

Preliminary Results



European Union Reference Laboratory for Pesticide Residues in Fruits & Vegetables

EUPT-FV-17

European Proficiency Test FV-17

DG SANTÉ

Mrs. Almut Bitterhof

Mrs. Veerle Vanheusden

ORGANISING GROUP

Amadeo R. Fernández-Alba
Milagros Mezcua
Octavio Malato
Carmen Ferrer

STATISTICAL GROUP

Carmelo Rodríguez

QUALITY CONTROL GROUP

Antonio Valverde
Stewart Reynolds

ADVISORY GROUP

Michelangelo Anastassiades
Miguel Gamón
Magnus Jezussek
André de Kok
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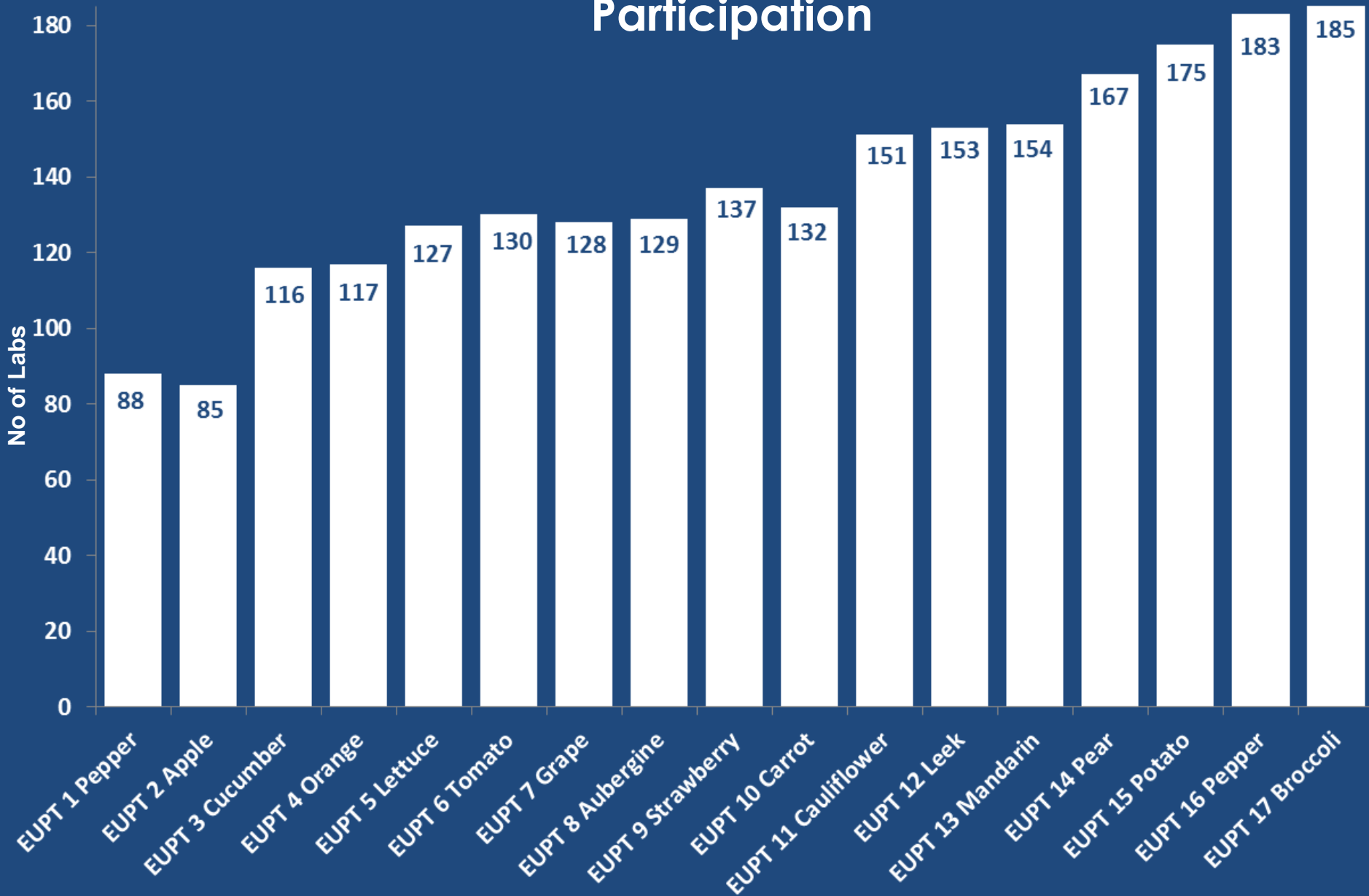
QUALITY CONTROL GROUP

Antonio Valverde
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ADVISORY GROUP

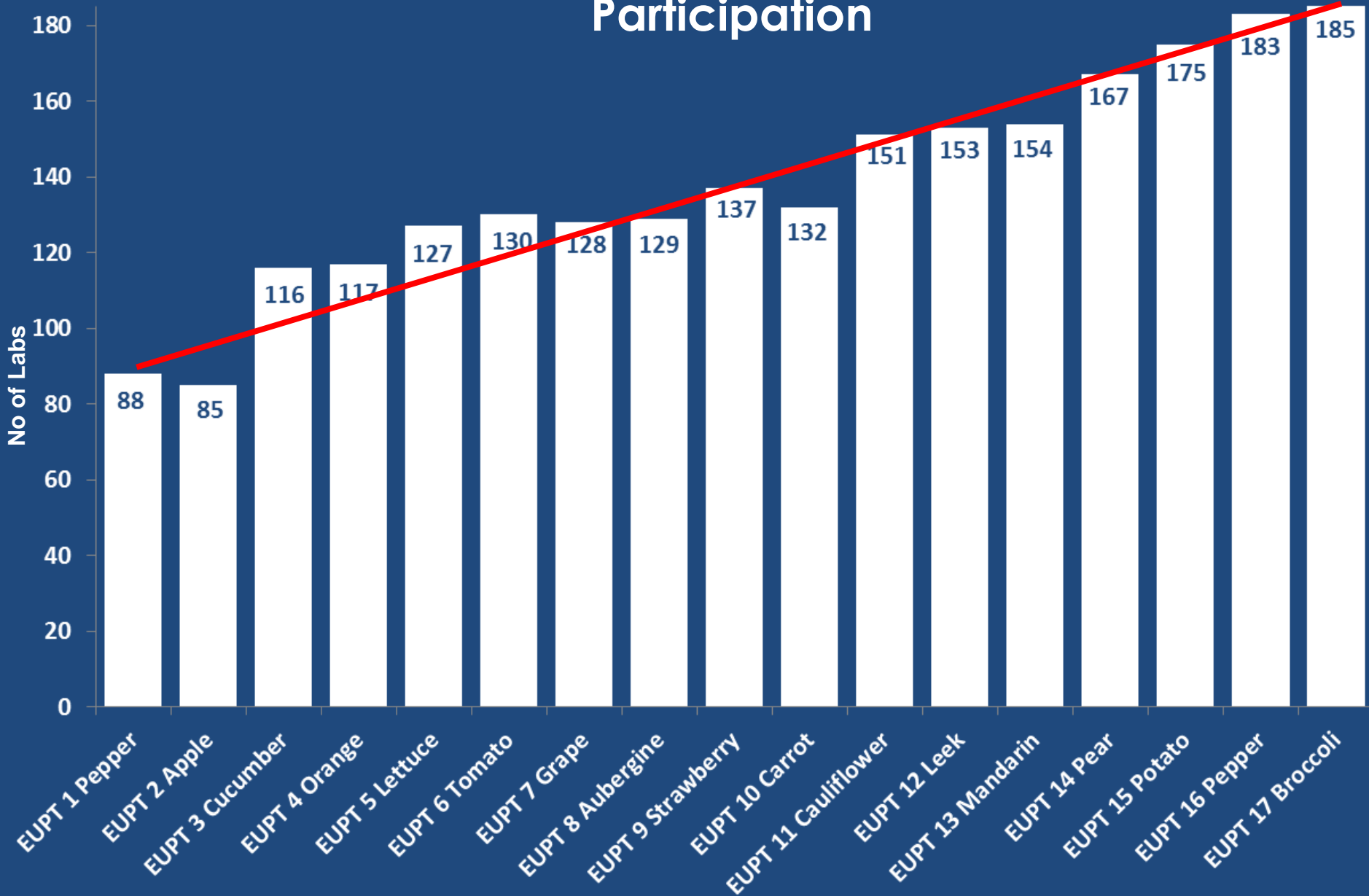
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Participation



20th May 2015

Participation



20th May 2015

Country	No. of Labs	Country	No. of Labs	Country	No. of Labs
Austria	2	Hungary	4	Serbia	2
Belgium	3	Iceland	1	Singapore	1
Brazil	2	Ireland	1	Slovakia	2
Bulgaria	2	Italy	23	Slovenia	3
China	1	Jamaica	1	Spain	32
Costa Rica	1	Kenya	2	Sweden	2
Croatia	6	Latvia	1	Switzerland	2
Cyprus	1	Lebanon	1	Thailand	1
Czech Republic	3	Lithuania	1	The Netherlands	4
Denmark	2	Luxembourg	1	Turkey	1
Egypt	1	Norway	1	United Kingdom	4
Estonia	2	Peru	1	Uruguay	1
Finland	2	Poland	11	Total No. of Labs = 185 Total No. of Non-EU countries Labs = 17 Total No. of Countries = 44 EU countries, EFTA countries , Other countries	
France	7	Portugal	2		
Germany	29	Romania	4		
Greece	10	Saudi Arabia	1		

Participation

Total No. of Labs = 185

EU/EFTA Labs = 168

Other countries Labs = 17

Total No. of Countries = 44

EU/EFTA countries = 30

Other countries = 14

(27 EU + 3 EFTA + 2 Candidate countries + 12 other countries)

183 pesticides

Target List

3-hydroxy-carbofuran
 Acephate
 Acetamiprid
 Acrinathrin
 Aldicarb
 Aldicarb Sulfone
 Aldicarb Sulfoxide
 Azinphos-methyl
 Azoxystrobin
 Benfuracarb
 Benomyl
 Bifenthrin
 Bitertanol
 Boscalid
 Bromopropylate
 Bromuconazole
 Bupirimate
 Buprofezin
 Cadusafos
 Carbaryl
 Carbendazim
 Carbofuran
 Carbosulfan
 Chlorfenapyr
 Chlorfenvinphos
 Chlorobenzilate
 Chlorothalonil
 Chlorpropham (only parent compound)
 Chlorpyrifos
 Chlorpyrifos-methyl
 Clofentezine (only parent compound)
 Clothianidin
 Cyfluthrin (cyfluthrin and its isomers)
 Cypermethrin
 Cyproconazole
 Cyprodinil

Deltamethrin
 Demeton-S-methylsulfon
 Desmethyl-pirimicarb
 Diazinon
 Dichlofluanid (only parent compound)
 Dichlorvos
 Dicloran
 Dicofol
Diethofencarb
 Difenconazole
 Diflubenzuron
 Dimethoate
 Dimethomorph
 Dimethylaminosulfotoluid
Diniconazole
 Diphenylamine
 Endosulfan alpha
 Endosulfan beta
 Endosulfan sulfate
 EPN
 Epoxiconazole
 Ethion
Ethirimol
 Ethoprophos
 Etofenprox
Fenamidone
 Fenamiphos
 Fenamiphos sulfone
 Fenamiphos sulfoxide
 Fenarimol
 Fenazaquin
 Fenbuconazole
 Fenhexamid
 Fenitrothion
 Fenoxycarb
 Fenpropathrin

Fenpropimorph
Fenpyroximate
 Fenthion
 Fenthion oxon
 Fenthion oxon sulfone
 Fenthion oxon sulfoxide
 Fenthion sulfone
 Fenthion sulfoxide
Fenvalerate and Esfenvalerate
 Fipronil (only parent compound)
 Fludioxonil
 Flufenoxuron
 Fluopicolide
Fluopyram
 Fluquinconazole
 Flusilazole
 Flutolanil
 Flutriafol
 Fosthiazate
 Hexaconazole
 Hexythiazox
 Imazalil
 Imidacloprid
 Indoxacarb (Indoxacarb)
 Iprodione
 Iprovalicarb
Isocarbophos
 Isofenphos-methyl
Isoprothiolane
 Kresoxim-methyl
 Lambda-Cyhalothrin
 Linuron
 Lufenuron
 Malaoxon
 Malathion

Mandipropamid
 Mepanipyrim (only parent compound)
 Metaflumizone
 Metalaxyl and metalaxyl-M
 Metconazole
 Methamidophos
 Methidathion
 Methiocarb
 Methiocarb sulfone
 Methiocarb sulfoxide
 Methomyl
 Methoxyfenozide
 Monocrotophos
 Myclobutanil
 Omethoate
 Orthophenylphenol
 Oxadixyl
 Oxamyl
 Oxydemeton-methyl
 Paclobutrazole
 Paraoxon-methyl
 Parathion-ethyl
 Parathion-methyl
 Penconazole
 Pencycuron
 Pendimethalin
Permethrin
 Phenthoate
 Phosalone
 Phosmet
 Phosmet oxon
 Phoxim
 Pirimicarb
 Pirimiphos-methyl
 Prochloraz (only parent compound)
 Procymidone

Profenofos
 Propargite
 Propiconazole
 Propyzamide
 Prothioconazole (Prothioconazole-desthio)
 Prothiofos
 Pyraclostrobin
 Pyridaben
 Pyrimethanil
 Pyriproxyfen
 Quinoxifen
 Spinosad (sum of spinosyn A and spinosyn B)
 Spirodiclofen
 Spiroxamine
 Tau-Fluvalinate
 Tebuconazole
 Tebufenozide
 Tebufenpyrad
 Teflubenzuron
 Tefluthrin
Terbuthylazine
 Tetraconazole
 Tetradifon
 Thiabendazole
 Thiacloprid
 Thiamethoxam
 Thiodicarb
 Thiophanate-methyl
 Tolclofos-methyl
 Tolyfluanid
 Triadimefon
 Triadimenol
 Triazophos
 Trichlorfon (only parent compound)
 Trifloxystrobin
 Triflumuron
 Trifluralin
 Triticonazole
 Vinclozolin (only parent compound)
 Zoxamide

12 New compounds

Pesticides used for the treatment

Bupirimate	Pendimetalin
Carbendazim	Permethrin*
Diazinon	Spinosad
Difenoconazole	Thiabendazole
Diflubenzuron	Trifloxystrobin
Methoxyfenozide	Total: 11

ALL INCLUDED IN MACP

COMMISSION IMPLEMENTING REGULATION (EU)
No 400/2014 of 22 April 2014
concerning a coordinated multiannual control programme of the Union for 2015, 2016 and 2017 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 .

*** New in the target list**

Calendar

ACTIVITY	DATE
- Publishing the Target Pesticide List, Calendar and Matrix on the Web page.	15 th December 2014
- Receiving Application Form from invited laboratories.	12 th Jan-13 th Feb 2015
- Specific Protocol published on the Web site.	23 rd Feb 2015 at the latest
- Deadline for receiving Laboratory scope: Form 0	23 rd Feb-6 th March 2015
- Sample distribution.	16 th March 2015
- Deadline for receiving sample acceptance: Form 1	20 th March 2015
- Deadline for receiving results: Forms 2, 3 and 4	13 th April 2015
- Filling in Form 5	20 th -24 th April 2015
- Preliminary Report: only results, no statistical treatment.	May 2015
- Final Report distributed to the Laboratories.	December 2015

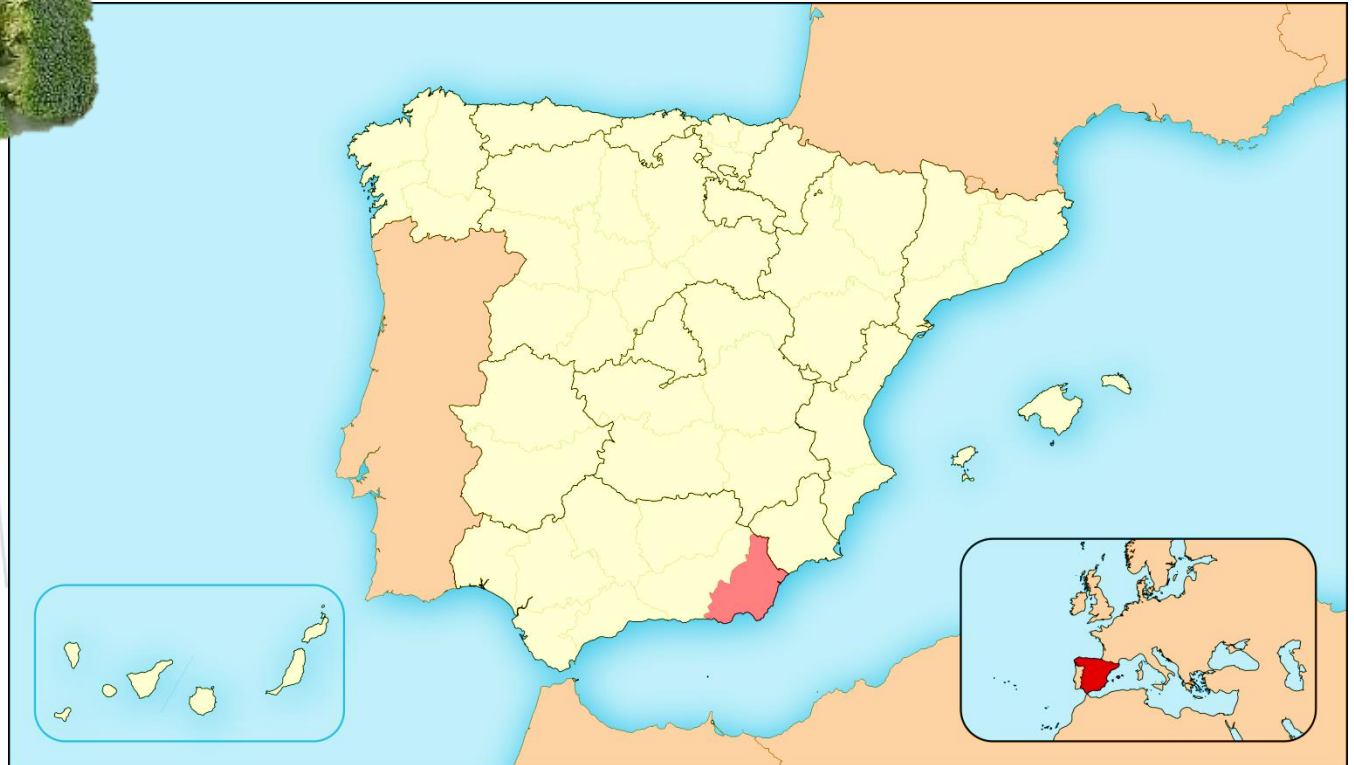
EUPT-FV-17

European Proficiency Test FV-17

Broccoli



Organic broccolis were grown in a greenhouse in Almería, Spain.





