

# 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  
FOR PESTICIDE RESIDUES IN FRUITS  
AND VEGETABLES  
SCREENING METHODS 05**



European  
Commission

# EURL

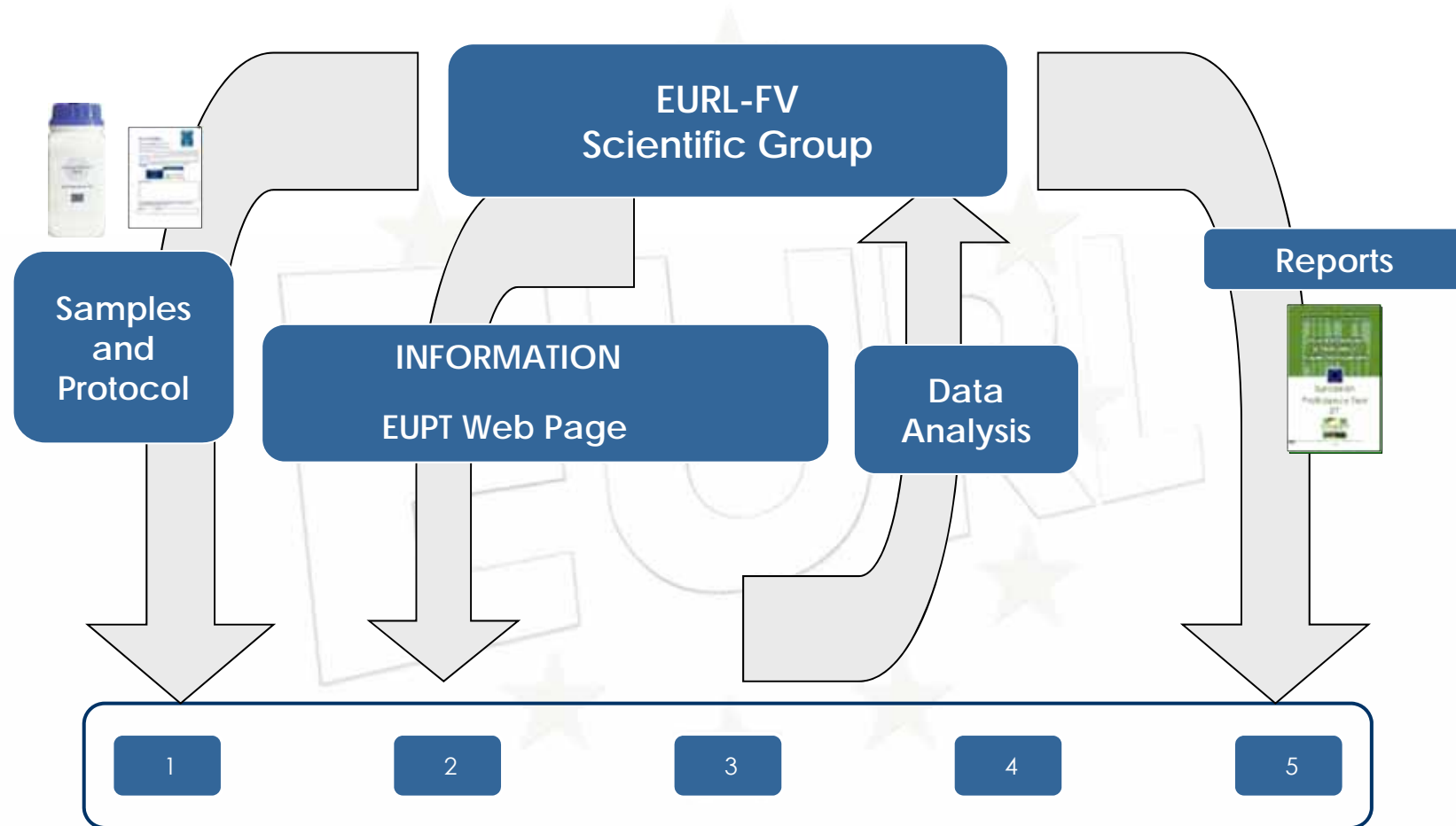


## AMADEO R. FERNÁNDEZ-ALBA

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*	Spain	6
Denmark	1*	Sweden	1*+1
Estonia	1*	Switzerland	1*+1
Finland	1*	The Netherlands	1*+1
France	1*+7	Turkey	1
Germany	7	UK	1*+2
Greece	1		
Hungary	1*+2		

