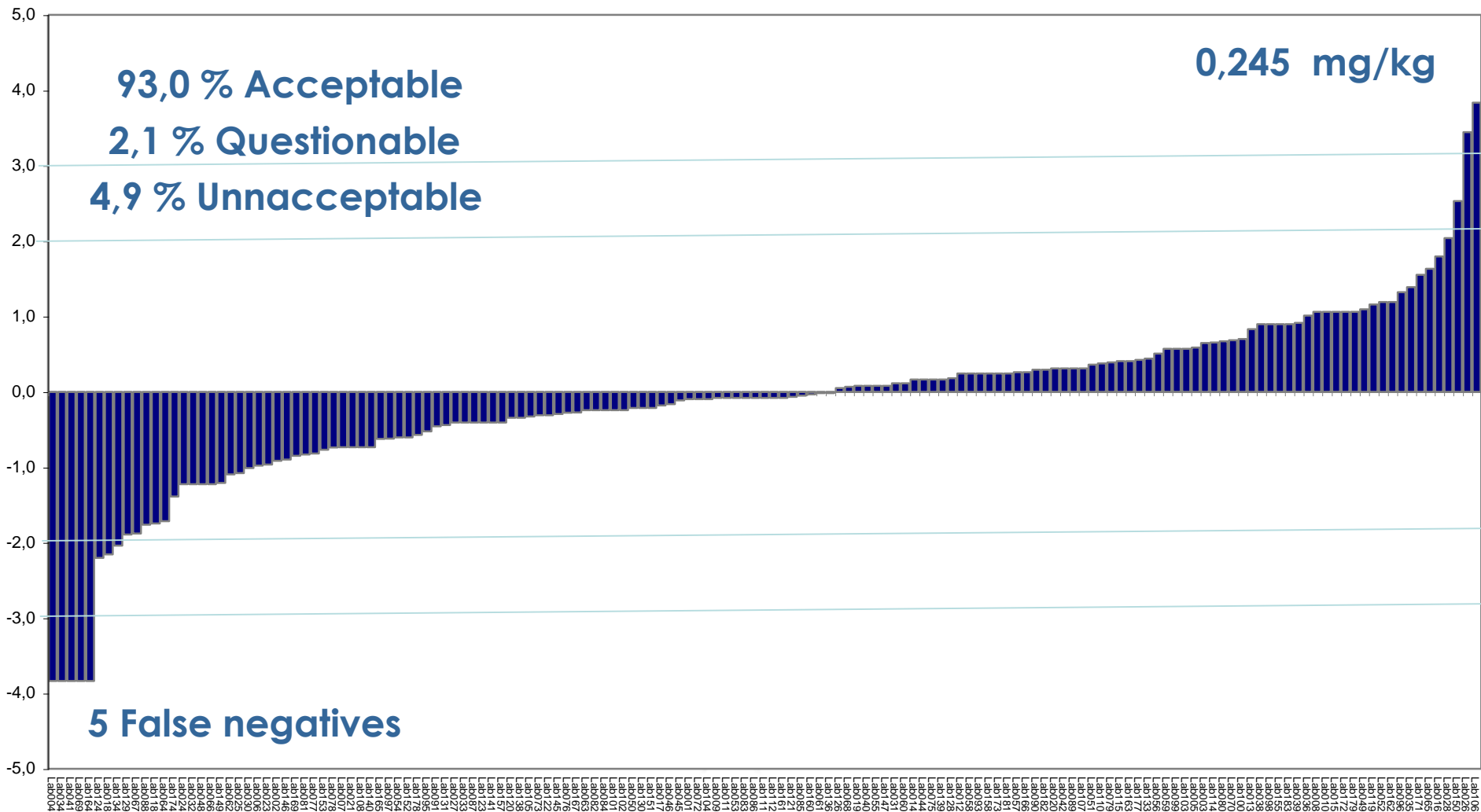


Fenhexamid

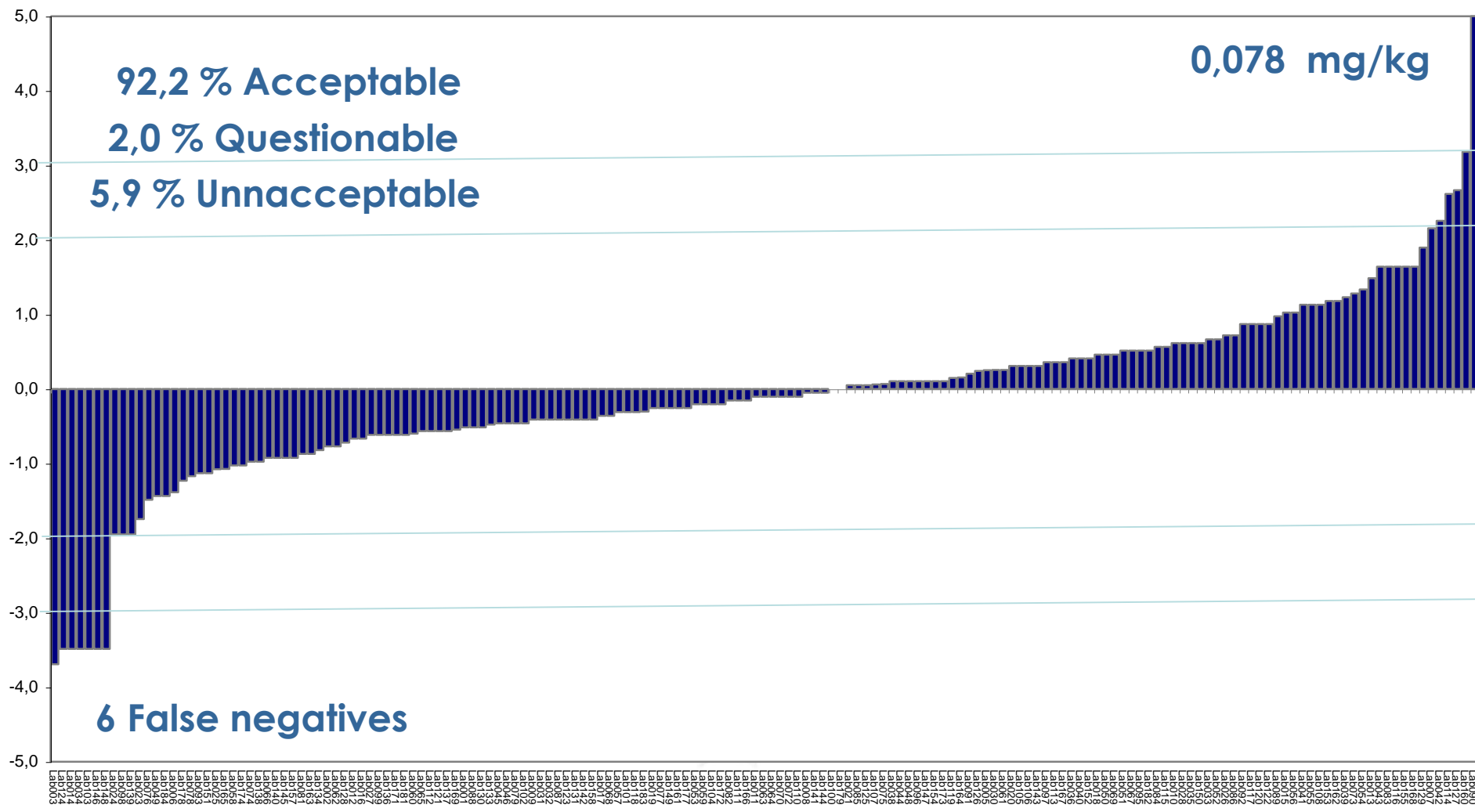




Fludioxonil