Before harvest, the broccolis were treated with commercial formulations



Commercial products used to treat the sample

Pesticide	Type	Commercial Product
Bupirimate	Commercial Formulation	Nimrod Quattro
Carbendazim	Commercial Formulation	Bengu 50 Flow
Diazinon	Commercial Formulation	Zoogama-D
Difenoconazole	Commercial Formulation	Core
Diflubenzuron	Commercial Formulation	Dimilin 25
Methoxyfenozide	Commercial Formulation	Runner
Pendimetalin	Commercial Formulation	Stonp LE
Permethrin	Commercial Formulation	Defendog
Spinosad	Commercial Formulation	Spintor 480 SG
Thiabendazole	Commercial Formulation	Textar 60
Trifloxystrobin	Commercial Formulation	Flint

Preparation of the test item





Homogeneity

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

Stability

1st Analysis - prior to the sample shipment

2nd Analysis - after the deadline for reporting results

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

All the pesticides passed the homogeneity test

All the pesticides passed the stability test

Results



Assigned values

Pesticides	Robust mean (mg/kg)
Spinosad	0.051
Pendimethalin	0.062
Diazinon	0.071
Bupirimate	0.16
Permethrin	0.17
Diflubenzuron	0.32
Methoxyfenozide	0.35
Trifloxystrobin	0.47
Carbendazim	0.51
Difenocoazole	0.53
Thiabendazole	1.90

0.051-0.100 mg/kg

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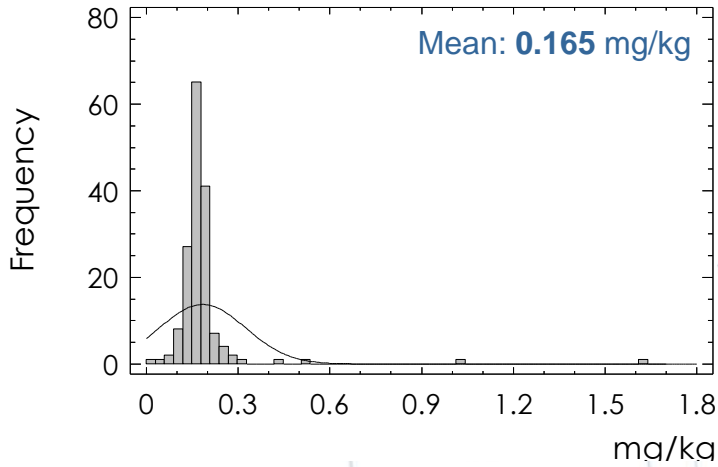
0.100-0.500 mg/kg

> 0.500 mg/kg

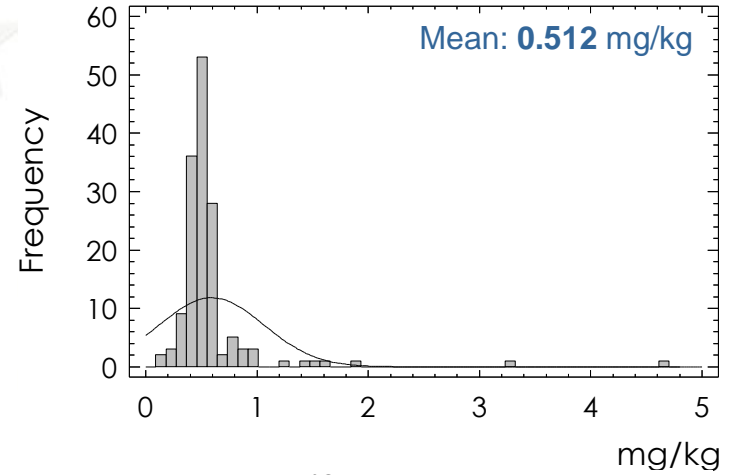
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Distribution of data-Histograms

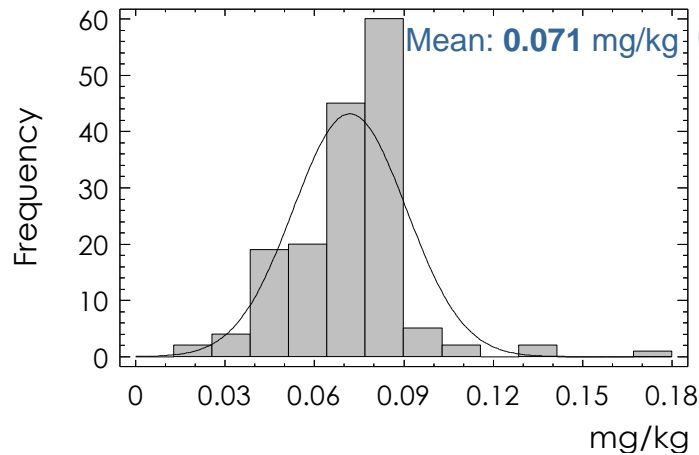
Bupirimate



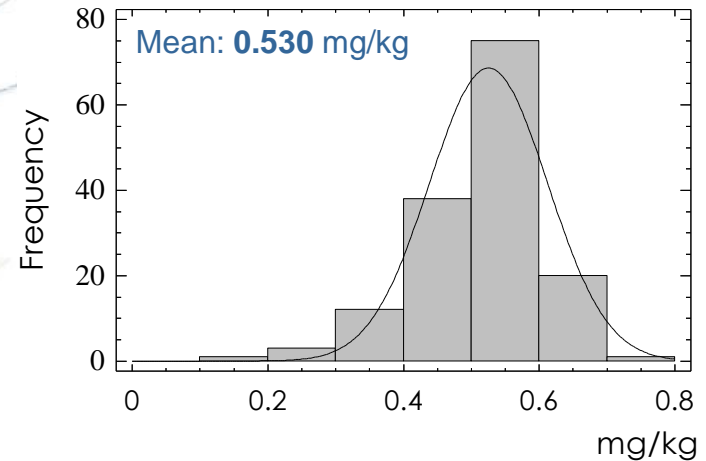
Carbendazim



Diazinon

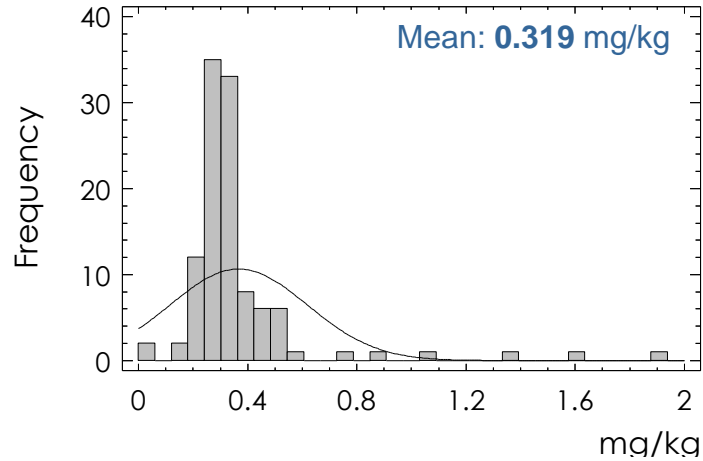


Difenocoazole

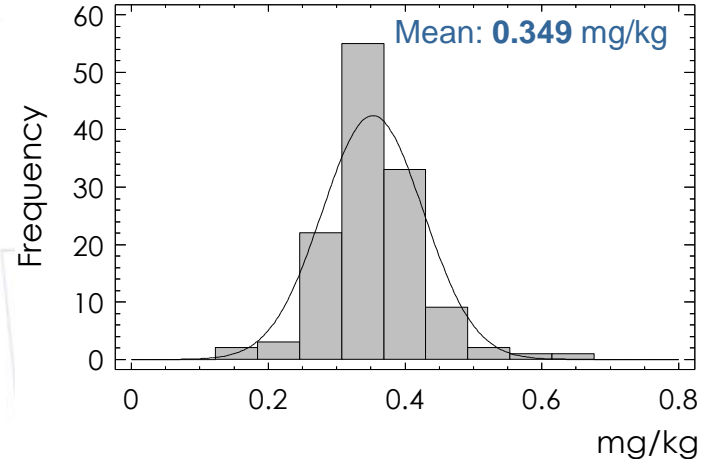


Distribution of data-Histograms

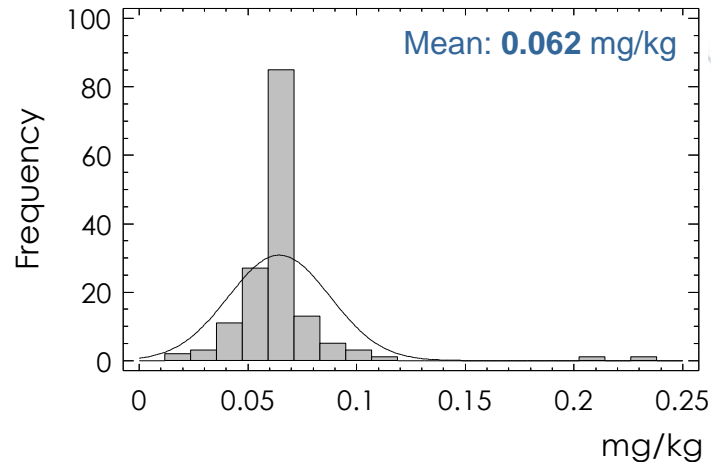
Diflubenzuron



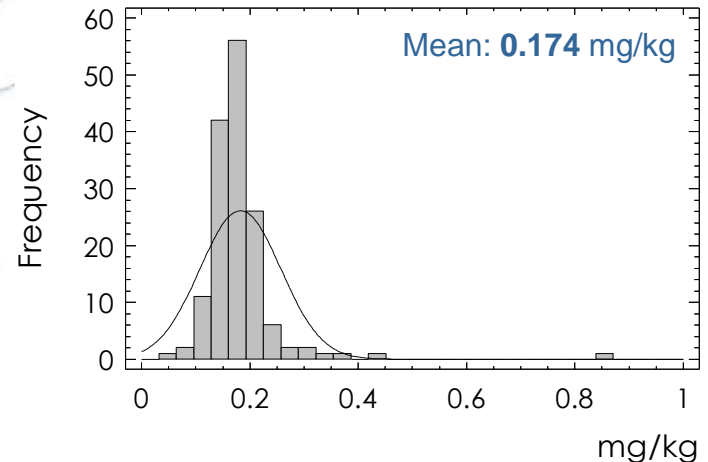
Methoxyfenozide



Pendimethalin

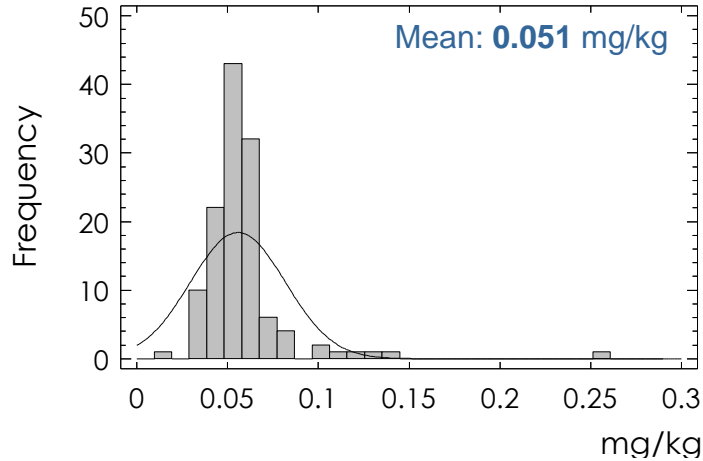


Permethrin

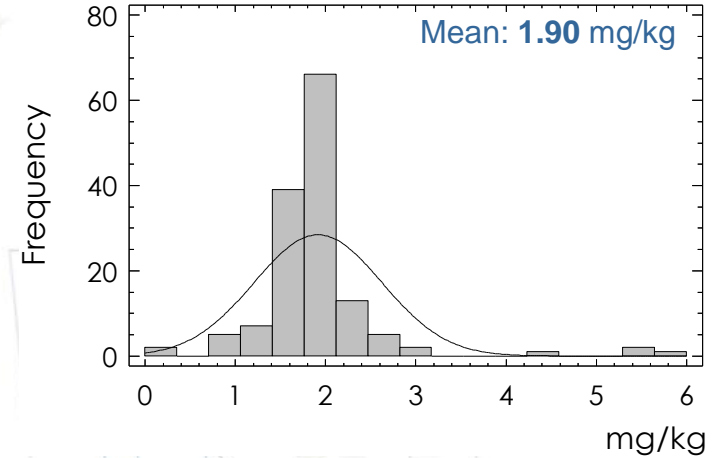


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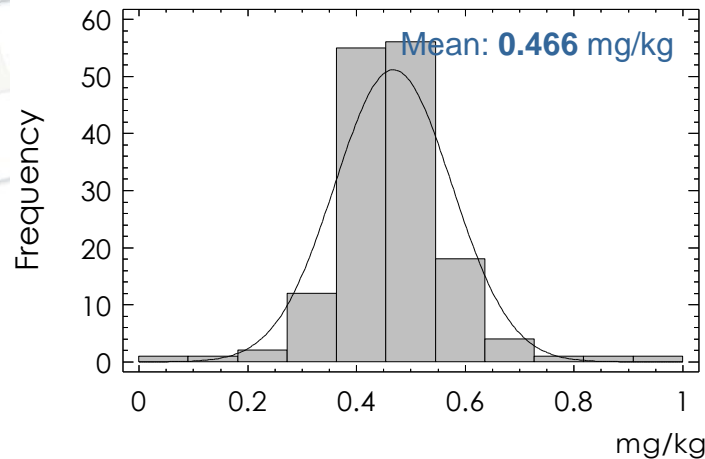
Spinosad



