

# 4<sup>th</sup>

## Joint Workshop of the

# EUROPEAN UNION REFERENCE LABORATORIES



Almeria  
(SPAIN) 23<sup>rd</sup>-25<sup>th</sup> OCT  
**2013**

*EUROPEAN COMMISSION*

*PROFICIENCY TEST-T01*

*PESTICIDE RESIDUES IN  
TEA HOMOGENATE*



European  
Commission

# EURL



## AMADEO R. FERNÁNDEZ-ALBA

EUROPEAN UNION REFERENCE LABORATORIES



# Calendar

ACTIVITY	DATE
Sending Application Form to laboratories	9th May 2013
Sending calendar and pesticides target list to participant laboratories.	14th May 2013
Deadline for receiving Application Form from laboratories.	17th May 2013
Sample distribution.	10th June 2013
Deadline for receiving results	28th June 2013
Preliminary Report: only results, no statistical treatment.	July 2013
Final Report	October 2013



# Participants

## EU and EFTA countries

1	Austria
4	Belgium
1	Denmark
1	Finland
3	France
8	Germany
2	Hungary
1	Ireland
8	Italy
1	Norway*
1	Poland
1	Romania
1	Slovakia
8	Spain
1	Sweden
2	Switzerland*
2	The Netherland

\*EFTA countries

## Non EU and EFTA countries

1	China
1	Egypt
1	Israel
1	Saudi Arabia
1	Serbia
1	Uruguay

52 laboratories in total (16 of them NRLs)

# Incurred Compounds

## 20 LC<sup>a</sup>- and GC<sup>b</sup>- amenable pesticides

### 15 pesticides evaluated

Acetamiprid  
Buprofezin  
Carbendazim  
Chlorpyrifos  
Cypermethrin  
Difenoconazole  
Endosulfan beta  
Ethofenprox  
Fenpropathrin  
Imidacloprid  
Lambda cyhalothrin  
Methomyl  
Parathion ethyl  
Pyridaben  
Tebuconazole

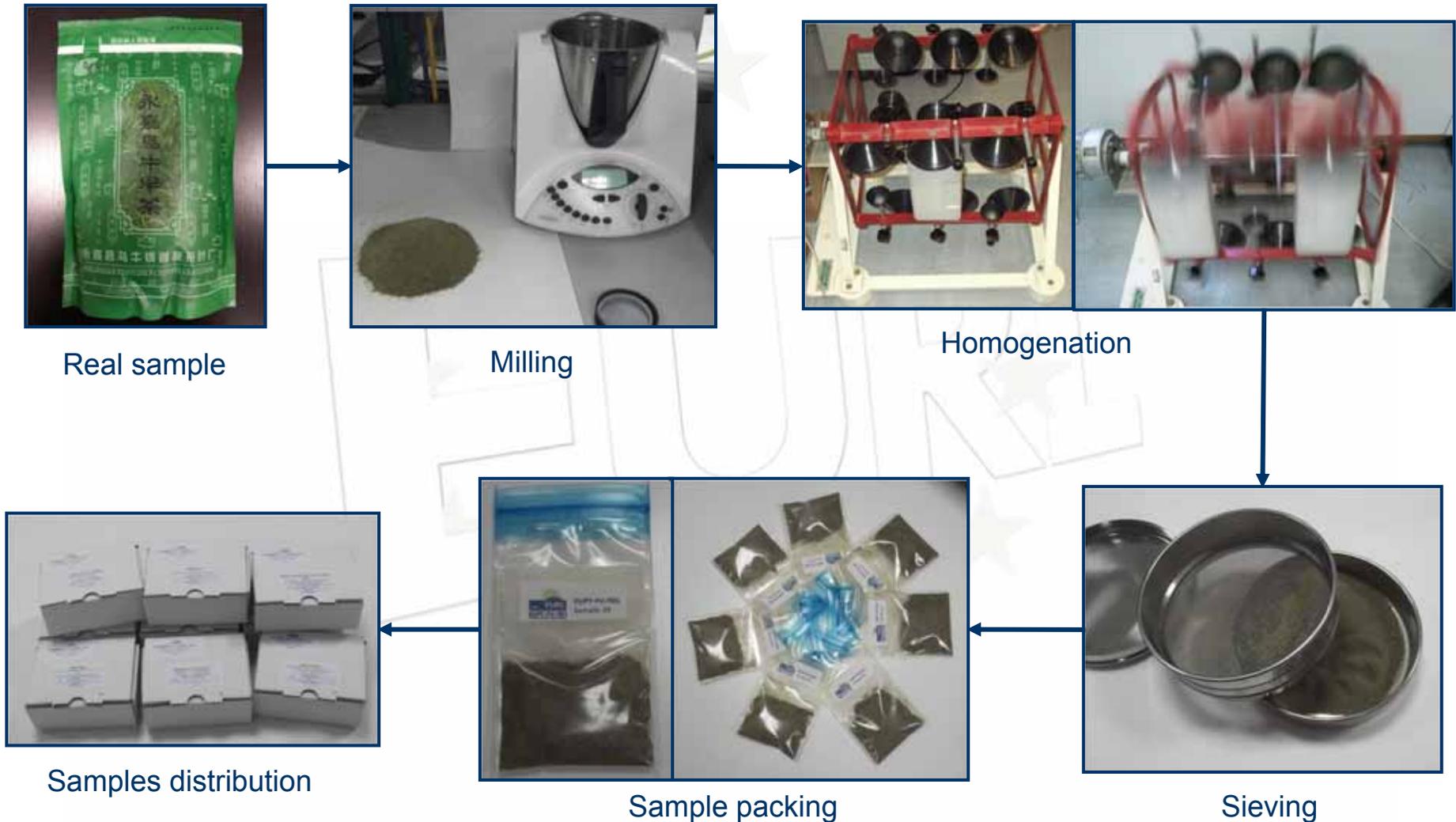
### Not evaluated (Median < 4MRRL)

Bifenthrin  
Endosulfan alpha  
Endosulfan sulphate  
Omethoate

### Not evaluated (Homogeneity test fails)

Thiophanate methyl

# Preparation of the test item



# Homogeneity

Pesticides	Homogeneity median concentration (mg/kg)	Assigned value (mg/kg)
Acetamiprid	0.148	0.108
Buprofezin	0.213	0.180
Carbendazim	0.271	0.250
Chlorpyrifos	0.466	0.364
Cypermethrin	0.145	0.112
Difenoconazole	0.380	0.367
Endosulfan beta	0.096	0.081
Ethofenprox	0.234	0.170
Fenpropathrin	0.258	0.188
Imidacloprid	0.108	0.081
Lambda cyhalothrin	0.175	0.141
Methomyl	0.113	0.089
Parathion ethyl	0.436	0.372
Pyridaben	0.338	0.268
Tebuconazole	0.423	0.338
Thiophanate methyl*	0.259	0.190

\*Homogeneity test fails



**EURL-FV**



Almería 23<sup>rd</sup>-25<sup>th</sup> October 2013

# Results

## Summary of reported results

Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Reported Results (out of 46)*
Acetamiprid	36	4	6	78
<b>Bifenthrin*</b>	<b>19</b>	<b>22</b>	<b>5</b>	<b>41</b>
Buprofezin	41	3	2	89
Carbendazim	42	0	4	91
Chlorpyrifos	43	1	2	93
<b>Cypermethrin</b>	<b>31</b>	<b>6</b>	<b>9</b>	<b>67</b>
Difenoconazole	40	1	5	87
Endosulfan alpha*	36	5	5	78
Endosulfan beta	38	3	5	83
Endosulfan sulfate*	39	2	5	85
Ethofenprox	36	2	8	78
Fenpropathrin	40	1	5	87
<b>Imidacloprid</b>	<b>33</b>	<b>7</b>	<b>6</b>	<b>72</b>
Lambda cyhalothrin	44	0	2	96
<b>Methomyl</b>	<b>29</b>	<b>9</b>	<b>8</b>	<b>63</b>
<b>Omethoate*</b>	<b>20</b>	<b>19</b>	<b>7</b>	<b>43</b>
Parathion ethyl	40	2	4	87
Pyridaben	44	1	1	96
Tebuconazole	42	1	3	91
Thiophanate methyl**	35	2	9	76

\*Median < 4 MRRL

\*\*Homogeneity test fails

## False positives

Laboratory Code	Pesticide	Concentration (mg/kg)	Determination Technique	RL (mg/Kg)	MRRL (mg/Kg)	Extraction method used
Lab007	Orthophenylphenol	0.020	LC-MS/MS (QQQ)	0.01	0.02	QuEChERS
Lab009	Penconazole	0.071	LC-MS/MS (QQQ)	0.01	0.02	Modified QuEChERS with CaCl <sub>2</sub>
Lab011	Amitraz	0.126	LC-MS/MS (QQQ)	0.02	0.02	Modified QuEChERS with CaCl <sub>2</sub>
Lab012	Methidathion	0.041	GC-MS/MS (QQQ)	0.01	0.02	L-L extraction
Lab013	Carbaryl	0.042	Other	0.02	0.02	Modified QuEChERS with CaCl <sub>2</sub>
Lab034	Folpet	0.056	GC-MS/MS (QQQ), FPD, ECD	0.02	0.02	miniLuke
Lab035	Ethoprophos	0.155	LC and GC MS/MS (QQQ)	0.01	0.02	QuEChERS
Lab043	Ethoprophos	0.177	LC-MS/MS (QQQ)	0.01	0.02	QuEChERS
Lab043	Triazophos	0.196	LC-MS/MS (QQQ)	0.01	0.02	QuEChERS

# False negatives

Laboratory Code	Acetamiprid	Buprofezin	Chlorpyrifos	Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))	Difenoconazole	Endosulfan beta	Etofenprox	Fenpropathrin	Imidacloprid	Methomyl	Parathion-ethyl	Pyridaben	Tebuconazole	Thiophanate-methyl
Lab003									ND	ND				
Lab006	ND													
Lab007				ND						ND				ND
Lab010		ND												
Lab011									ND	ND				
Lab013			ND											
Lab016				ND		ND			ND	ND				
Lab018				ND										
Lab020									ND					
Lab022											ND	ND	ND	
Lab025				ND						ND				
Lab026										ND				
Lab027	ND								ND	ND	ND			
Lab033		ND												
Lab034		ND						ND						
Lab035							ND							
Lab039				ND										
Lab043				ND		ND	ND							
Lab047						ND								
Lab050														ND
Lab051	ND								ND	ND				
Lab054	ND				ND				ND	ND				

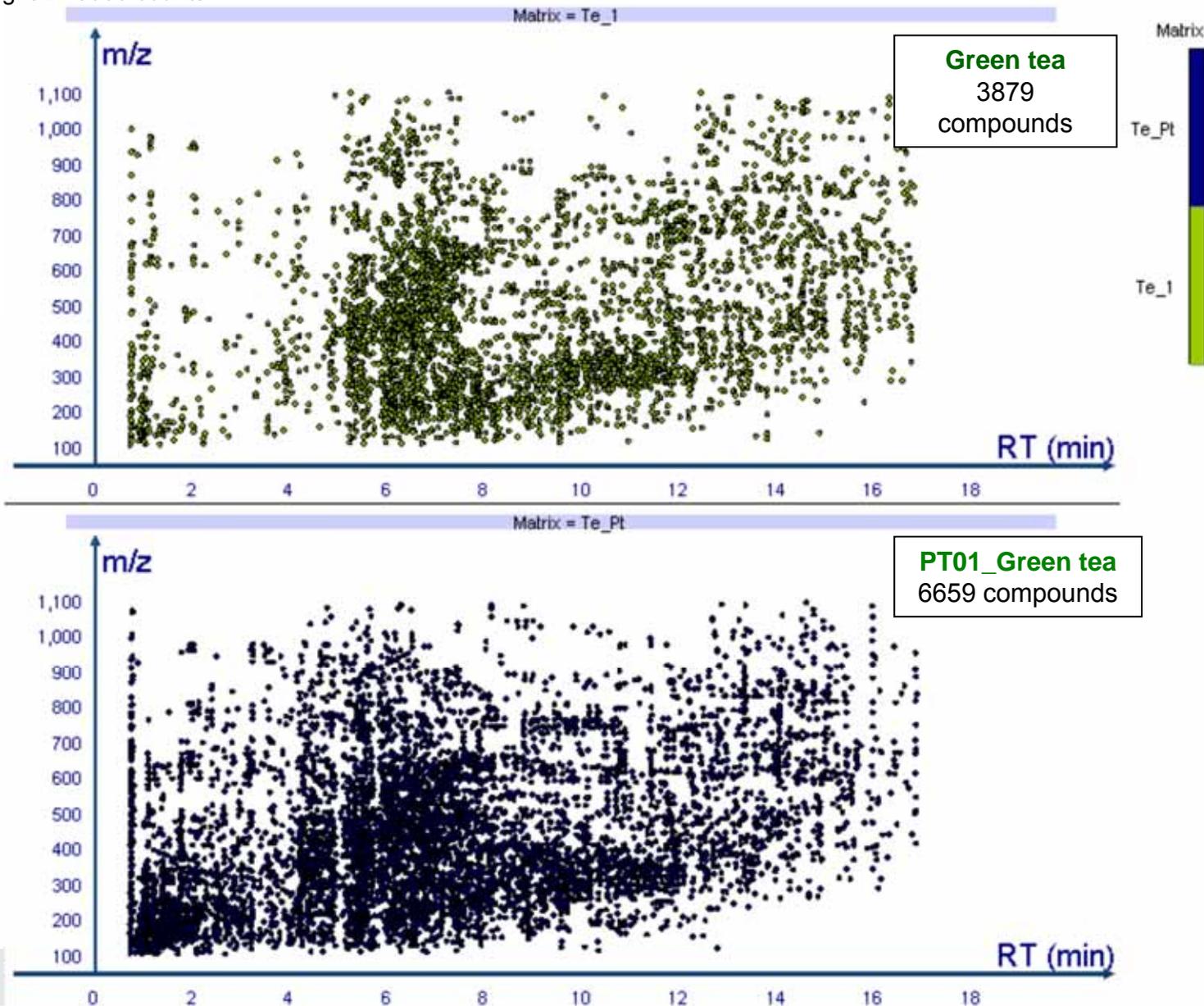
- QuEChERS
- CaCl<sub>2</sub>
- EtAc
- MiniLuke
- MeOH
- Other

False negatives (%) over the total number of results per extraction method

QuEChERS: 6.9%  
 CaCl<sub>2</sub>: 6.3%  
 Ethyl Acetate method: 2.3%  
 MeOH extraction: 6.7%  
 miniLuke: 6.1%  
 Other methods: 2.2%



Tea matrix compounds. QuEChERS\_CaCl<sub>2</sub> extraction method. Dilution:5 LC-TOF-MS  
Absolute height  $\geq 10000$  counts





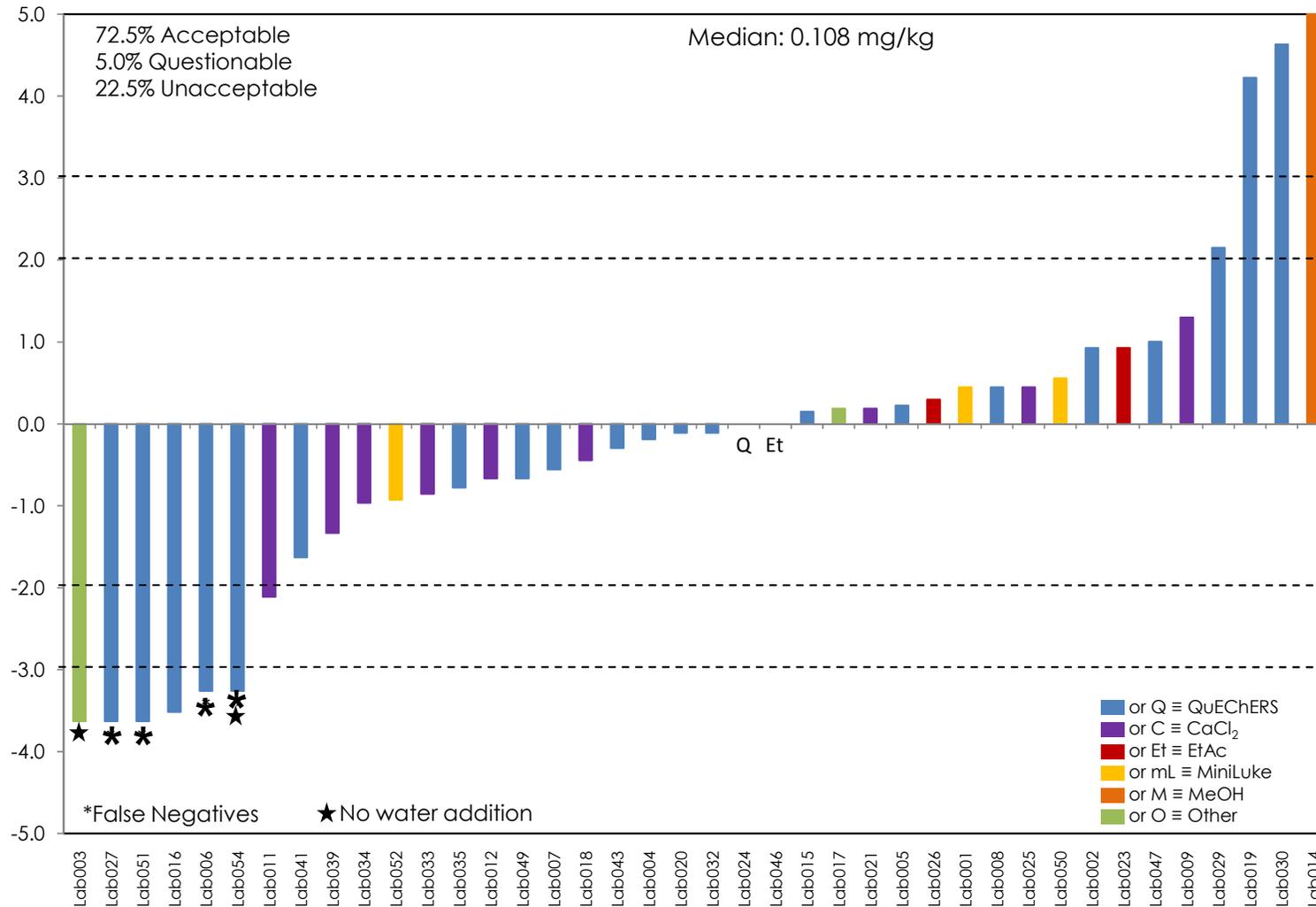
## Median values, uncertainty and %RSDs for all pesticides present in the test item

Pesticides	MRRL (mg/Kg)	Median (mg/Kg)	u (mg/kg)	FFP RSD (%)	Qn RSD (%)
Acetamiprid	0.02	0.108	0.007	25	31
Buprofezin	0.02	0.180	0.014	25	39
Carbendazim	0.02	0.250	0.021	25	44
Chlorpyrifos	0.02	0.364	0.021	25	35
Cypermethrin	0.02	0.112	0.011	25	44
Difenoconazole	0.02	0.367	0.018	25	25
Endosulfan beta	0.02	0.081	0.007	25	44
Ethofenprox	0.01	0.170	0.012	25	34
Fenpropathrin	0.02	0.188	0.008	25	22
Imidacloprid	0.02	0.081	0.006	25	36
Lambda cyhalothrin	0.02	0.141	0.009	25	33
Methomyl	0.02	0.089	0.007	25	32
Parathion ethyl	0.02	0.372	0.021	25	29
Pyridaben	0.02	0.268	0.017	25	32
Tebuconazole	0.02	0.338	0.017	25	26