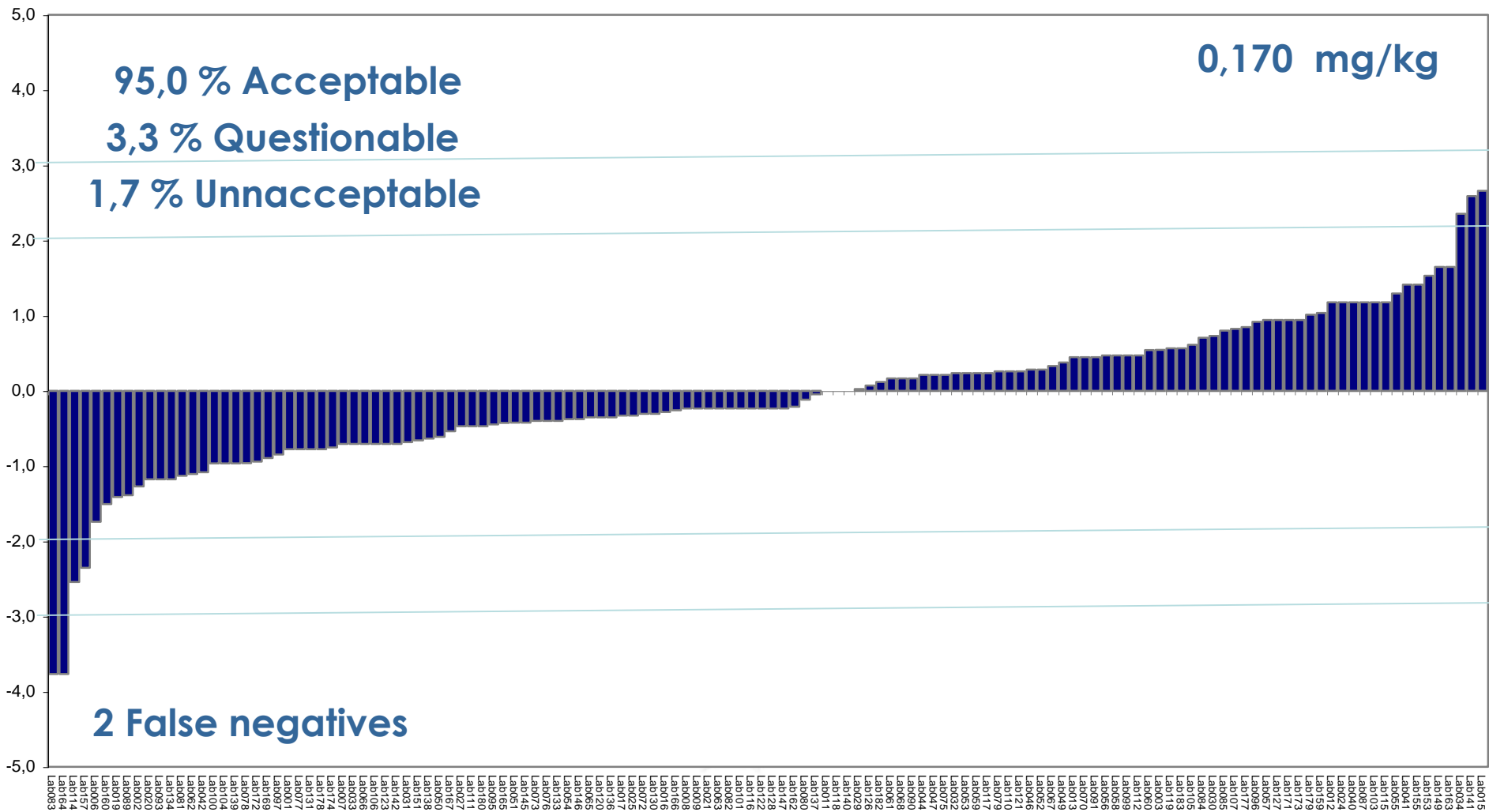


Lambda-Cyhalothrin



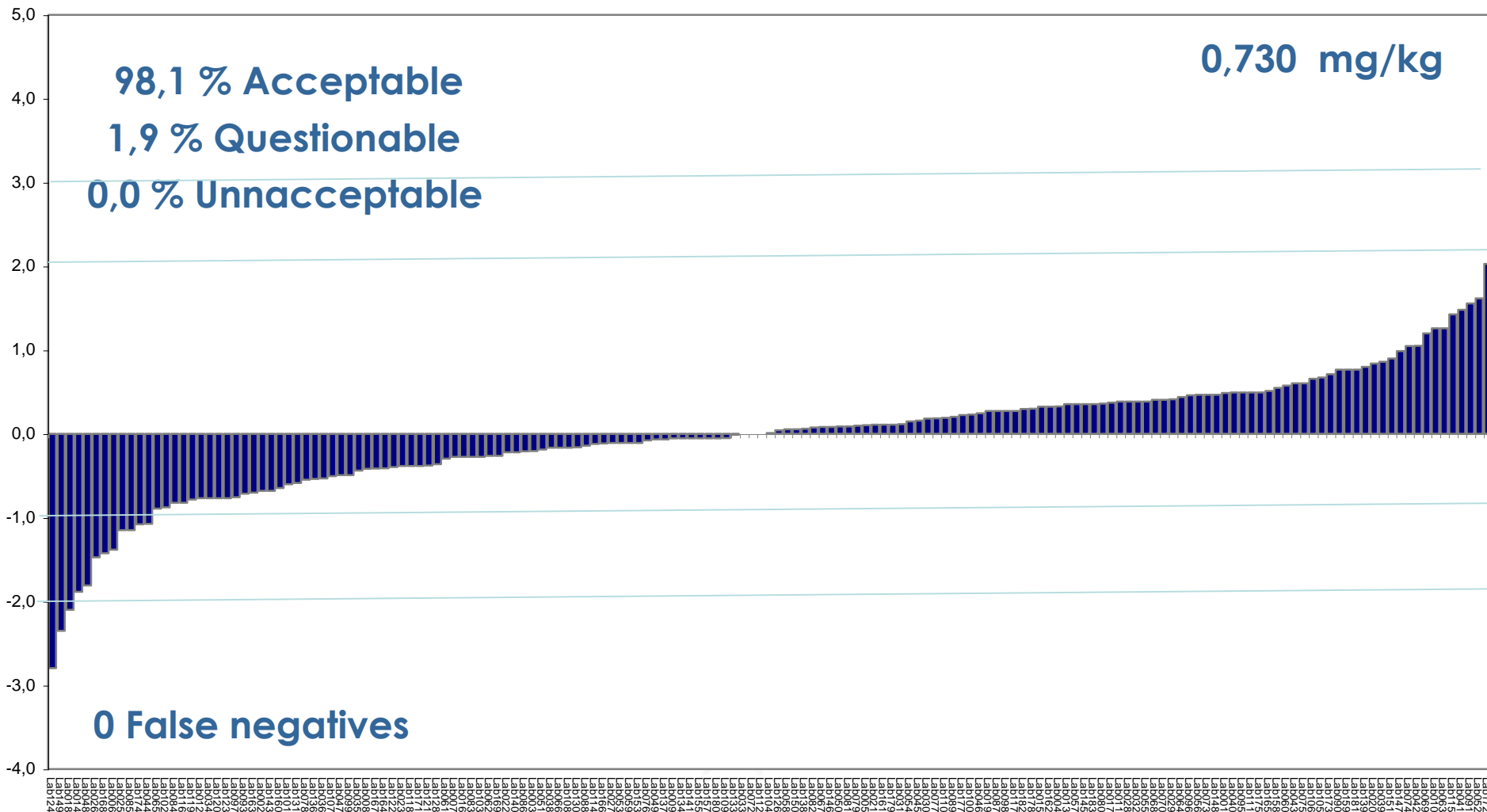


Methoxyfenozide



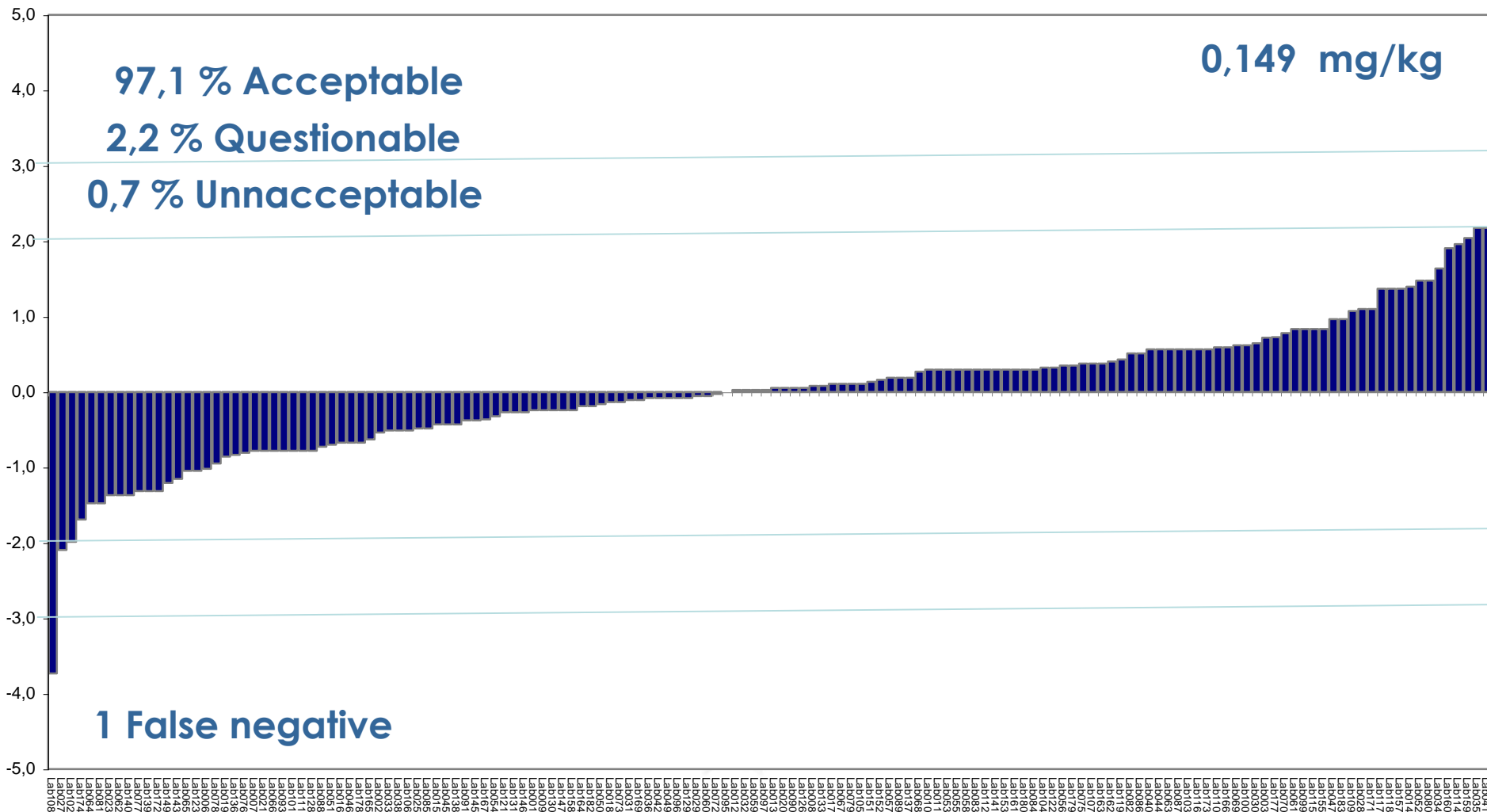