Thiabendazole



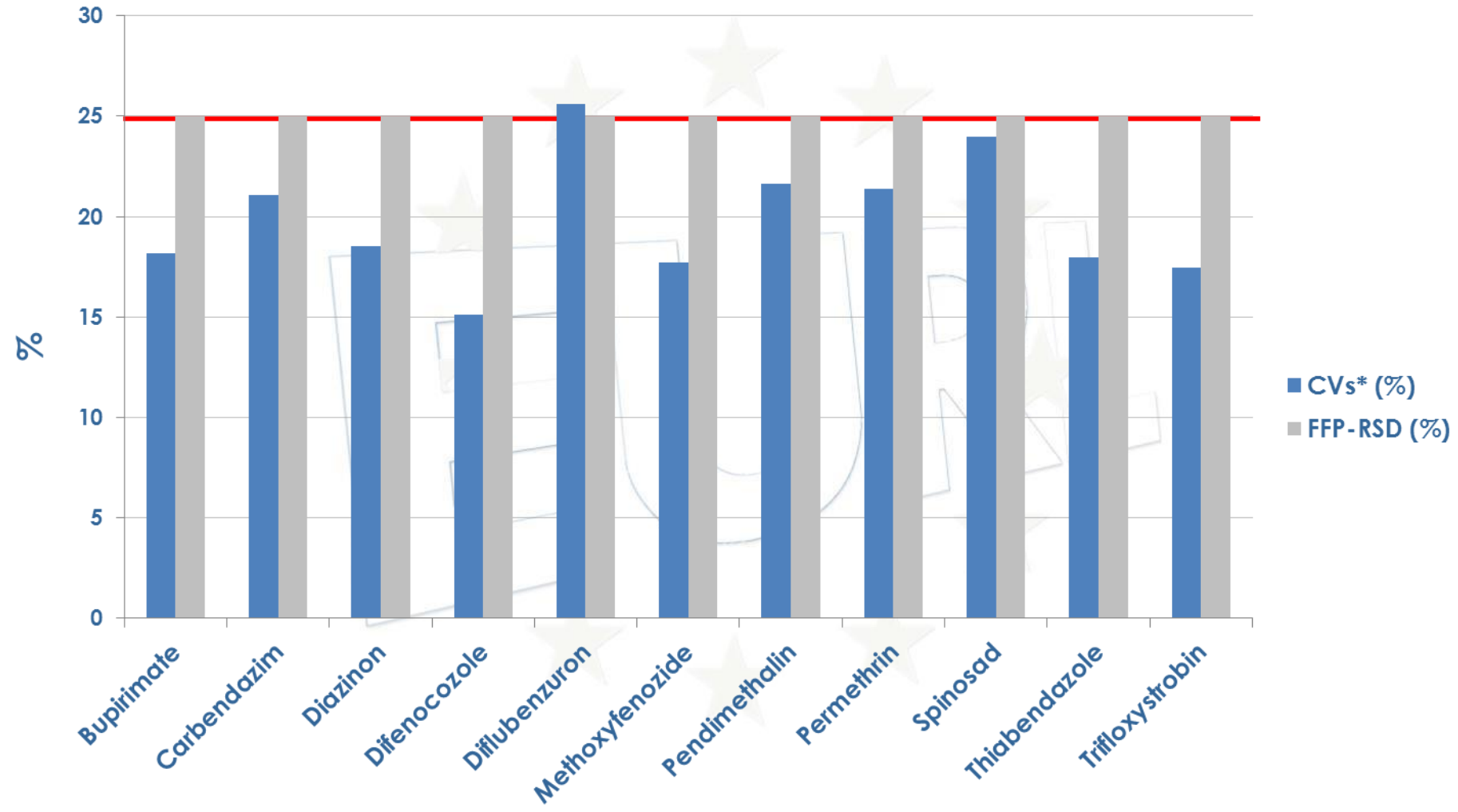
Trifloxystrobin





Pesticides	MRRL (mg/kg)	Robust mean (mg/kg)	CV (%)
Bupirimate	0.01	0.165	18.2
Carbendazim	0.01	0.512	21.1
Diazinon	0.01	0.071	18.5
Difenocoazole	0.01	0.530	15.1
Diflubenzuron	0.01	0.319	25.6
Methoxyfenozide	0.01	0.349	17.7
Pendimethalin	0.01	0.062	21.6
Permethrin	0.01	0.174	21.4
Spinosad (sum of spinosyn A a spinosyn D. expr. as spinosad)	0.01	0.051	24.0
Thiabendazole	0.01	1.900	18.0
Trifloxystrobin	0.01	0.466	17.5

Dispersion of Results

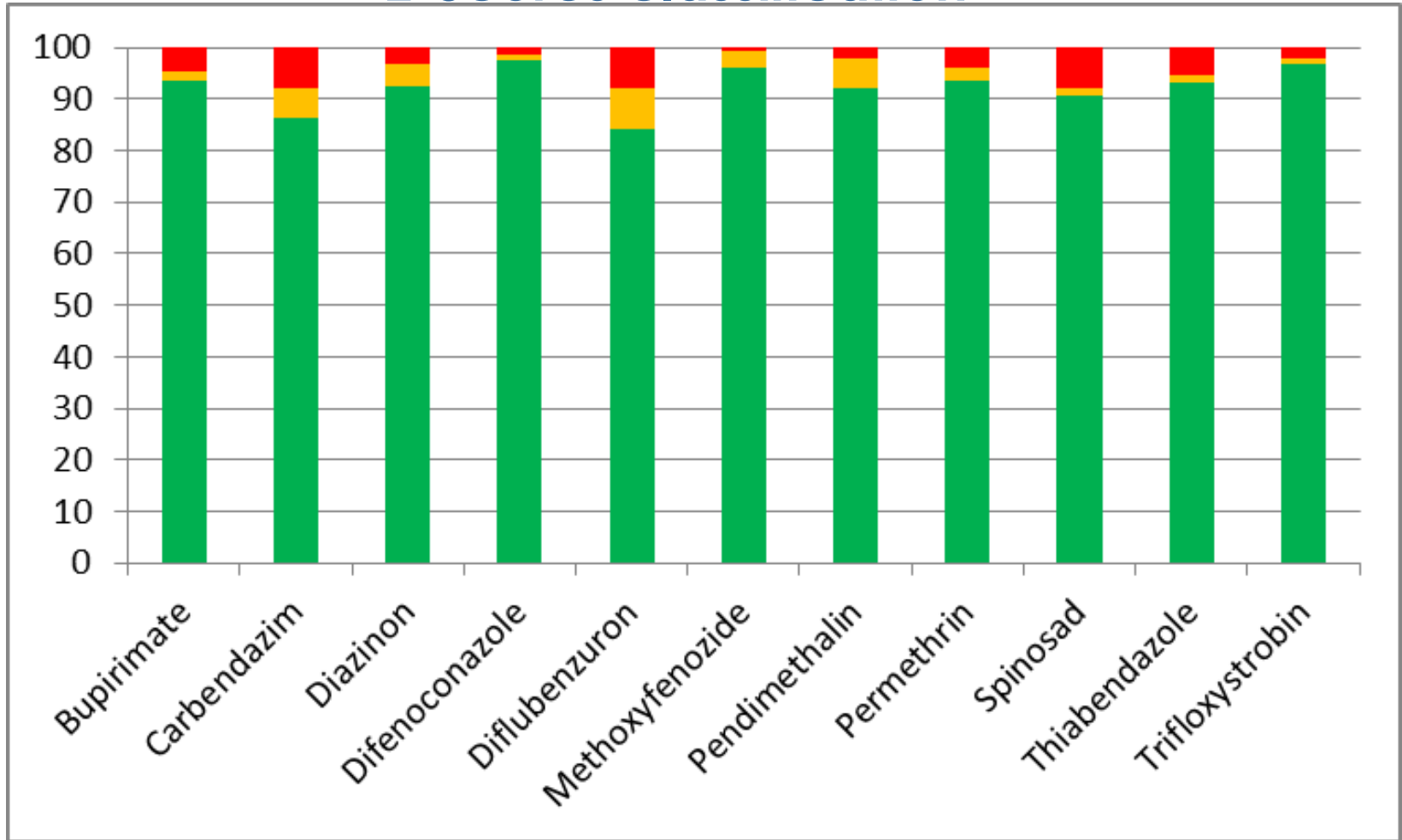




Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results (out of 165)
Bupirimate	154	0	11	93
Carbendazim	140	0	25	85
Diazinon	160	2	3	97
Difenocoazole	151	1	13	92
Diflubenzuron	111	2	52	67
Methoxyfenozide	128	0	37	78
Pendimethalin	152	1	12	92
Permethrin	152	1	12	92
Spinosad	125	2	38	76
Thiabendazole	143	2	20	87
Trifloxystrobin	152	0	13	92

z-Scores

z-Scores classification



Acceptable

Questionable

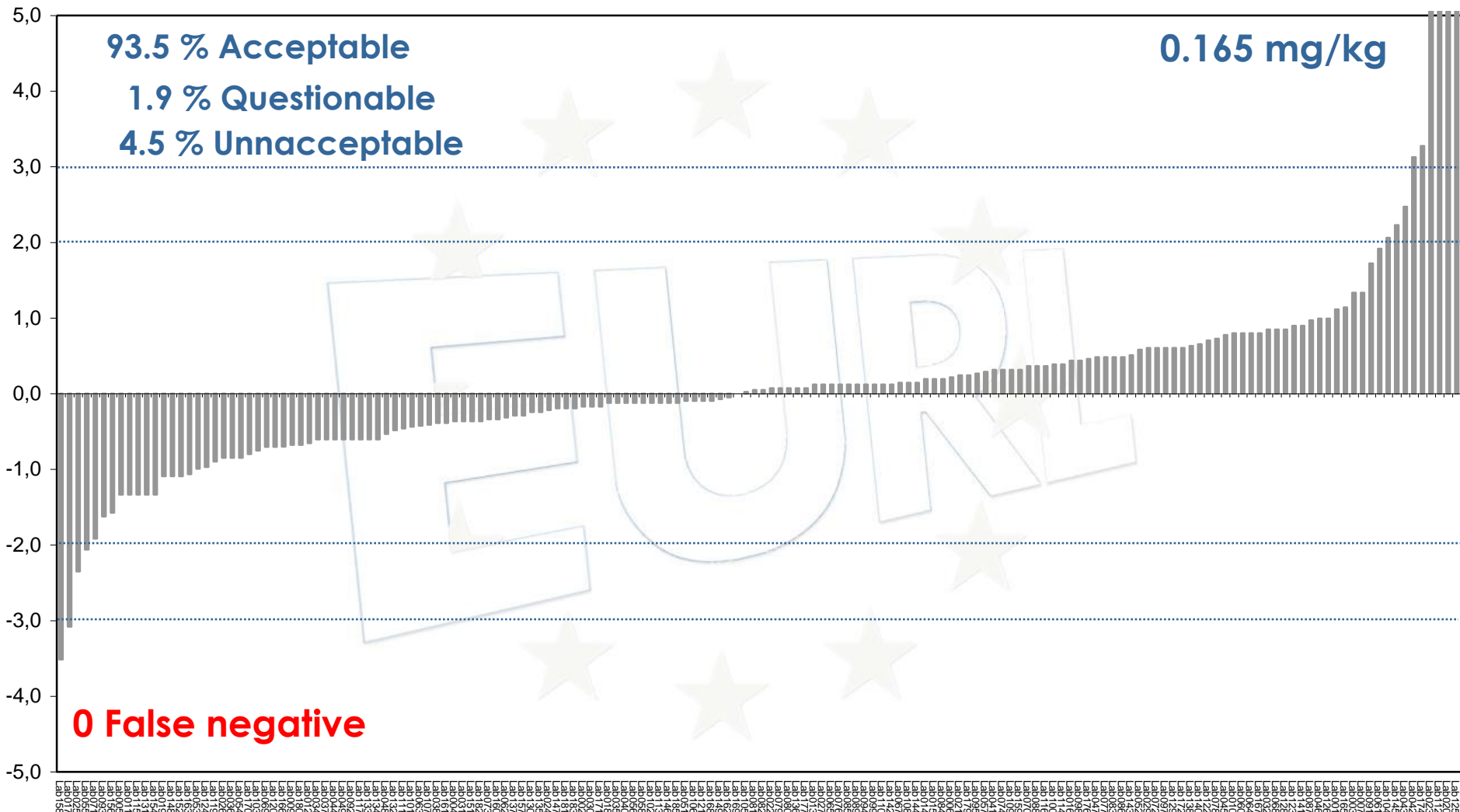
Unacceptable

EU/EFTA Laboratories

Pesticides	Robust Mean (mg/kg)	% Acceptable z-scores	% Questionable z-scores	% Unacceptable z-scores
Bupirimate	0.165	93,5	1,9	4,5
Carbendazim	0.512	86,4	5,7	7,9
Diazinon	0.071	92,6	4,3	3,1
Difenocoazole	0.530	97,4	1,3	1,3
Diflubenzuron	0.319	84,1	8,0	8,0
Methoxyfenozide	0.349	96,1	3,1	0,8
Pendimethalin	0.062	92,2	5,9	2,0
Permethrin	0.174	93,5	2,6	3,9
Spinosad (sum of spinosyn A a spinosyn D. expr. as spinosad)	0.051	90,6	1,6	7,9
Thiabendazole	1.900	93,1	1,4	5,5
Trifloxystrobin	0.466	96,7	1,3	2,0