# Z-Scores

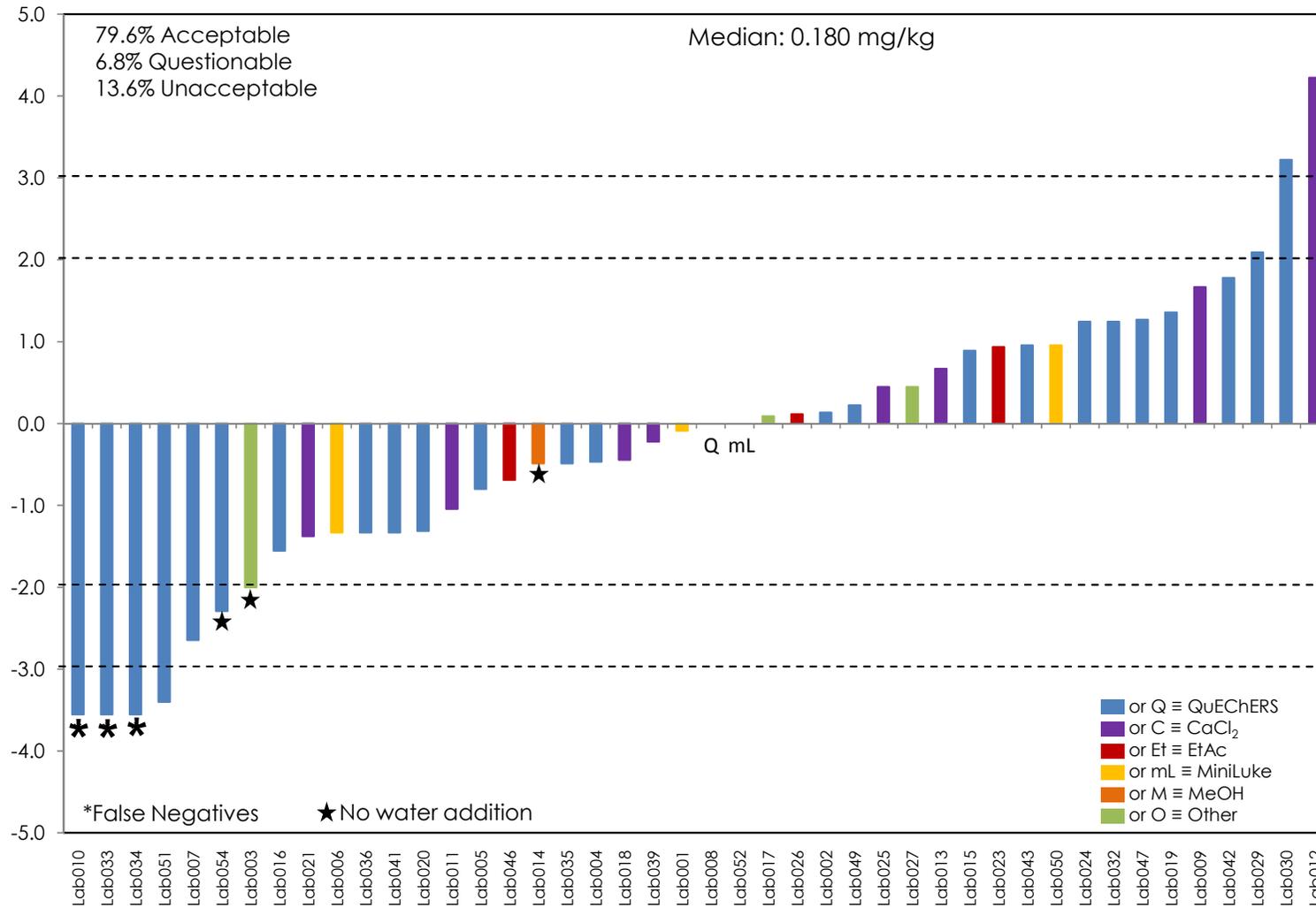
## Acetamiprid





# Z-Scores

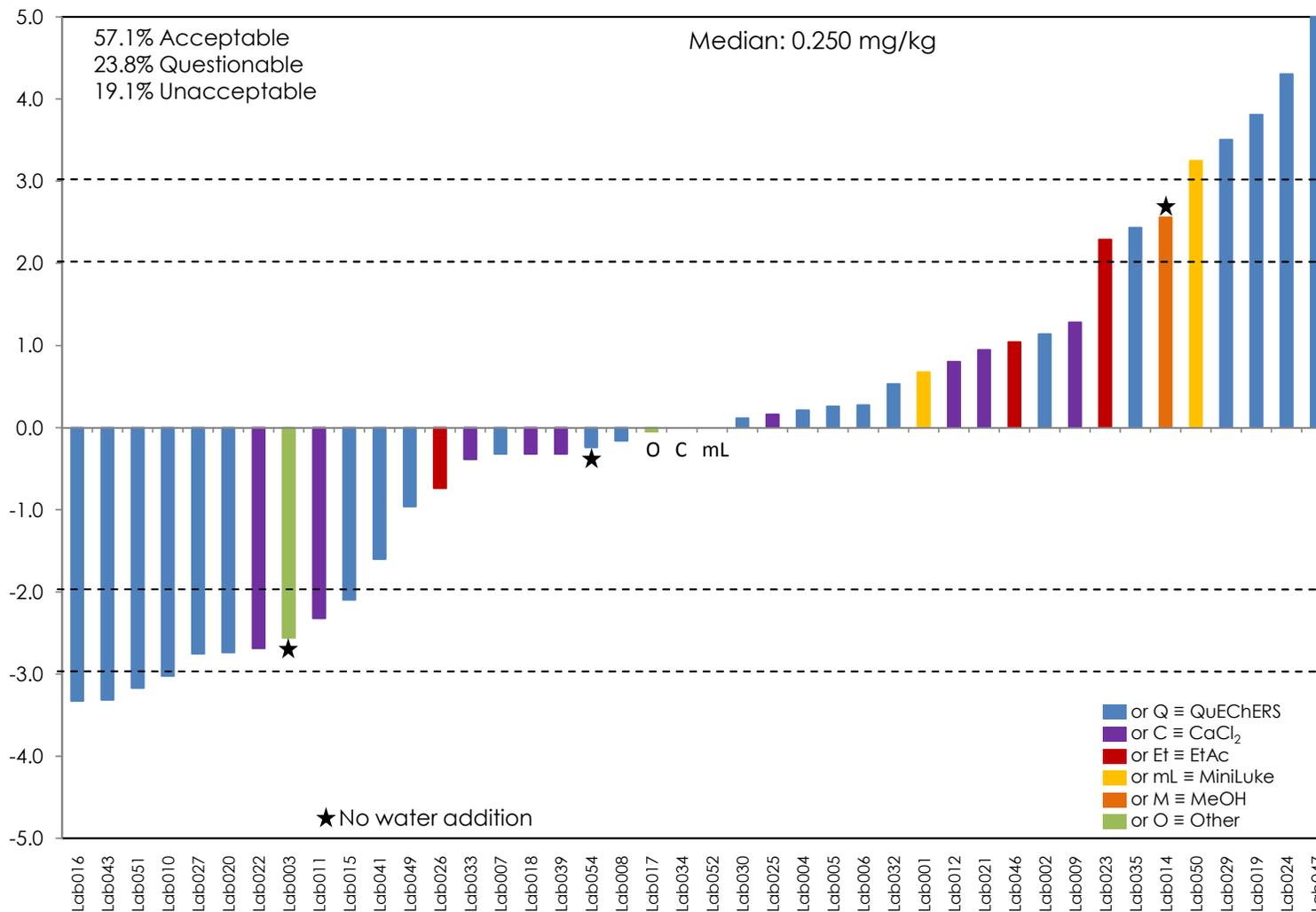
## Buprofezin





# Z-Scores

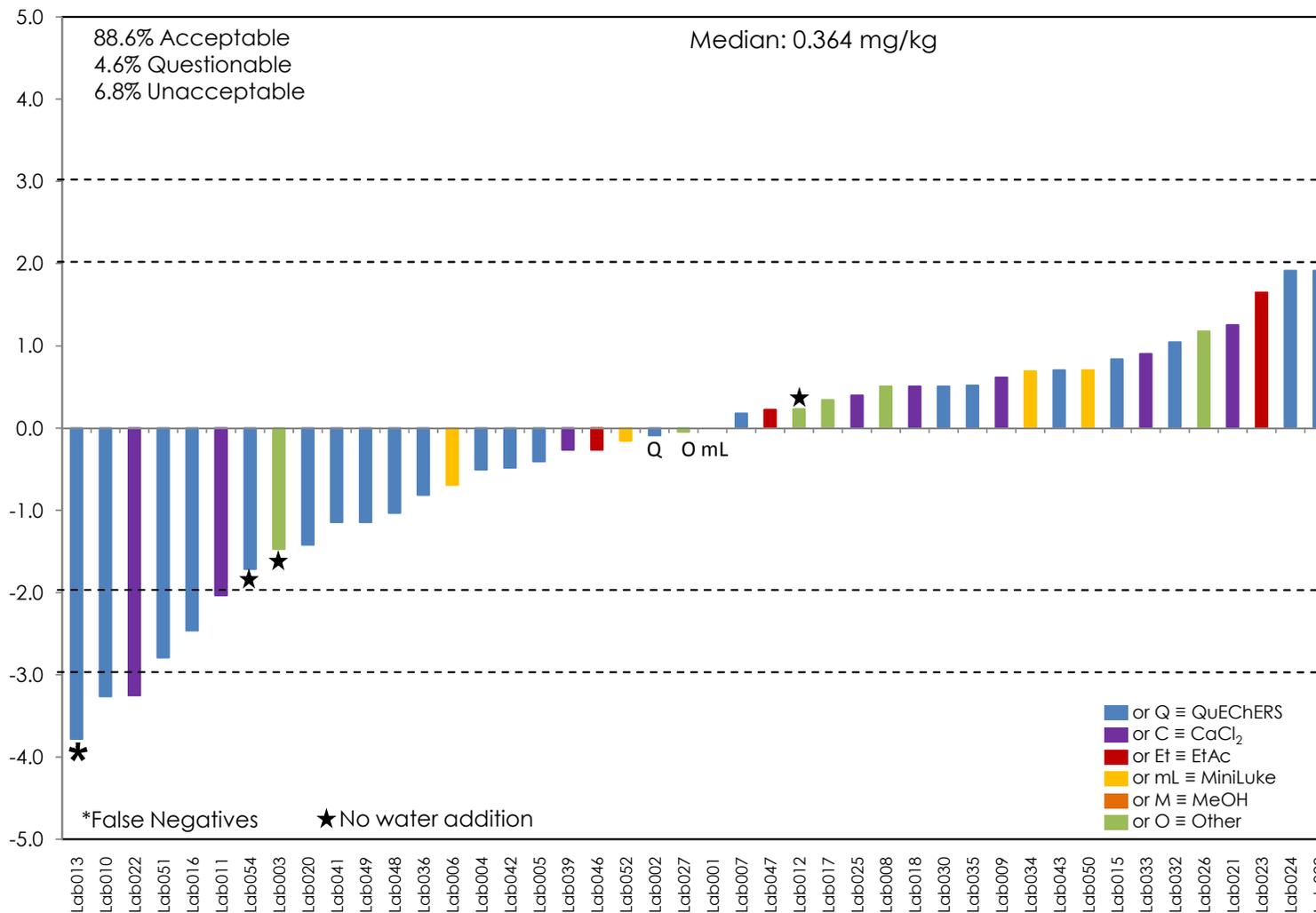
## Carbendazim





# Z-Scores

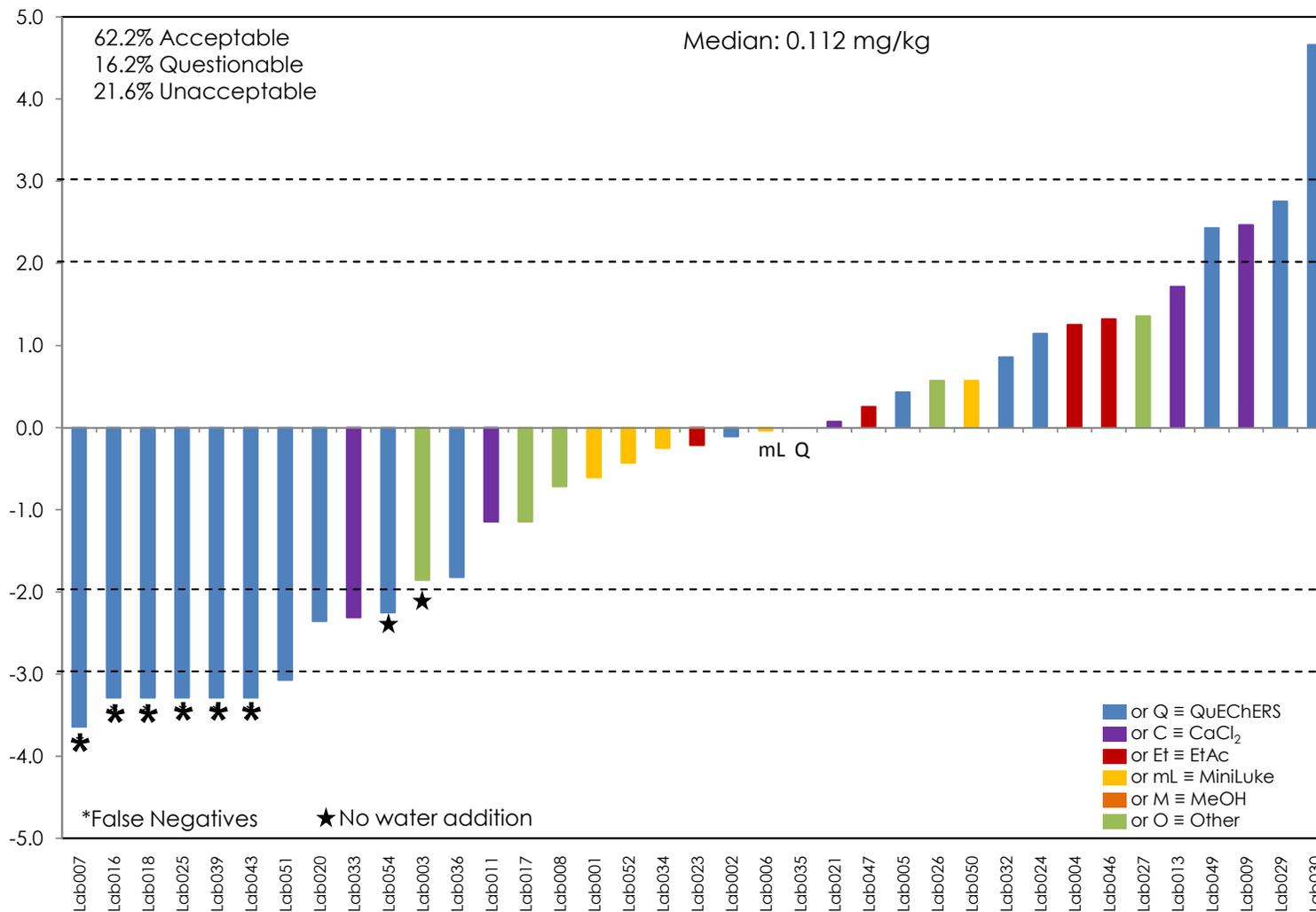
## Chlorpyrifos





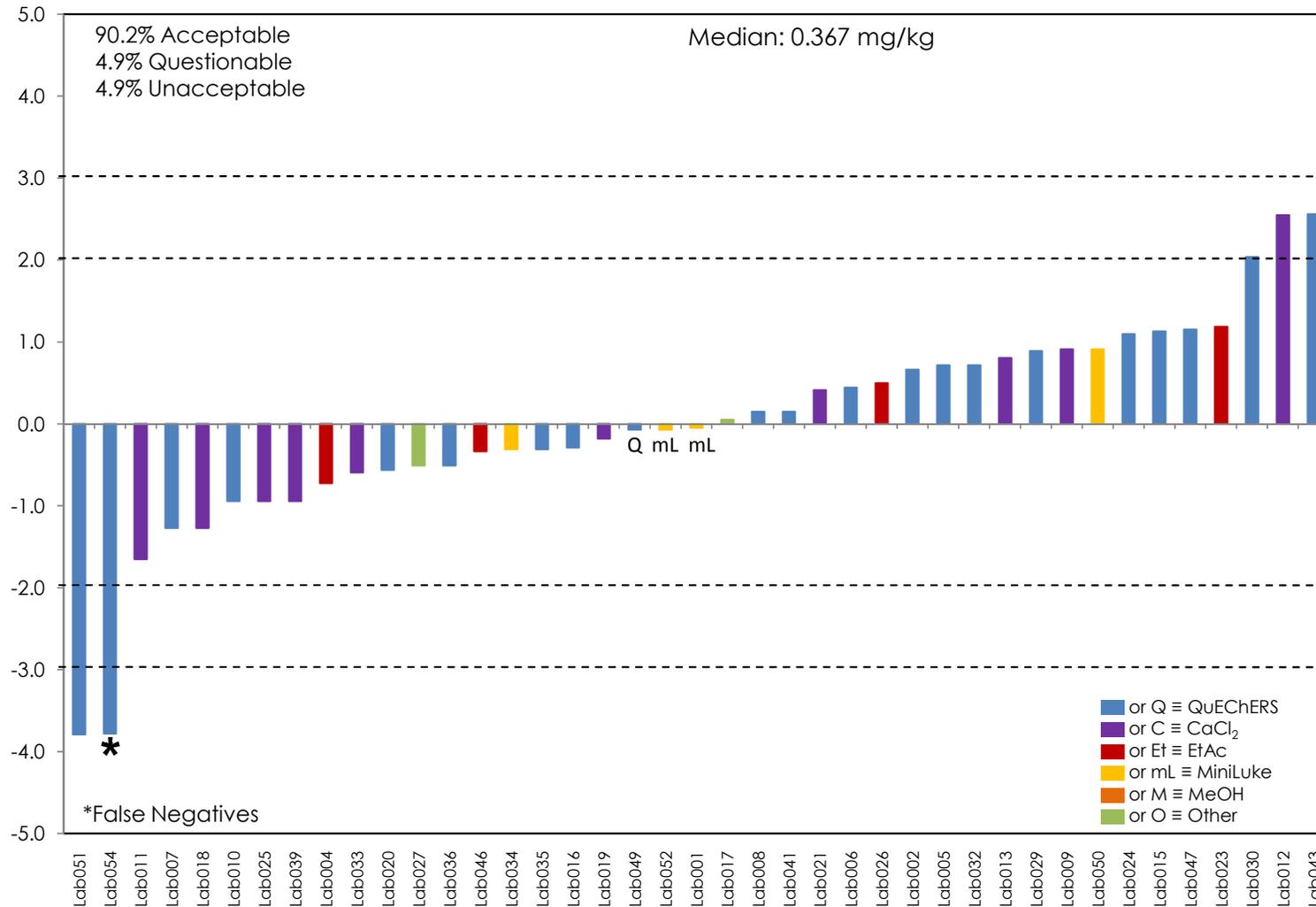
# Z-Scores

## Cypermethrin



# Z-Scores

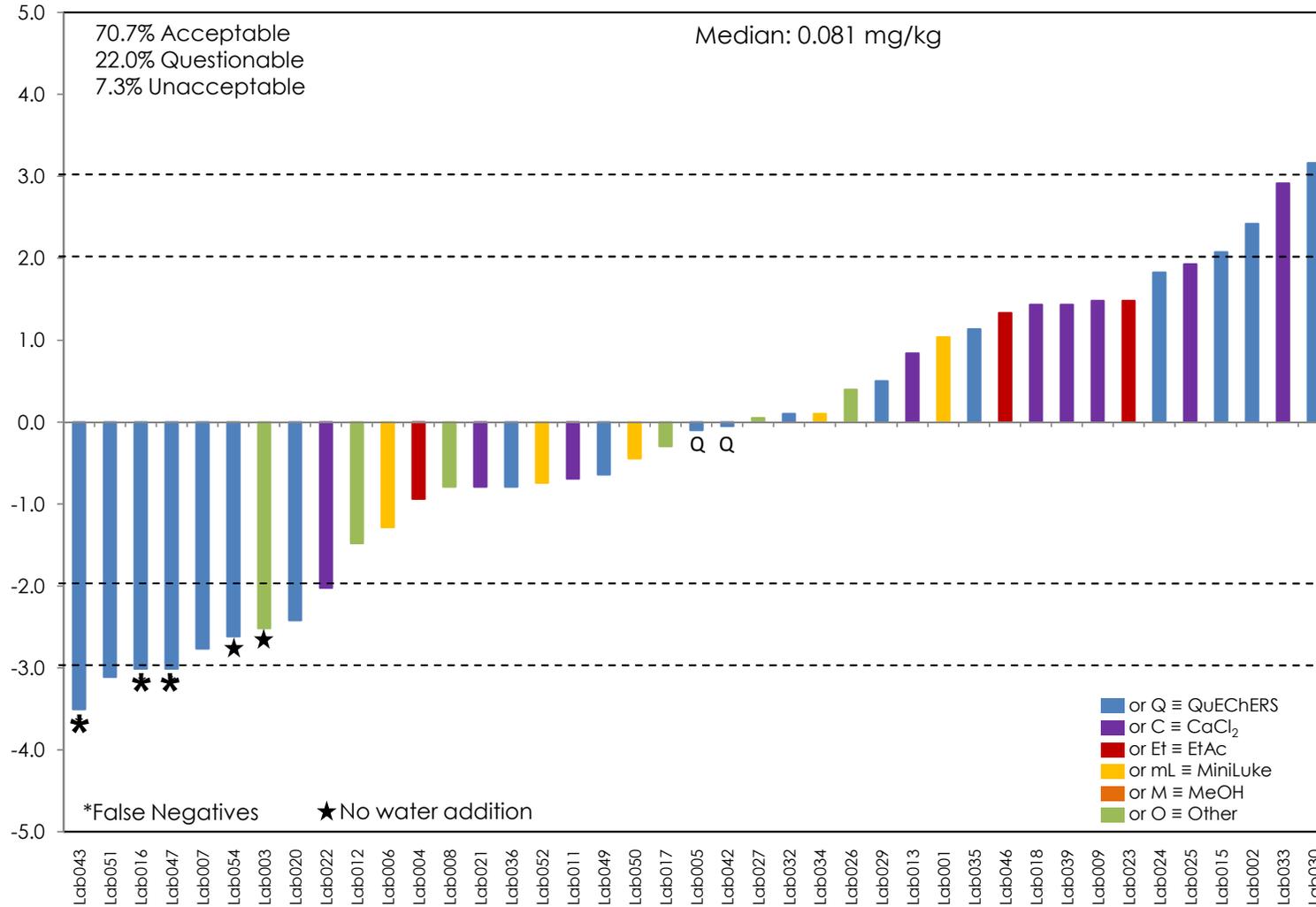
## Difenoconazole





# Z-Scores

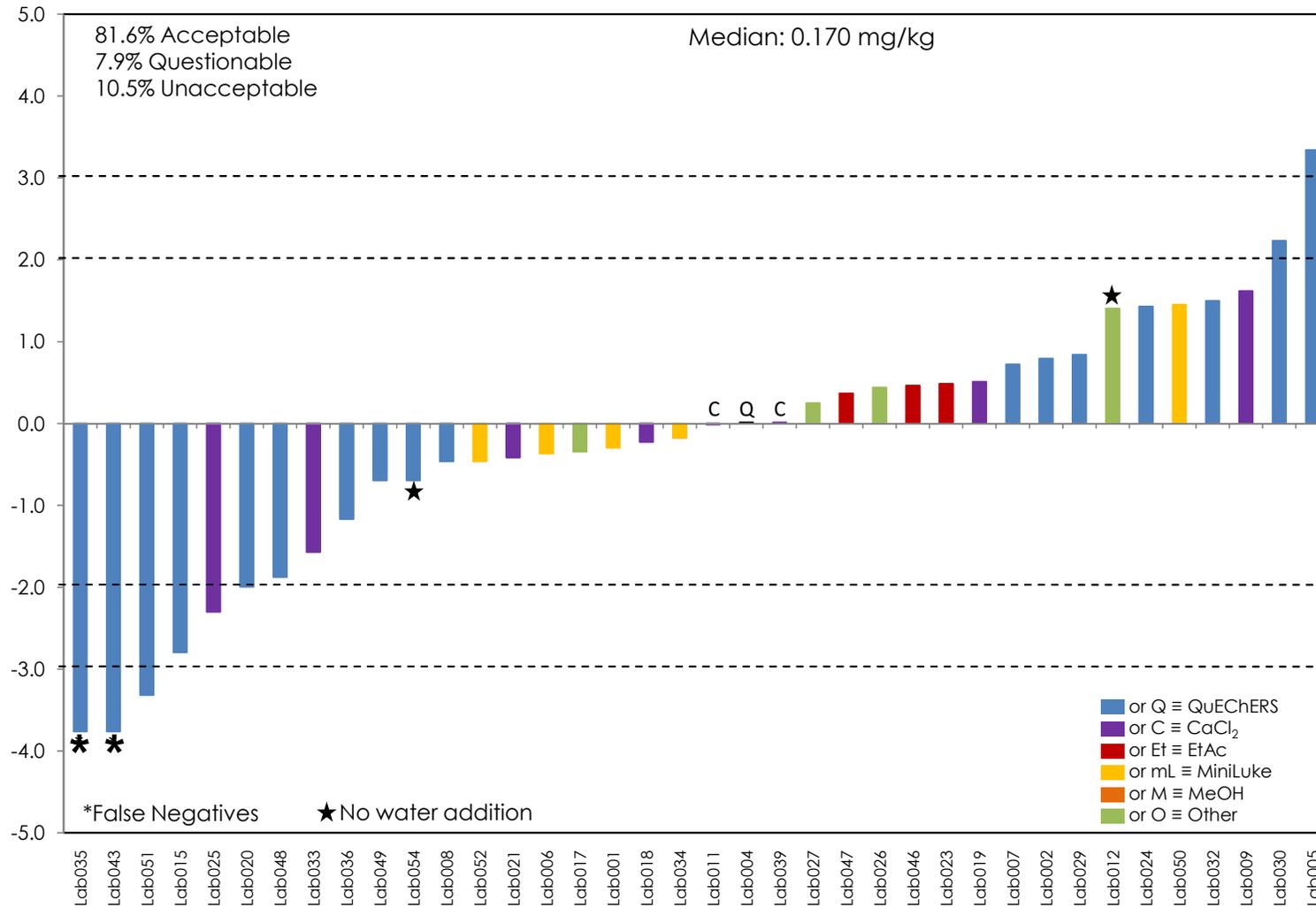
## Endosulfan beta





# Z-Scores

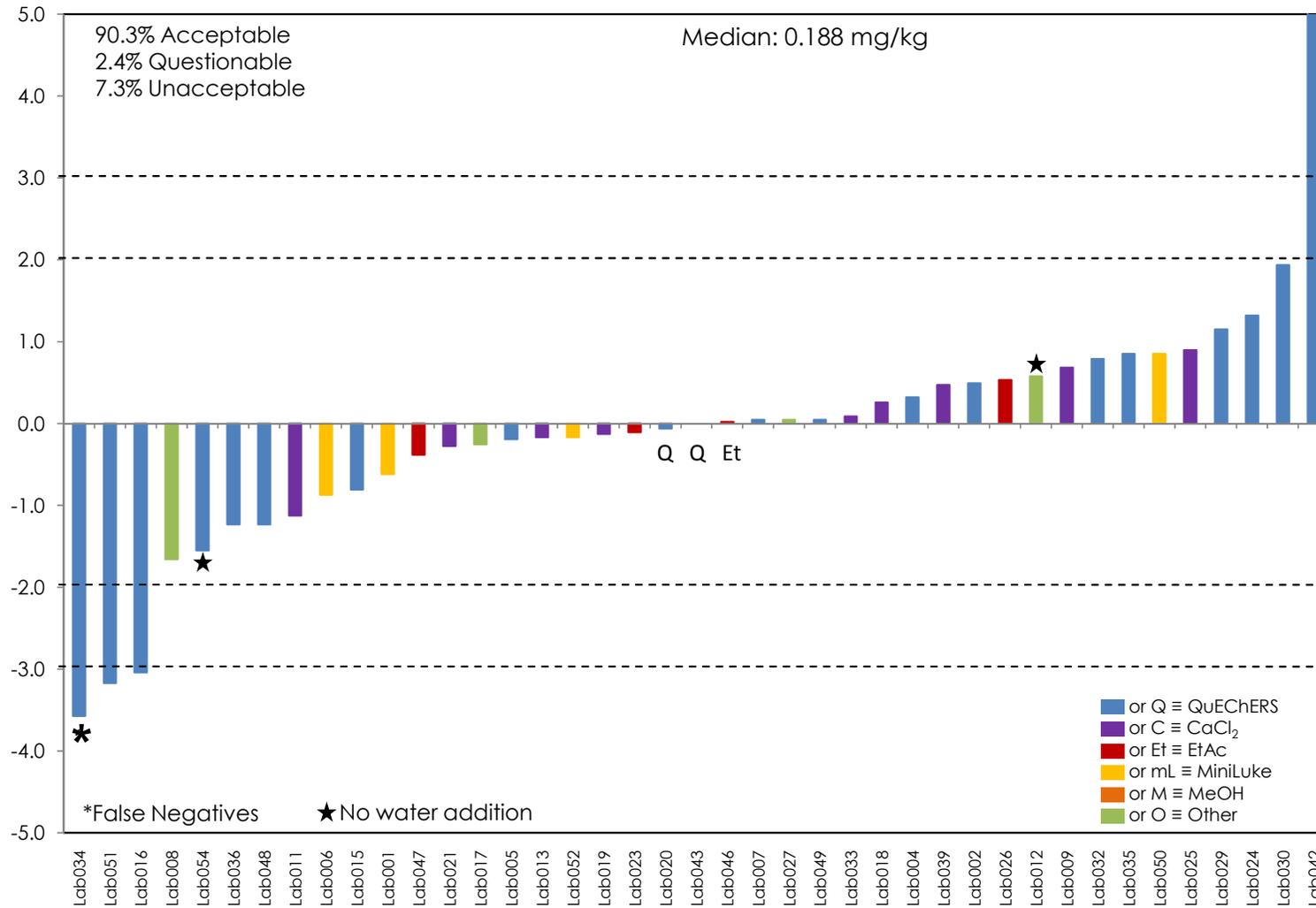
## Etofenprox





# Z-Scores

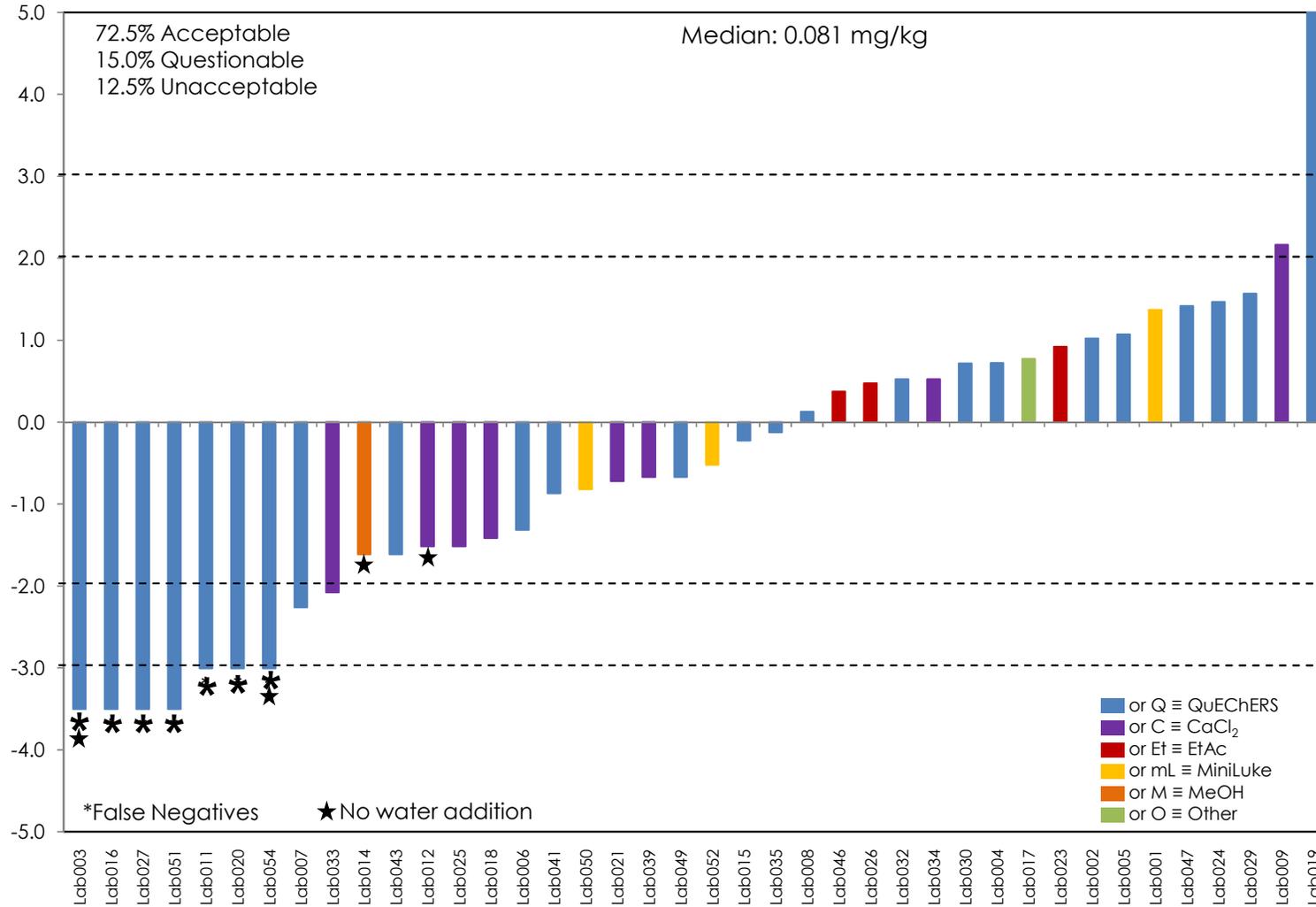
## Fenpropathrin





# Z-Scores

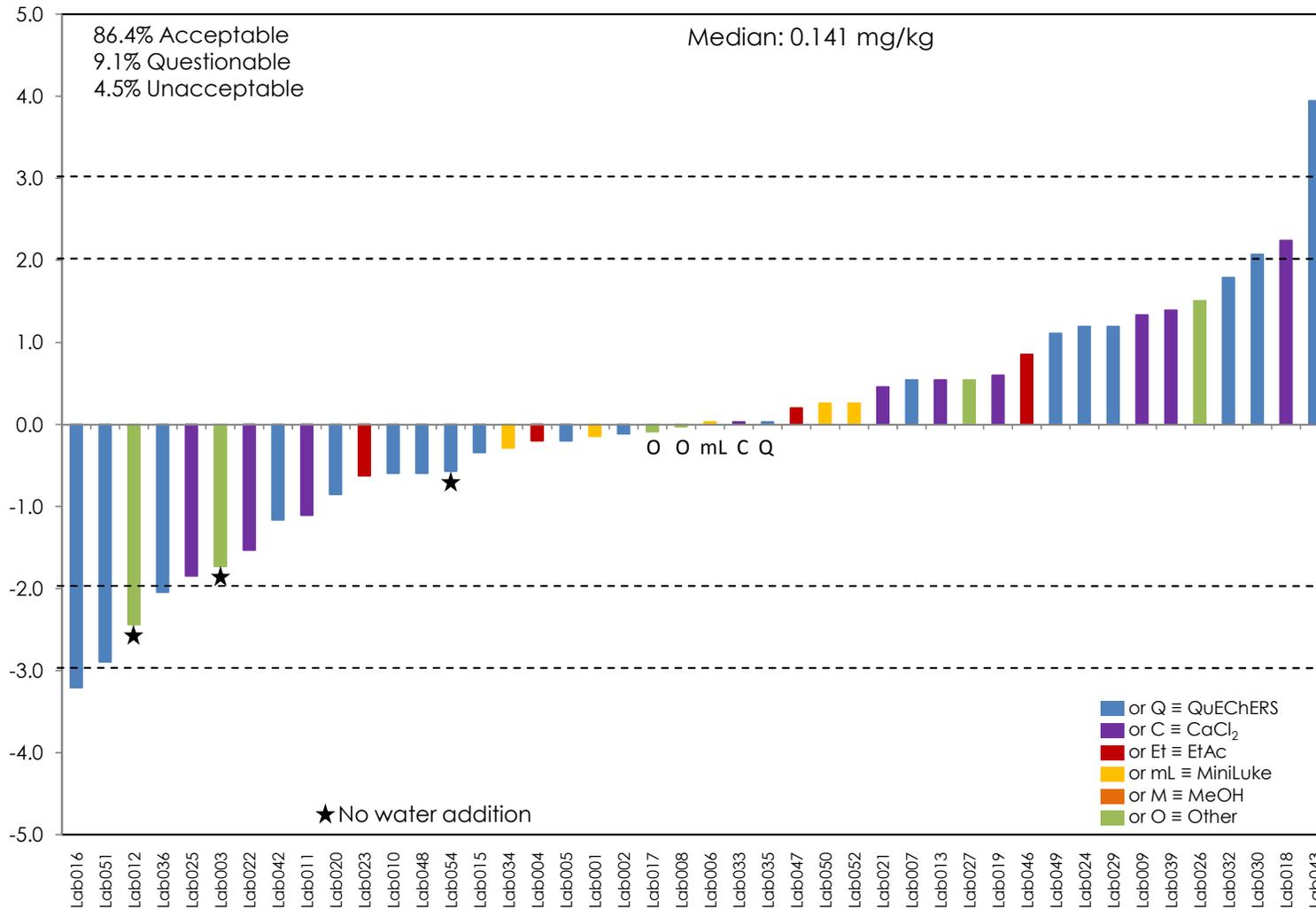
## Imidacloprid





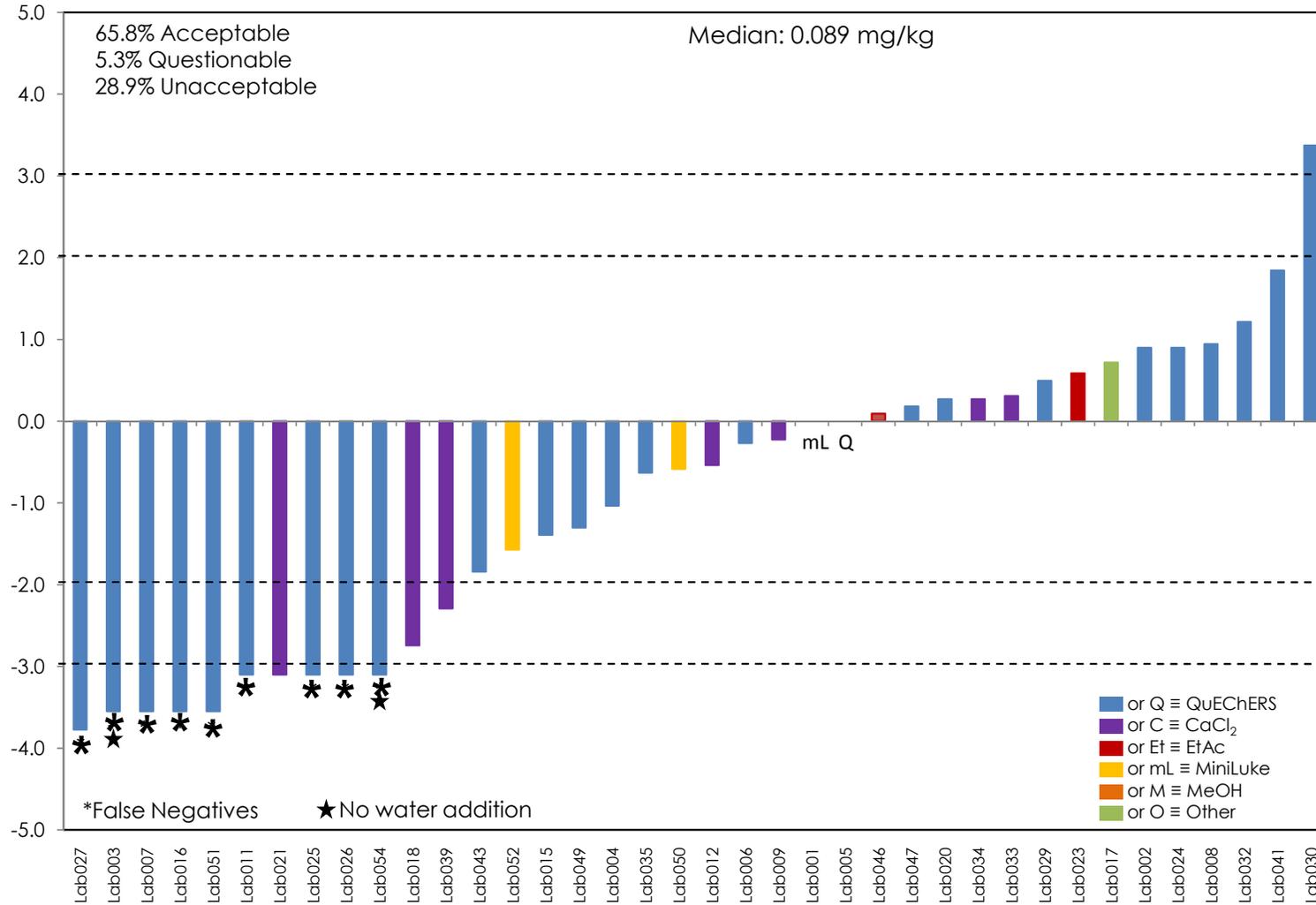
# Z-Scores

## Lambda cyhalothrin



# Z-Scores

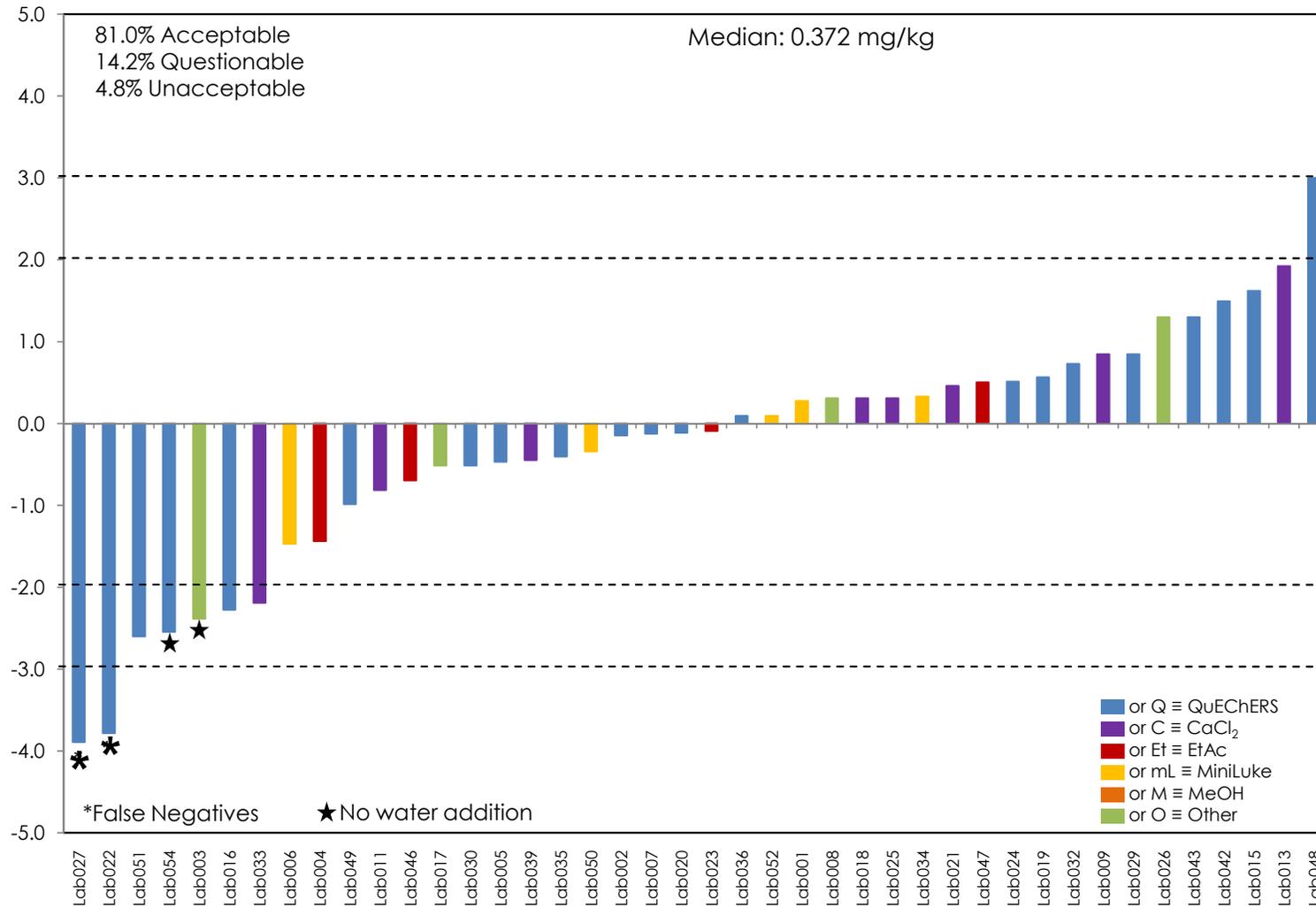
## Methomyl





# Z-Scores

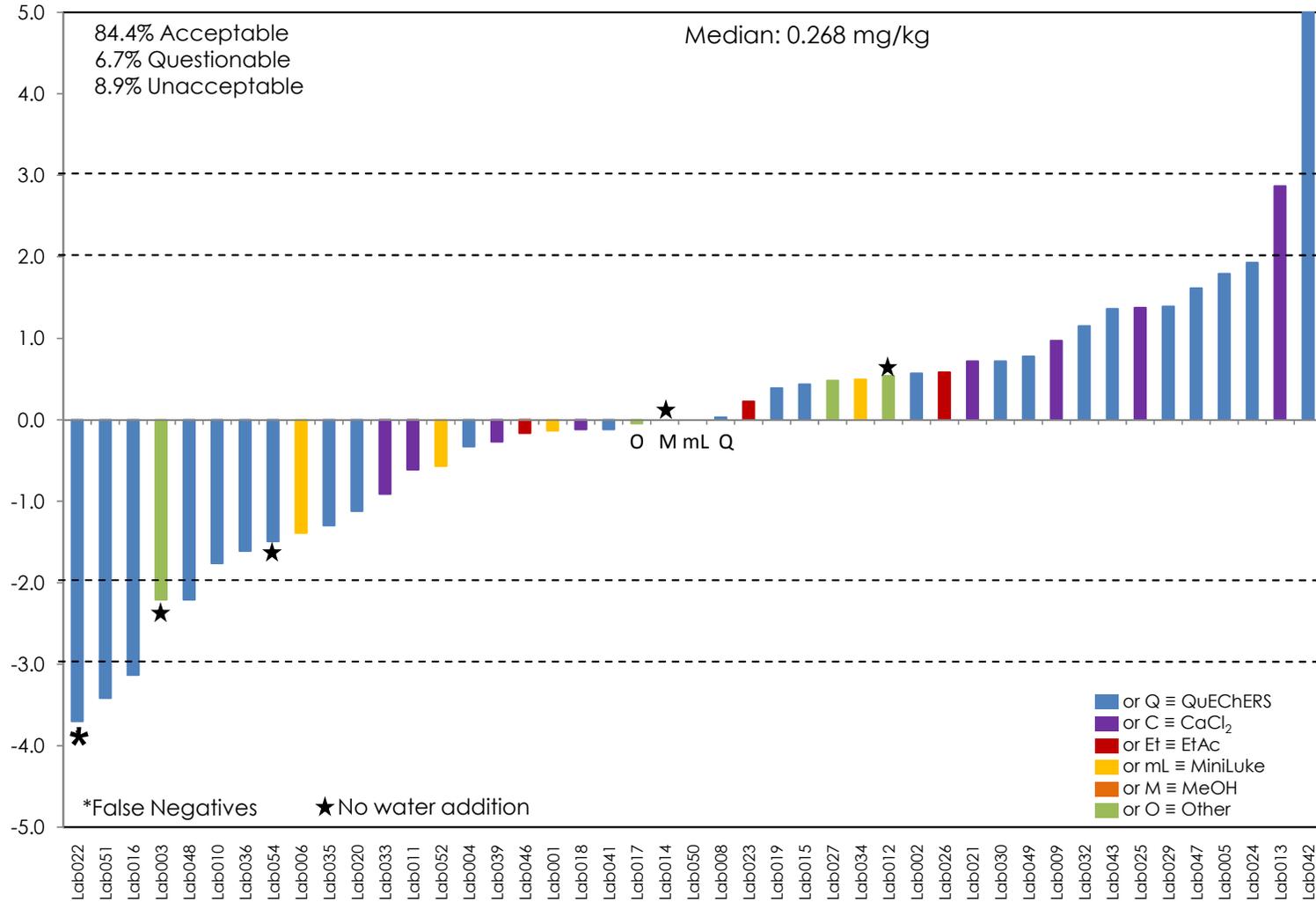
## Parathion-ethyl





# Z-Scores

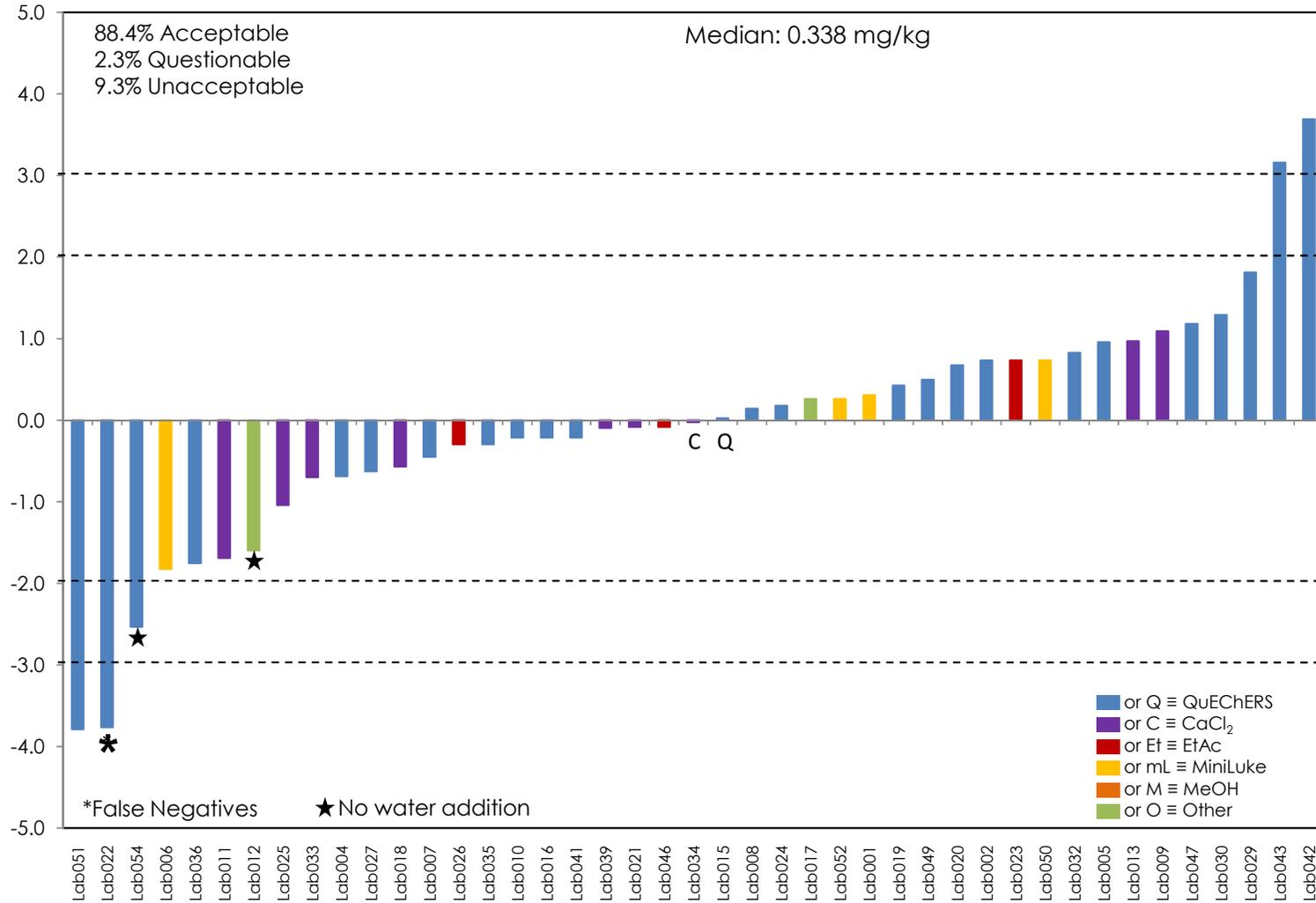
## Pyridaben





# Z-Scores

## Tebuconazole

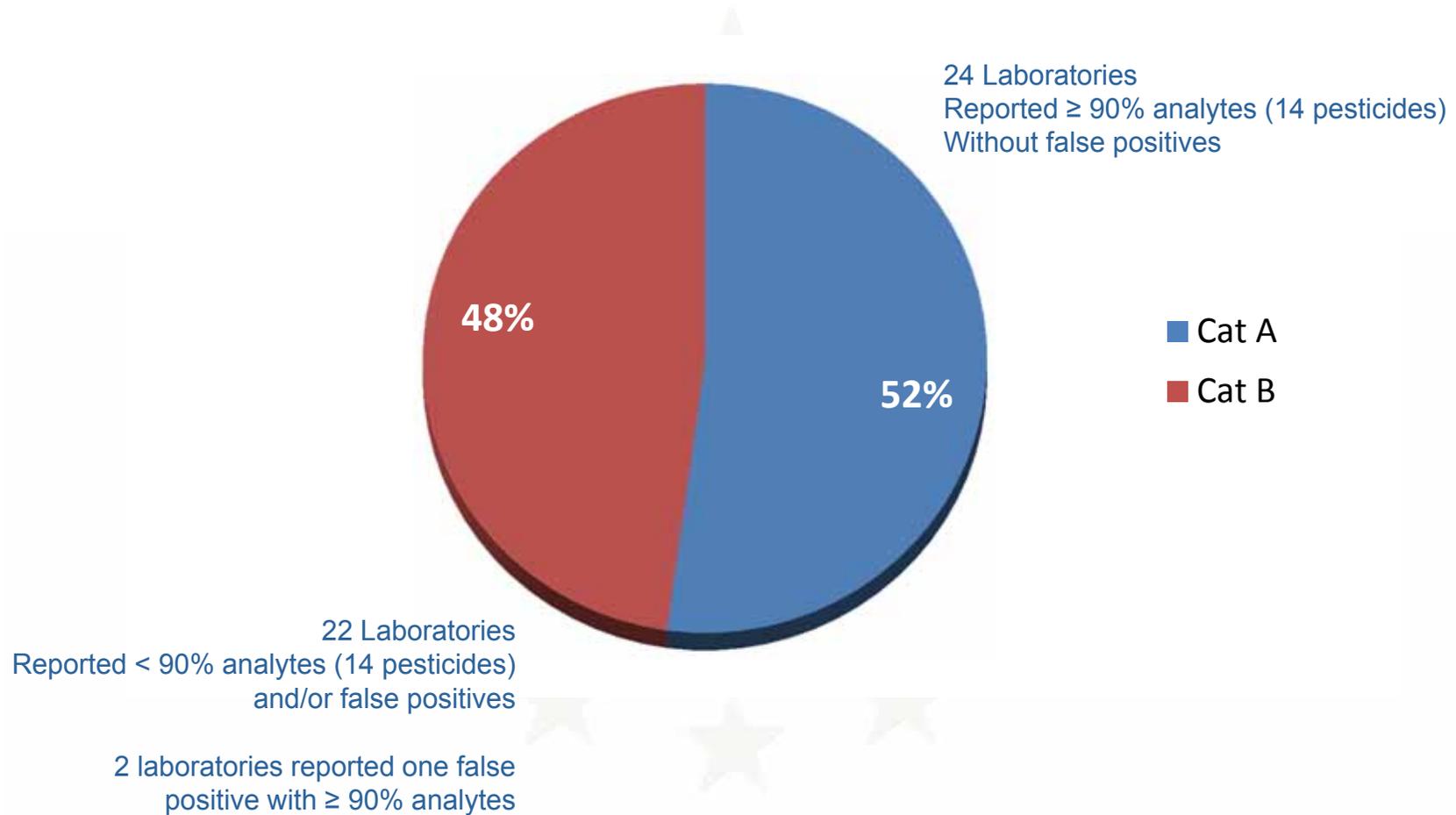


## Classification of Z-scores for the pesticides reported

Pesticides	Acceptable (%)	Questionable (%)	Unacceptable (%)