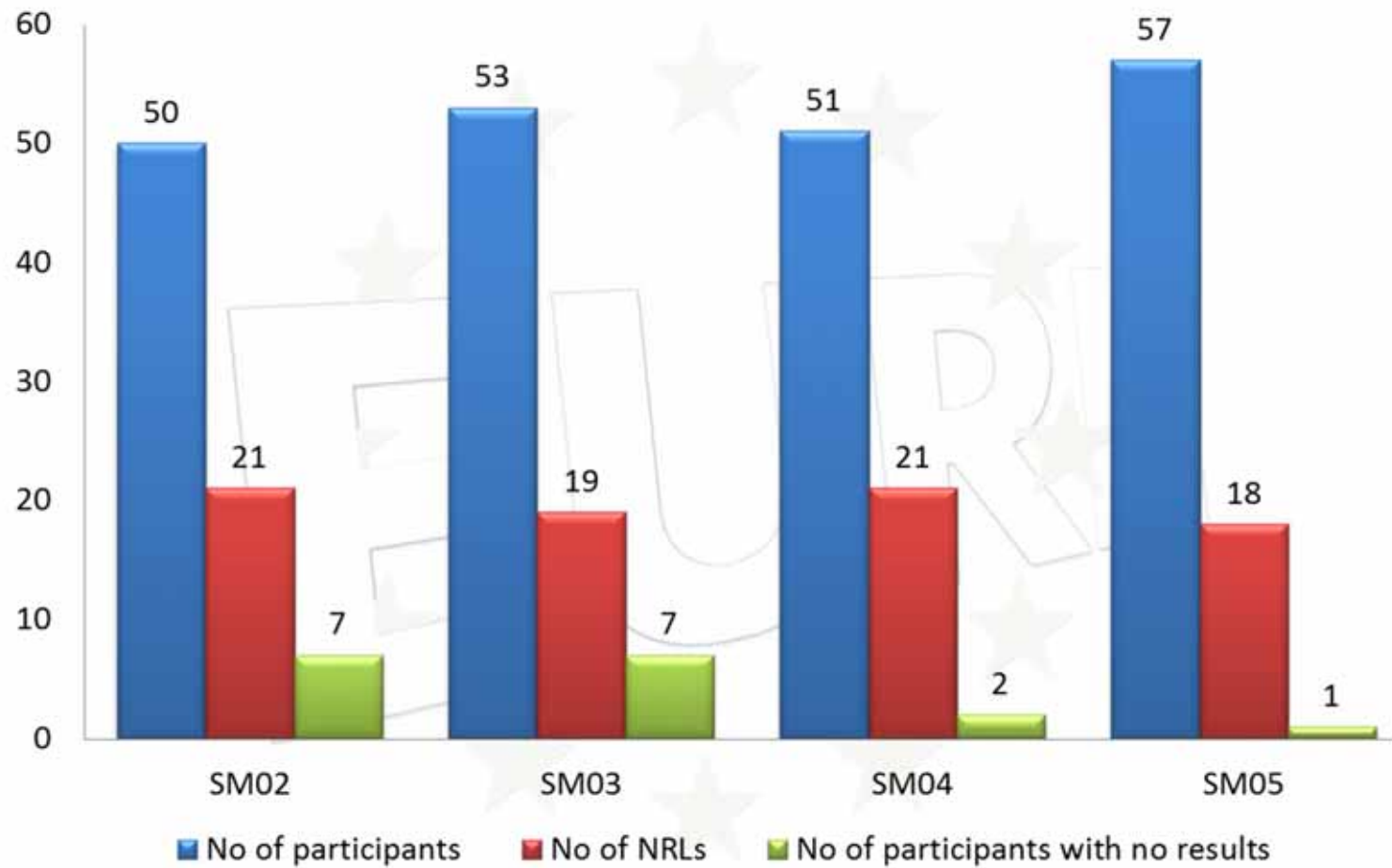
58

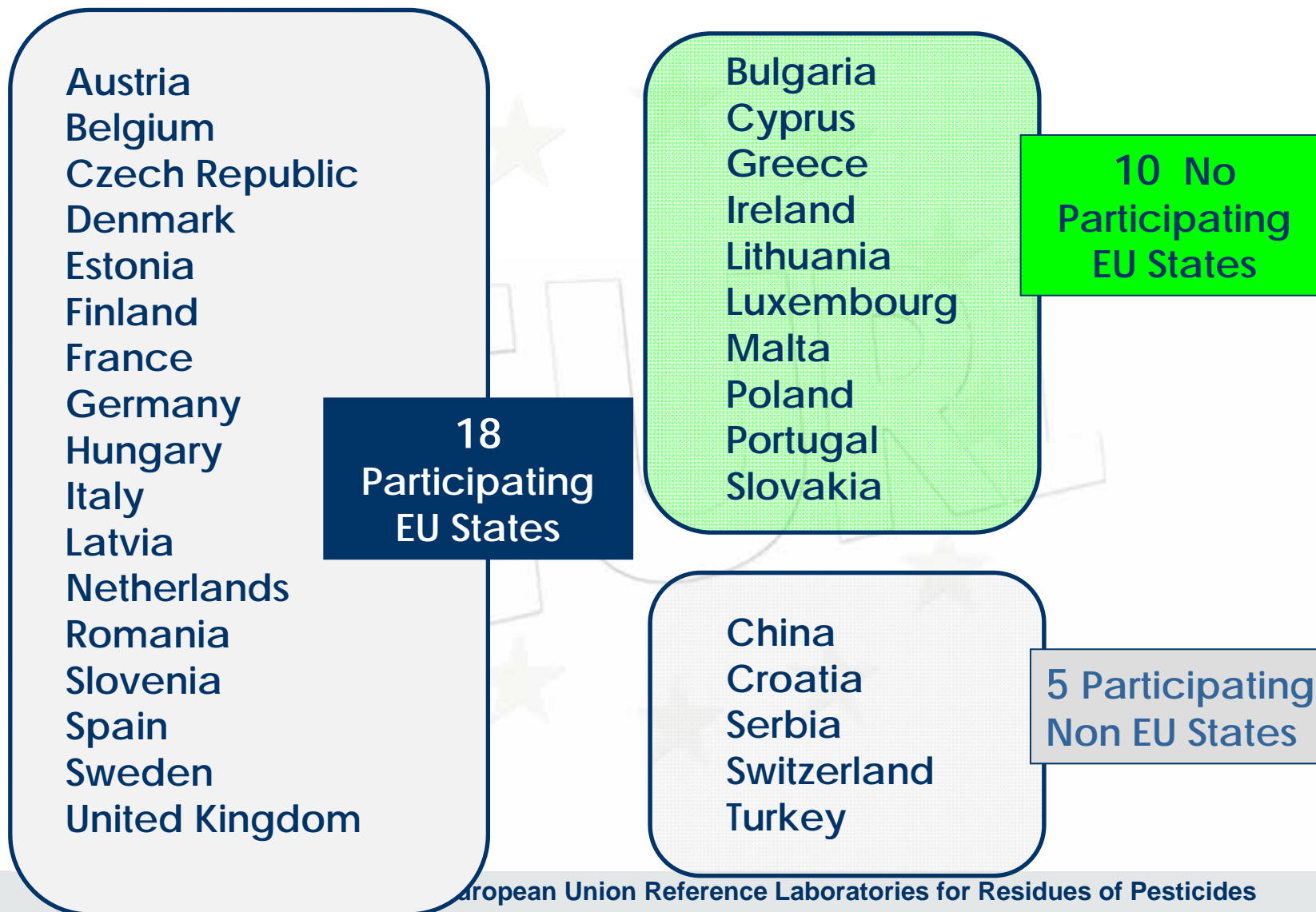
58

## 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	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	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	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	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	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	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	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	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	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	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	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	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	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	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	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	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	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	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	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	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	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	Melicafe	Oxamyl	Picloram	Piccolinifen	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		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	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	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		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	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



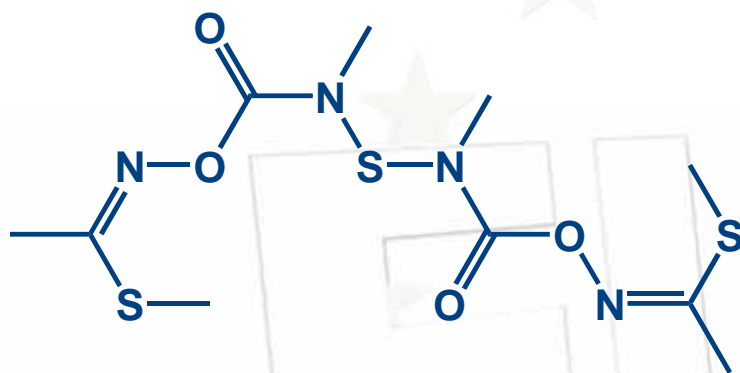
Laboratory Code (Total No of Reporting Laboratories = 58 [1=EU-FV])	Evaluated Pesticides (22) (in green pesticides with concentration <100 ppb)																					Reported Pesticides by Laboratory	% of Reported Pesticides by Laboratory	
	Aldicarb sulfone	Aldicarb sulfoxide	Alazine	Beta-Cyfluthrin	Bromoxynil	Clomazone	Diethrin	Duron	Fuaznham	Flufenacet	Metamifron	Methomyl#	Metribuzin	Molindate	Oxamyl	Picbryam	Picolinifen	Propamocarb	Prothiofocarb	Quinoclamine	Thiodicarb#			Triflumizole
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	R	R				11	50
Lab037	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		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.

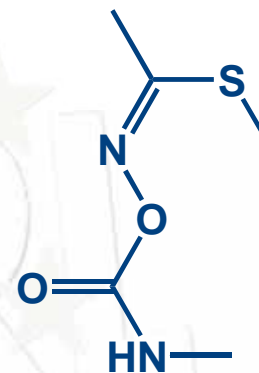
# The test material was spiked/treated with thiodicarb, which degrades to methomyl. The test material was not spiked/treated with methomyl.

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.



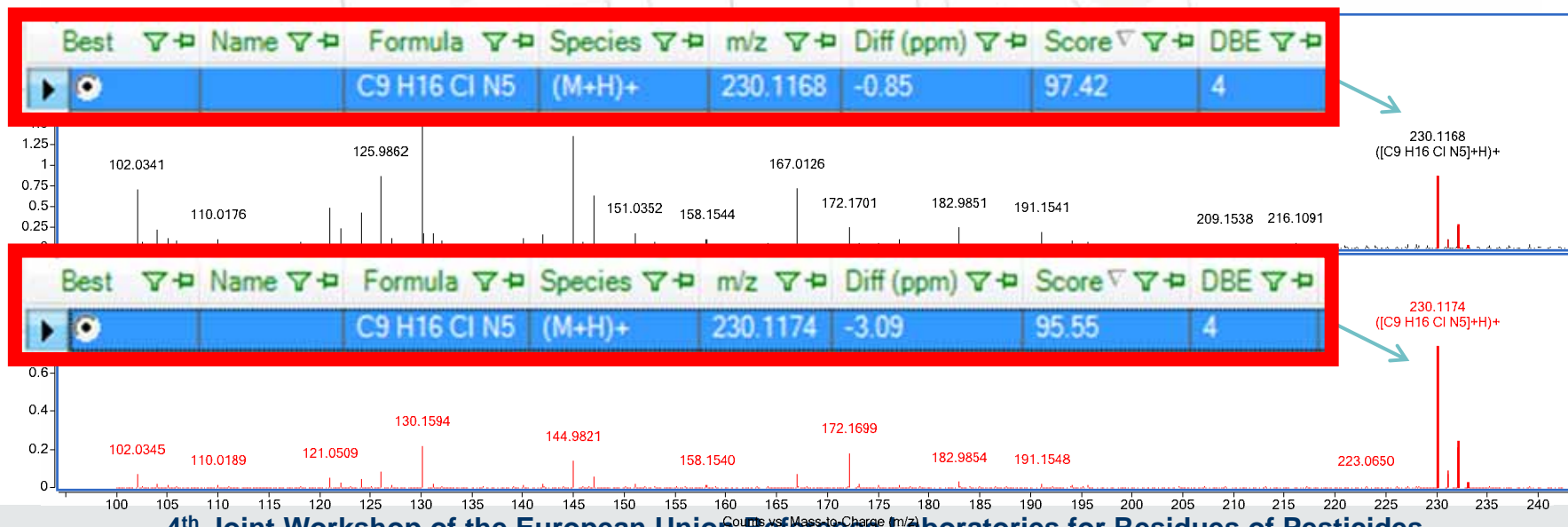
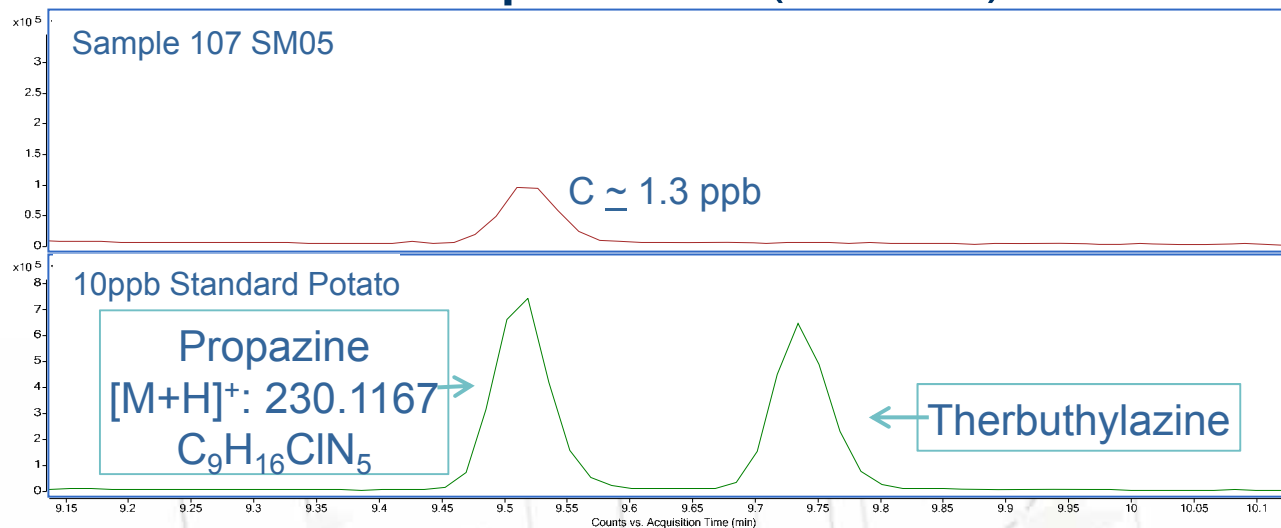
Thiodicarb