Bupirimate

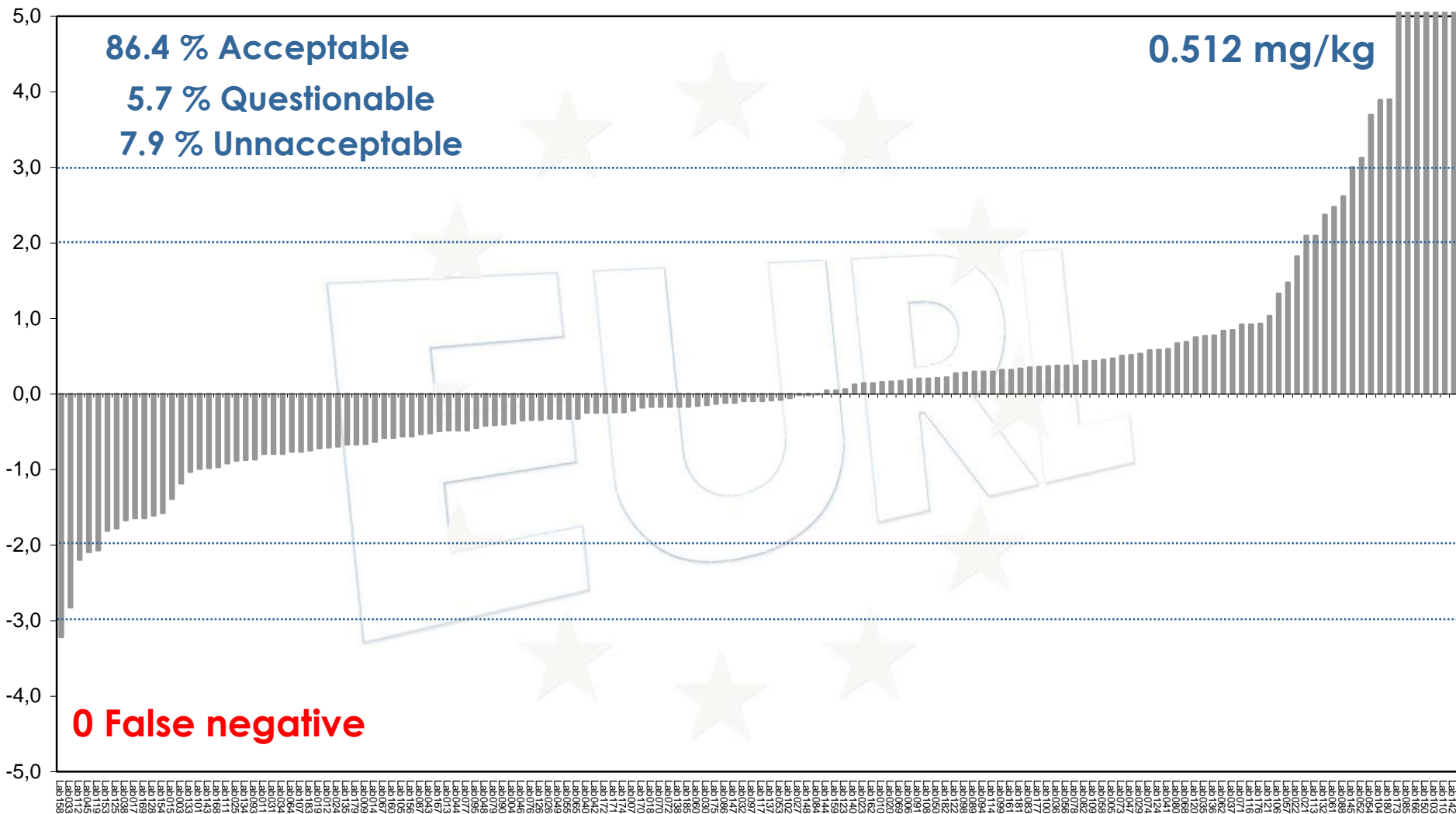
EU/EFTA Laboratories





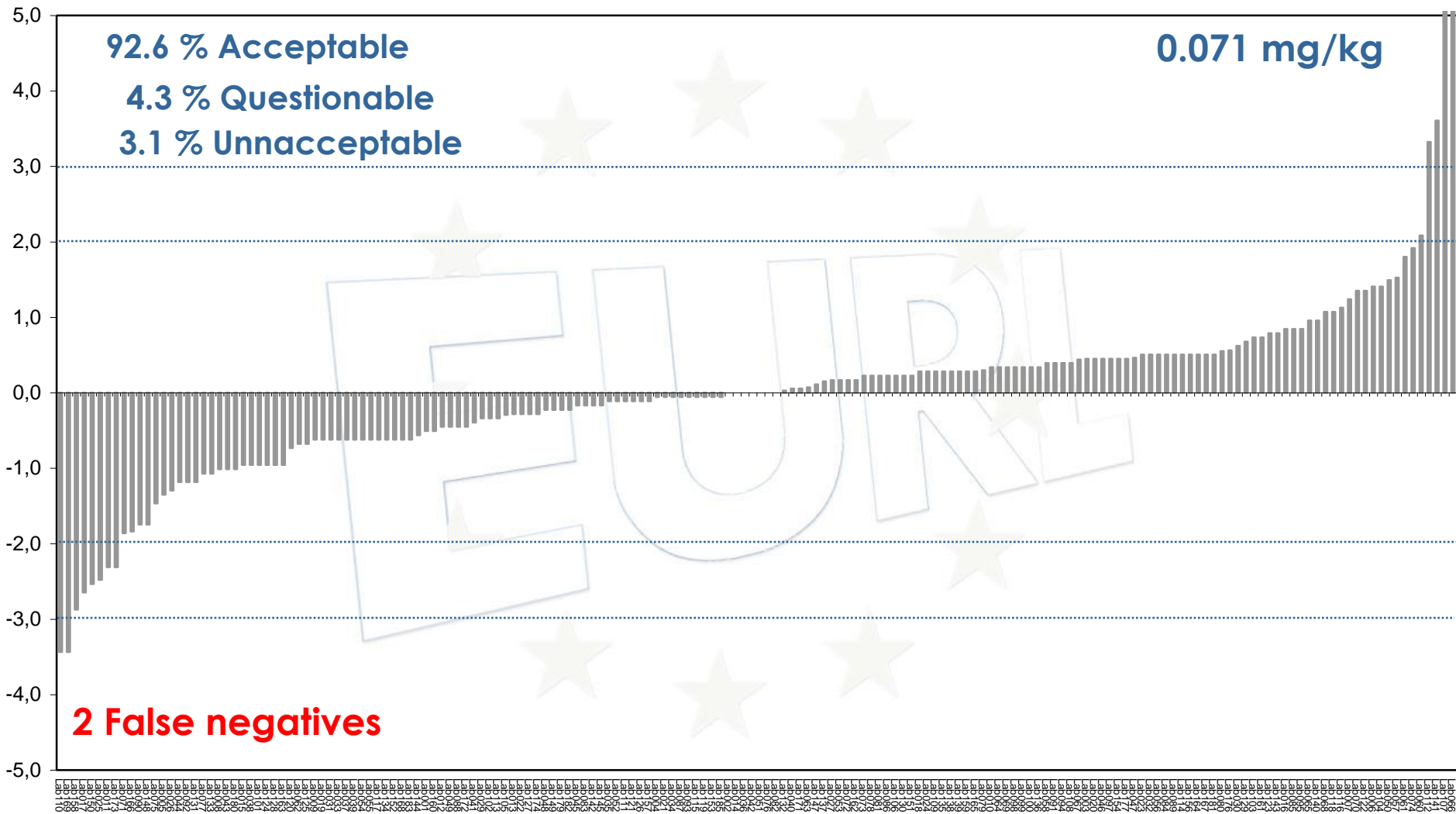
Carbendazim

EU/EFTA Laboratories



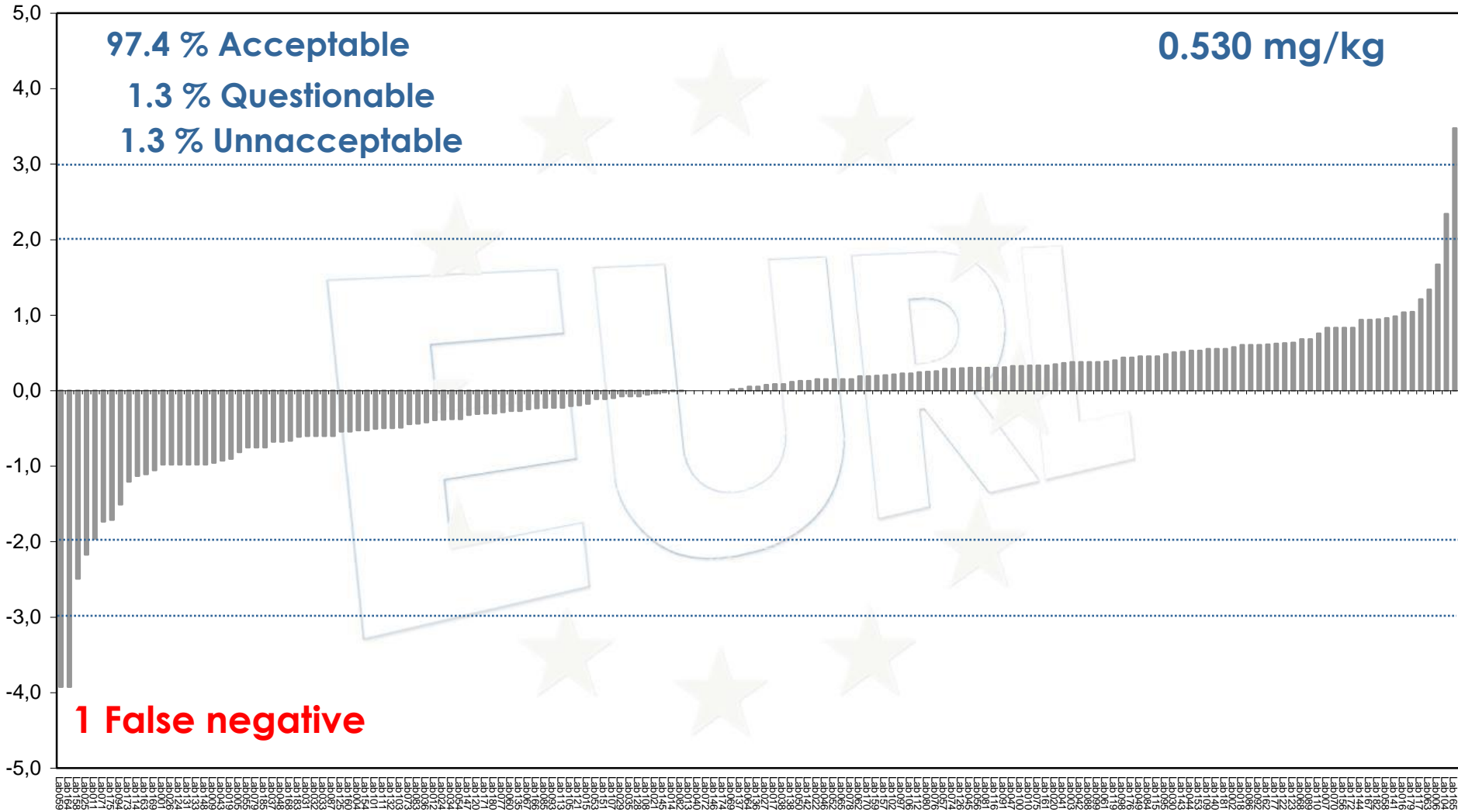
Diazinon

EU/EFTA Laboratories



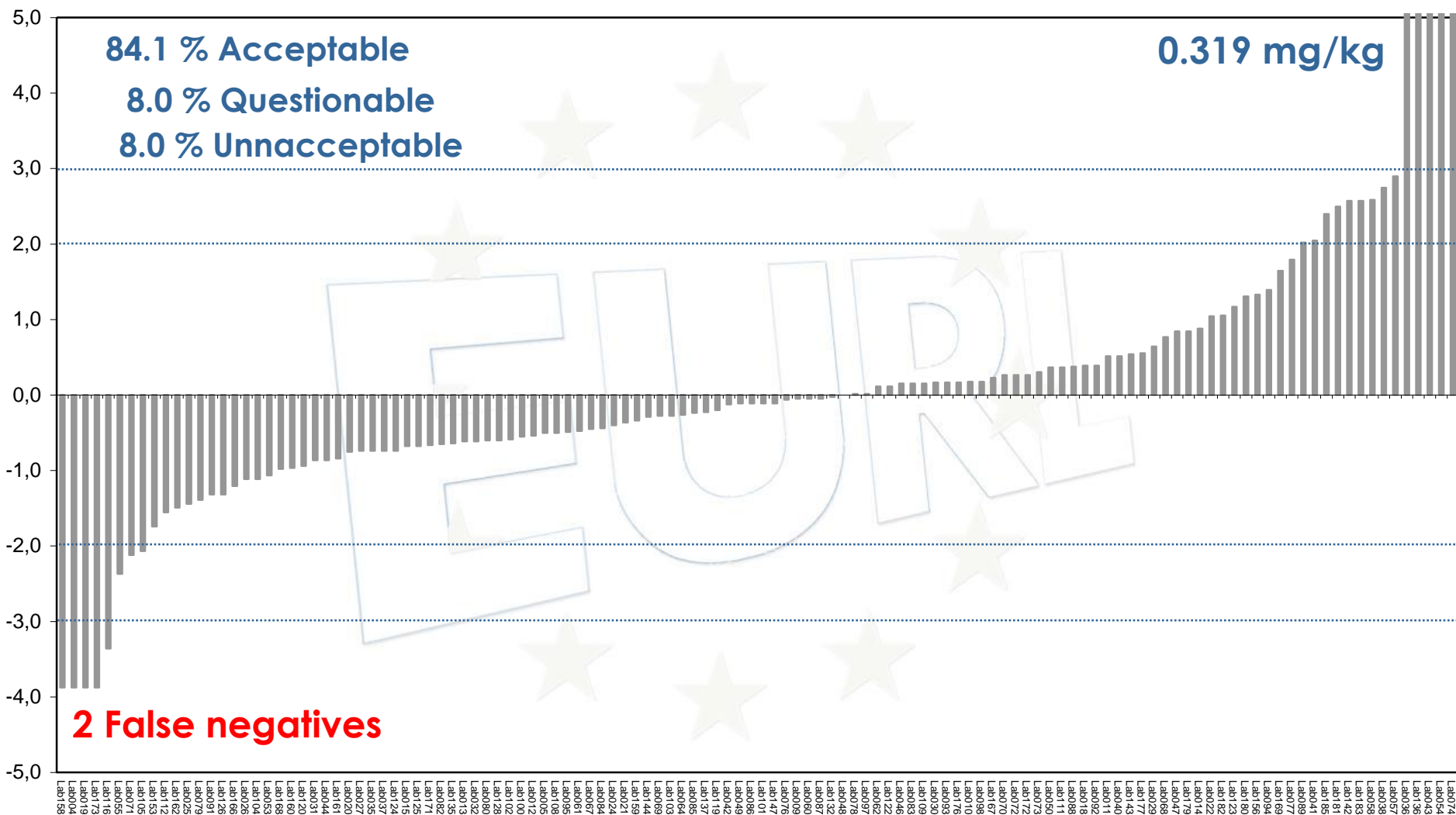
Difenoconazole

EU/EFTA Laboratories



Difflubenzuron

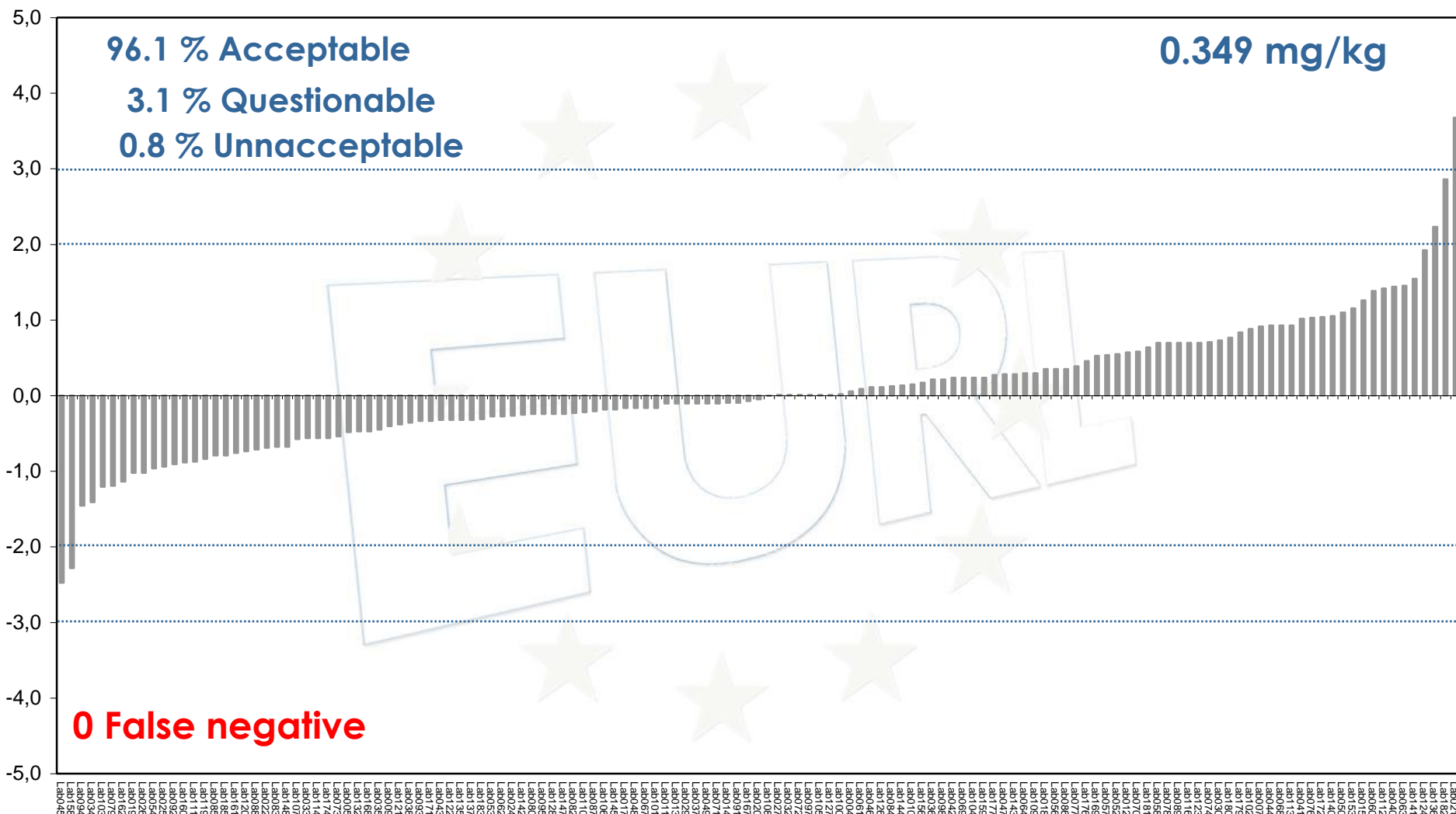
EU/EFTA Laboratories





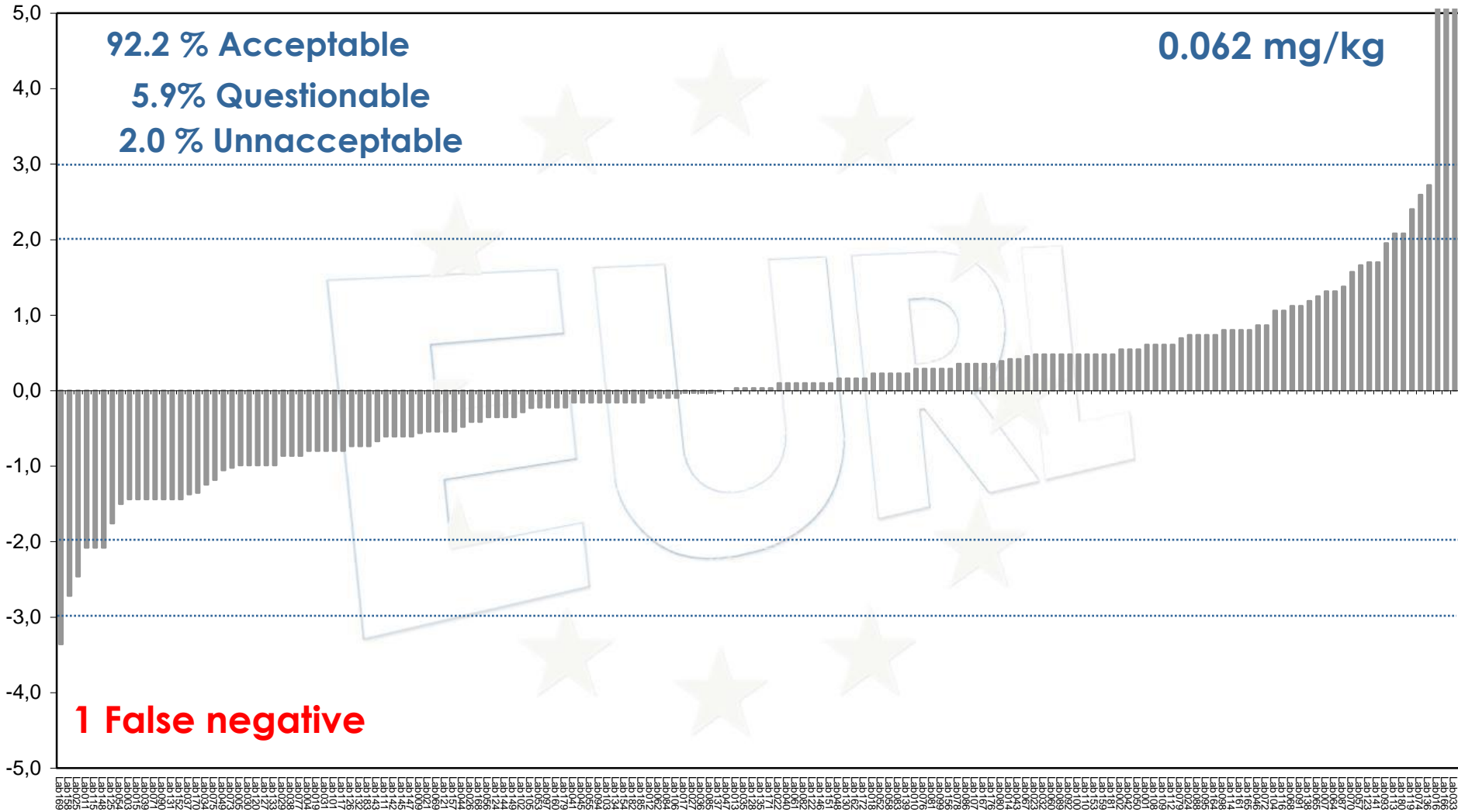
Methoxyfenozide

EU/EFTA Laboratories



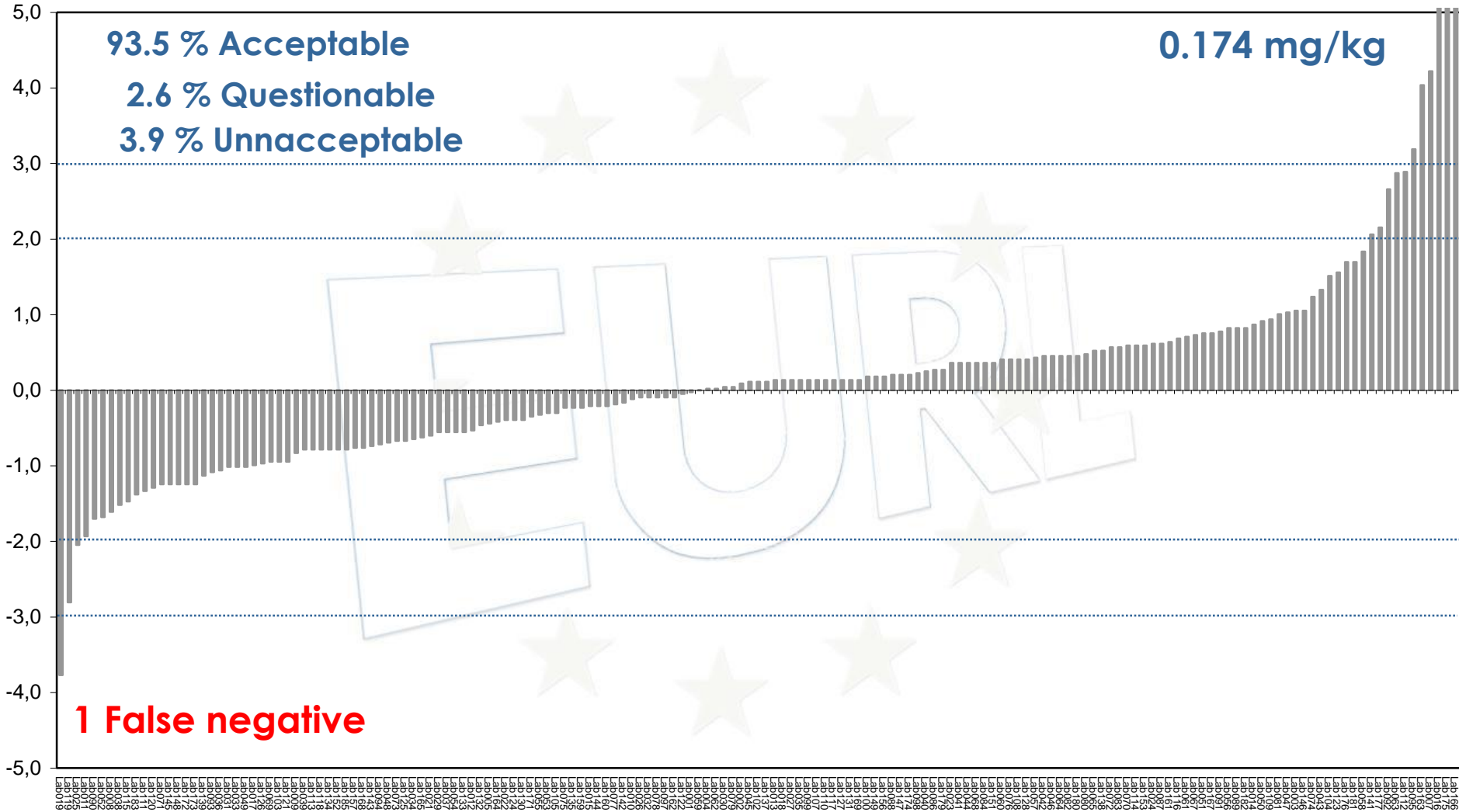