Acetamiprid	72.5	5.0	<b>22.5</b>
Buprofezin	79.6	6.8	13.6
Carbendazim	57.1	23.8	<b>19.1</b>
Chlorpyrifos	88.6	4.6	6.8
Cypermethrin	62.2	16.2	<b>21.6</b>
Difenoconazole	90.2	4.9	4.9
Endosulfan beta	70.7	22.0	7.3
Ethofenprox	81.6	7.9	10.5
Fenpropathrin	90.3	2.4	7.3
Imidacloprid	72.5	15.0	12.5
Lambda cyhalothrin	86.4	9.1	4.5
Methomyl	65.8	5.3	<b>28.9</b>
Parathion ethyl	81.0	14.2	4.8
Pyridaben	84.4	6.7	8.9
Tebuconazole	88.4	2.3	9.3



## Category A and B





**EURL-FV**

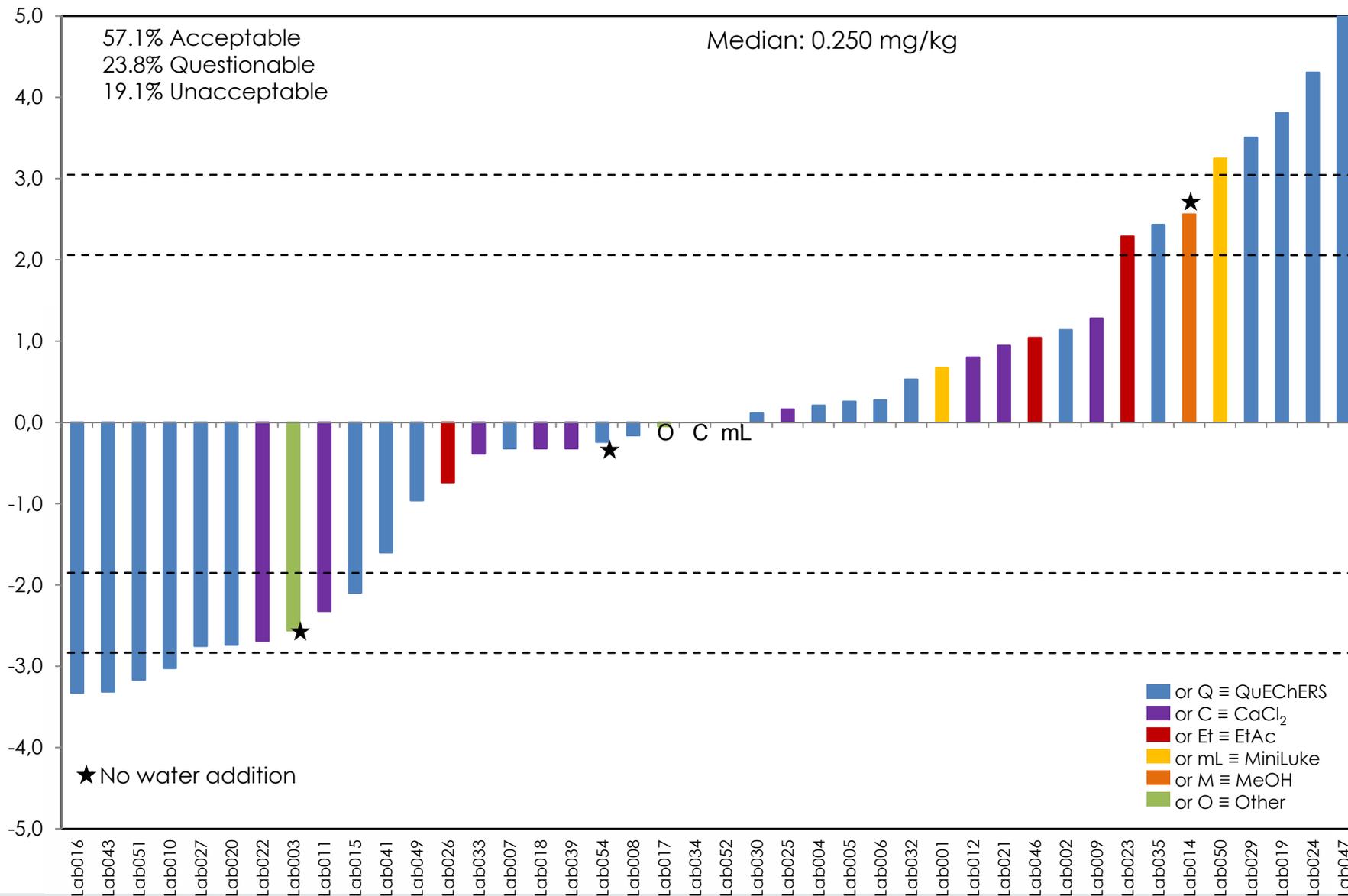


Almería 23<sup>rd</sup>-25<sup>th</sup> October 2013

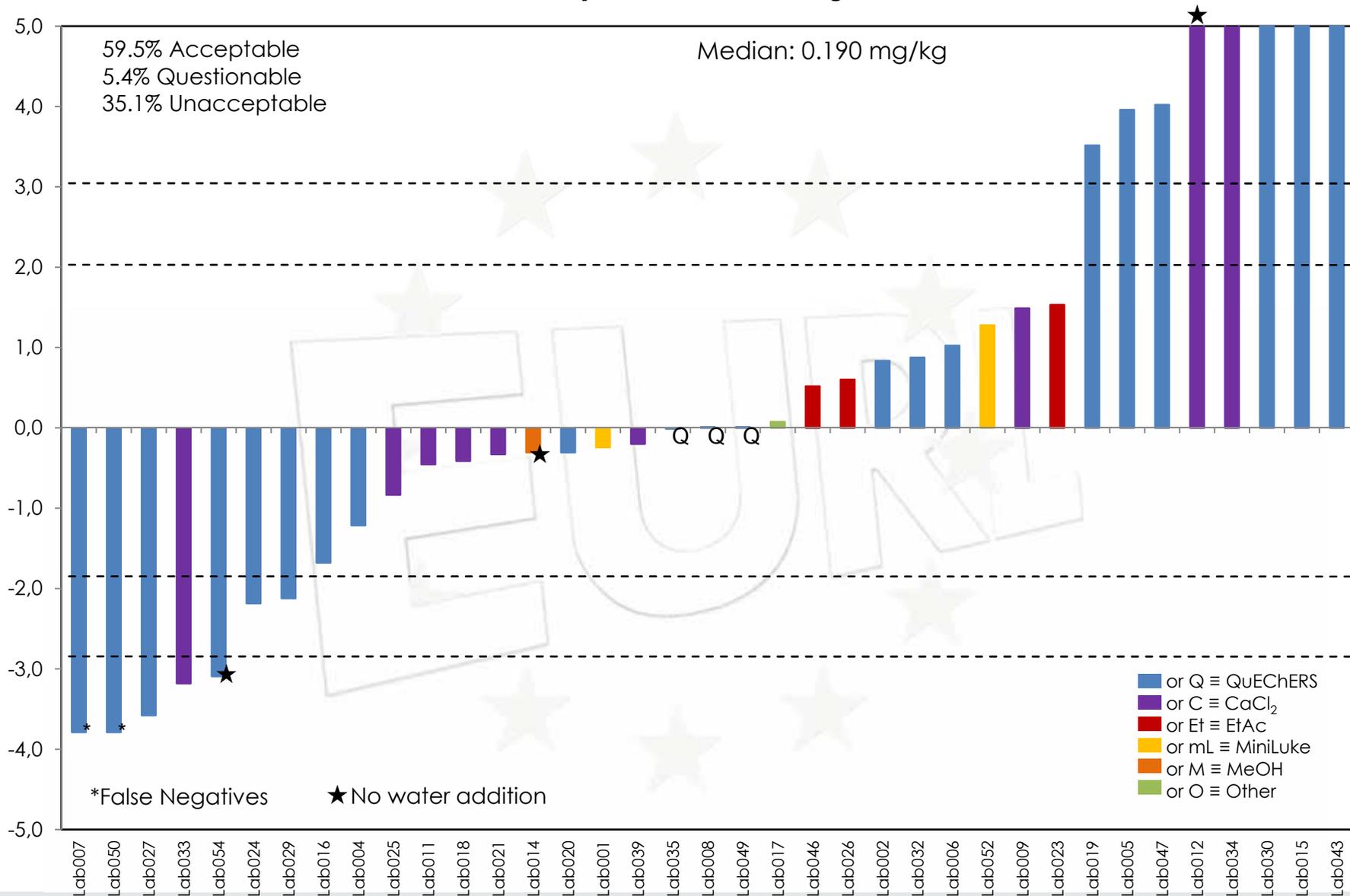
# Special case



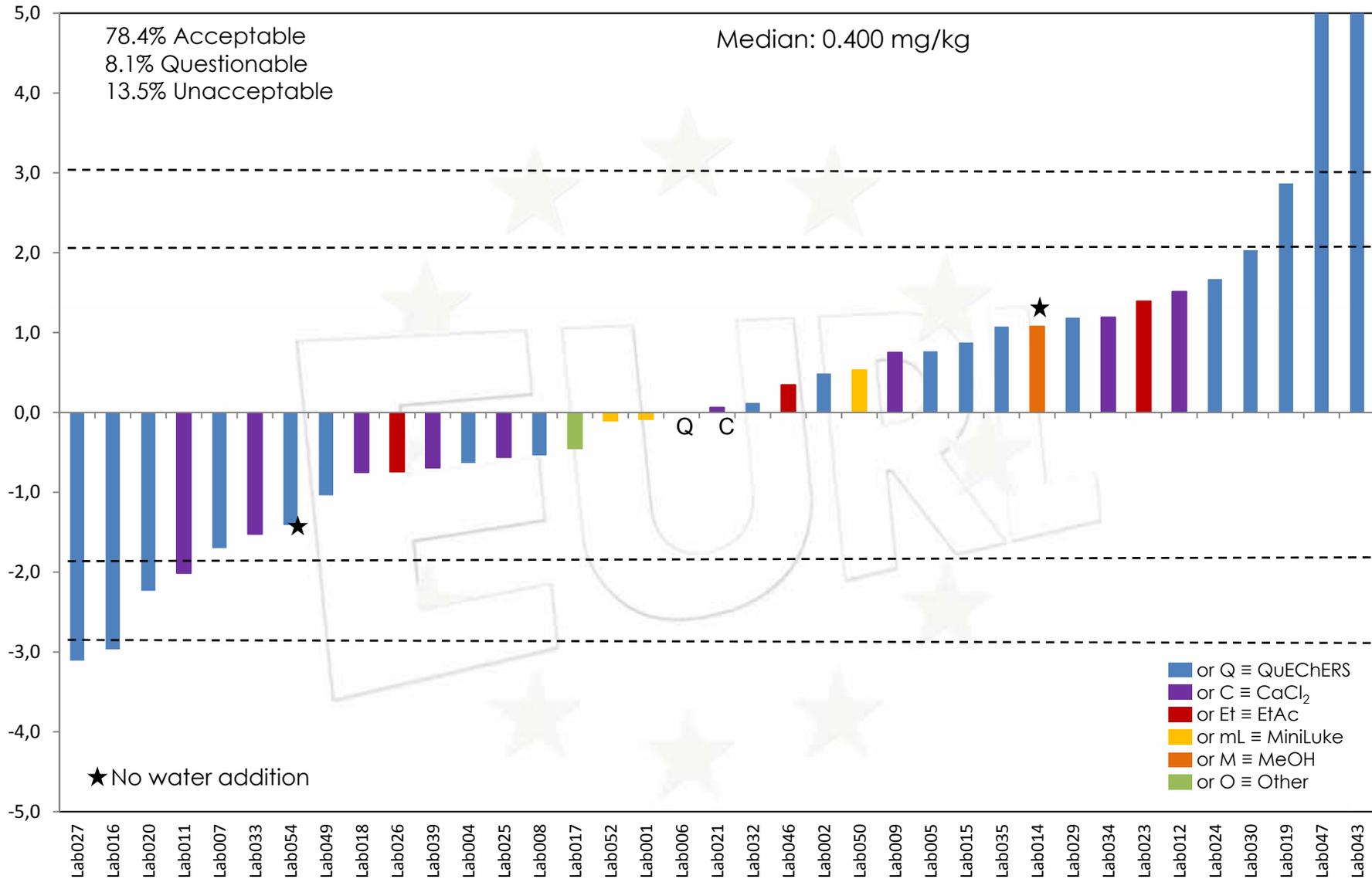
# Carbendazim



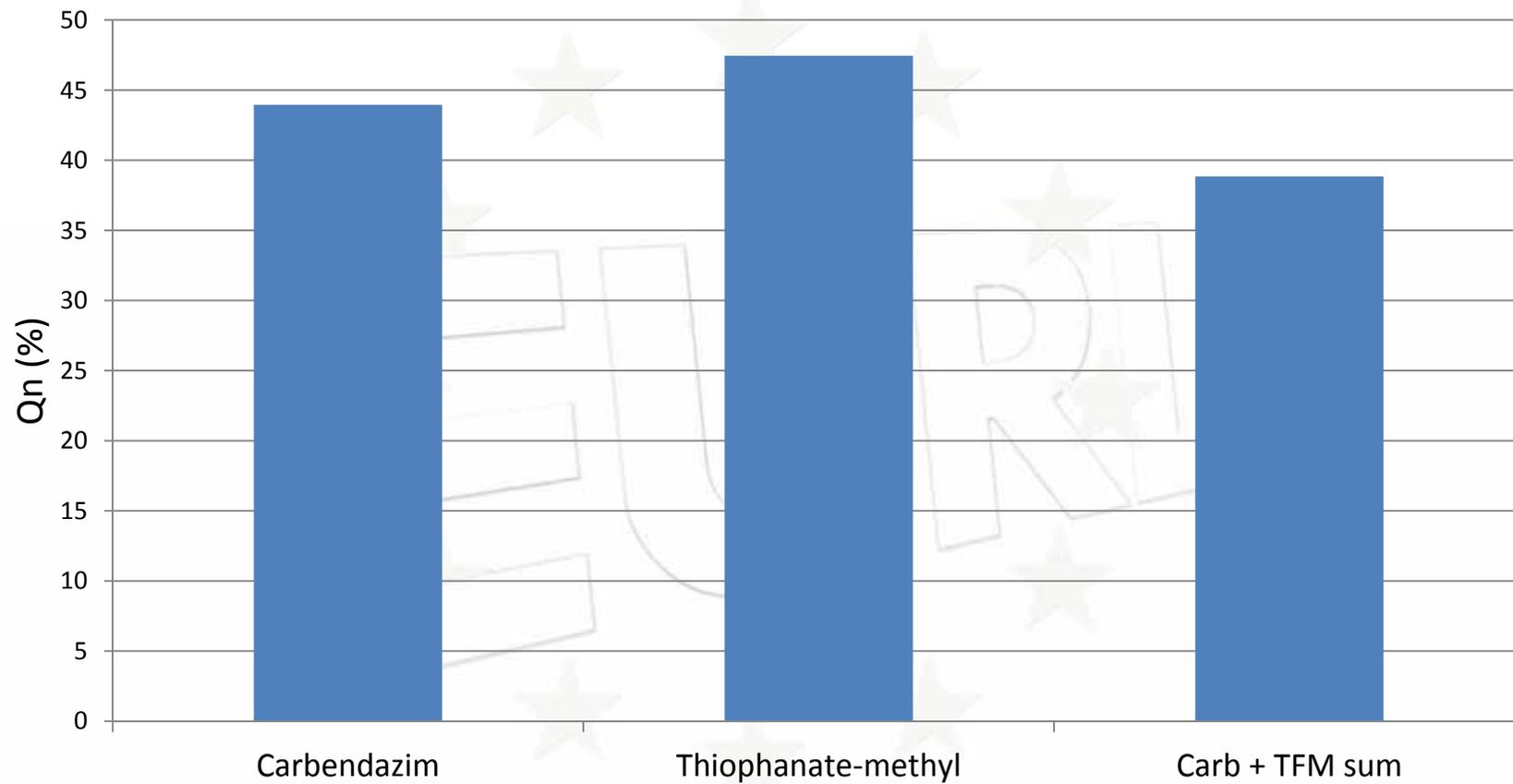
# Thiophanate methyl



### Carbendazim + Thiophanate methyl expressed as Carbendazim

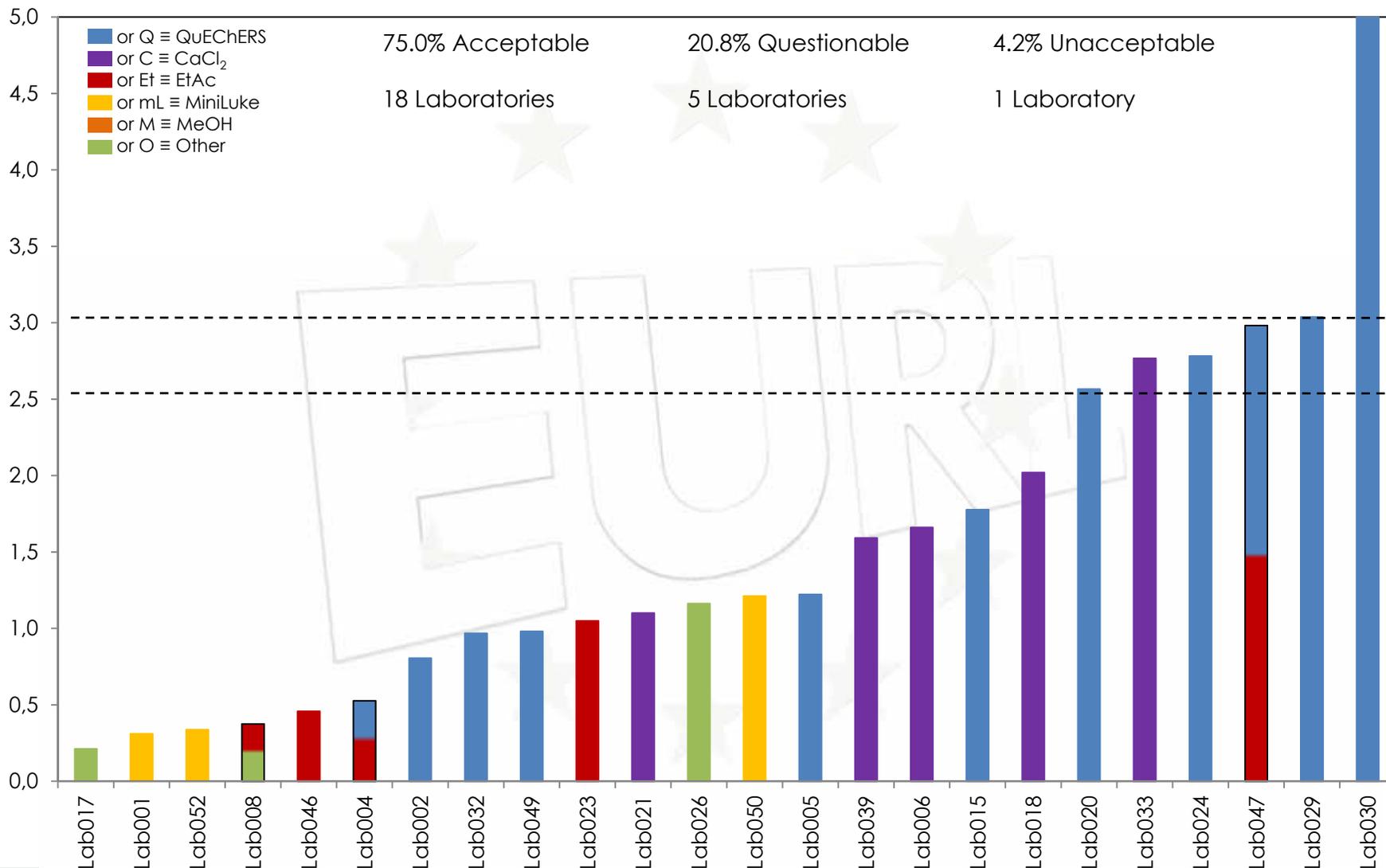


## Qn (%)



## Graph using individual values for Carbendazim and Thiophanate methyl

EUPT-PT01 Graphical representation for laboratories in Category A





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Almería 23<sup>rd</sup>-25<sup>th</sup> October 2013

# Conclusions

# Conclusions

- Fifty-two laboratories participated in EUPT-T01 (Six from non EU or EFTA countries, 16 NRLs).
- Pesticides considered as positives were those which were reported by both the Organiser and the majority of participants.
- A higher dispersion (Qn) than in most fruits and vegetables have been observed (As maximum 44%).
- For carbendazim and thiophanate methyl, the results were influenced by the different degrees of degradation obtained by participants during the sample handling.
- QuEChERS without calcium chloride addition modification, presented a higher dispersion accounted as Qn than for the whole data reported (average Qn for QuEChERS was 41% whereas for the combined data, it was 34%).
- One compound did not pass the homogeneity test (thiophanate methyl). This fact is clearly as a consequence of some degradation to carbendazim.
- Overall, the results can be considered to be good with regard to the z-scores for each pesticide present in the test item, except for some pesticides which obtained a value more than 20% of unacceptable results (3 of them).
- It would appear that multiresidue methods such as QuEChERS have improved following modification (CaCl<sub>2</sub> addition).
- All of the laboratories applying the miniLuke method obtained acceptable results in terms of z-scores, except for carbendazim.