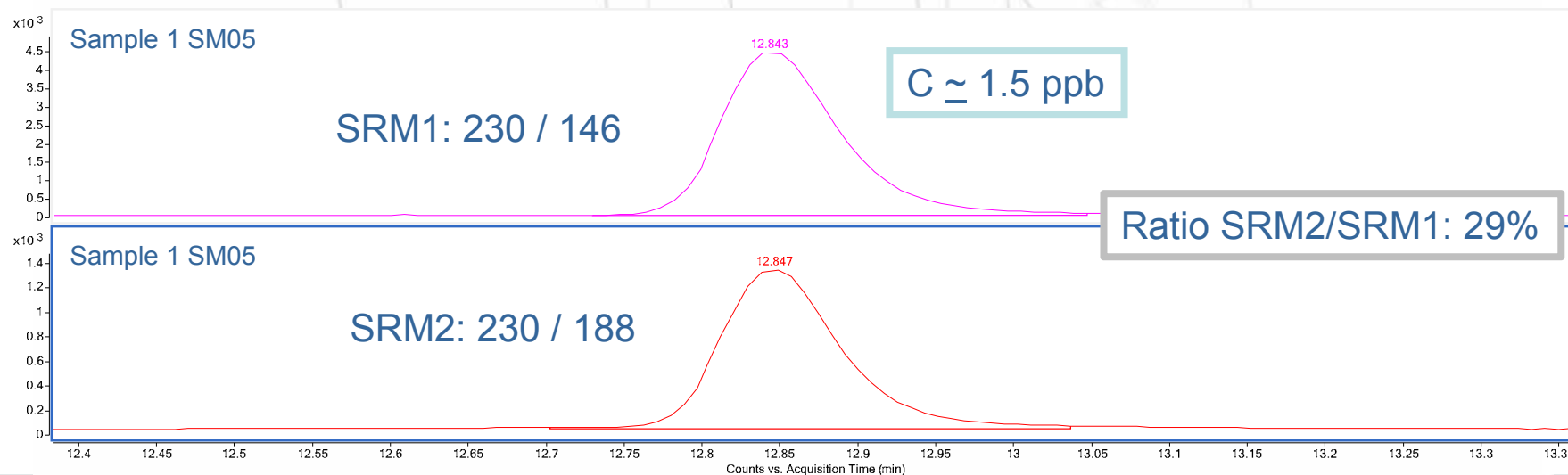
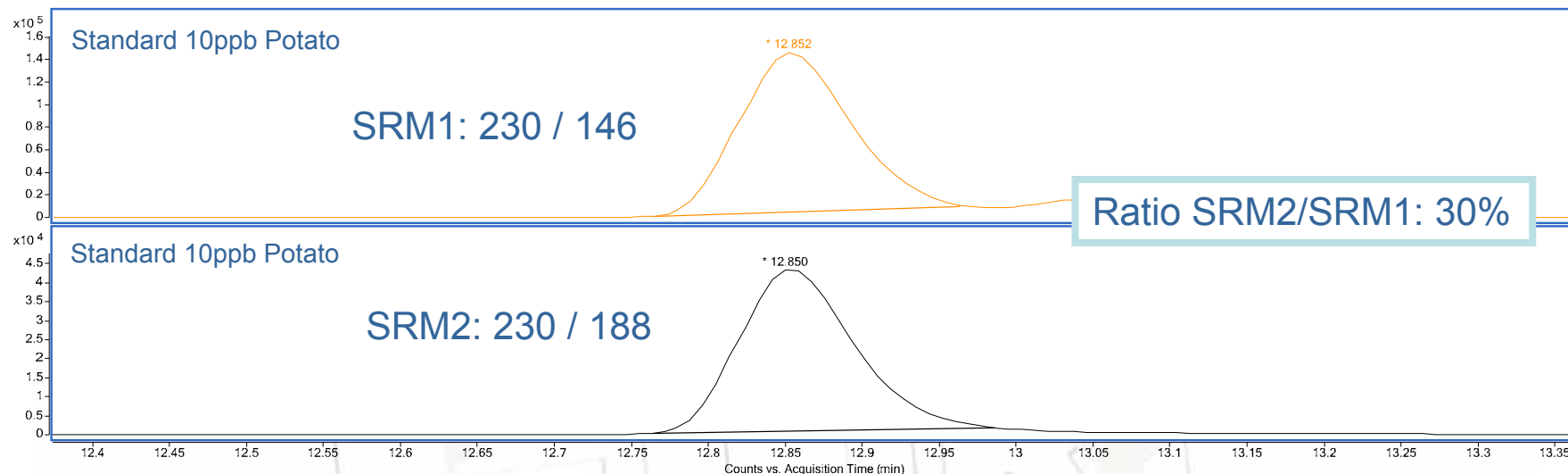
Methomyl

**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.

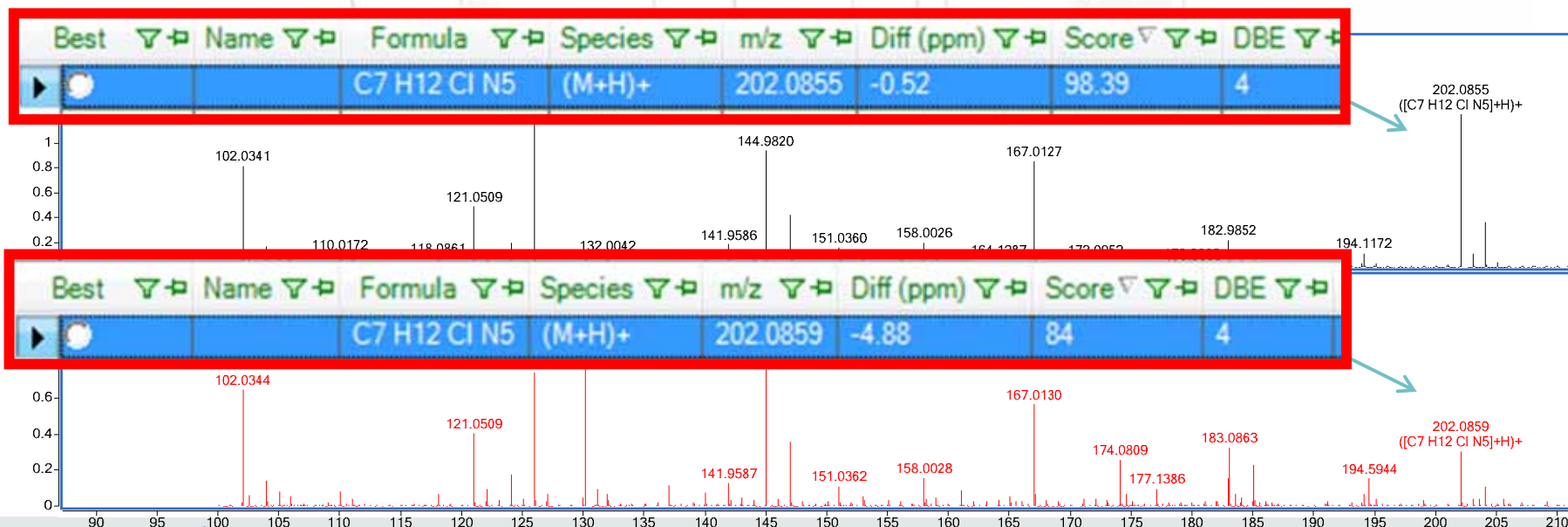
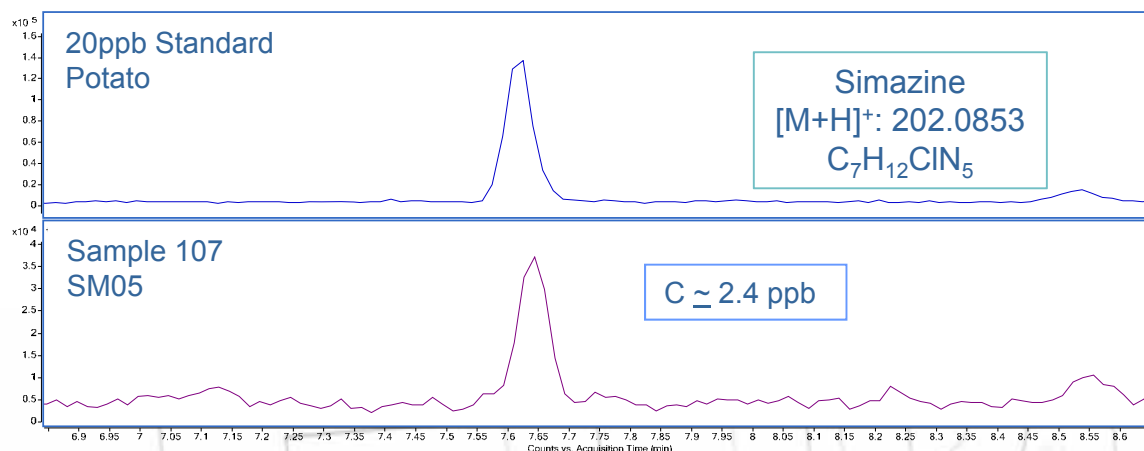
# Propazine (Q-TOF)



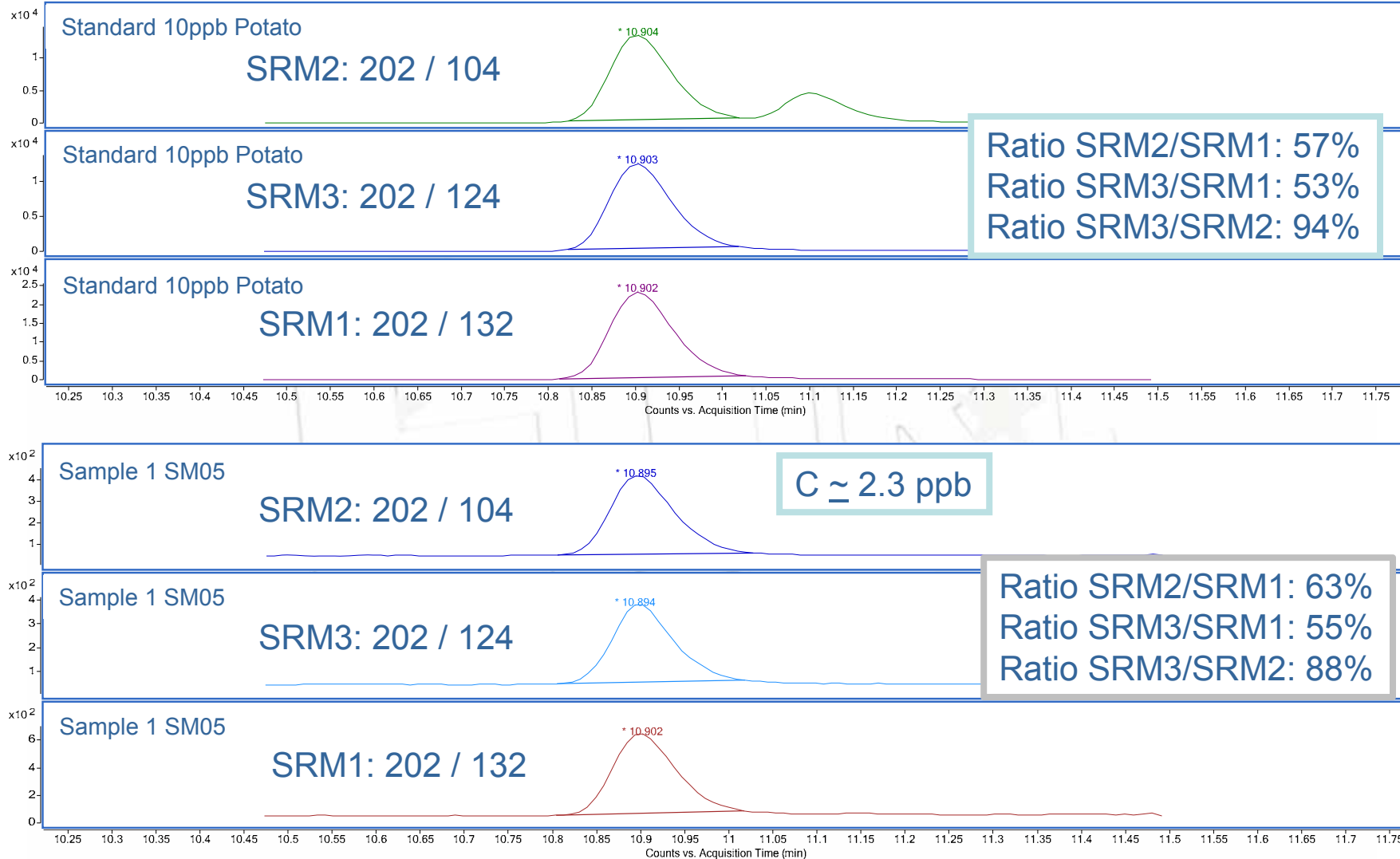
# Propazine (LC-QQQ)