Pendimethalin

EU/EFTA Laboratories



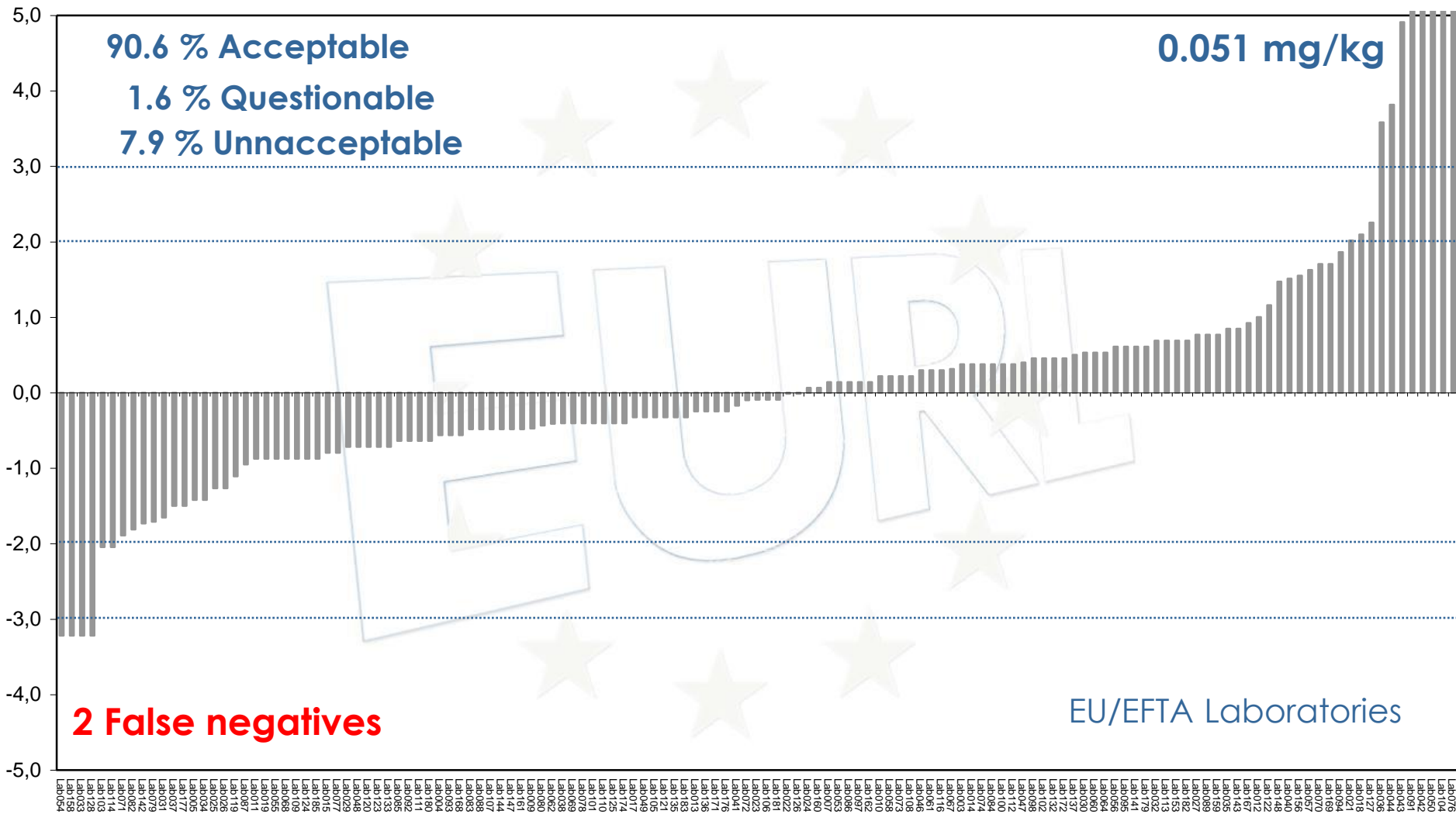
Permethrin

EU/EFTA Laboratories



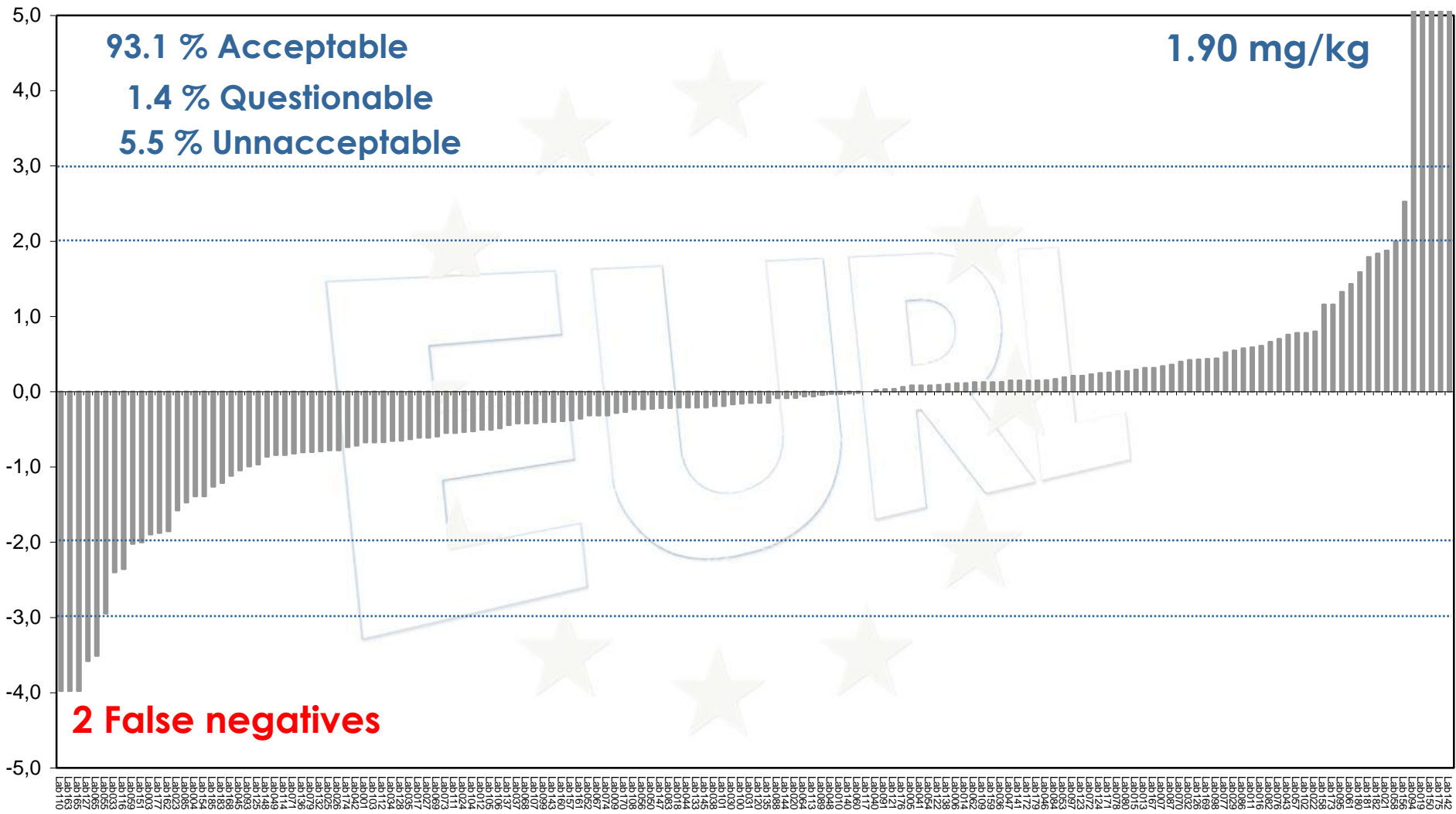


Spinosad (sum of spinosyn A a spinosyn D. expr. as spinosad)



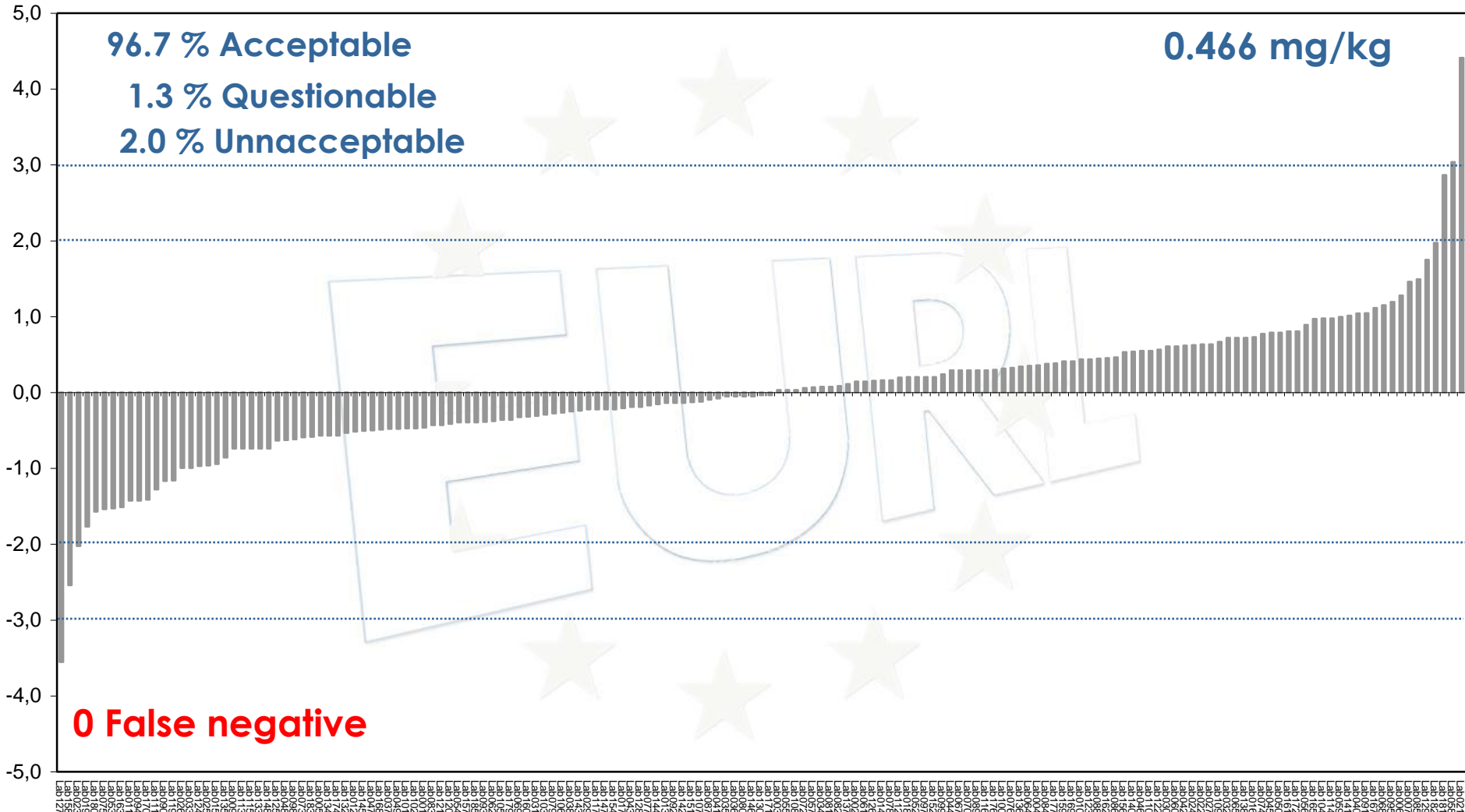
Thiabendazole

EU/EFTA Laboratories



Trifloxystrobin

EU/EFTA Laboratories



Combined z-Scores

Average of Squared z-Scores

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

$AZ^2 \leq 2.0$	Good
$2.0 < AZ^2 < 3.0$	Satisfactory
$AZ^2 \geq 3.0$	Unsatisfactory