# 4<sup>th</sup>

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**2013**

**EUROPEAN COMMISSION  
PROFICIENCY TEST  
FOR PESTICIDE RESIDUES IN FRUITS  
AND VEGETABLES  
SCREENING METHODS 05**



European  
Commission

# EURL

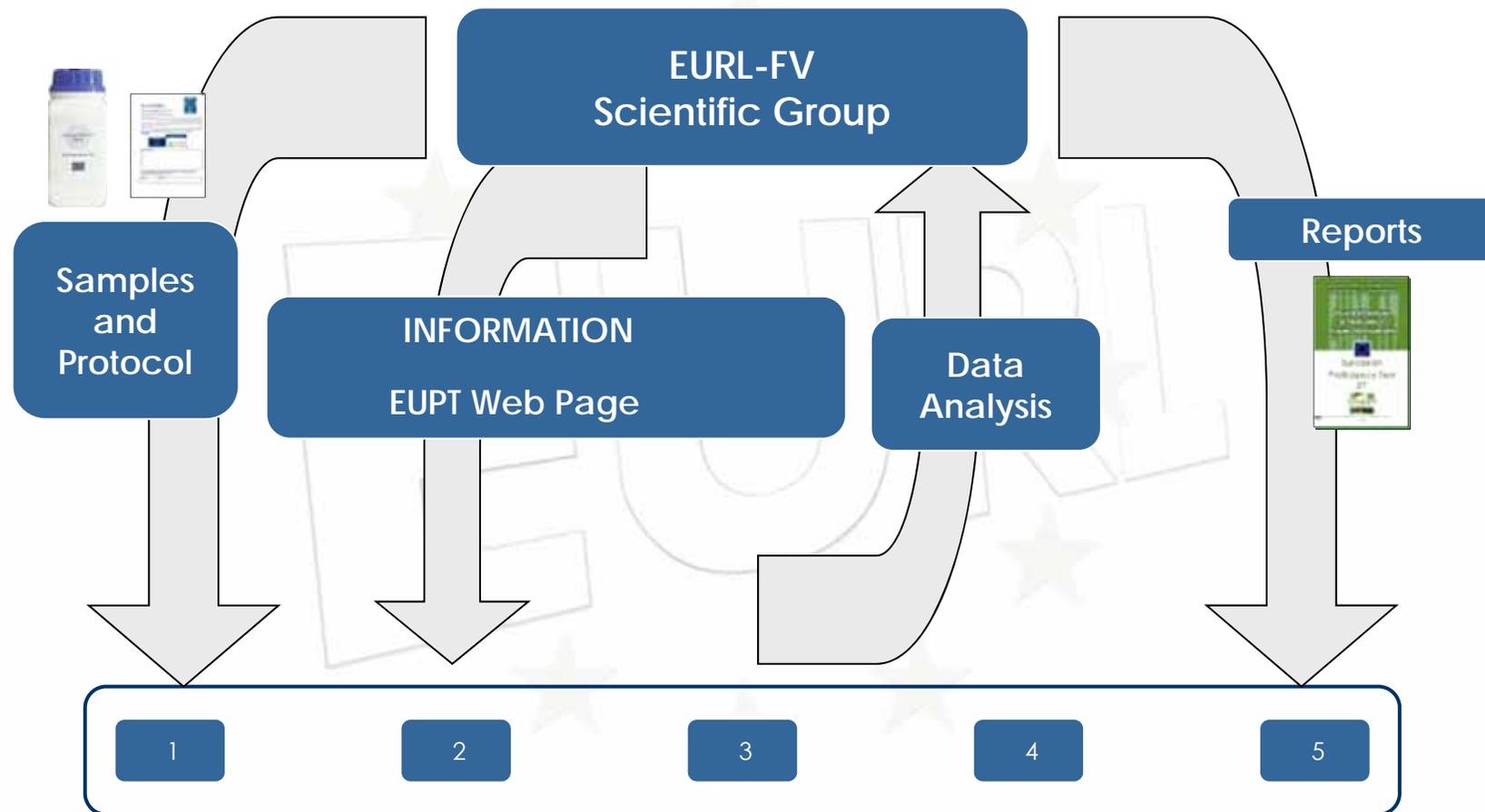


## AMADEO R. FERNÁNDEZ-ALBA

EUROPEAN UNION REFERENCE LABORATORIES



## General European Commission Proficiency Test Scheme



ACTIVITY	DATE
Publishing the Calendar and Matrix on the EURL-FV Web page.	23 <sup>rd</sup> October 2012
Submission of Application Form by invited laboratories.	3 <sup>rd</sup> Dec. 2012 - 7th Jan 2013
Sample distribution.	21 <sup>st</sup> January 2012
Submission of sample receipt and acceptance - Form 0.	As soon as sample is received
Deadline for receiving results - Form 1	<b>72 hours after receiving the sample</b>
Preliminary Report.	March 2013
Final Report distributed to the Laboratories.	December 2013

**EUROPEAN PROFICIENCY TEST - FRUITS AND VEGETABLES -  
MASS SCREENING 04- Participants** 17 NRLs\*

COUNTRY	No.	COUNTRY	No.
Austria	1*	Italy	1*+5
Belgium	1*+2	Latvia	1*
China	2	Romania	2*
Croatia	1	Serbia	1
Czech Republic			1*
Denmark	1*	Spain	6
Estonia	1*	Sweden	1*+1
Finland	1*	Switzerland	1*+1
France	1*+7	The Netherlands	1*+1
Germany	7	Turkey	1
Greece	1	UK	1*+2
Hungary	1*+2		

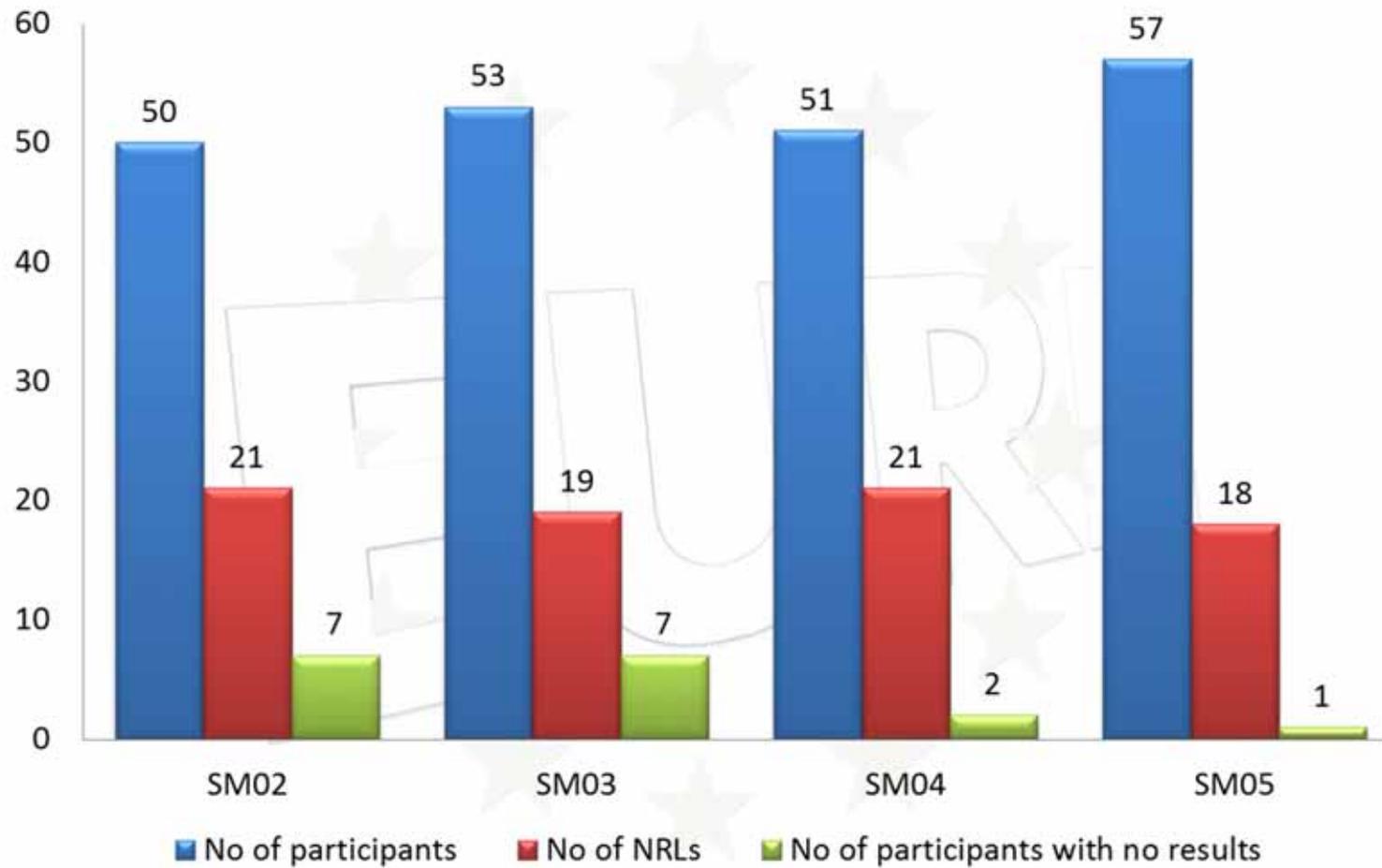
58

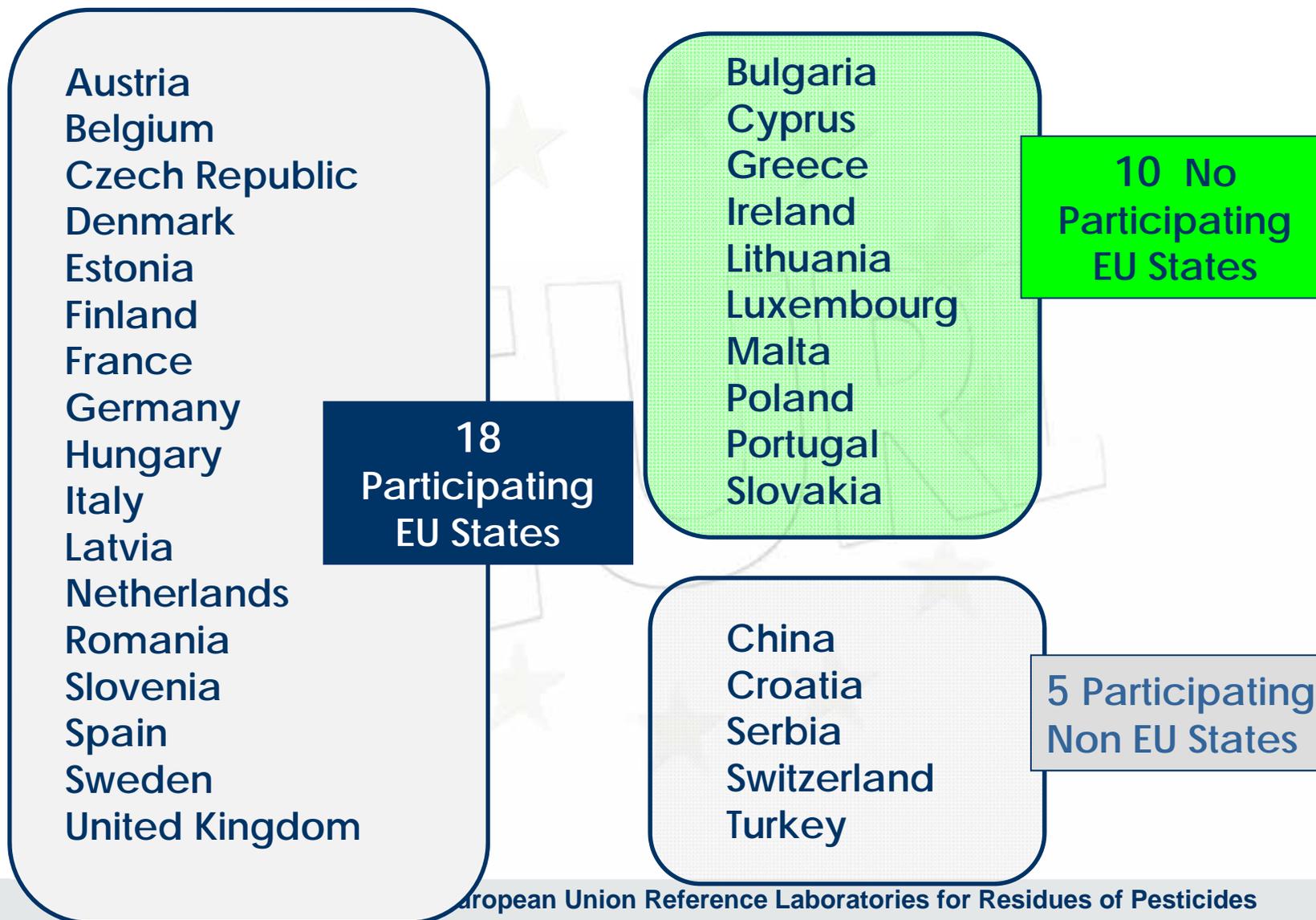
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## EUPT-FV-SCREENING METHODS 5

Participants with no results

COUNTRY	No. of laboratory
Slovenia (results after the deadline)	3





# Homogeneity test

Test item No.	004 a	004 b	007 a	007 b	023 a	023 b	064 a	064 b	066 a	066 b	076 a	076 b	101 a	101 b	122 a	122 b	144 a	144 b	148 a	148 b	A. Cc* (mg/kg)	RSD (%)
Aldicarb sulfone	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.210	14.7
Aldicarb Sulfoxide	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.426	9.0
Atrazine	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.301	1.2
Beta-Cyfluthrin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.201	9.1
Bromoxynil	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.386	7.8
Clomazone	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.083	6.1
Dieldrin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.400	6.4
Diuron	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.935	4.5
Fluazinam	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.030	13.6
Flufenacet	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.126	7.3
Metamitron	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.184	6.8
Methomyl#	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.037	9.4
Metribuzin	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.225	4.4
Molinate	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.060	12.3
Oxamyl	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.092	7.4
Picloram	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.098	13.1
Picolinafen	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.488	13.7
Propamocarb	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.082	7.7
Prosulfocarb	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.247	9.3
Quinoclamine	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.073	9.6
Thiodicarb#	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.068	10.1
Triflumizole	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	0.022	14.9

D: Detected  
 A. Cc: Average Concentration  
 # Thiodicarb degradation product (not spiked)  
 \* Concentration evaluated by Organiser

RSD: Relative Standard Deviation (n=20)



European Commission Proficiency Test for Pesticide Residues in Fruits and Vegetables Screening Methods 05  
Preliminary Report

Laboratory Code Total No of Reporting Laboratories = 38 ( <sup>1</sup> NB: FV)	Evaluated Pesticides (22) (in green pesticides with concentration <100 ppb)																				Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory		
	Aldicarb sulfone	Aldicarb sulfoxide	Atrazine	Beta-Cyfluthrin	Bromoxynil	Clomazone	Dieldrin	Diuron	Fluazinam	Flufenacet	Metamitron	Methomyl#	Methibuzin	Mellicafe	Oxamyl	Picloram	Picolinifen	Propamocarb	Prosulfocarb	Quinocloramine			Thiodicarb#	Trifluralin
Lab001	R	R	R	R	R	R	R	R		R	R	R	R	R	R		R	R	R	R	R	R	18	82
Lab002	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
Lab003*																							0	0
Lab004*	R	R				R	R	R		R	R	R	R		R		R	R	R	R	R	R	16	73
Lab005*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	21	95
Lab006*	R	R	R	R		R	R		R		R	R	R	R	R		R	R	R		R	R	15	68
Lab007	R	R		R			R		R		R	R	R	R	R		R	R	R		R	R	14	64
Lab008	R	R	R	R	R	R	R	R		R		R	R	R	R		R	R	R	R	R	R	16	73
Lab009	R	R	R		R	R		R		R		R	R	R	R		R	R	R		R		15	68
Lab010*		R	R	R	R	R	R	R		R		R	R	R	R		R		R		R	R	15	68
Lab011	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	21	95
Lab012*	R	R	R	R		R	R		R		R	R	R	R	R		R	R	R		R	R	13	59
Lab013	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	20	91
Lab014*	R	R	R	R	R	R	R	R		R	R	R		R	R		R	R	R	R	R	R	19	86
Lab015*	R	R	R	R			R	R				R	R	R	R		R	R	R		R	R	11	50
Lab016*	R	R	R	R	R	R	R	R		R	R	R	R	R	R		R	R	R		R	R	19	86
Lab017	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	20	91
Lab018		R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R		R	R	17	77
Lab019	R	R	R	R		R	R	R	R	R	R	R	R	R	R		R	R	R		R	R	14	64
Lab020*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	18	82
Lab021	R					R	R	R		R	R	R	R	R	R		R	R	R	R	R	R	18	82
Lab022	R	R	R	R			R		R		R	R	R	R	R		R	R	R	R	R	R	15	68
Lab023	R	R	R	R		R	R	R	R	R	R	R	R	R	R		R	R	R		R	R	18	82
Lab024*	R	R	R	R	R	R	R	R		R	R	R	R	R	R		R	R	R	R	R	R	19	86
Lab025*	R	R	R		R		R	R				R	R	R	R		R	R	R		R	R	12	55
Lab026	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R		R	R	20	91
Lab028			R	R																			4	18
Lab029	R	R	R	R	R	R	R	R							R		R	R	R	R	R	R	19	86
Lab030	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	21	95
Lab031	R	R	R	R		R	R	R	R	R		R	R	R	R		R	R			R	R	15	68
Lab032			R																				2	9
Lab033*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100



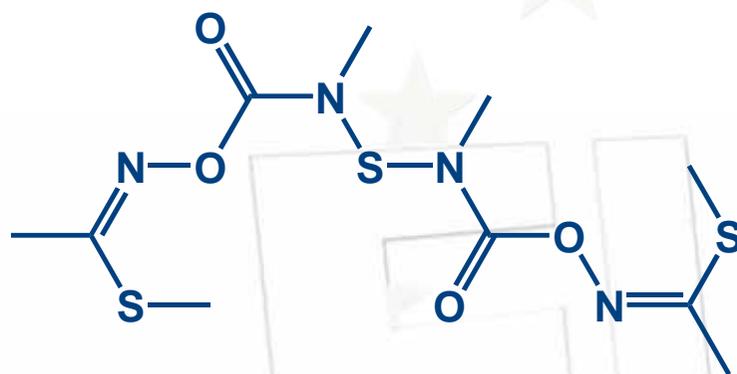
Laboratory Code (Total No of Reporting Laboratories = 58 [1=EU-FV])	Evaluated Pesticides (22) (in green pesticides with concentration <100 ppb)																					Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory	
	Aldicarb sulfone	Aldicarb sulfoxide	Alazine	Beta-Cyfluthrin	Bromoxynil	Clomazone	Diethrin	Duron	Fuaznham	Flufenacet	Metamifron	Methomyl#	Metribuzin	Molindde	Oxamyl	Picbryam	Picolinifen	Propamocarb	Prothiocarb	Quinoclamine	Thiodicarb#			Triflurzinole
Lab034*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	95
Lab035	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	19	86
Lab036			R	R	R	R	R	R		R	R	R	R				R	R					11	50
Lab037	R	R	R	R	R	R	R	R		R	R	R	R	R		R	R	R	R	R	R	R	19	86
Lab038	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	95
Lab039	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	95
Lab040	R	R	R	R		R	R	R		R	R	R	R	R	R		R	R	R	R	R	R	16	73
Lab041	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	21	95
Lab042	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
Lab043	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	21	95
Lab044	R	R	R		R	R	R	R		R	R	R	R	R	R	R	R	R	R		R	R	17	77
Lab046*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	20	91
Lab047	R		R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	19	86
Lab048			R	R	R	R	R	R	R	R		R	R			R	R	R	R	R	R	R	12	55
Lab049	R	R	R	R		R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	19	86
Lab050	R	R		R							R						R				R		7	32
Lab051	R	R					R				R												5	23
Lab053	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	22	100
Lab054			R	R	R	R	R	R		R			R	R		R	R	R				R	12	55
Lab055*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R		R	R	18	82
Lab056	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	21	95
Lab057	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R		R	R	18	82
Lab058*	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	21	95
Lab059	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R			R	R	19	86
Lab060	R	R	R	R			R				R	R	R	R							R		9	41
Lab061	R	R	R	R	R	R	R				R	R					R			R	R		13	59
Reported Pesticides	50	50	53	51	40	49	53	46	31	45	39	50	51	37	52	10	39	47	46	22	50	42		
% of Reported Pesticides	85	85	90	86	68	83	90	78	53	76	66	85	86	63	88	17	66	80	78	37	85	71		

R: Reported Pesticide.

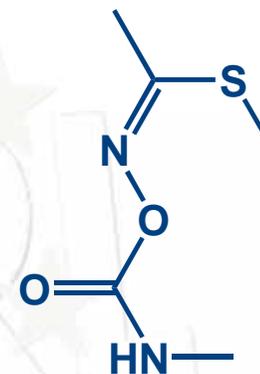
# The test material was spiked/treated with thiodicarb, which degrades to methomyl. The test material was not spiked/treated with methomyl.

Propazine, simazine and thiabendazole were not intentionally used to spike/treat the test material, but they were detected by the organisers at concentrations below 10 ppb due to impurities of the commercial formulations.

The test material was spiked/treated with **thiodicarb**, which degrades to **methomyl**. The test material **was not** spiked/treated with methomyl.