# Simazine (Q-TOF)

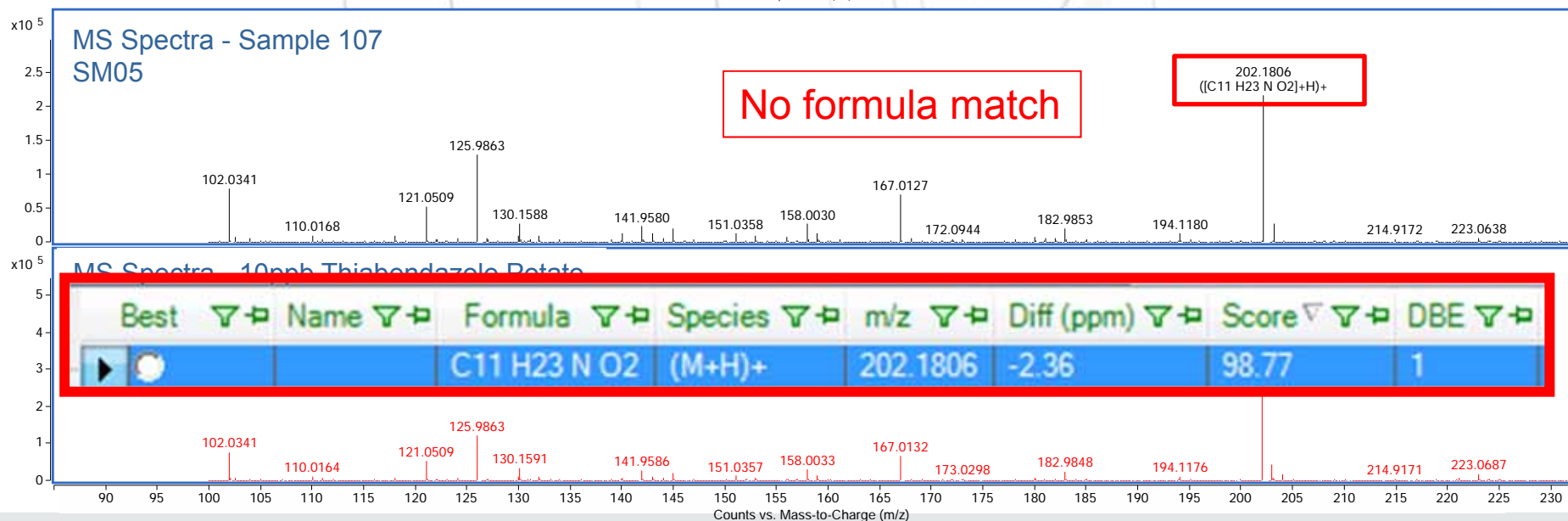
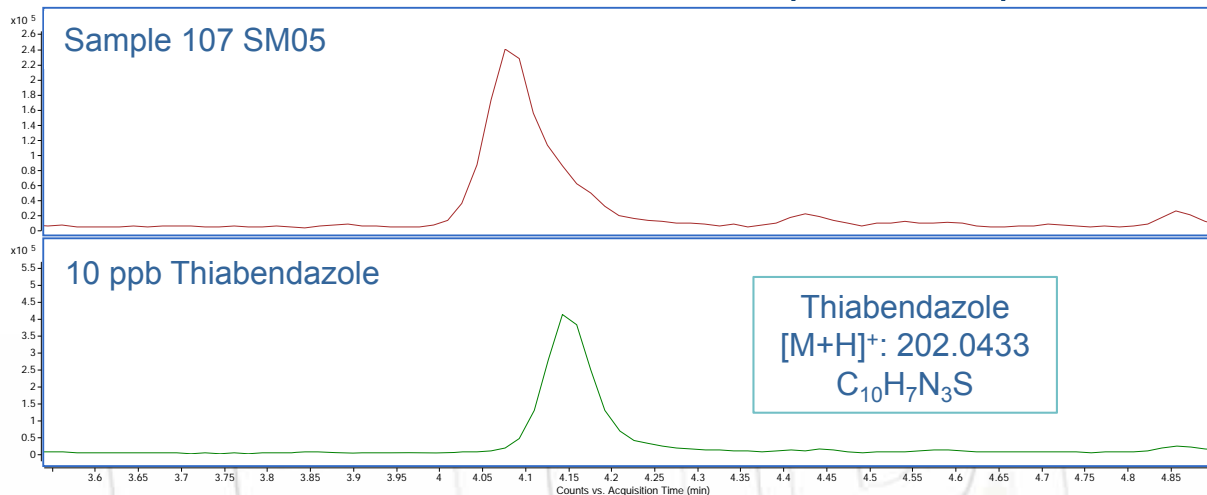