EU/EFTA Laboratories

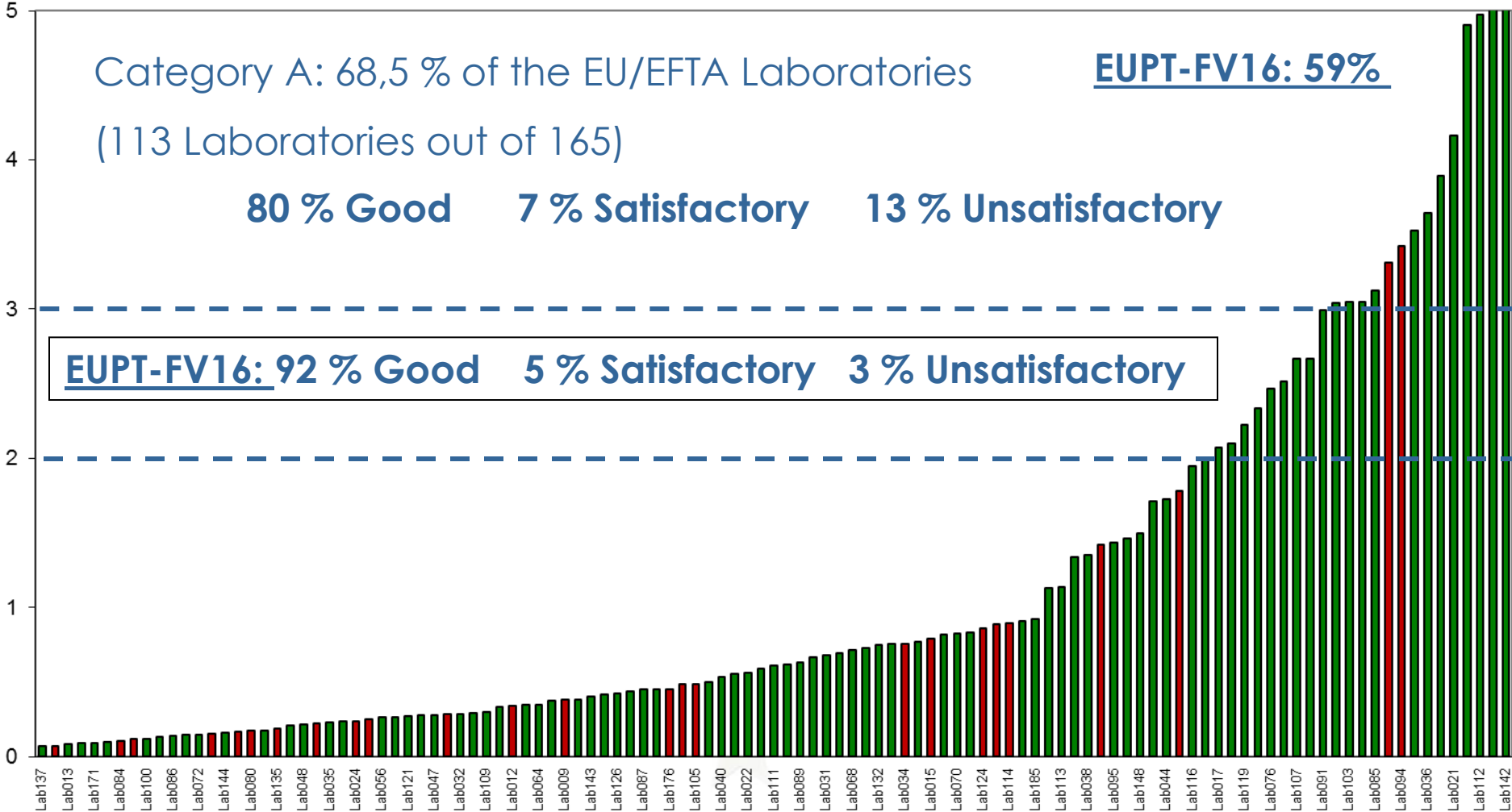
■ NRLs ■ OfLs

Category A: 68,5 % of the EU/EFTA Laboratories
(113 Laboratories out of 165)

EUPT-FV16: 59%

80 % Good 7 % Satisfactory 13 % Unsatisfactory

EUPT-FV16: 92 % Good 5 % Satisfactory 3 % Unsatisfactory



Preliminary False Positives

6 different laboratories from EU/EFTA countries reported 6 pesticides as false positives

Preliminary False Positives

Laboratory Code	Pesticide	Concentration (mg/kg)	Determination Technique	RL (mg/Kg)	MRRL (mg/Kg)
Lab027	Bromopropylate	0.014	GC-MS/MS	0.01	0.01
Lab098	Bromopropylate	0.021	GC-MS	0.01	0.01
Lab051	Bromopropylate	0.039	GC-MS	0.01	0.01
Lab173	Carbaryl	0.020	GC-MS/MS	0.01	0.01
Lab158	Fenpropathrin	0.020	GC-MS	0.01	0.01
Lab164	Kresoxim-methyl	0.015	GC-MS/MS	0.01	0.01
Lab173	Orthophenylphenol	2.95	HPLC-UV	0.1	0.01
Lab173	Tetradifon	0.014	GC-MS/MS	0.01	0.01

Preliminary False Positives

GC-MS or GC-MS/MS

Bromopropylate

Carbaryl

Fenpropathrin

Kresoxim-methyl

Tetradifon

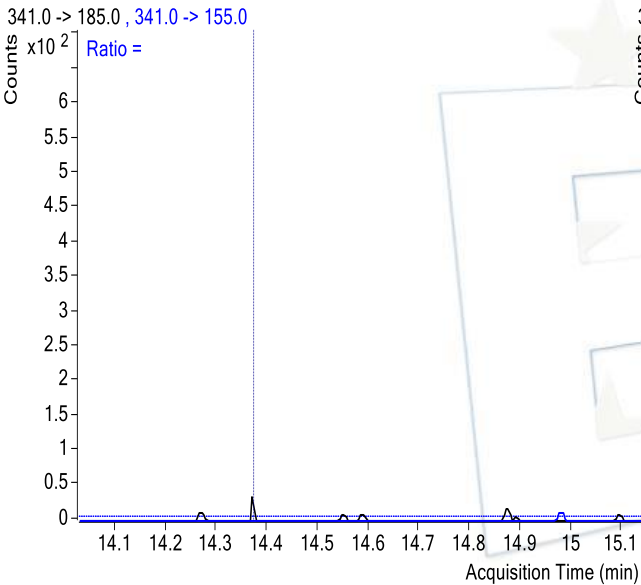
HPLC-UV

Orthophenylphenol

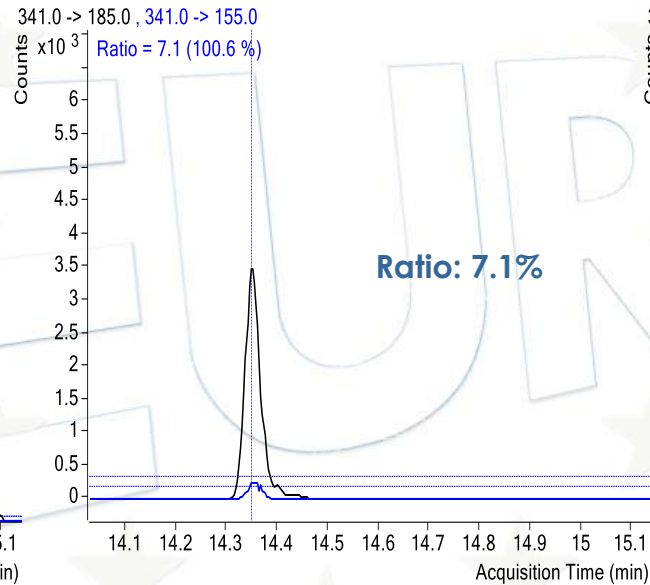


Bromopropylate in FV17

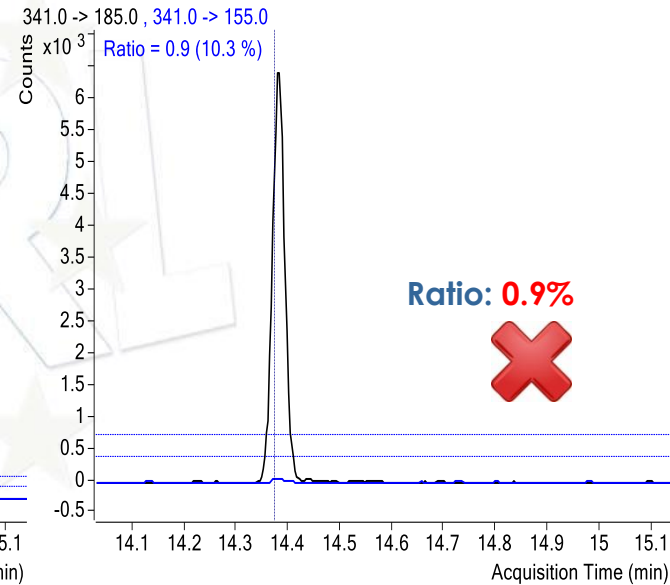
EUPT-FV17 Blank



10 µg/kg Std in Broccoli



EUPT-FV17 Sample



MRM1: 341 > 185
MRM2: 341 > 155

Bupirimate Carbendazim Diazinon
Difenoconazole Diflubenzuron
Methoxyfenozide Pendimetalin
Permethrin Spinosad
Thiabendazole Trifloxystrobin



Cyprodinil

Ethirimol

Fludioxonil

Concentrations below MRRL (0,01 mg/kg)



EURL

European Union Reference Laboratory for Pesticide Residues in Fruits & Vegetables

EUPT-FV-SM07

European Proficiency Test FV-SM07

**EUROPEAN COMMISSION PROFICIENCY TEST
FOR PESTICIDE RESIDUES IN FRUITS AND
VEGETABLES**

SCREENING METHODS 07

PRELIMINARY RESULTS WEBINAR



European
Commission

EURL



EUROPEAN UNION REFERENCE LABORATORIES

**OCTAVIO MALATO
AND CARMEN FERRER**



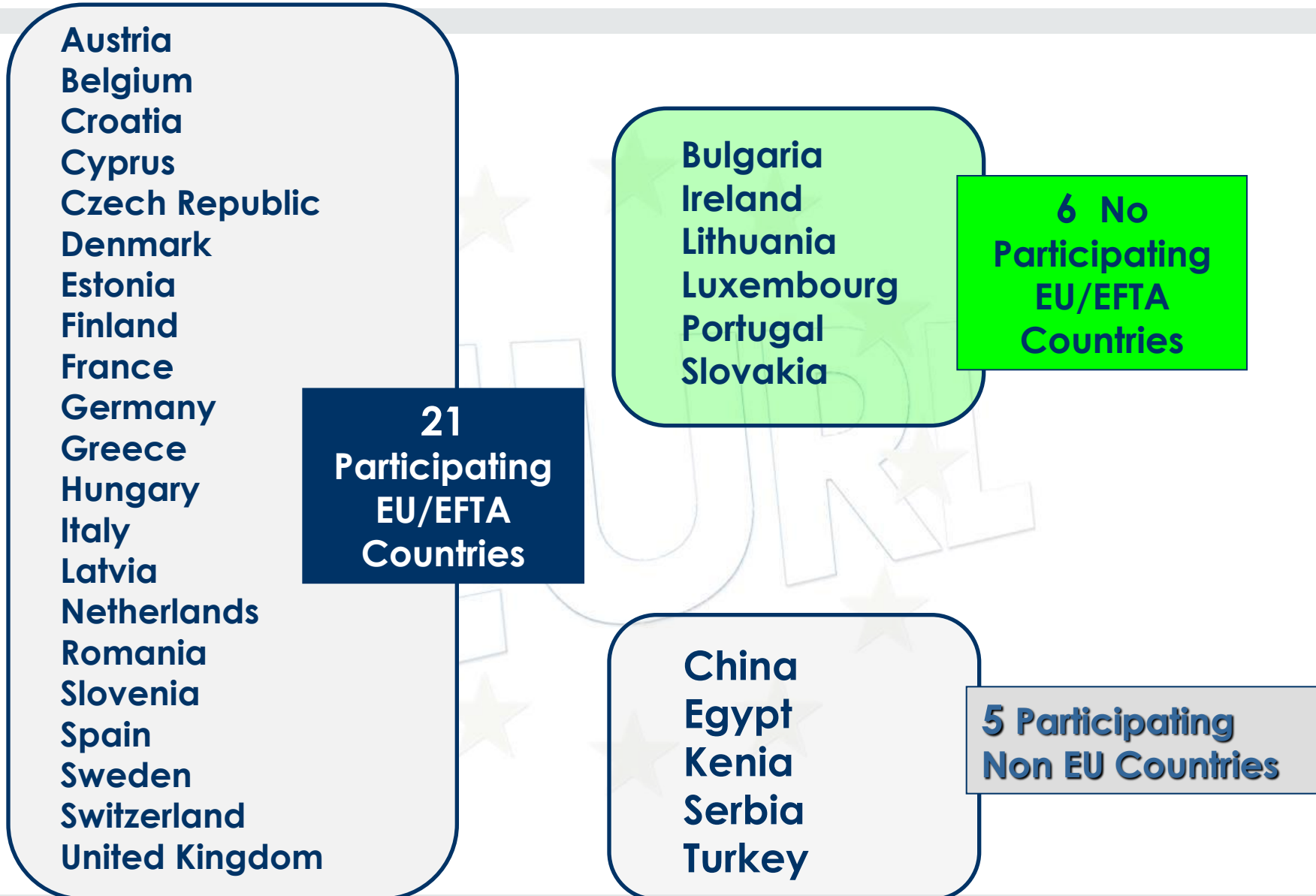
ACTIVITY	DATE
Publishing the Calendar and Matrix on the Web page.	17th December 2014
Receiving Application Form from invited laboratories.	12th Jan-13th Feb 2015
Specific Protocol published on the Web site.	23 rd Feb 2015 at the latest
Sample distribution.	16th March 2015
Deadline for receiving results: Fill in "Results Page"	72 hours after receiving the sample
Preliminary Report: only results, no statistical treatment.	April 2015
Final Report distributed to the Laboratories.	December 2015

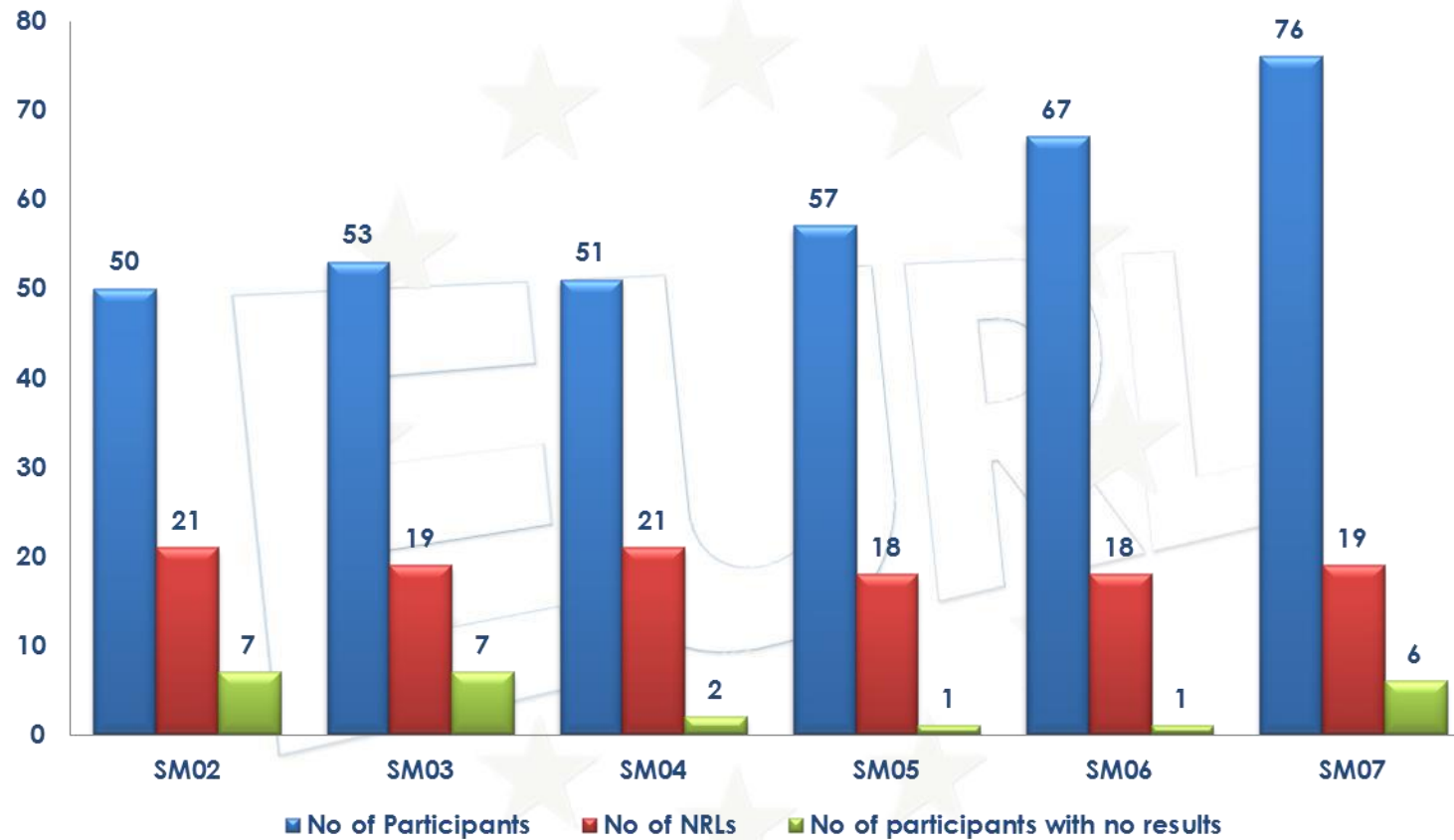
70 Participants

EUPT-FV-SM07- Participants with reported results

18 NRLs*

COUNTRY	No.	COUNTRY	No.
Austria	1*	Italy	1*+8
Belgium	2	Kenia	1
China	1	Latvia	1*
Croatia	1	Poland	2
Cyprus	1*	Romania	1*+1
Czech Republic	1*+2	Serbia	1
Denmark	1*	Slovenia	1*
Egypt	1	Spain	13
Estonia	1*	Sweden	1*+1
Finland	1*	Switzerland	1
France	1*+4	The Netherlands	1*+1
Germany	1*+8	Turkey	1
Greece	1*+1	UK	1*
Hungary	1*+3		