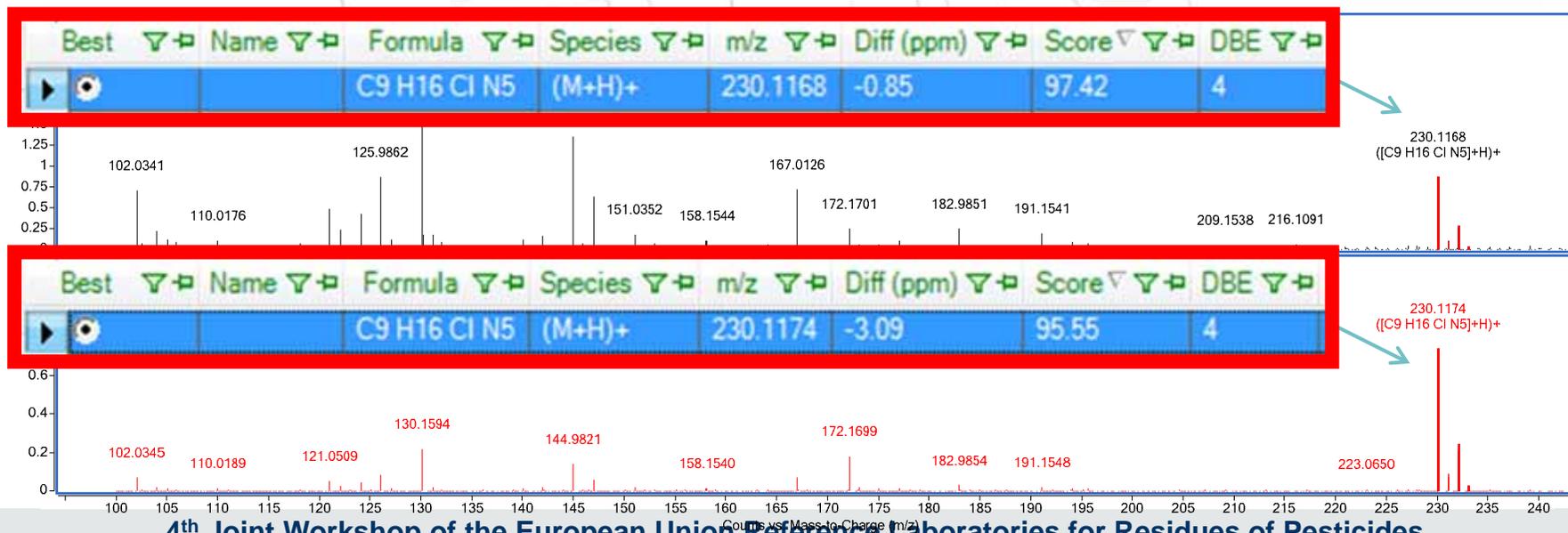
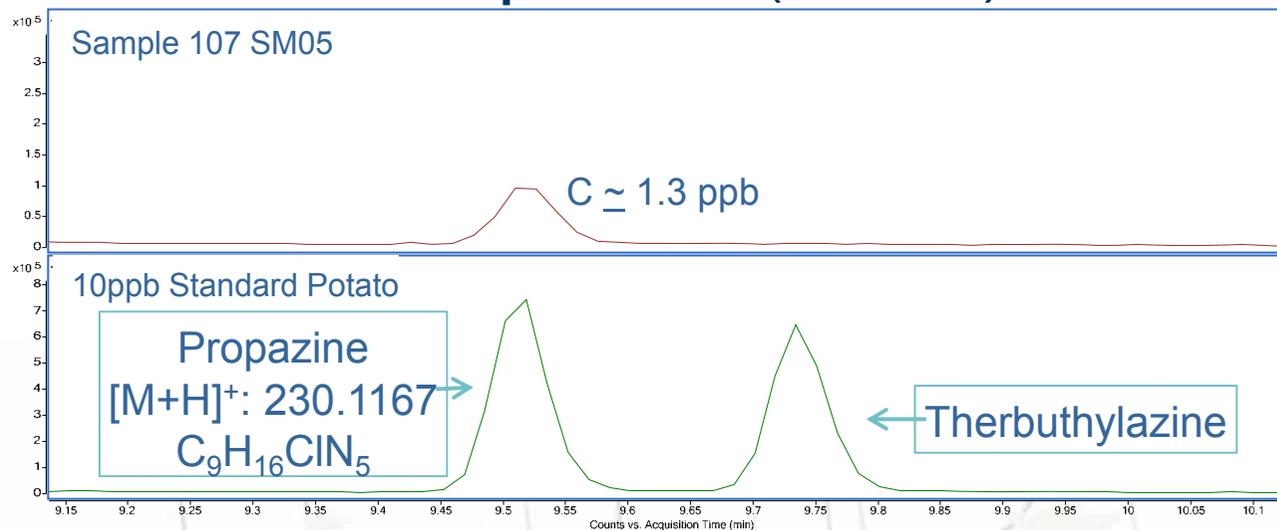
Thiodicarb



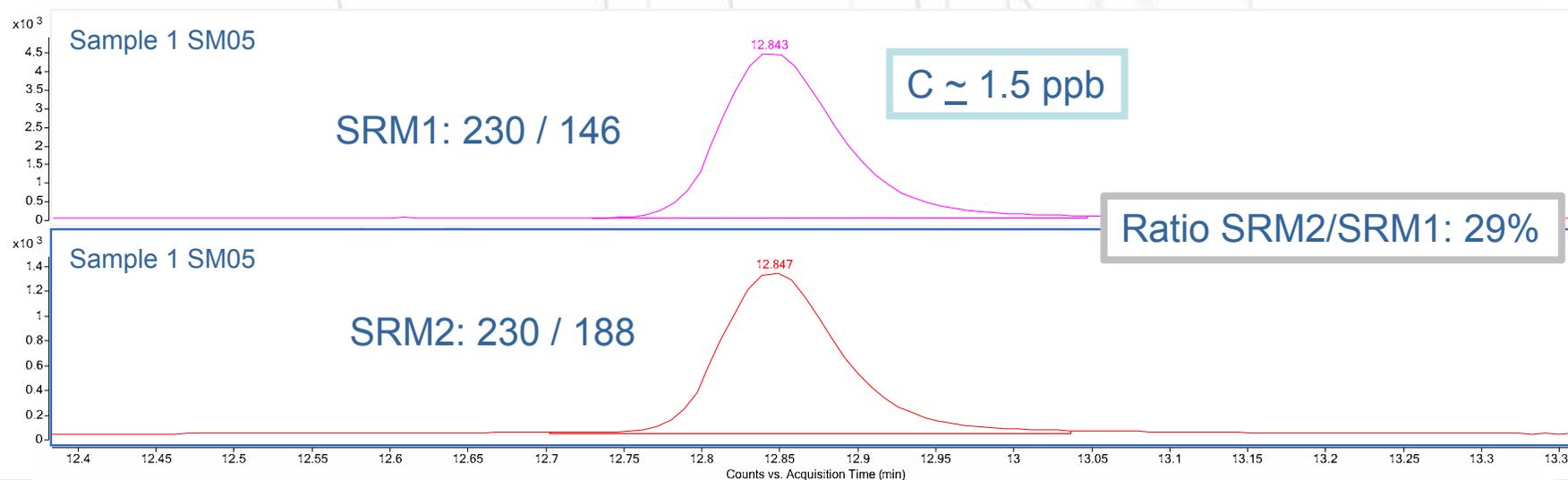
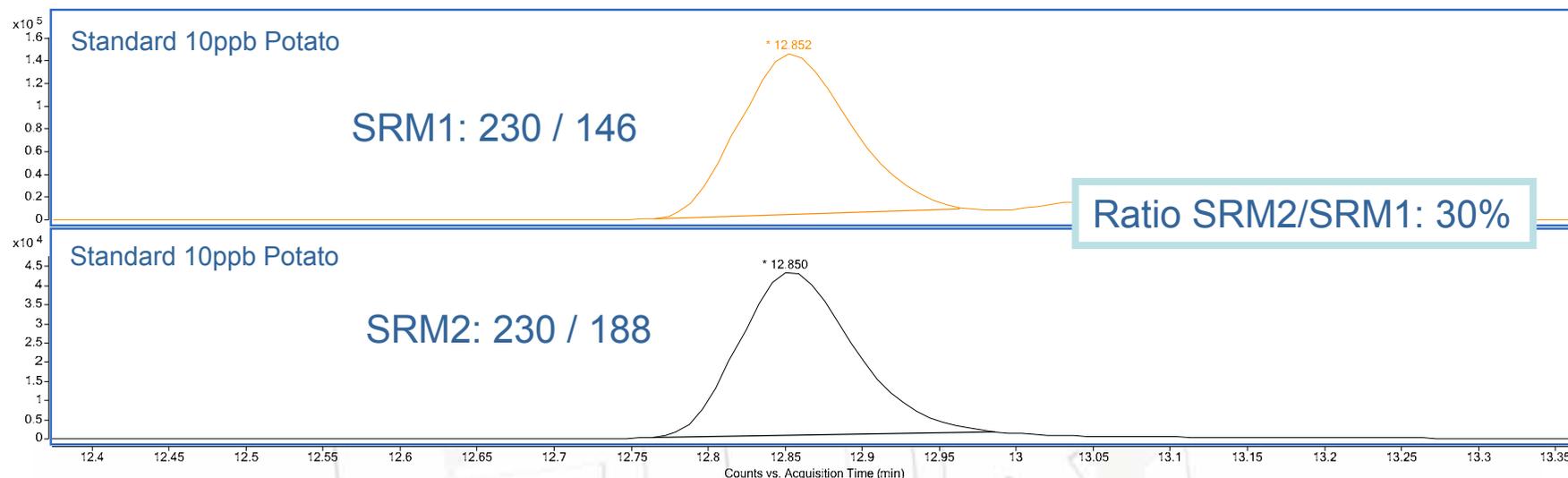
Methomyl

**Propazine, simazine and thiabendazole** were not intentionally used to spike/treat the test material, but they were detected by the organisers at concentrations below 10 ppb due to impurities of the commercial formulations.

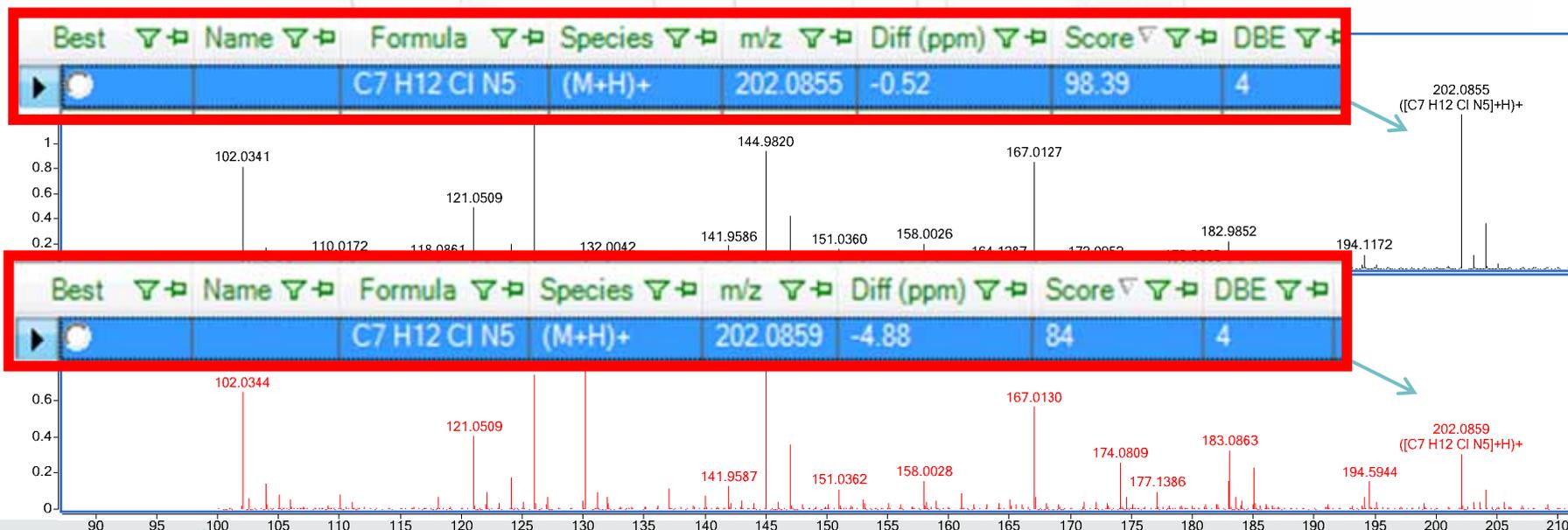
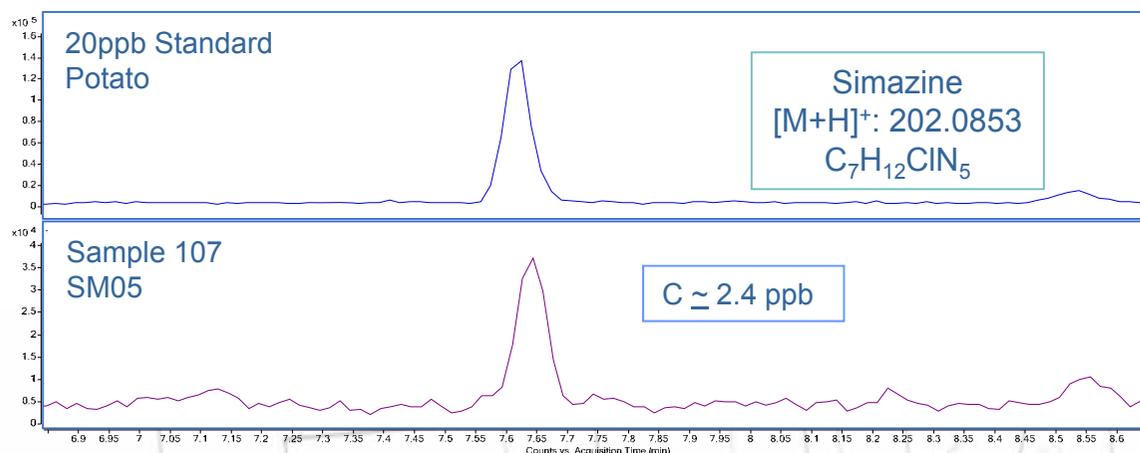
# Propazine (Q-TOF)



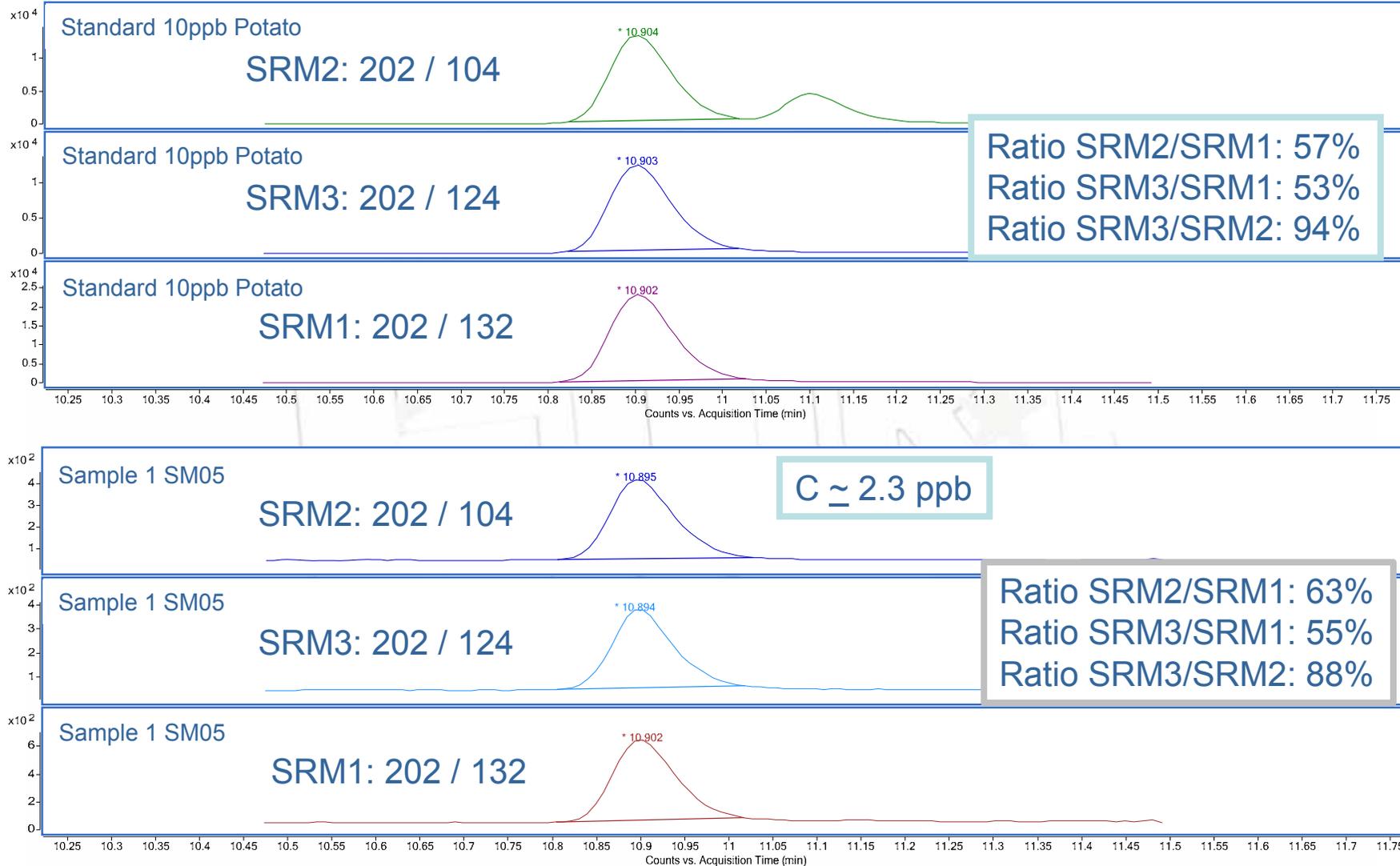
# Propazine (LC-QQQ)



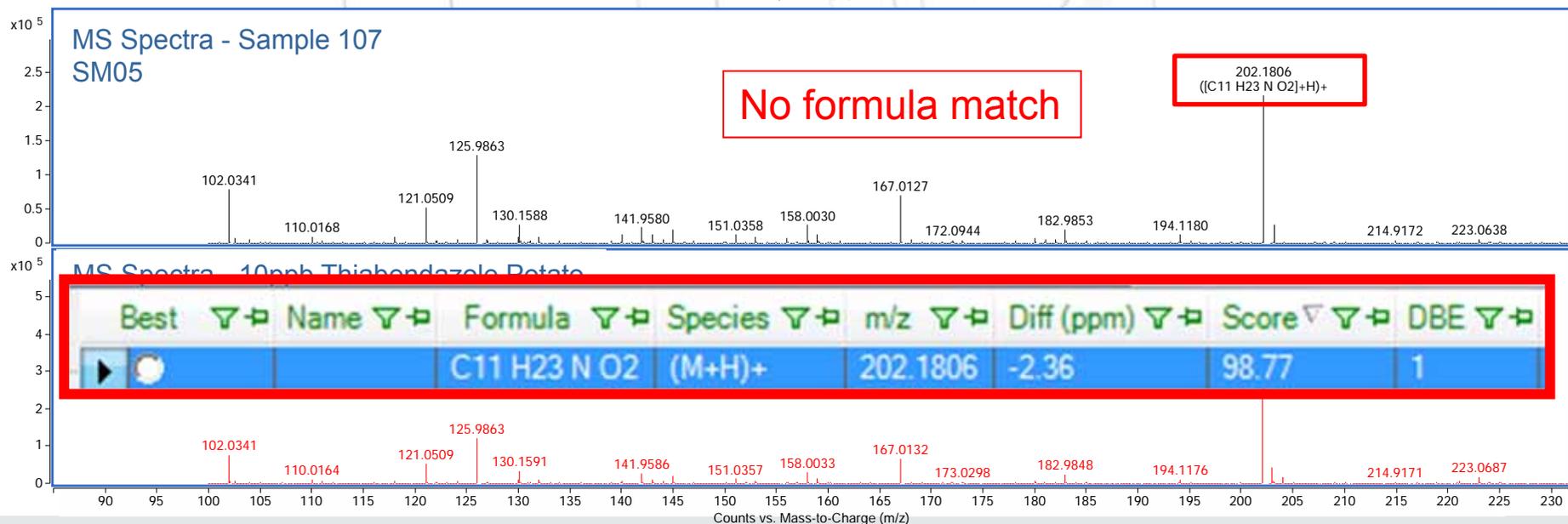
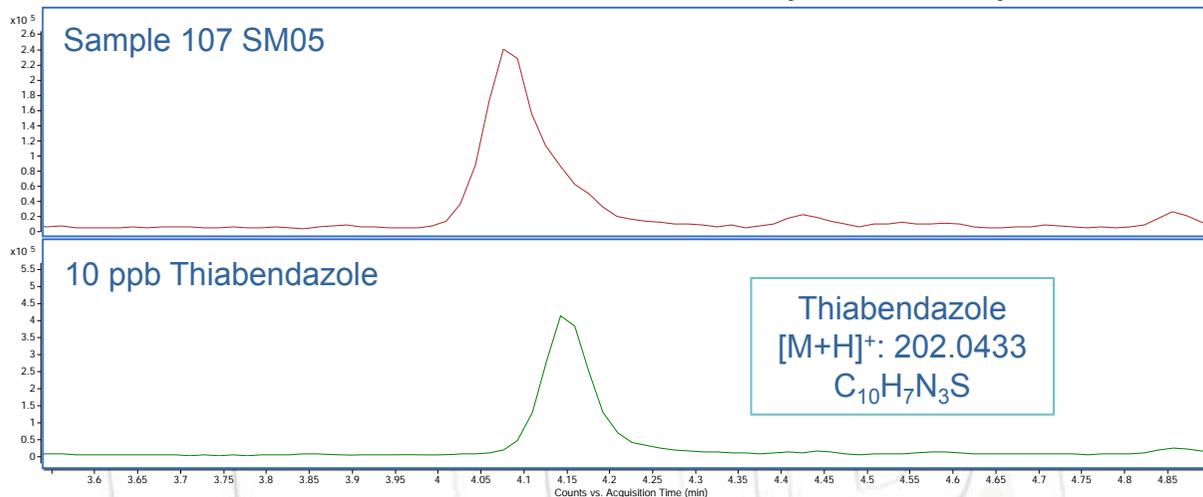
# Simazine (Q-TOF)