# Simazine (LC-QQQ)

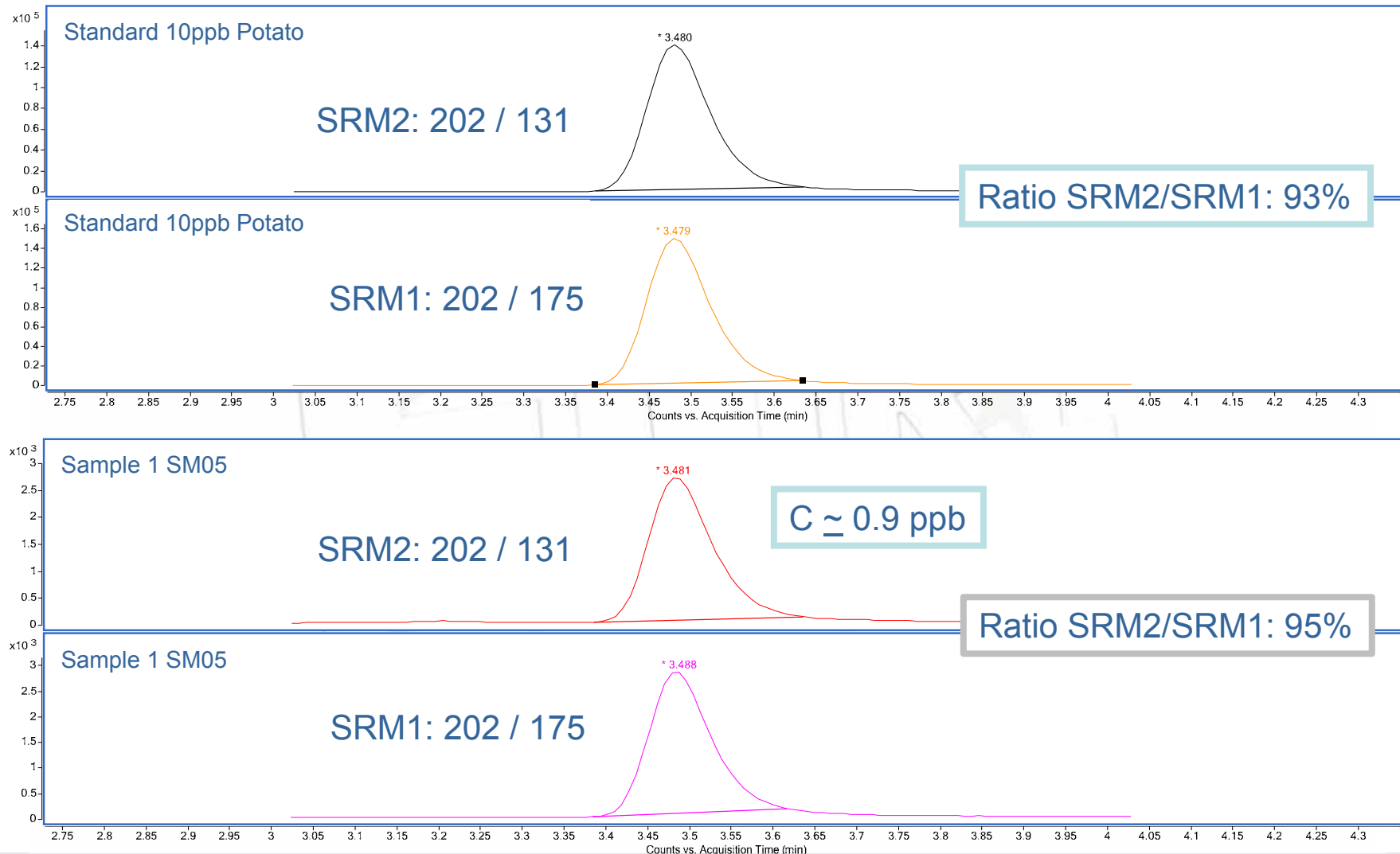




# Thiabendazole (Q-TOF)



# Thiabendazole (LC-QQQ)



22 Evaluated Pesticides (21 added )

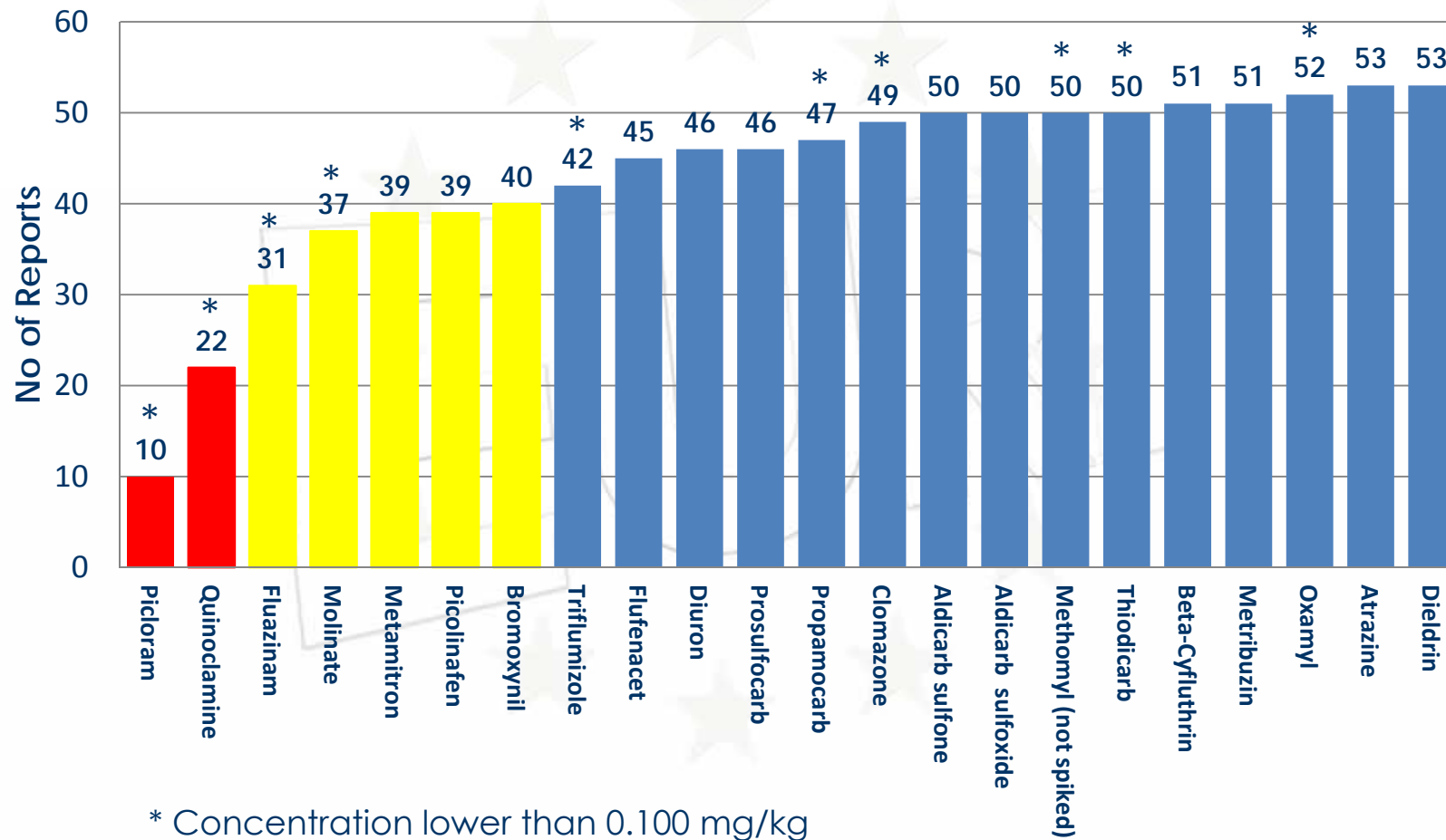
1 Degradation product #

(in green pesticides with concentration <100 ppb)

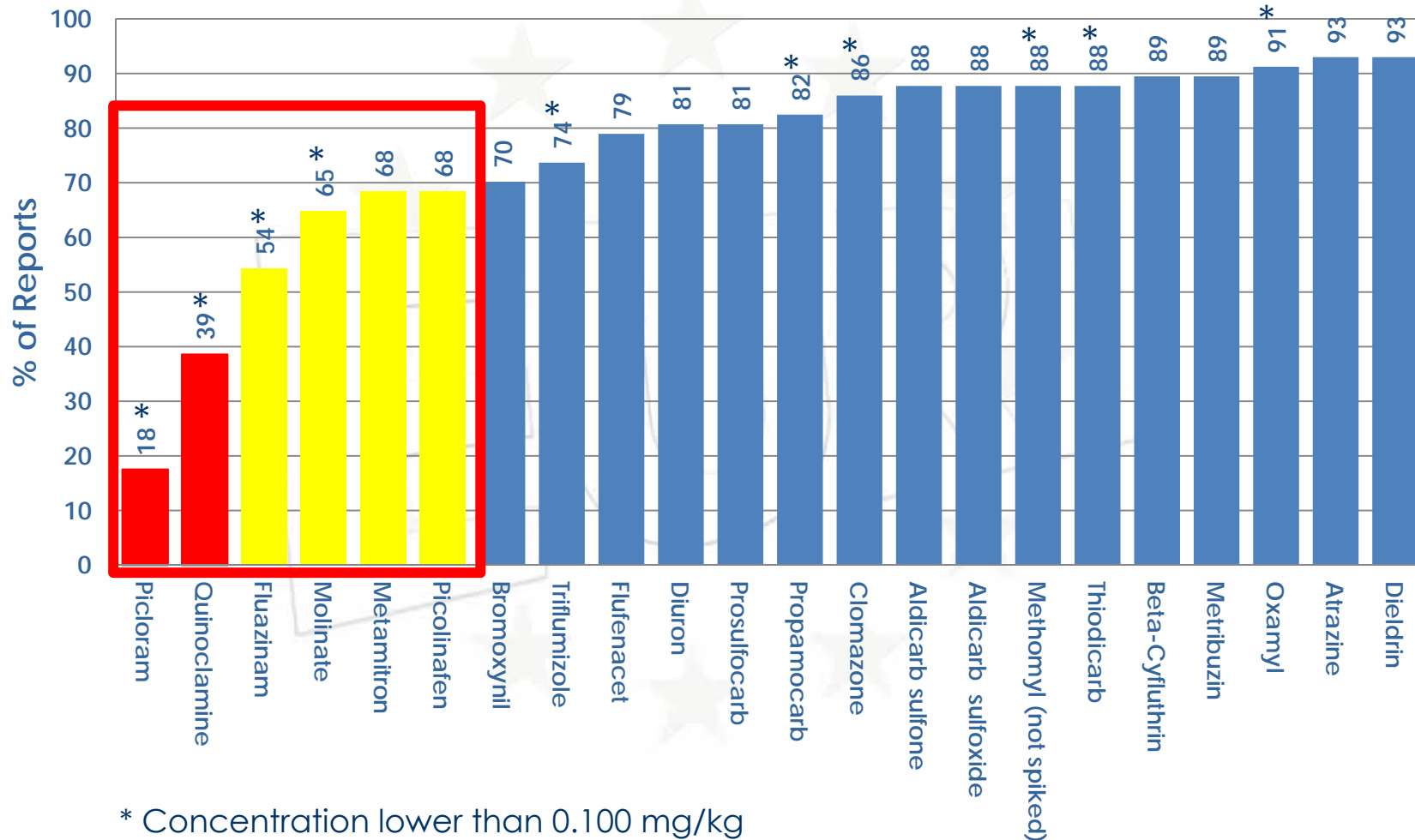
22 Pesticides = 2+5+15

	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)