Pesticides used for the treatment

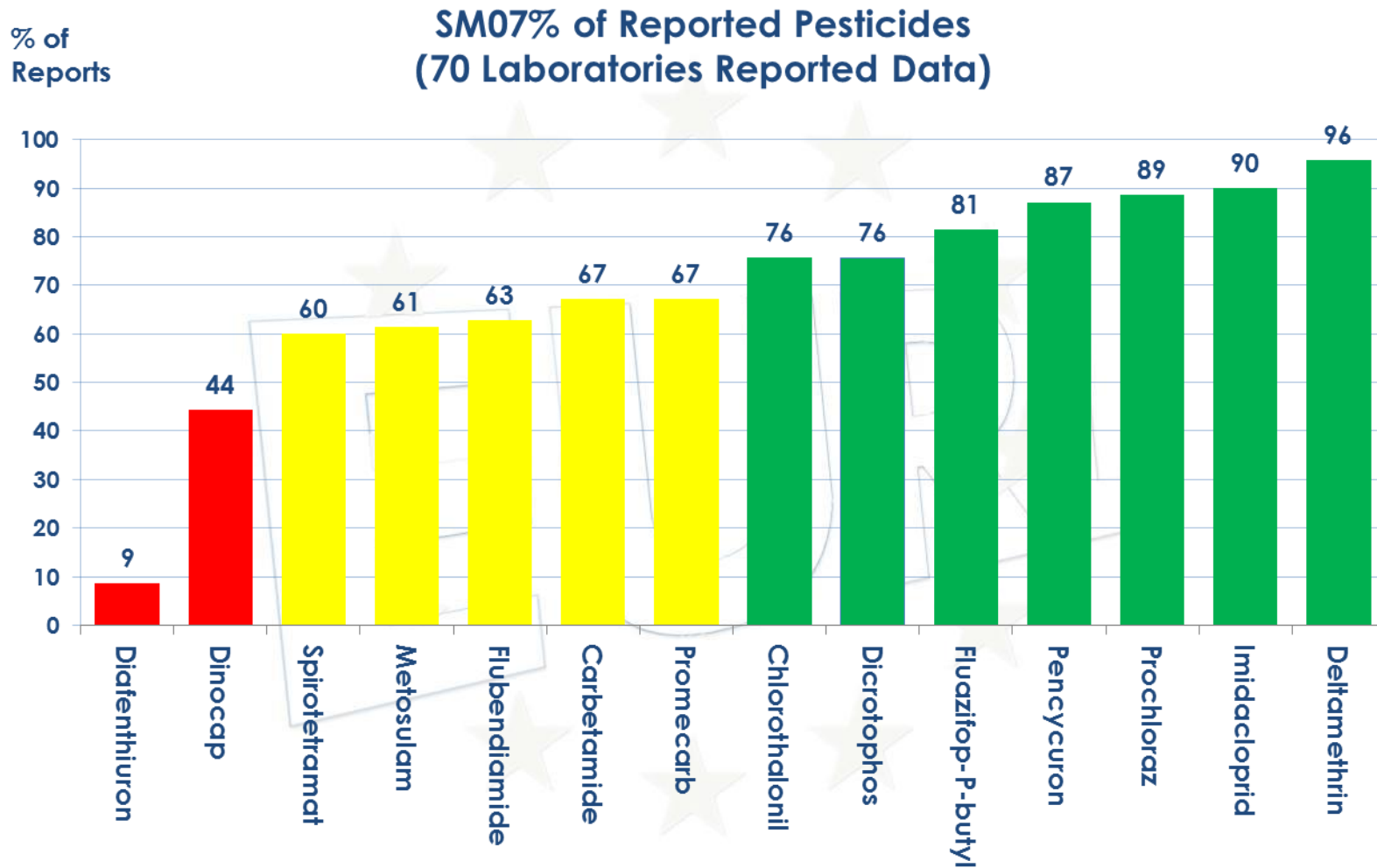


Carbetamide	Flubendiamide
Chlorothalonil	Imidacloprid
Deltamethrin	Metosulam
Diafenthiuron	Pencycuron
Dicrotophos	Prochloraz
Dinocap	Promecarb
Fluazifop-P-butyl	Spirotetramat

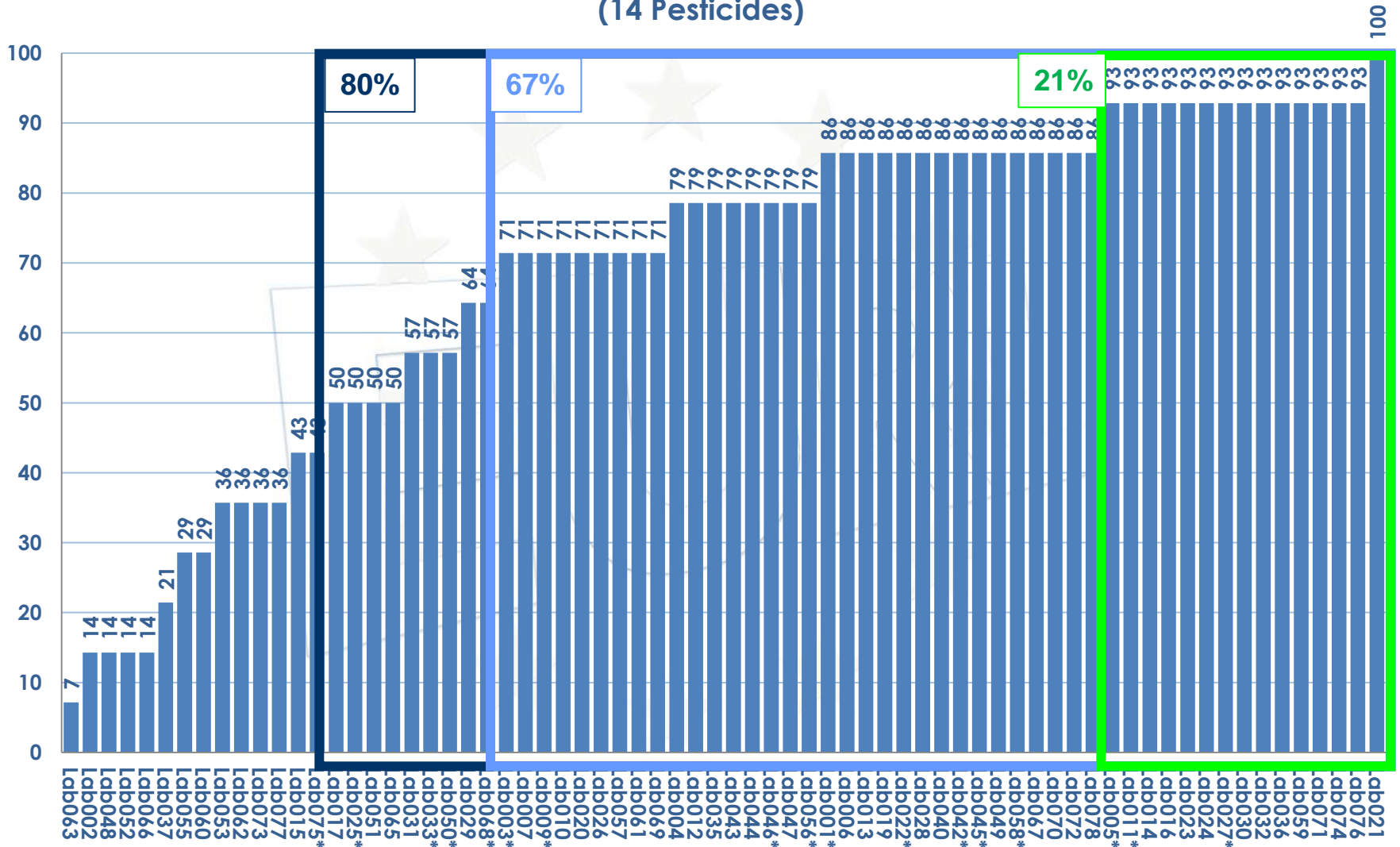
14 Evaluated Pesticides
 70 Laboratories

14 Pesticides = 2+5+7

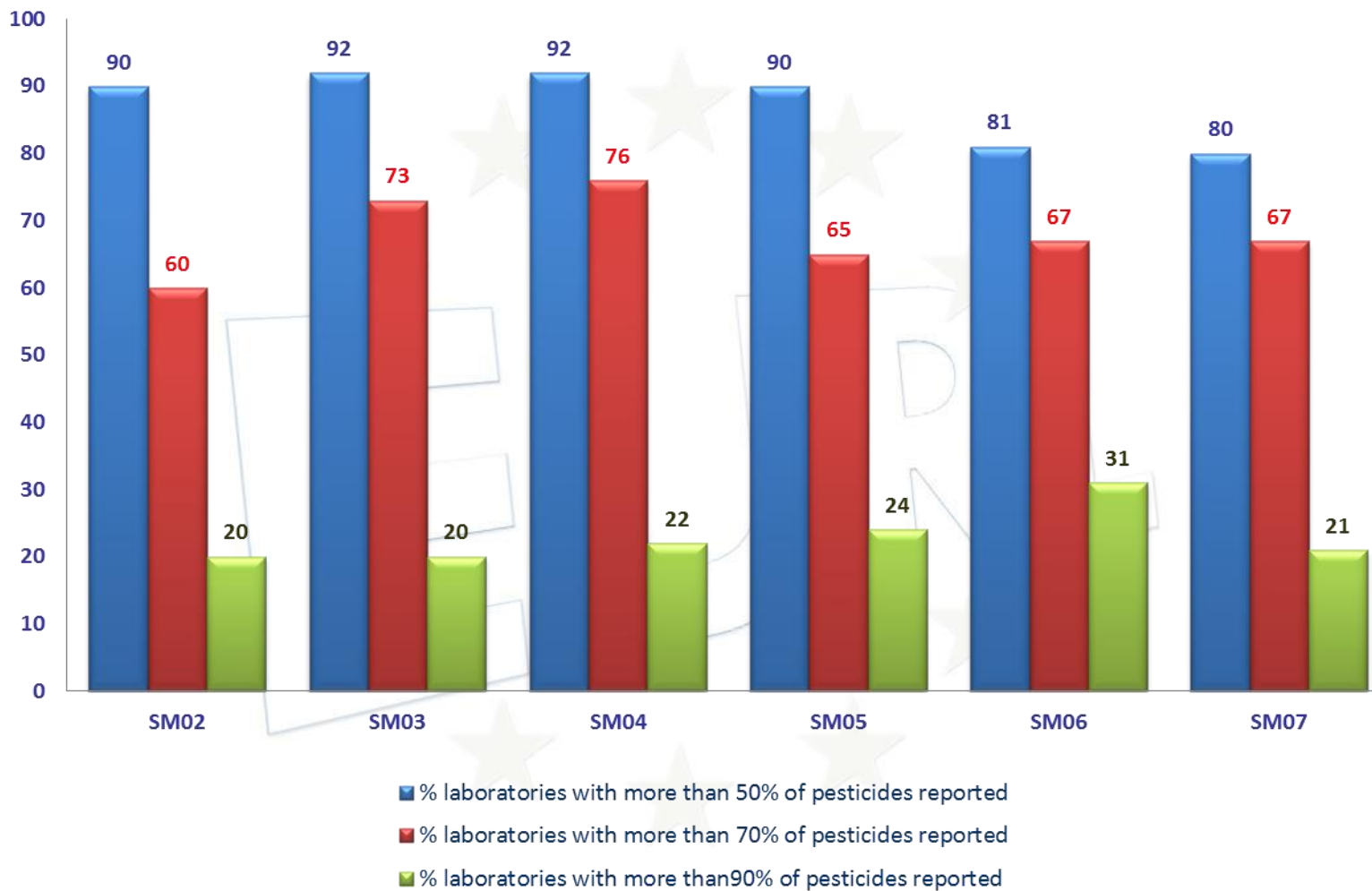
	Carbetamide	Chlorothalonil	Deltamethrin	Diafenthiuron	Dicrotophos	Dinocap	Fluazifop-P-butyl	Flubendiamide	Imidacloprid	Metosulam	Pencycuron	Prochloraz	Promecarb	Spirotetramat
Total Number of Reported Pesticides	47	53	67	6	53	31	57	44	63	43	61	62	47	42
% of Reported Pesticides	67	76	96	9	76	44	81	63	90	61	87	89	67	60



SM07 % of Reported Pesticides (14 Pesticides)



100





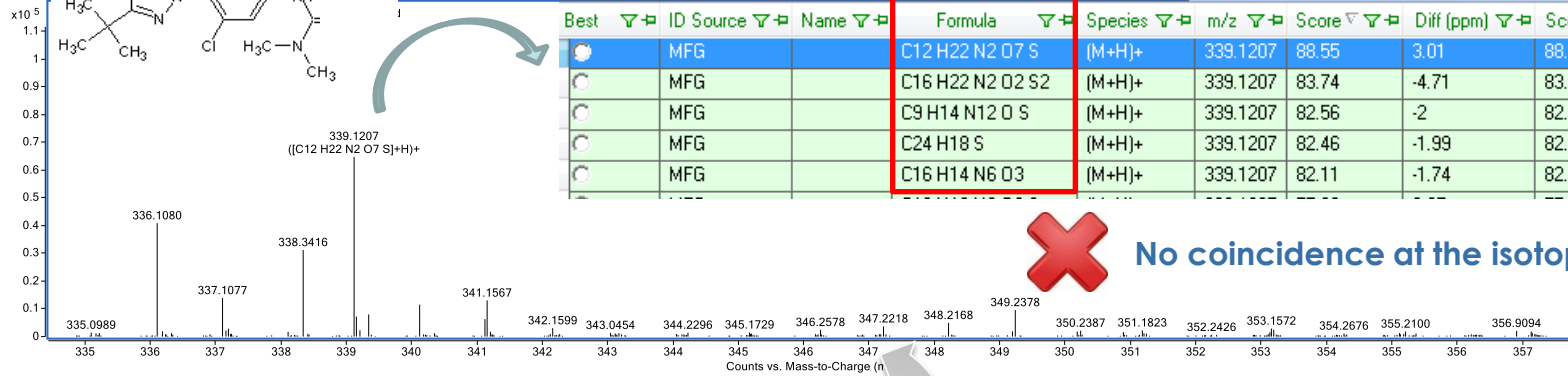
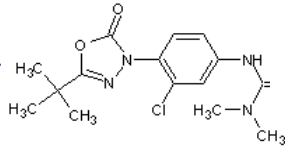
These pesticides have been reported by 3 or more
laboratories :

- 
- Dimefuron
 - Metamitron
 - Monochrotofos
 - Mepthyl Dinocap



Dimefuron

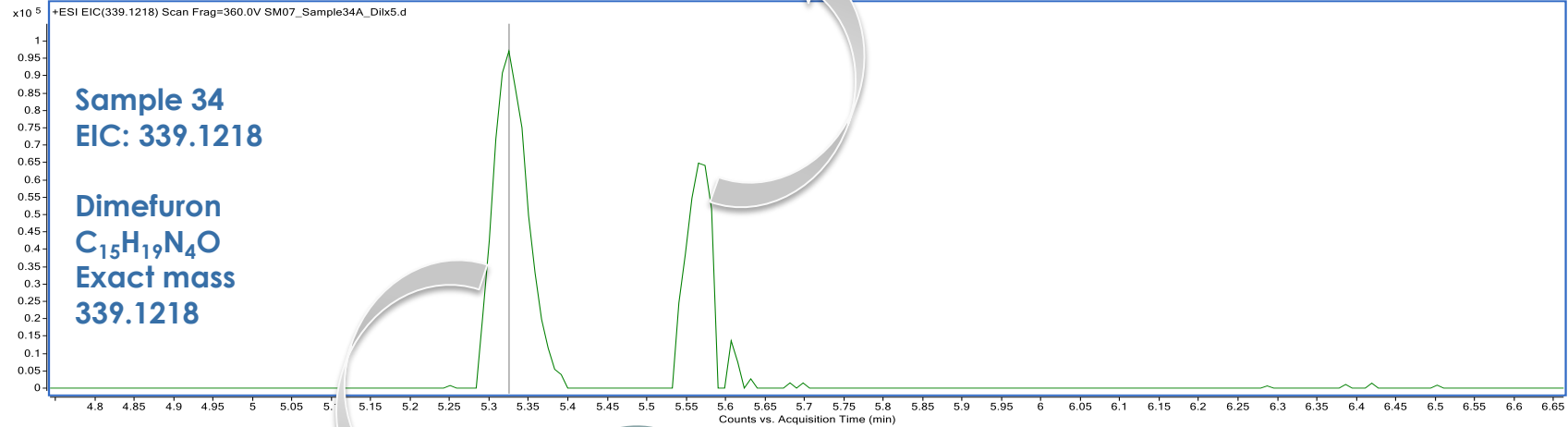
Dimefuron



Best	ID Source	Name	Formula	Species	m/z	Score	Diff (ppm)	Score (MFG)
●	MFG		C12H22N2O7S	(M+H)+	339.1207	88.55	3.01	88.55
○	MFG		C16H22N2O2S2	(M+H)+	339.1207	83.74	-4.71	83.74
○	MFG		C9H14N12O5	(M+H)+	339.1207	82.56	-2	82.56
○	MFG		C24H18S	(M+H)+	339.1207	82.46	-1.99	82.46
○	MFG		C16H14N6O3	(M+H)+	339.1207	82.11	-1.74	82.11