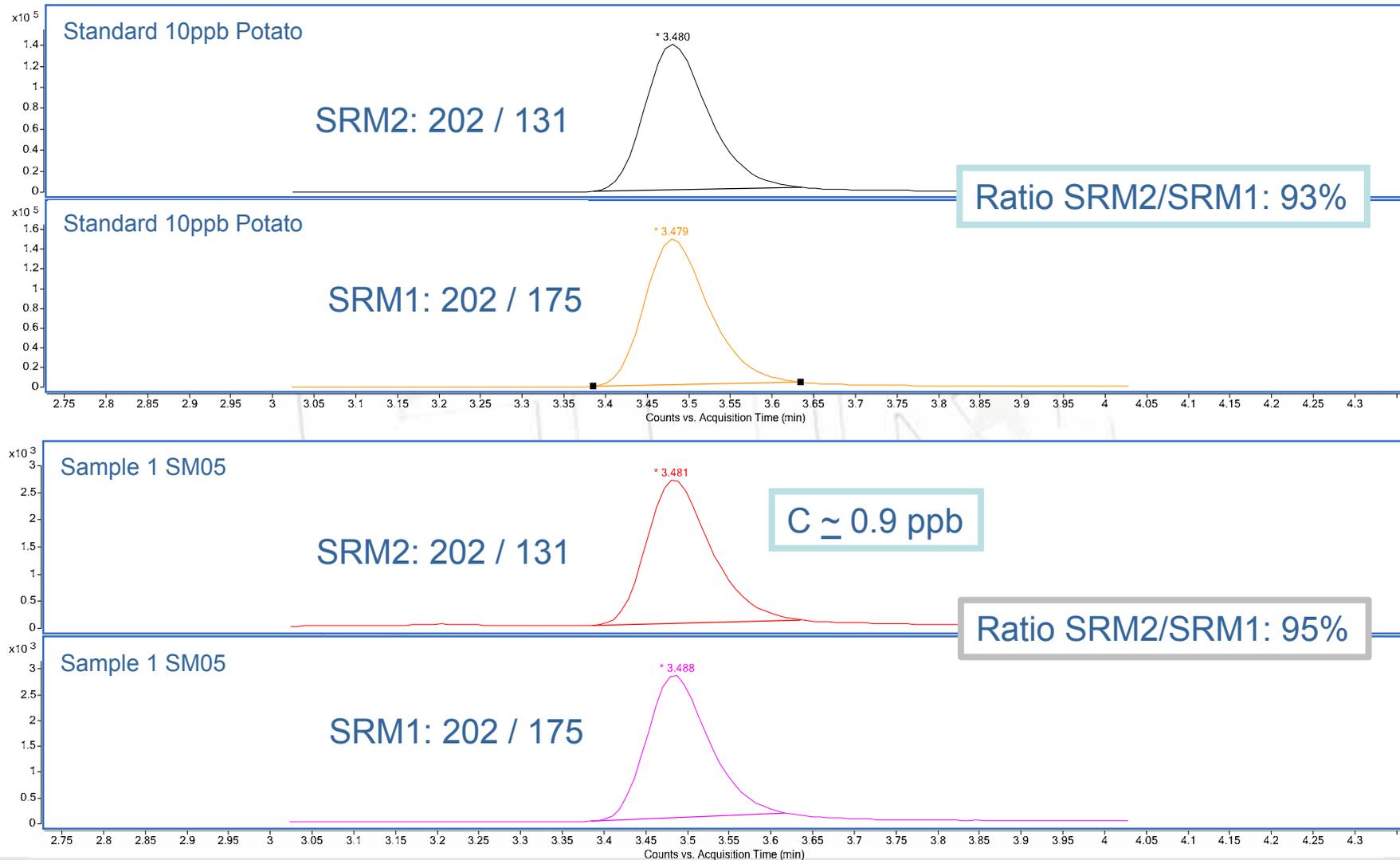
# Simazine (LC-QQQ)



# Thiabendazole (Q-TOF)



# Thiabendazole (LC-QQQ)



22 Evaluated Pesticides (21 added )

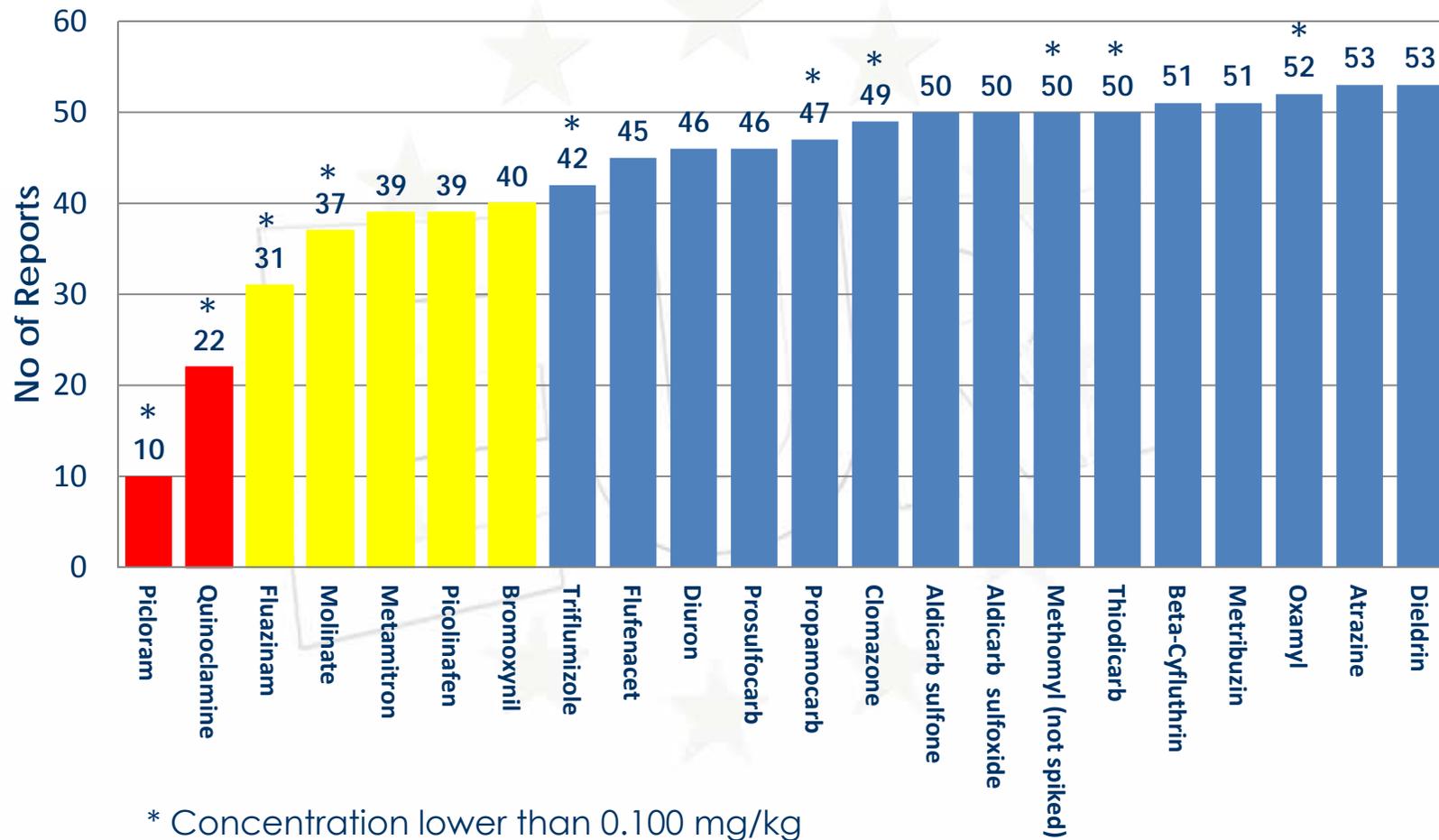
1 Degradation product #

(in green pesticides with concentration <100 ppb)

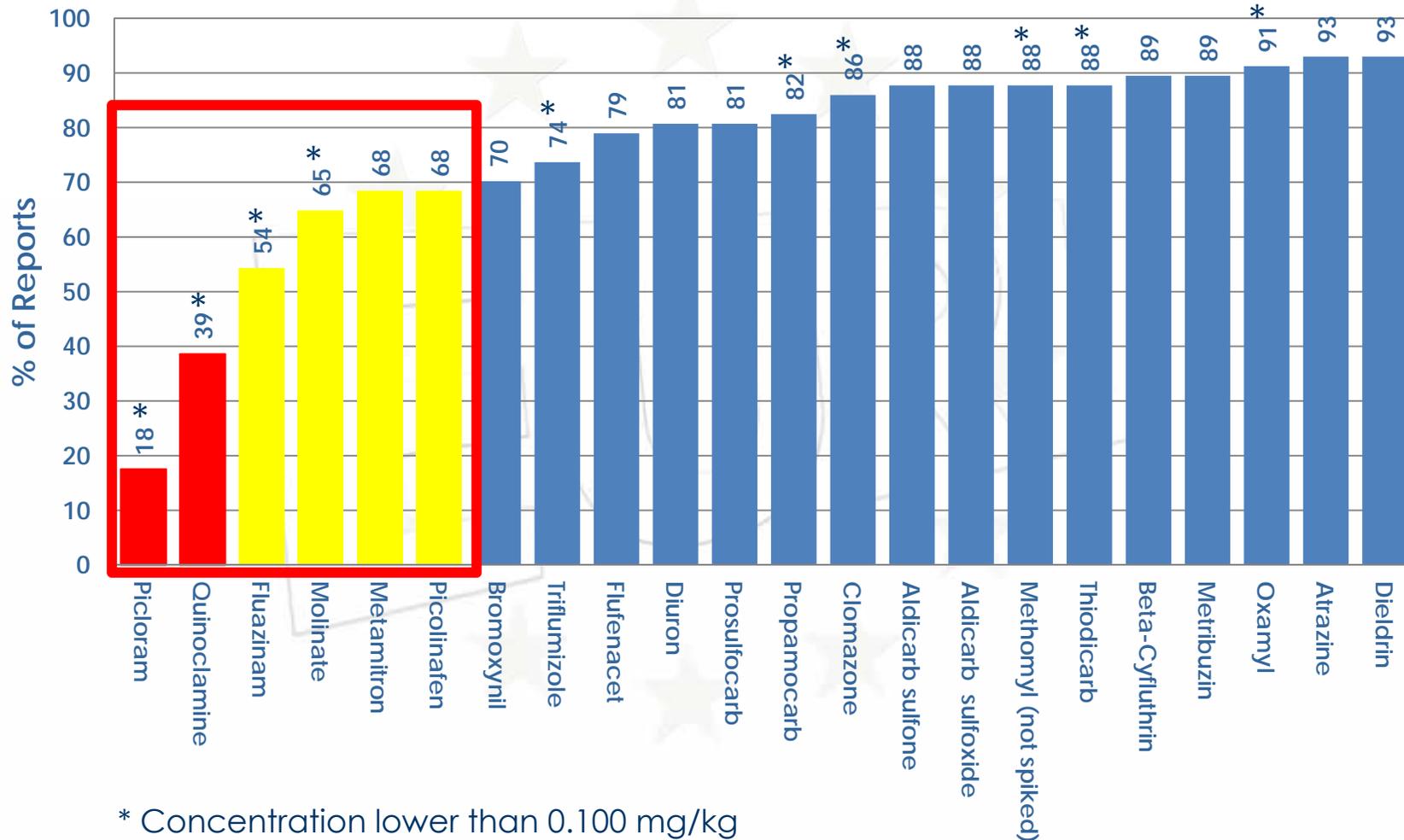
22 Pesticides = 2+5+15

	Aldicarb sulfone	Aldicarb sulfoxide	Atrazine	Beta-Cyfluthrin	Bromoxynil	Clomazone	Dieldrin	Diuron	Fluazinam	Flufenacet	Metamitron	Methomyl#	Metribuzin	Molinate	Oxamyl	Picloram	Picolinafen	Propamocarb	Prosulfocarb	Quinoclamine	Thiodicarb#	Triflumizole
Total Number of Reported Pesticides	50	50	53	51	40	49	53	46	31	45	39	50	51	37	52	10	39	47	46	22	50	42
% of Reported Pesticides	85	85	90	86	68	83	90	78	53	76	66	85	86	63	88	17	66	80	78	37	85	71

## SM05 No of Reported Pesticides (57 Laboratories Reported Data)

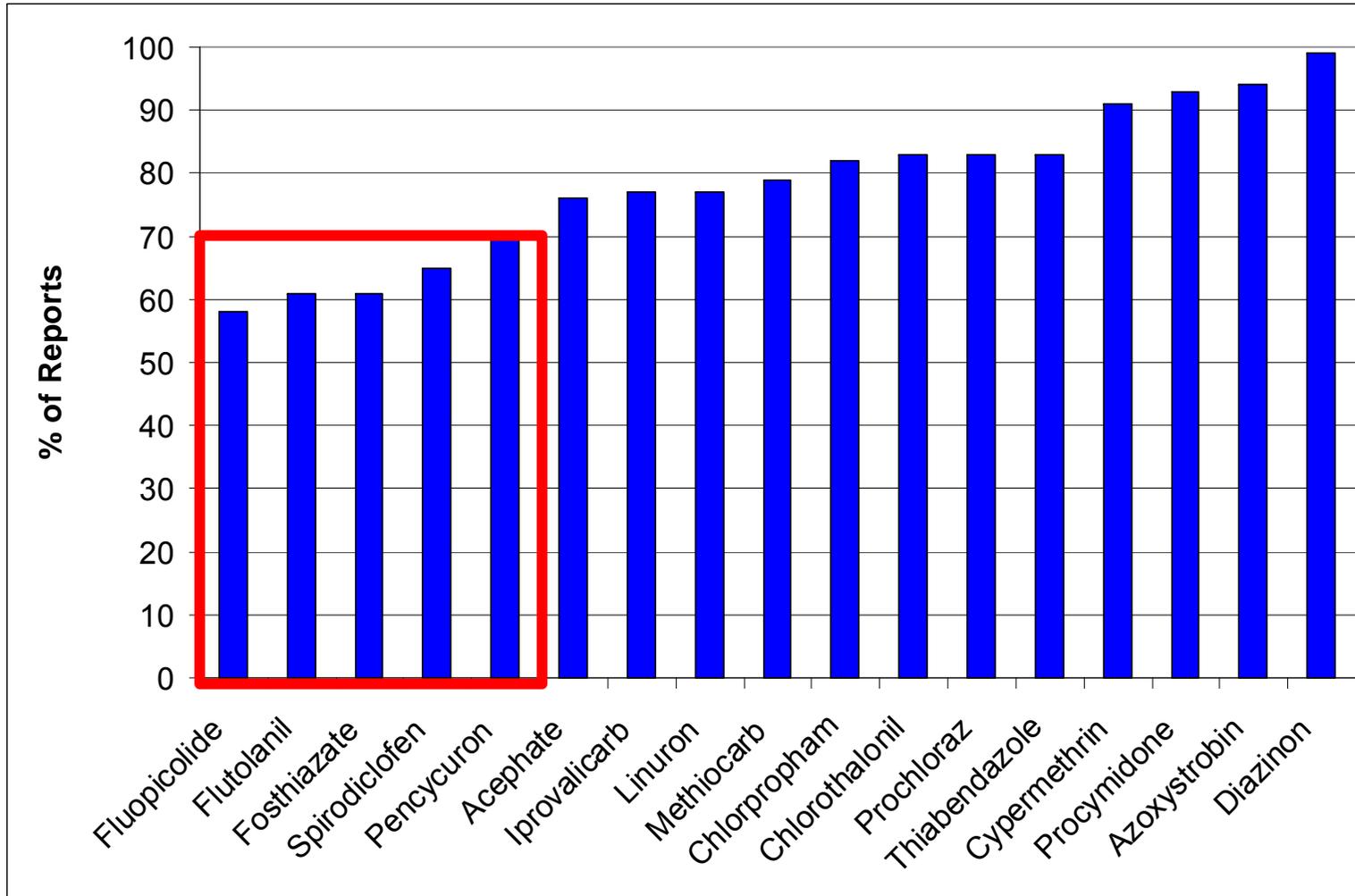


### SM05 % of Reported Data by Pesticide (57 Laboratories Reported Data)

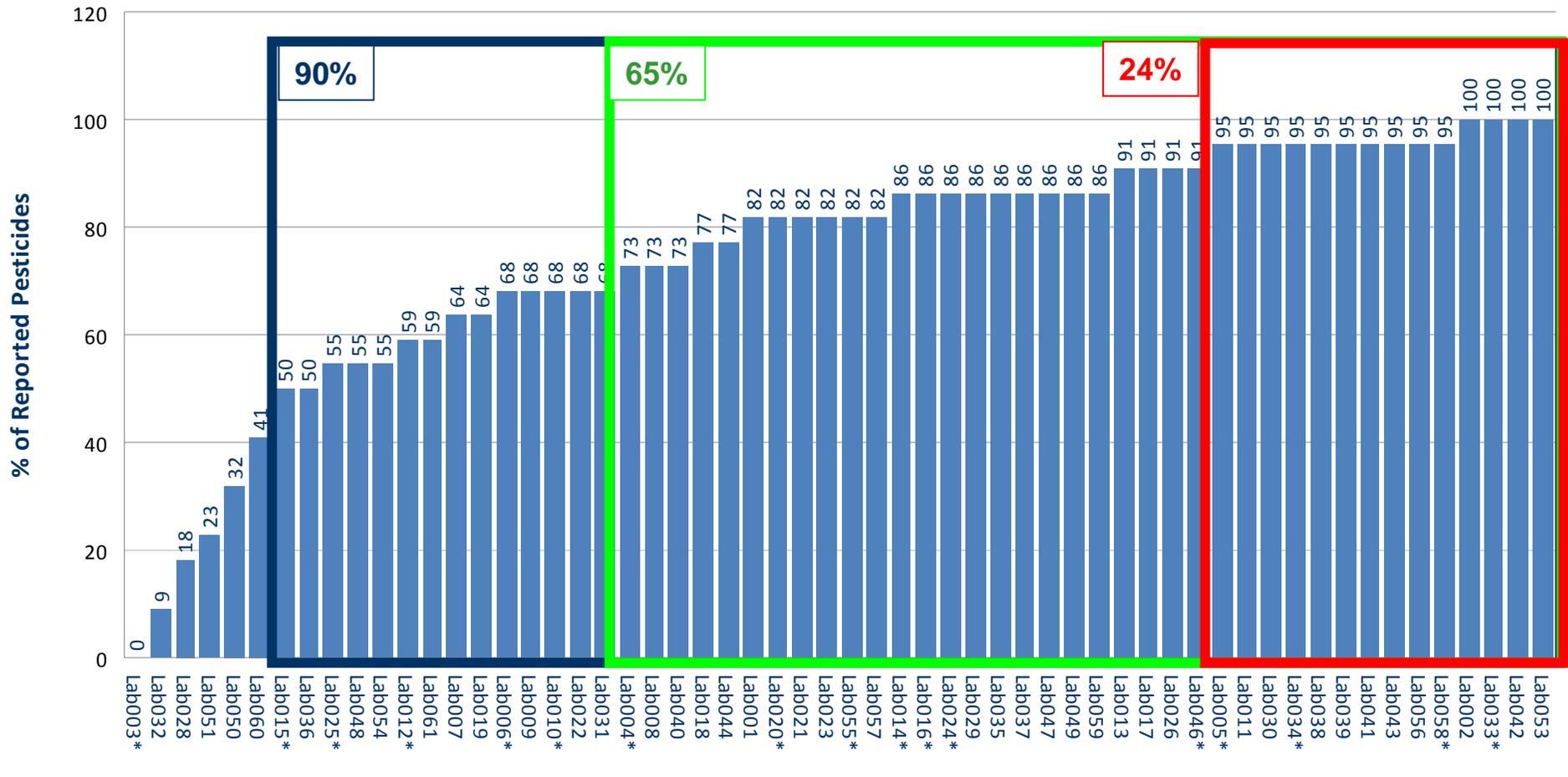


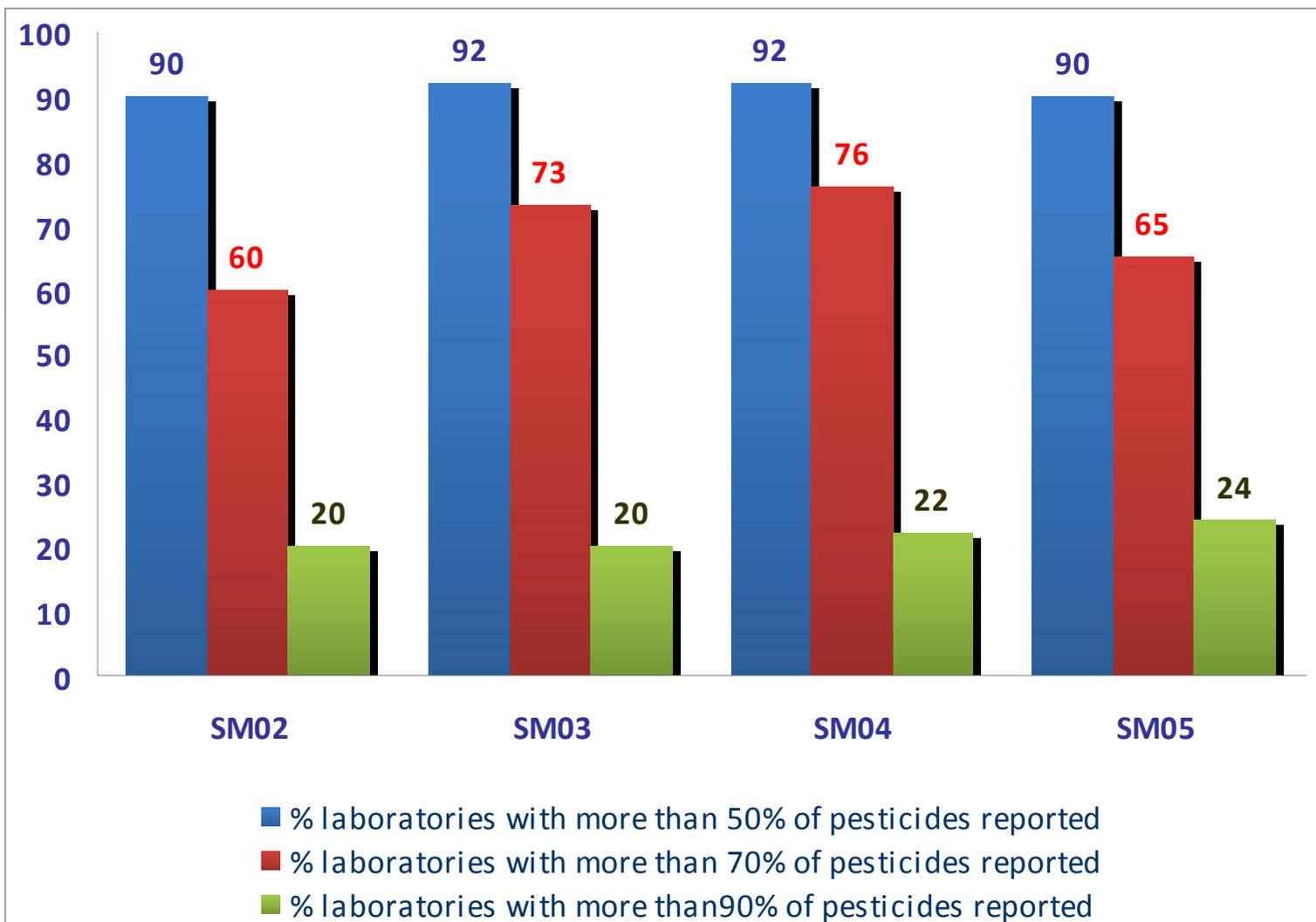
\* Concentration lower than 0.100 mg/kg

## Fv15 % of Reported Data by Pesticide



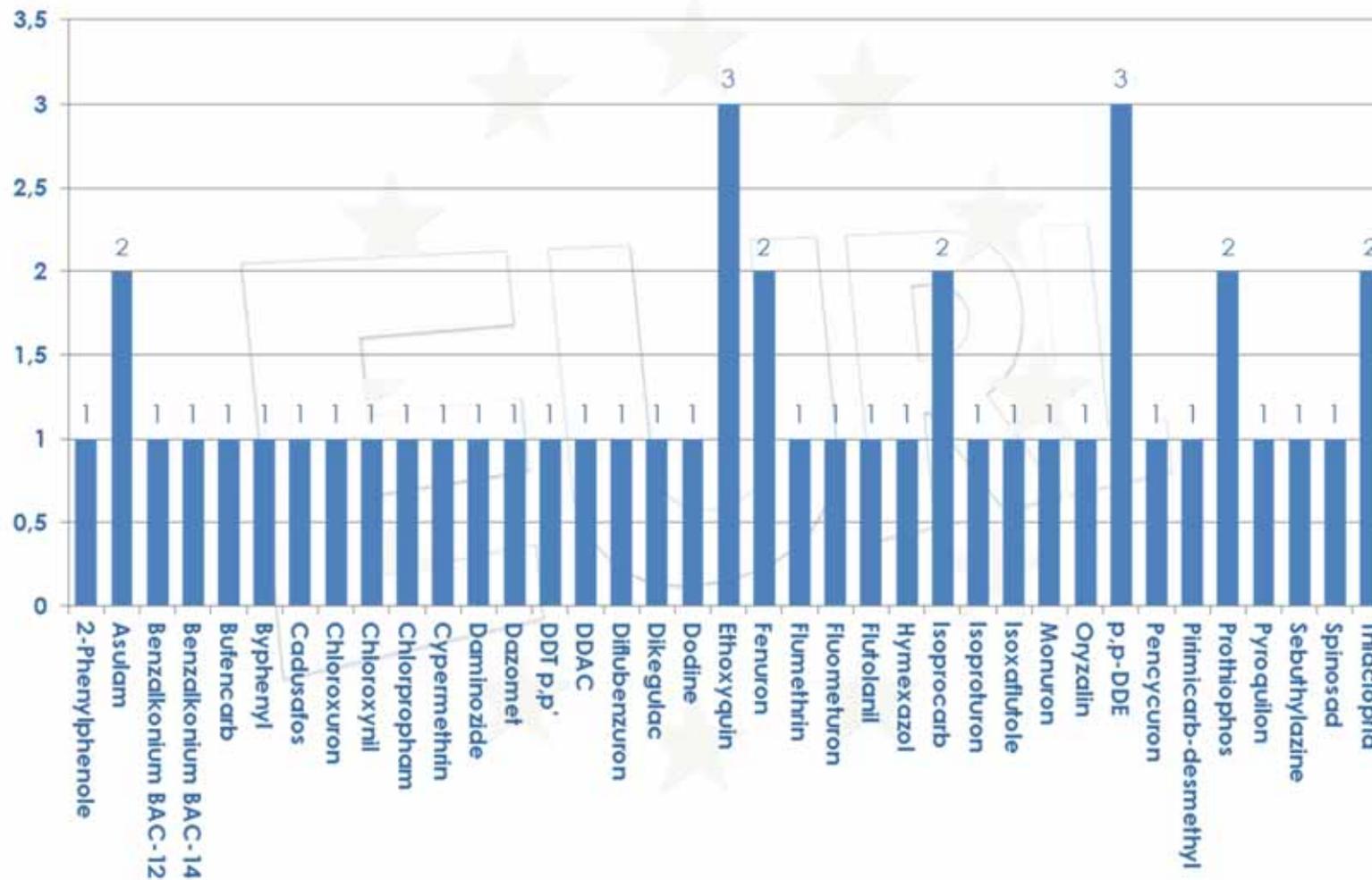
### % of Reported Data by Laboratory (\*NRL) (21+1 Pesticides Included)







### Other Reported Pesticides

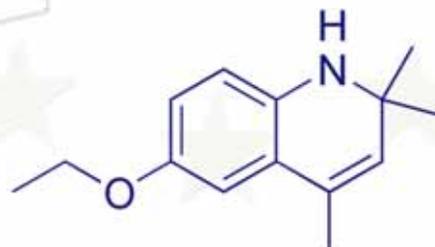


## Other reported pesticides

The test material was spiked/treated with **thiodicarb**, which degrades to **methomyl**. The test material **was not** spiked/treated with methomyl.