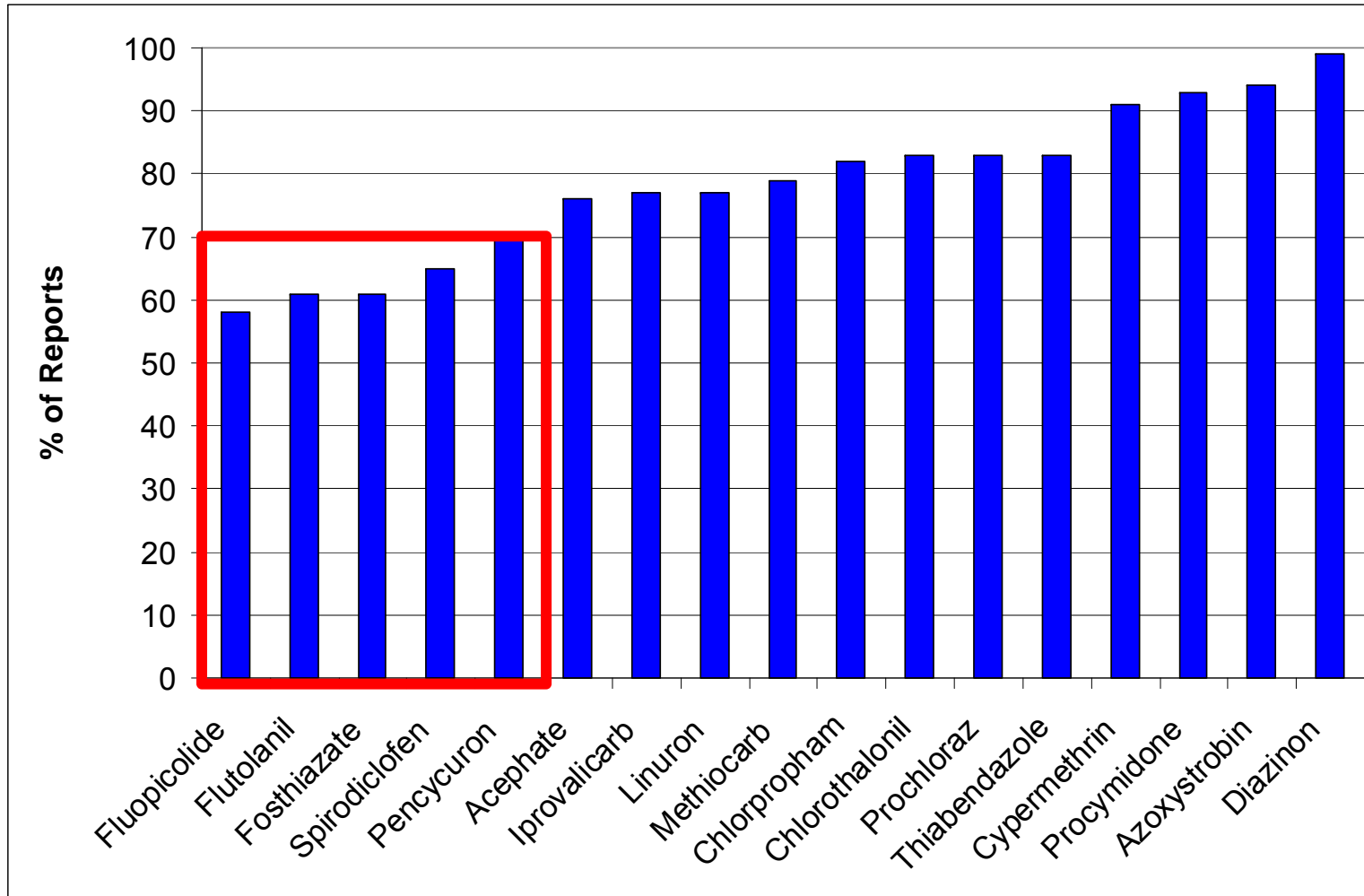


### SM05 % of Reported Data by Pesticide (57 Laboratories Reported Data)

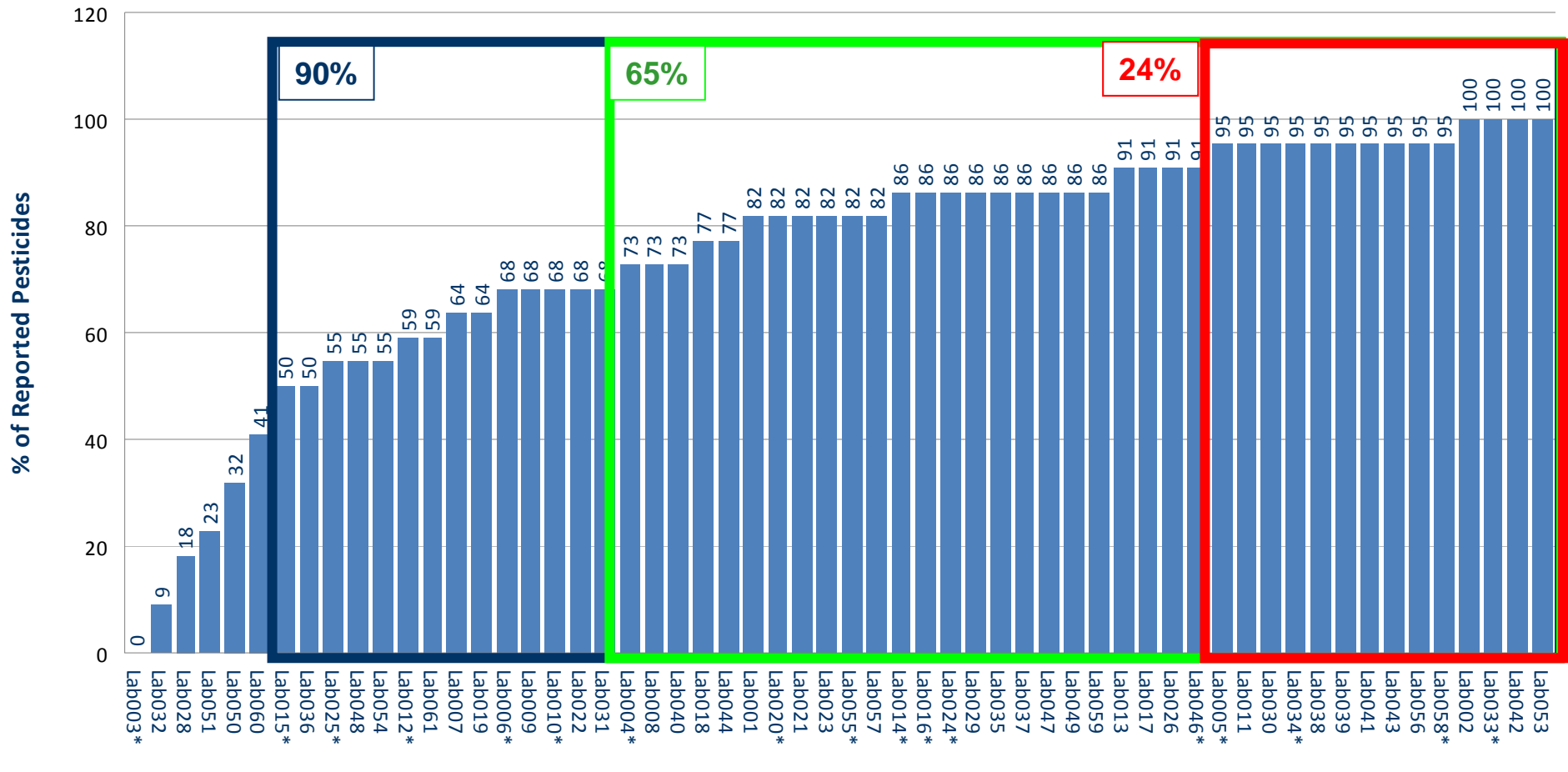


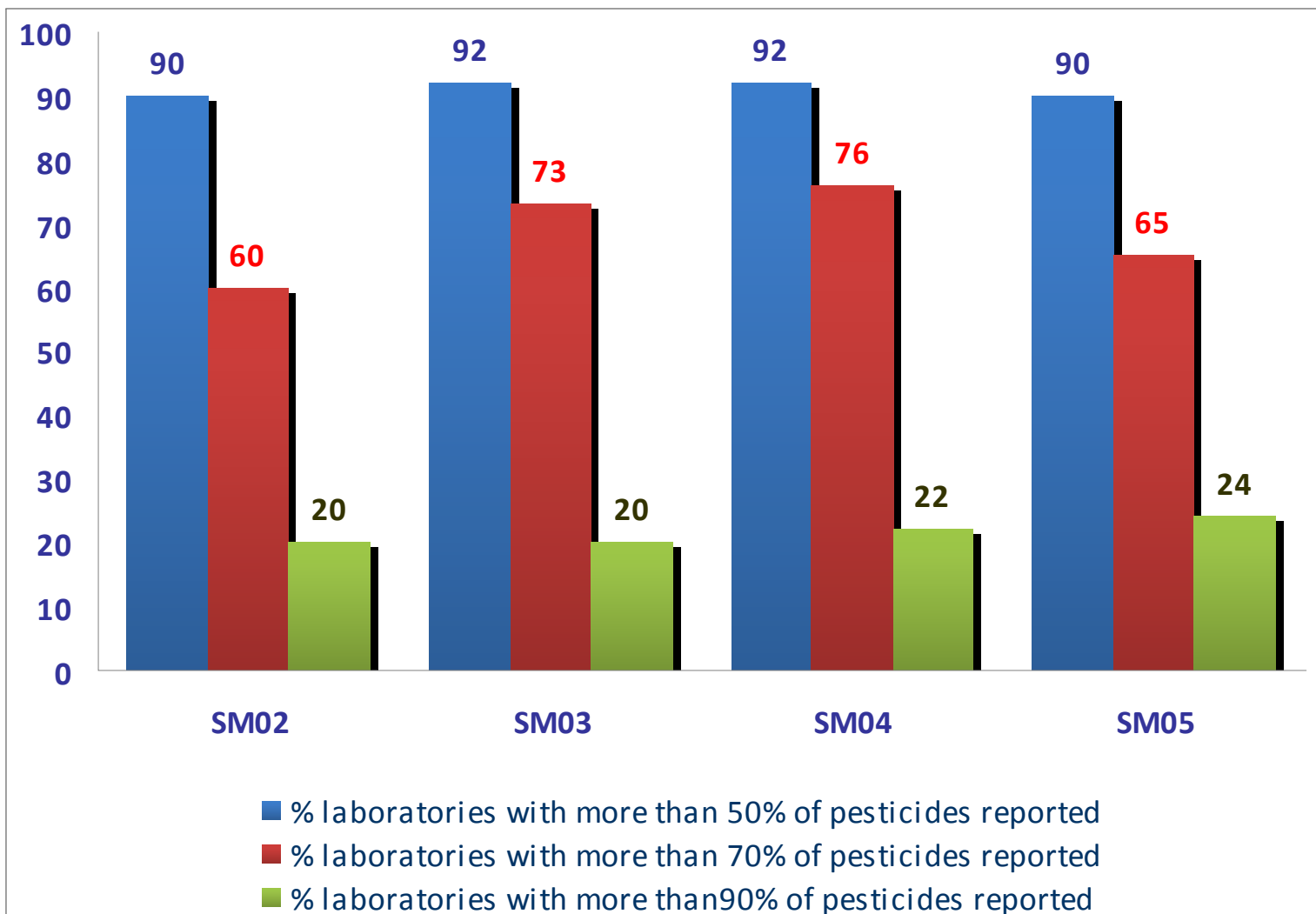
\* Concentration lower than 0.100 mg/kg

## Fv15 % of Reported Data by Pesticide



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

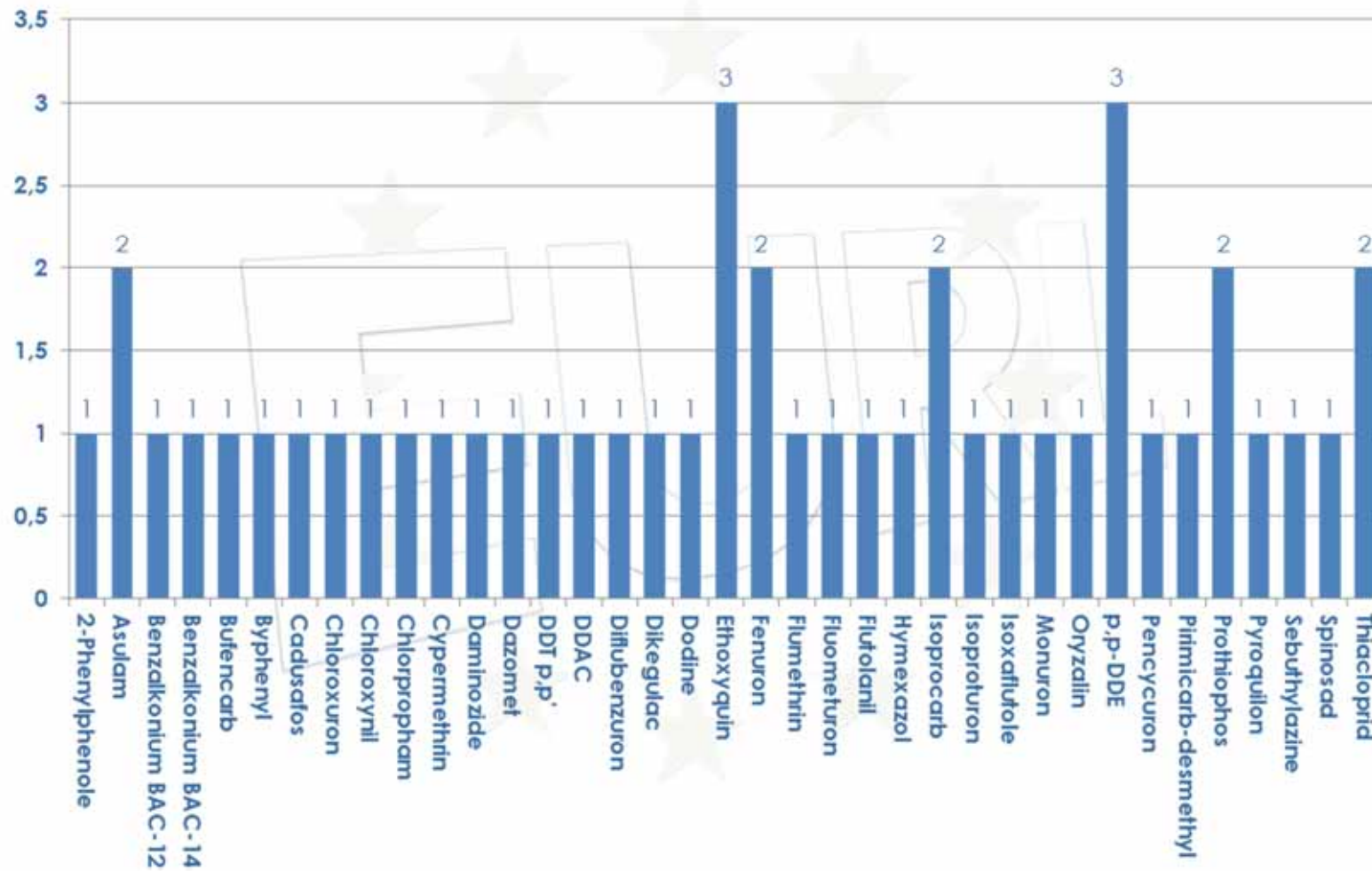








### Other Reported Pesticides

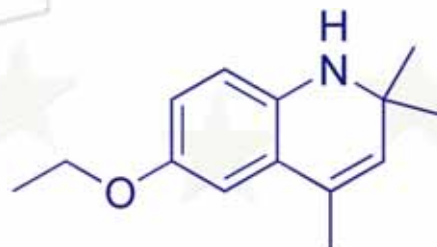