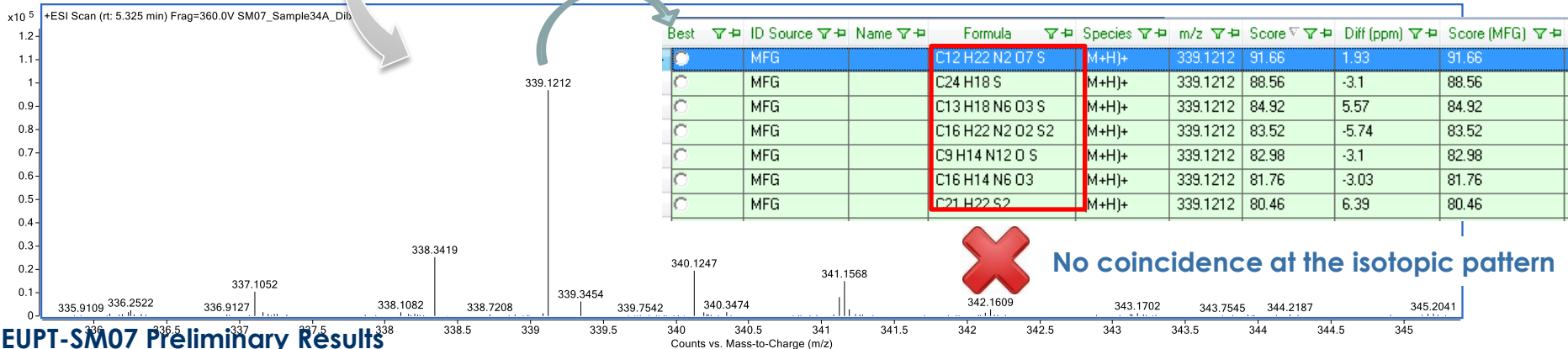


No coincidence at the isotopic pattern



Sample 34
EIC: 339.1218

Dimefuron
C₁₅H₁₉N₄O
Exact mass
339.1218

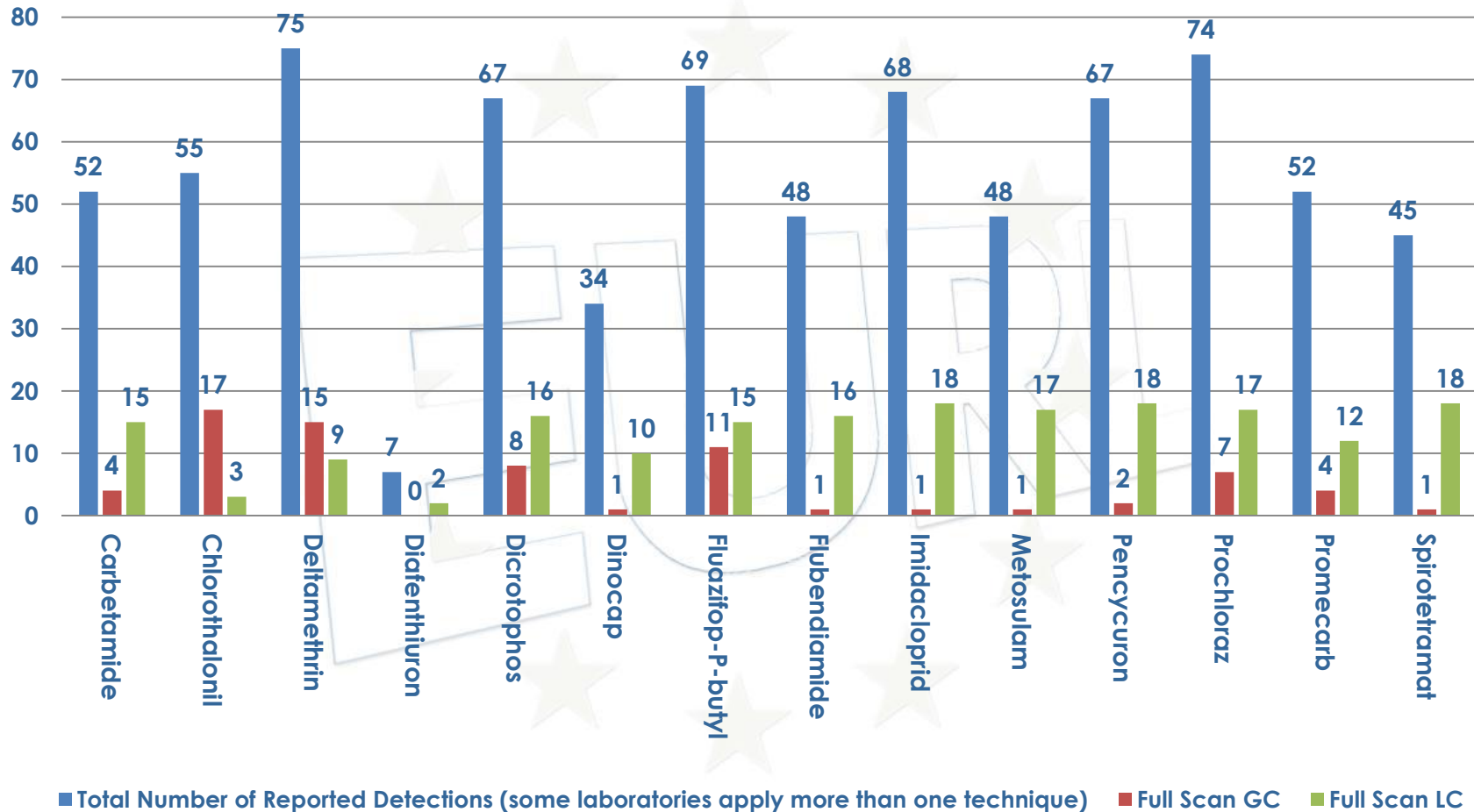


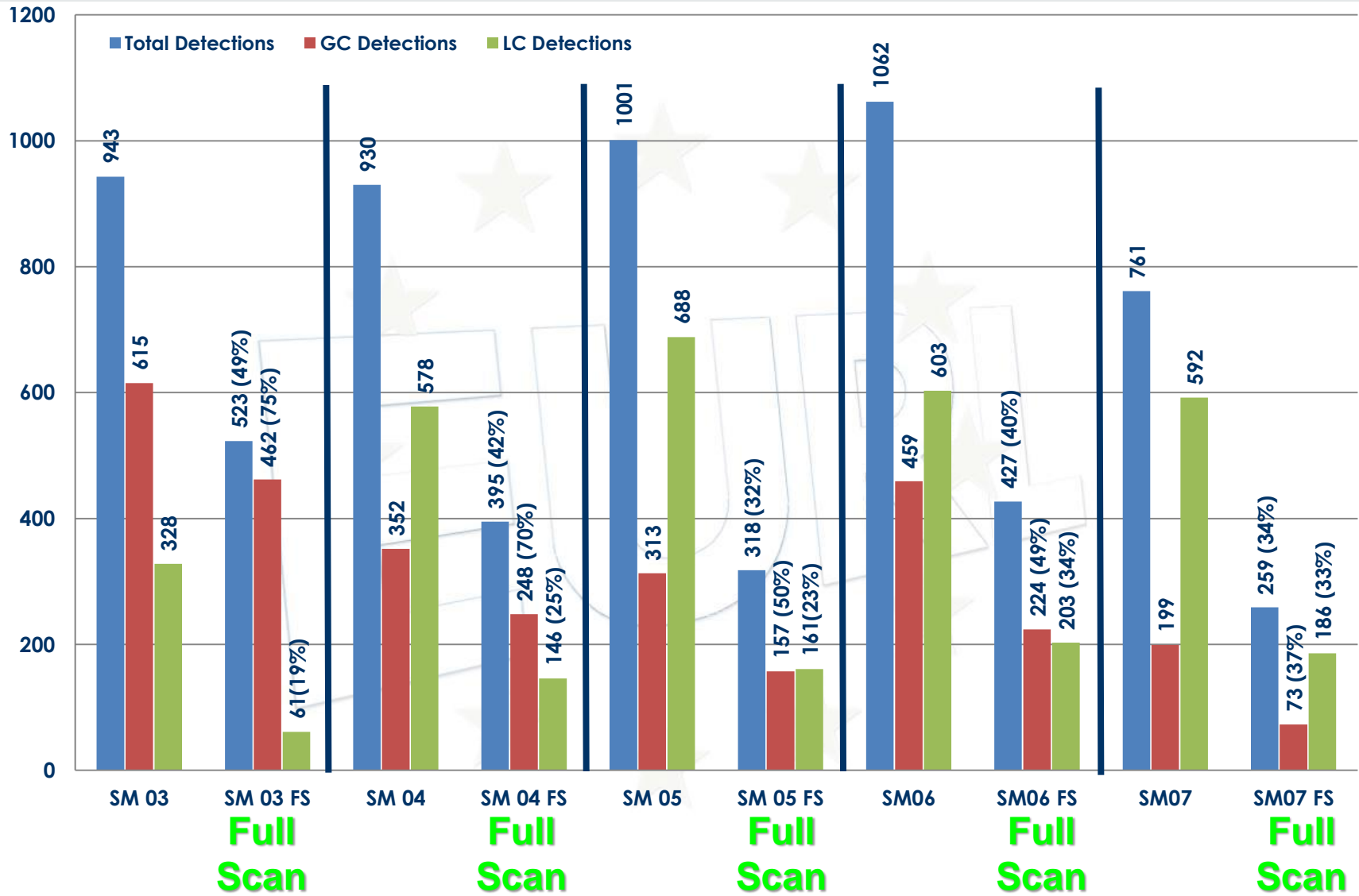
Best	ID Source	Name	Formula	Species	m/z	Score	Diff (ppm)	Score (MFG)
●	MFG		C12H22N2O7S	(M+H)+	339.1212	91.66	1.93	91.66
○	MFG		C24H18S	(M+H)+	339.1212	88.56	-3.1	88.56
○	MFG		C13H18N6O3S	(M+H)+	339.1212	84.92	5.57	84.92
○	MFG		C16H22N2O2S2	(M+H)+	339.1212	83.52	-5.74	83.52
○	MFG		C9H14N12O5	(M+H)+	339.1212	82.98	-3.1	82.98
○	MFG		C16H14N6O3	(M+H)+	339.1212	81.76	-3.03	81.76
○	MFG		C21H22S2	(M+H)+	339.1212	80.46	6.39	80.46



No coincidence at the isotopic pattern

SM-07 Techniques







LAB. CODE	Total Detections	GC Detections	LC Detections	Total Full Scan	GC Full Scan	LC Full Scan
Lab001*	12	2	10	2	2	10
Lab002	2	0	0	6	2	0
Lab003*	12	16	?	6	5	11
Lab004	13	?	?	?	?	?
Lab005*	12	10	10	12	5	10
Lab006	10	0	0	10	0	5
Lab007	10	0	0	0	0	0
Lab009*	11	0	0	0	0	0
Lab010	11	13	1	6	5	0
Lab011*	13	2	0	2	2	0
Lab012	11	3	0	3	3	0
Lab013	12	0	12	6	0	6
Lab014	25	6	0	6	0	0
Lab015	6	22	0	0	0	0
Lab016	22	7	0	0	0	0
Lab017	7	12	0	0	0	0
Lab019	12	11	0	0	0	0
Lab020	14	11	0	0	0	0
Lab021	12	14	0	0	0	0
Lab022*	14	13	?	0	?	?
Lab023	13	7	10	0	0	10
Lab024	7	10	0	0	0	0
Lab025*	10	0	0	0	0	0
Lab026	13	0	0	0	0	0
Lab027*	12	9	0	0	0	0
Lab028	24	11	0	11	0	11
Lab029	8	13	0	12	0	12
Lab030	14	14	6	11	0	11
Lab031	12	13	0	0	3	0
Lab032	14	12	0	0	0	0
Lab033*	20	13	0	2	1	0
Lab035	13	5	12	3	0	0
Lab036	22	4	0	0	0	0
Lab037	11	2	0	0	0	0
Lab040	12	24	0	0	0	0
Lab042*	11	11	0	0	0	0
Lab043	11	12	0	0	0	0
Lab044	12	20	0	0	0	0
Lab045*	11	11	0	0	0	0
Lab046*	20	11	0	0	0	0
Lab047	2	12	0	0	0	0
Lab048	12	8	0	0	0	0
Lab049	7	7	0	0	0	0
Lab050*	2	5	0	7	2	0
Lab051	5	4	0	2	0	0
Lab052	4	11	0	0	0	0
Lab053	10	10	0	0	0	0
Lab055	12	14	0	0	0	0
Lab056*	4	4	0	3	1	1
Lab057	1	3	0	1	1	0
Lab058*	2	2	0	2	2	0
Lab059	12	12	0	0	0	0
Lab060	14	10	0	0	0	0
Lab061	5	5	0	3	1	0
Lab062	1	1	0	1	1	0
Lab063	7	3	0	3	2	0
Lab065	2	12	0	2	12	0
Lab066	12	9	0	0	0	0
Lab067	9	10	0	7	0	0
Lab068*	10	12	0	0	0	0
Lab069	13	13	0	0	0	0
Lab070	21	24	0	13	11	0
Lab071	13	5	0	13	0	0
Lab072	24	13	0	0	0	0
Lab073	5	6	3	0	0	0
Lab074	13	6	7	0	0	0
Lab075*	6	2	3	0	0	0
Lab076	13	1	0	0	0	0
Lab077	5	2	3	0	0	0
Lab078	12	1	11	0	0	11

EUPT-FV-SM07
European Proficiency Test FV-SM07

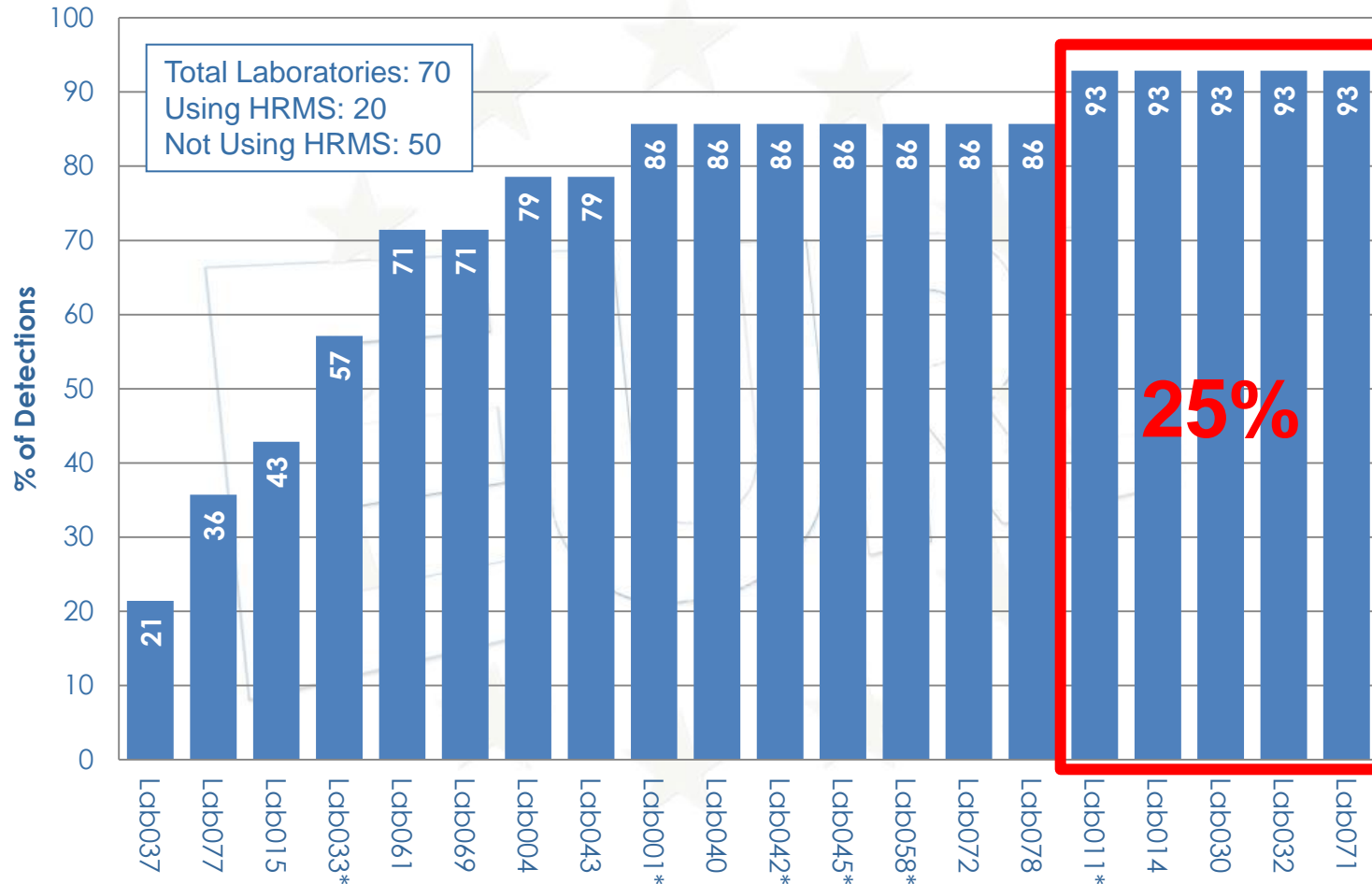
Total Detections: 761
Total Full Scan: 259
(34% of the Total)

GC Detections: 199
GC Full Scan: 73
(37% of the Full Scan)

LC Detections: 562
LC Full Scan: 186
(33% of the Full Scan)

Laboratories Using HRMS

Using HRMS: 20
 ≥ 90% Detections: 5



**Thank You
for Your Attention**



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LABORATORY