Pirimicarb

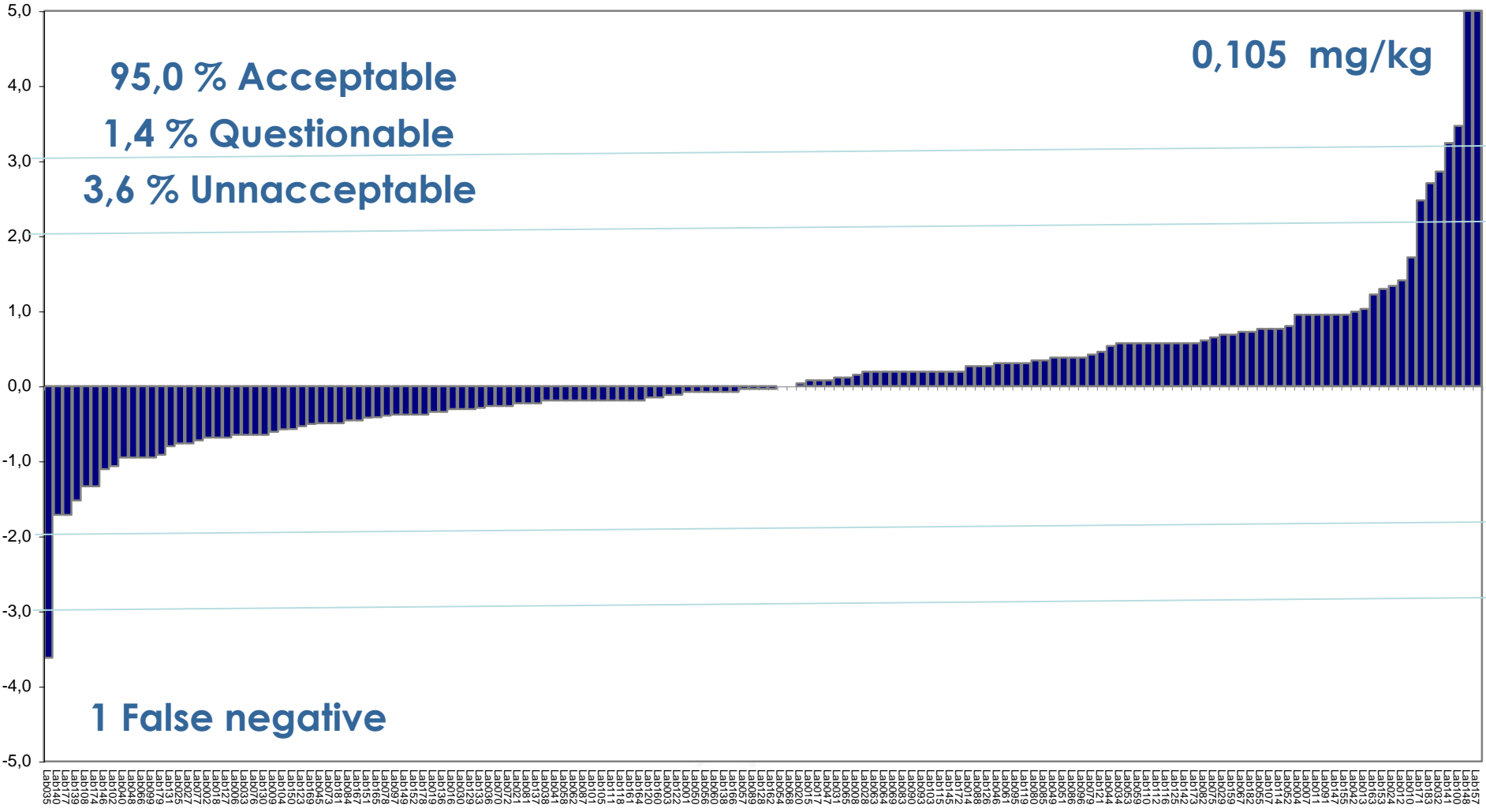




Pyridaben



Tetraconazole





False Positives

4 different laboratories from EU/EFTA countries reported 4 pesticides as false positives



False Positives

Laboratory Code	Pesticide	Concentration (mg/kg)	Determination Technique	RL (mg/Kg)	MRRL (mg/Kg)
Lab059	Fenpropathrin	0,980	LC-MS/MS (ESI+)	0.01	0,01
Lab160	Folpet	0,240	GC-MS/MS (EI)	0.02	0.01
Lab024	Metalaxyl and metalaxyl-M	0,010	LC-MS/MS (ESI+)	0.01	0.01
Lab003	Tefluthrin	0,012	GC-ECD	0.01	0.01