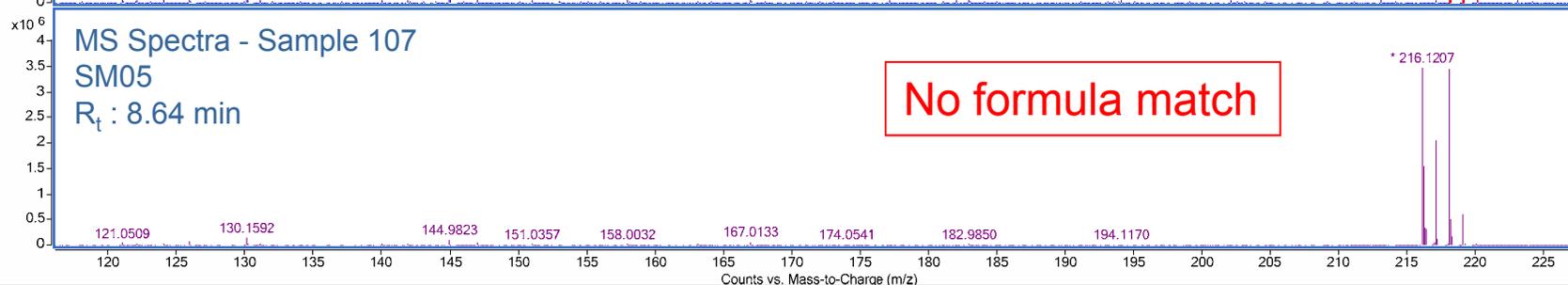
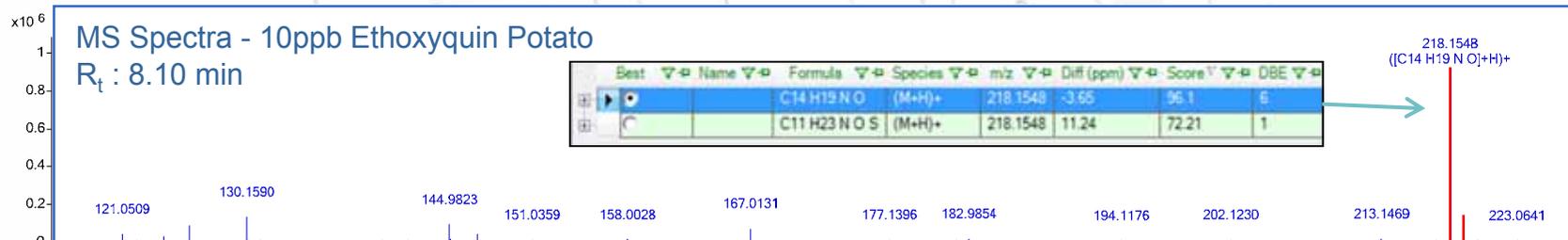
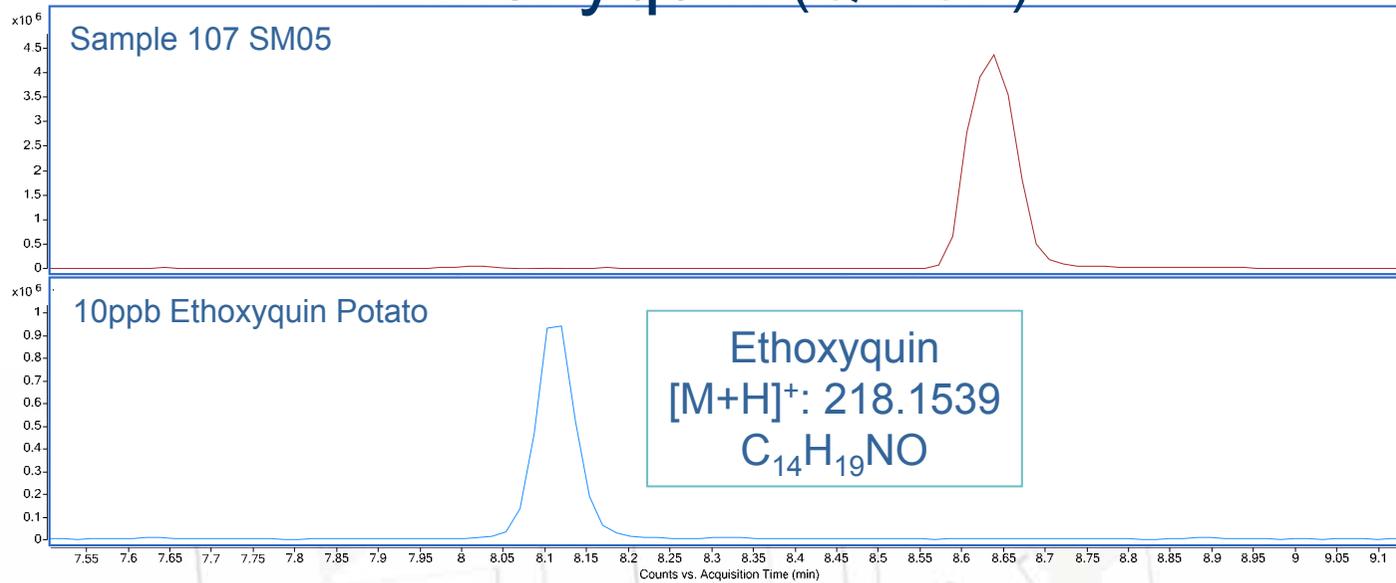
**Propazine, simazine and thiabendazole** were not intentionally used to spike/treat the test material, but they were detected by the organisers at concentrations below 10 ppb due to impurities of the commercial formulations.

**Ethoxyquin** has been reported by 3 laboratories but after the analysis by the organiser, it was not detected in the sample, all the other reported pesticides were detected only by one or two laboratories only.

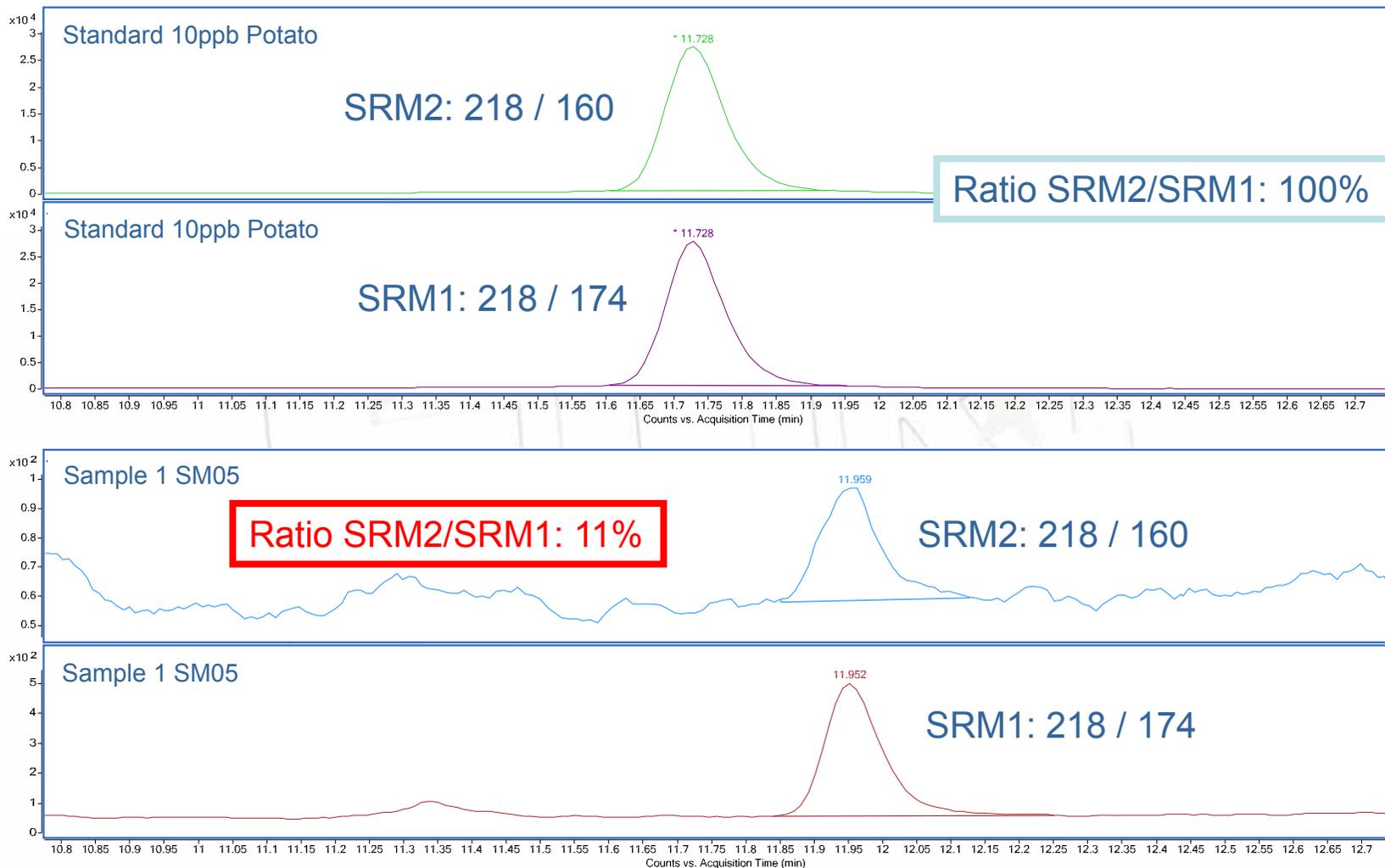


**Ethoxyquin**

# Ethoxyquin (Q-TOF)

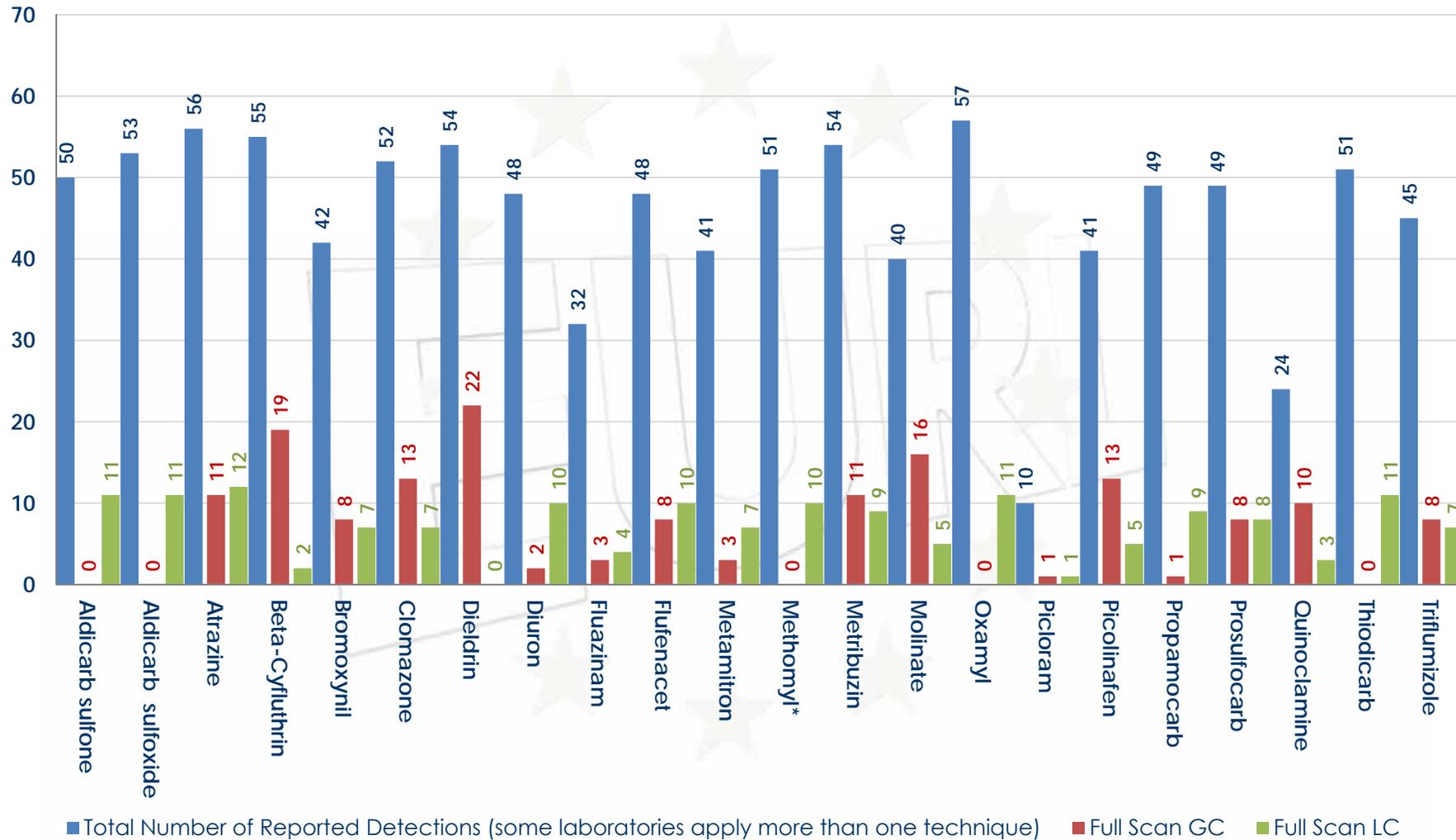


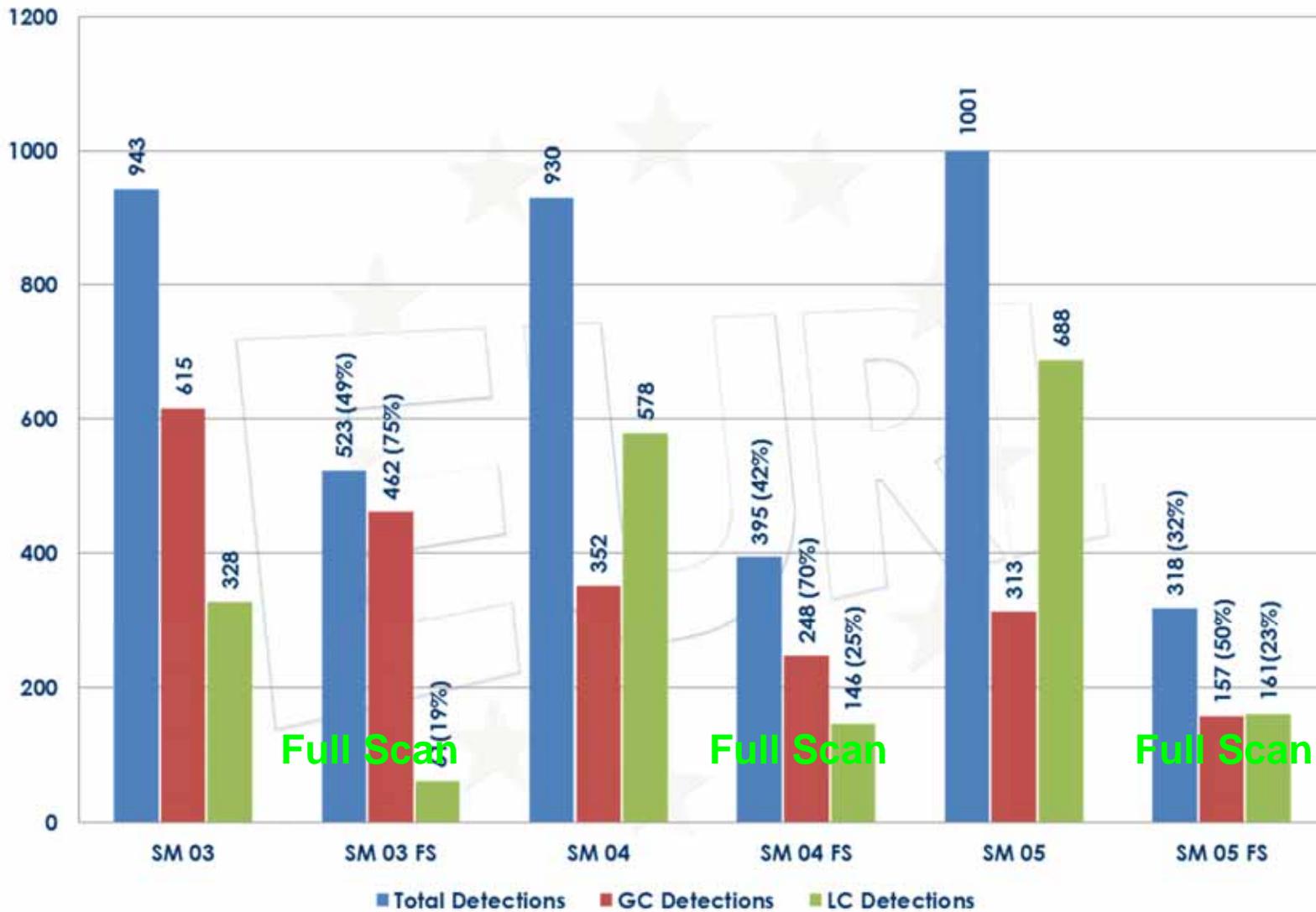
# Ethoxyquin (LC-QQQ)



## SM-05 Techniques (Full Scan)

\*not spiked







**EURL-FV**



Almería 23<sup>rd</sup>-25<sup>th</sup> October 2013

**EUPT-FV-SM05**  
European Proficiency Test FV-SM05

**Thanks for  
your Attention**



**EURL-FV**



Almería 23<sup>rd</sup>-25<sup>th</sup> October 2013

**Thanks for  
your Attention**



**EURL-FV**

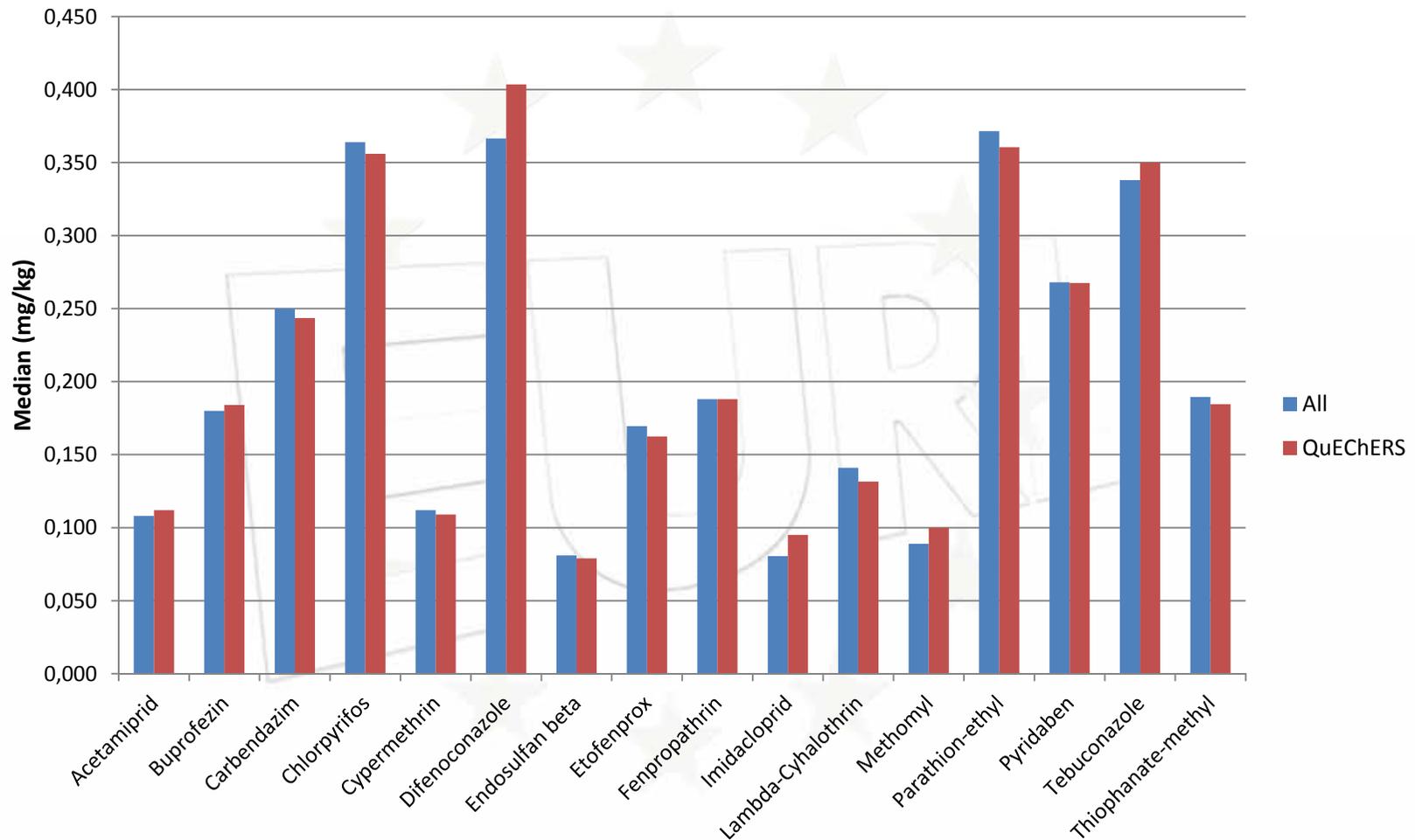


Almería 23<sup>rd</sup>-25<sup>th</sup> October 2013

# Method comparison



# Median comparison



# Qn comparison