## Other reported pesticides

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

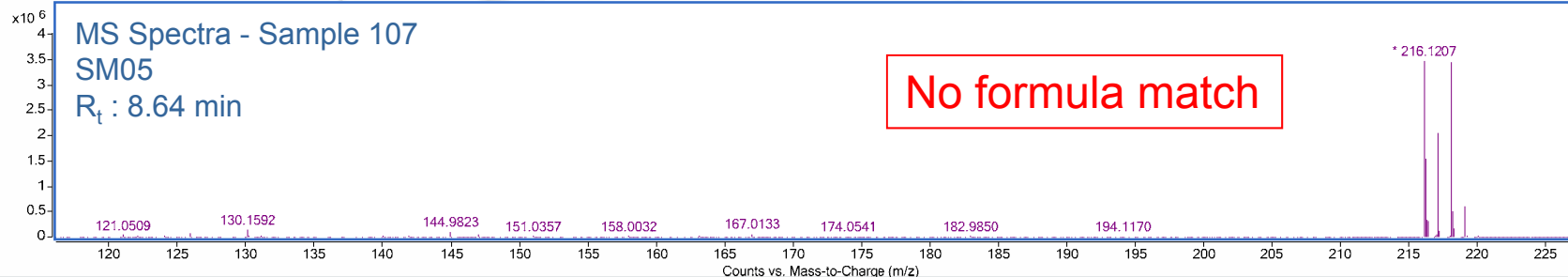
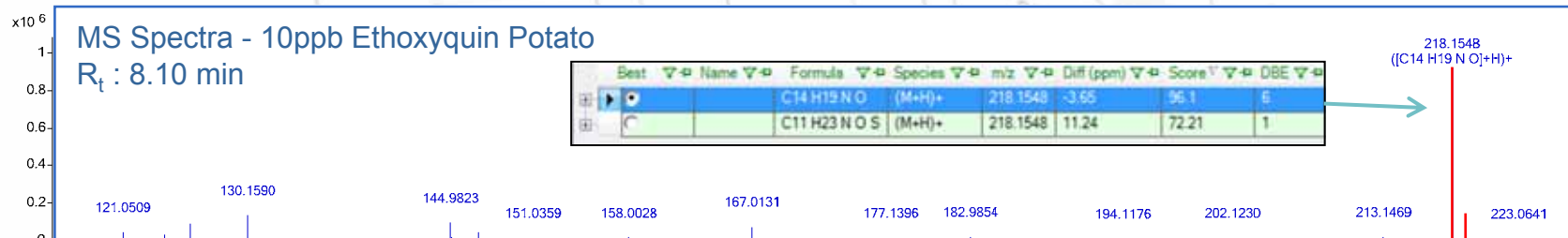
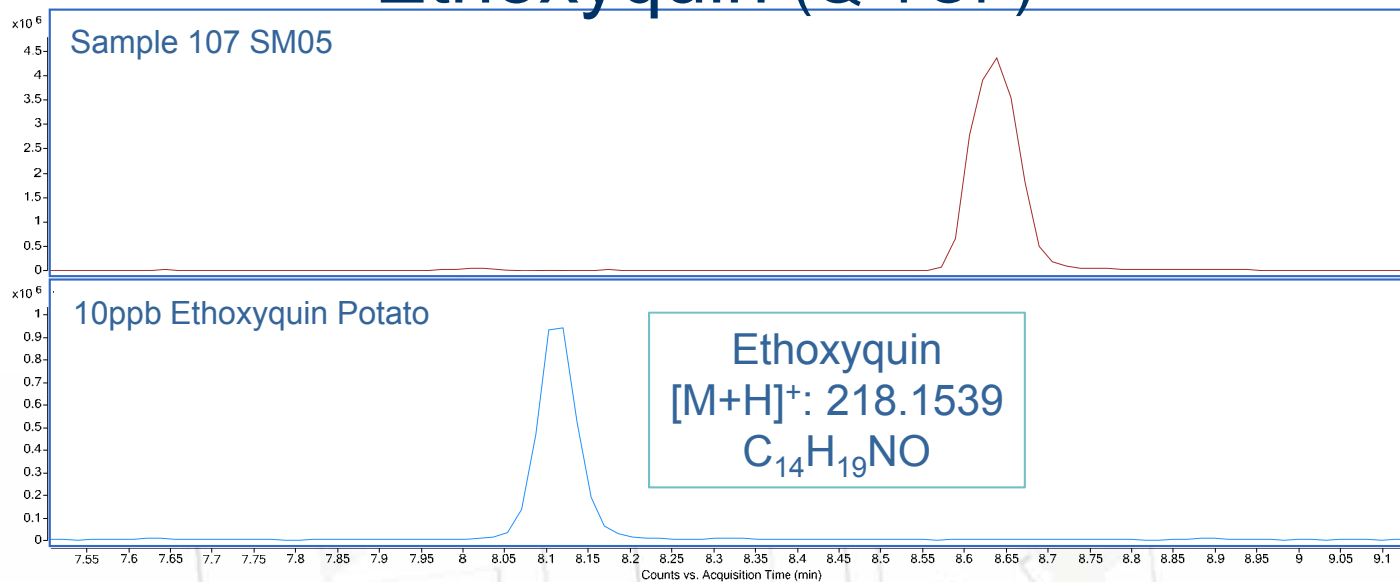
**Propazine, simazine and thiabendazole** were not intentionally used to spike/treat the test material, but they were detected by the organisers at concentrations below 10 ppb due to impurities of the commercial formulations.