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Combined z-Scores



Average of Squared z-Scores

$$AZ^2 = \frac{\sum_{i=1}^n |z_i| |z_i|}{n}$$

$AZ^2 \leq 2$	Good
$2 < AZ^2 \leq 3$	Satisfactory
$AZ^2 > 3$	Unsatisfactory

AZ² Representation

EU/EFTA Laboratories

92 % Good

5 % Satisfactory

3 % Unsatisfactory

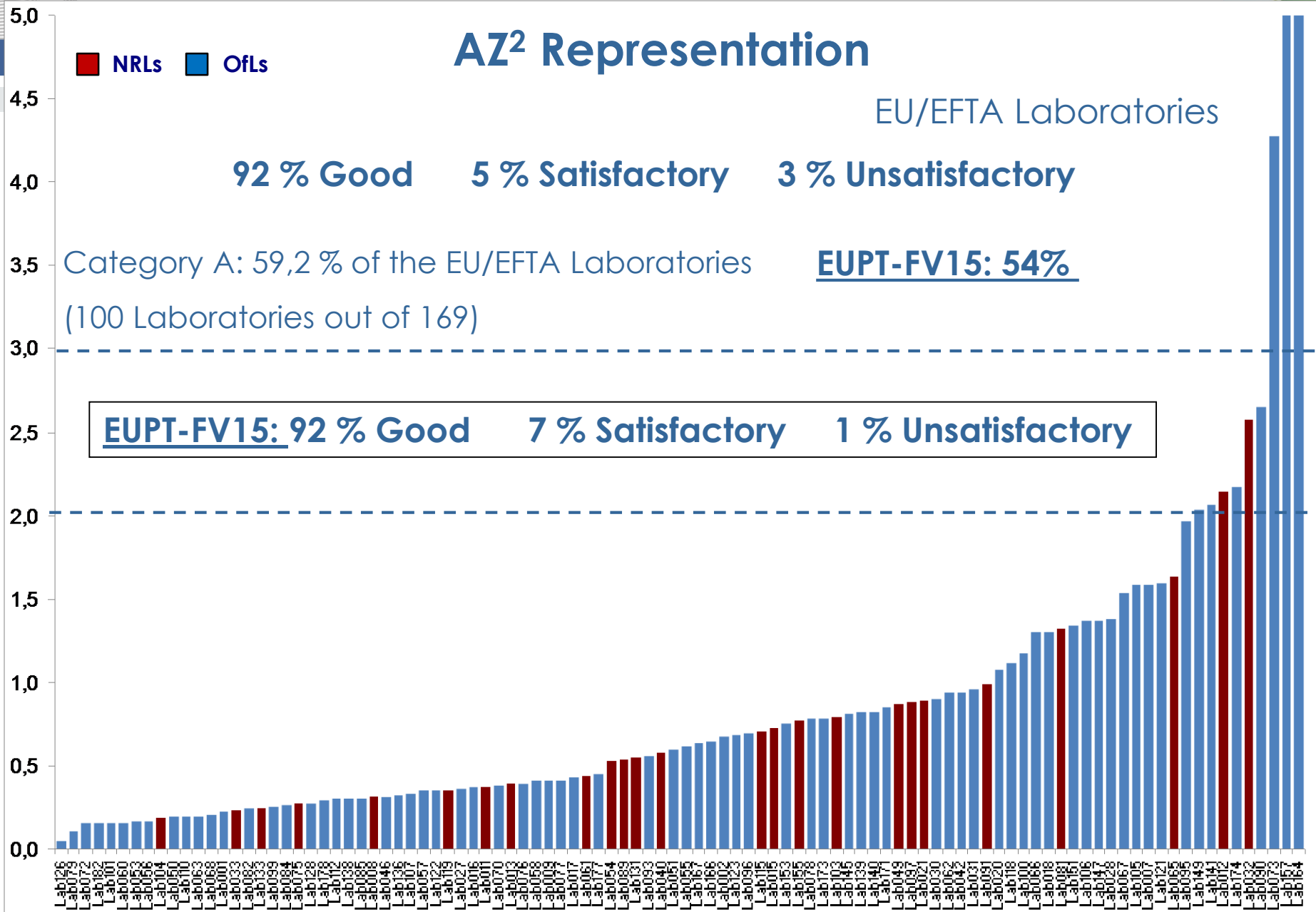
Category A: 59,2 % of the EU/EFTA Laboratories
(100 Laboratories out of 169)

EUPT-FV15: 54%

EUPT-FV15: 92 % Good

7 % Satisfactory

1 % Unsatisfactory





**Thank You
for Your Attention**



EURL EUROPEAN
UNION
REFERENCE
LABORATORY