**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.

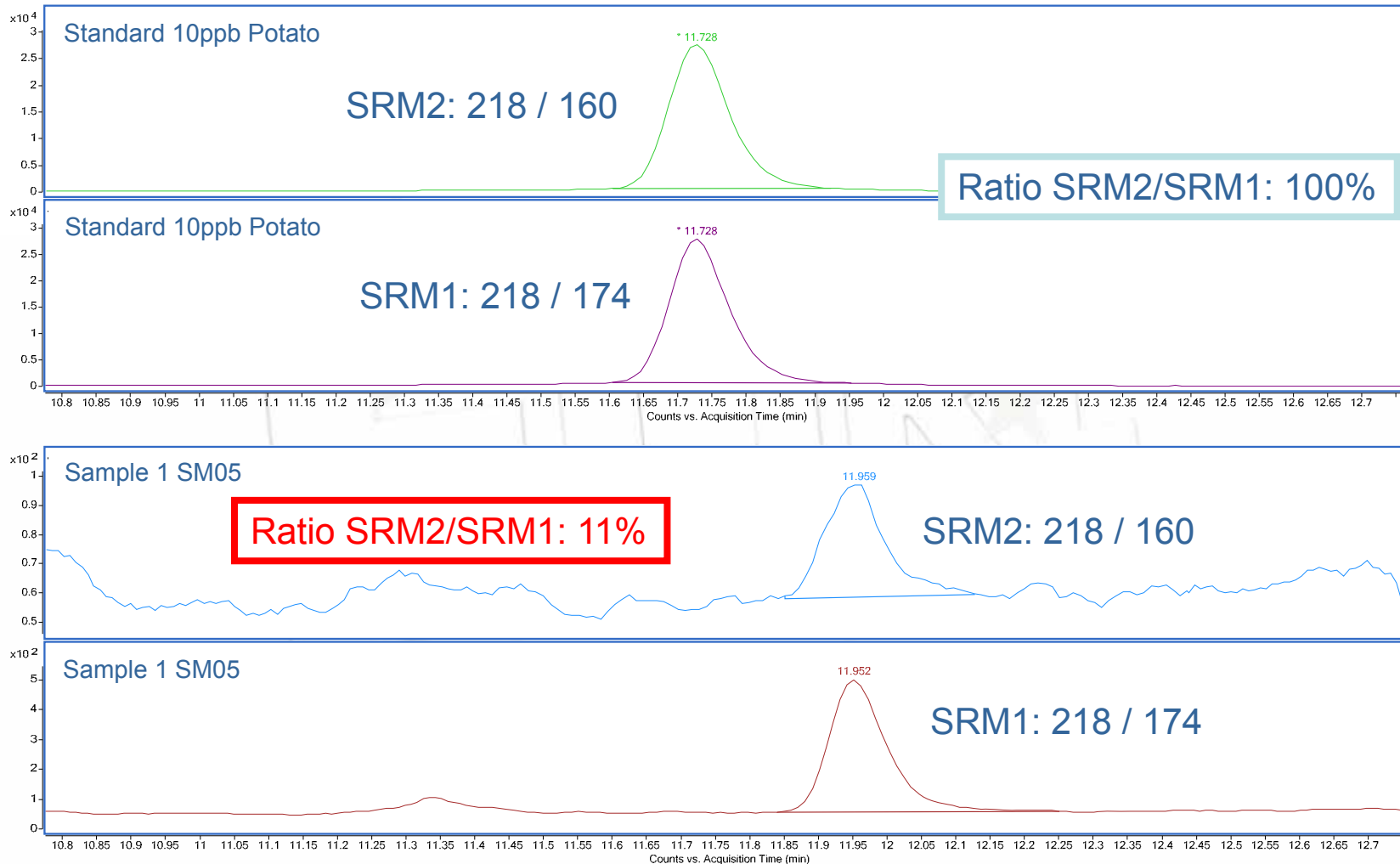


**Ethoxyquin**

# Ethoxyquin (Q-TOF)

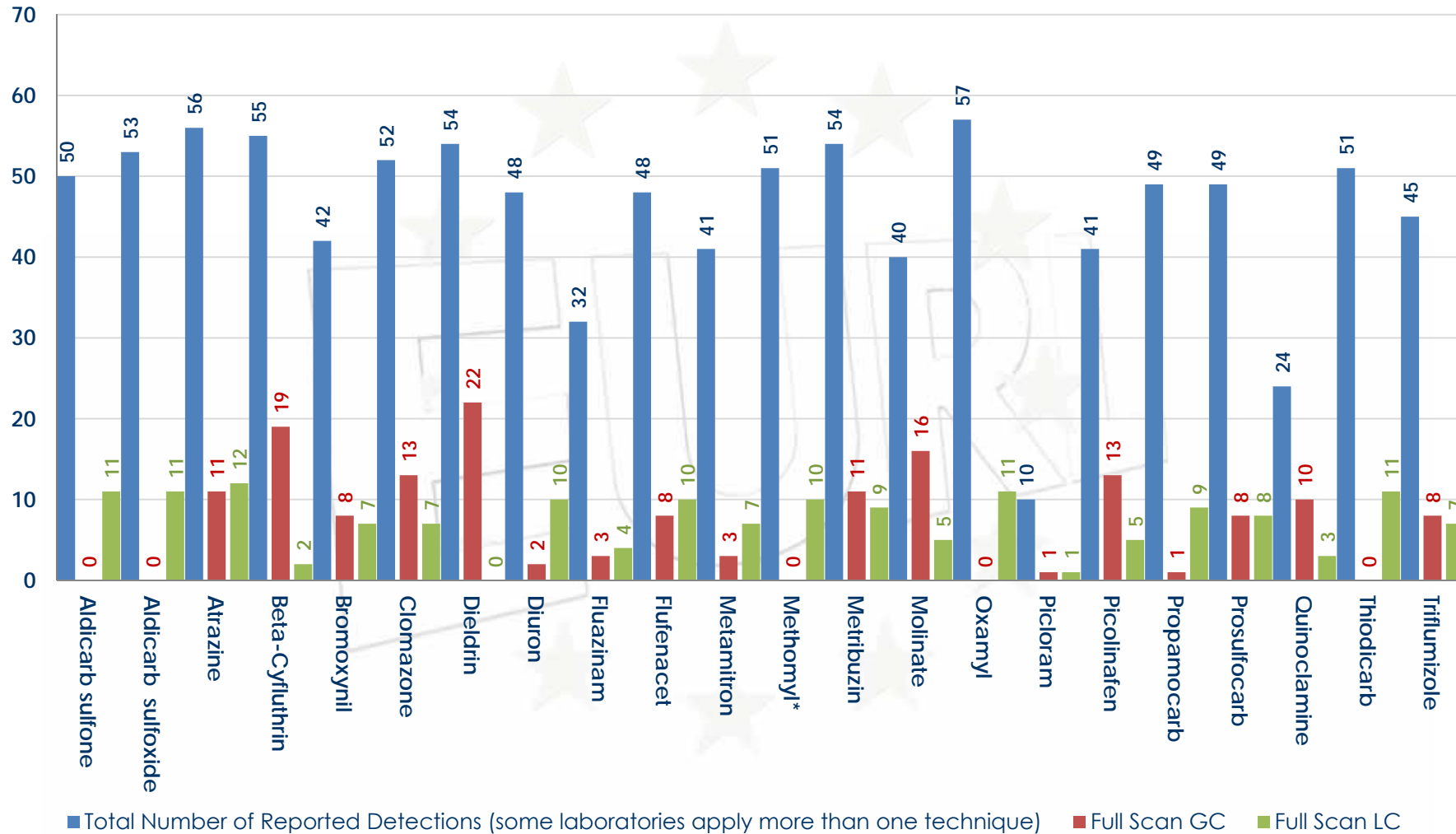


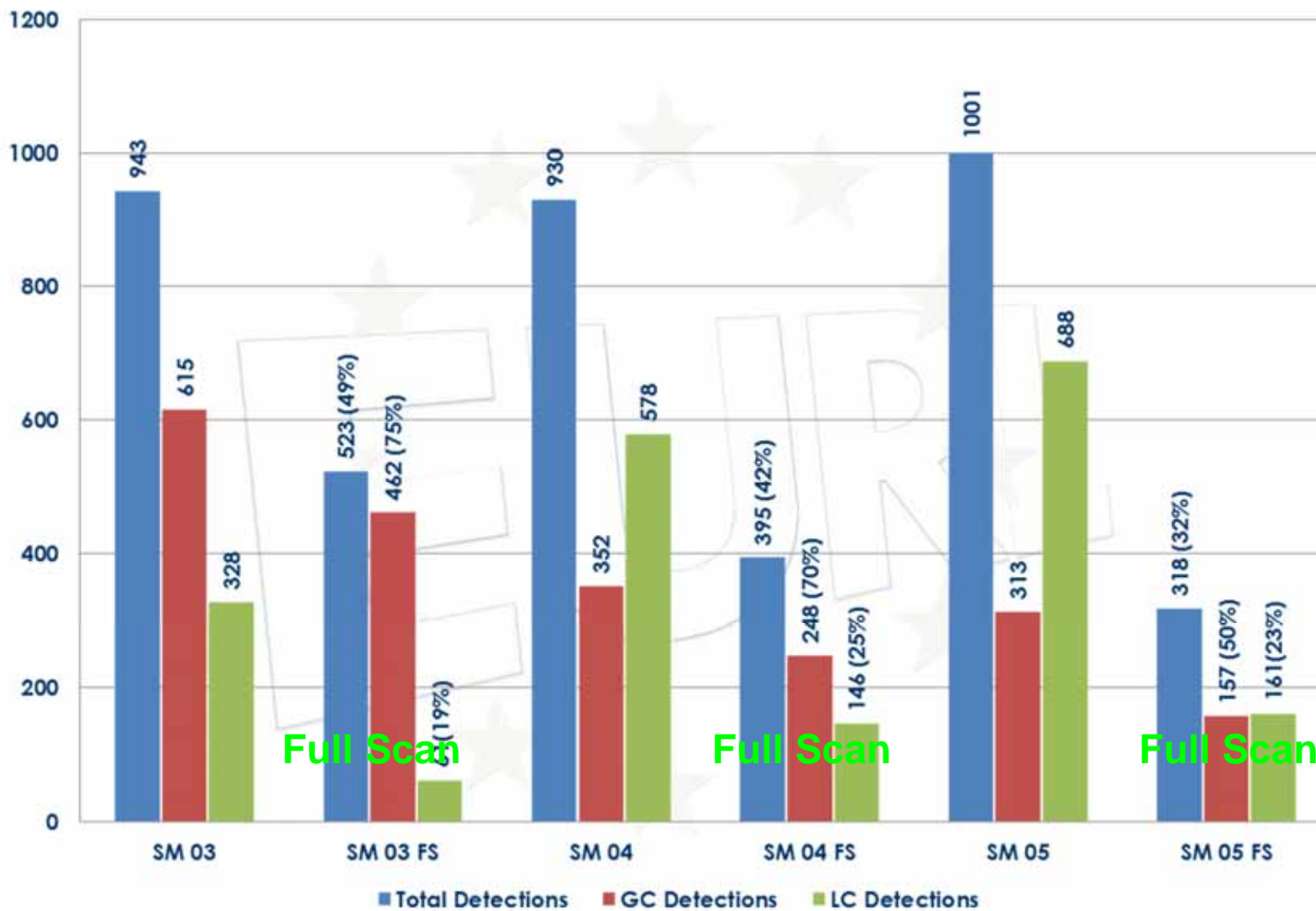
# Ethoxyquin (LC-QQQ)



## 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**



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

**Thanks for  
your Attention**