

# EURL-FV Webinar

## Results

### EUPT-SC02



# Calendar

ACTIVITY	DATE
Opening Registration period	9 <sup>th</sup> November 2018
Deadline for receiving Application Form from laboratories.	16 <sup>th</sup> November 2018
Sample distribution	3 <sup>rd</sup> December 2018
Deadline for receiving results	21 <sup>st</sup> January 2019
Preliminary Report: only results, no statistical treatment	4 <sup>th</sup> February 2018
Preliminary Report with statistical treatment	25 <sup>th</sup> February 2019
Final Report	July 2019



# Target List

**EUPT-FV20 Target List**  
**195 pesticides**

**3 new pesticides in 2018**

**Flonicamid**

**Formetanate**

**Prosulfocarb**

# EUPT-FV-SC02

## European Proficiency Test FV-SC02



# Ground Black Pepper



**Ground black pepper was purchased in bulk in  
the local market in Almería**



# Incurred pesticide residues in black pepper



Acetamiprid\*

Azoxystrobin\*

Carbendazim

Dimethomorph\*

Metalaxyl

Propamocarb\*

\*Residues were detected at concentration levels below the MRRL

## Pesticides used for the treatment

Acetamiprid	Dimethomorph
Azoxystrobin	Imidacloprid
Chlorpyrifos	Oxydemeton-methyl
Cypermethrin	Permethrin
Demeton-S-methylsulfone	Propamocarb

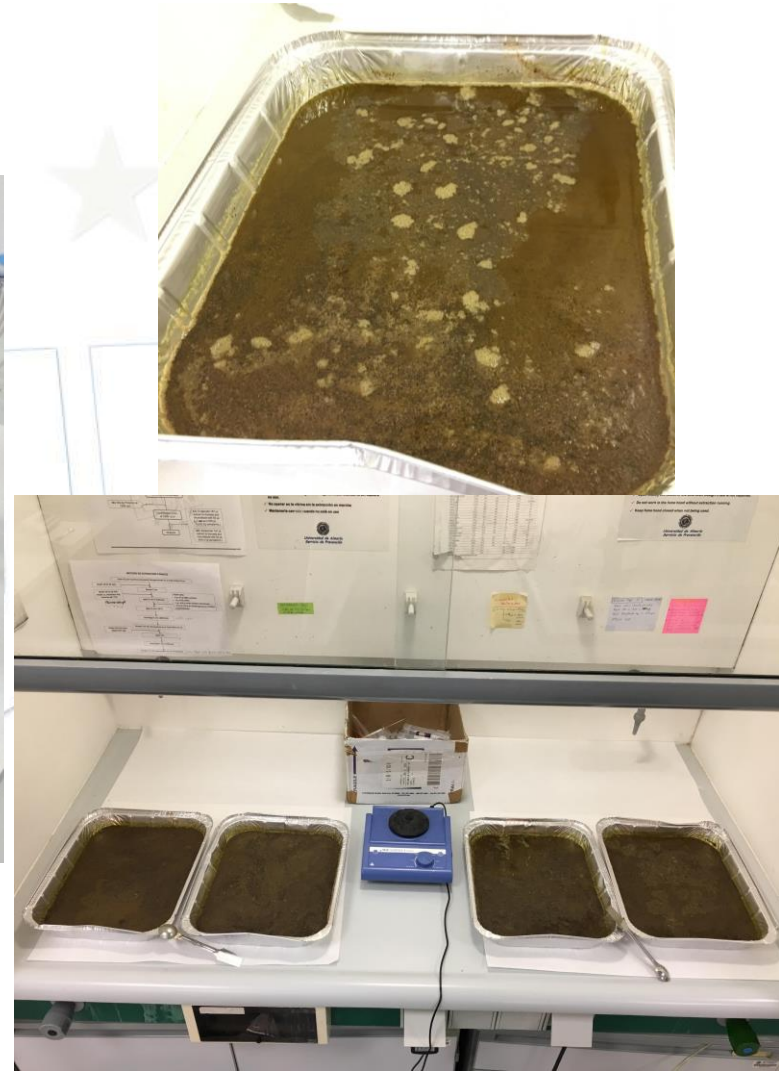
## Incurred pesticides

Carbendazim	Metalaxyl
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**Total: 12**



# Preparation of the test item





## Homogeneity

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

## Stability

*1<sup>st</sup> Analysis - prior to the sample shipment*

*2<sup>nd</sup> Analysis - after the deadline for reporting results*

*3<sup>rd</sup> Analysis - reproducing the delivery conditions that the samples experienced during 48 hours*

**All pesticides passed the homogeneity and stability tests**

# Participation

**Total No. of Labs = 41**

**EU/EFTA Labs = 39**

**Other countries Labs = 2**

**Total No. of Countries = 18**

**EU/EFTA countries = 16**

**Other countries = 2**

**9 NRLs**

**3 participants did not submit results**



**36 EU/EFTA Labs**

# Participation

Member State	Number of Labs
Austria	1
Belgium	3
Bulgary	1
Croatia	3
Cyprus	1
Estonia	1
Finland	1
Germany	6
Greece	2
Hungary	3
Ireland	1
Italy	4
Malta	1
Poland	3
Spain	7
The Netherlands	1

Non-EU/EFTA Country	No. of Labs
Costa Rica	1
Serbia	1



# Results

## Assigned values

	Robust Mean X* (mg/kg)
Imidacloprid	0,021
Carbendazim	0,037
Metalaxyl and metalaxyl-M	0,052
Dimethomorph	0,070
Permethrin	0,078
Propamocarb	0,093
Cypermethrin	0,094
Demeton-S-methylsulfone	0,097
Azoxystrobin	0,155
Oxydemeton-methyl	0,225
Acetamiprid	0,316
Chlorpyrifos	0,336

# Assigned values

0.021-0.097 mg/kg

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Imidacloprid	0,021
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0.021-0.097 mg/kg

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Metalaxyl and metalaxyl-M	0,052
Dimethomorph	0,070

**Assigned value < 3 x MRRL**

**Not included in the evaluation.  
Information shown only for informative purposes**

Azoxystrobin	0,155
Oxydemeton-methyl	0,225
Acetamiprid	0,316
Chlorpyrifos	0,336

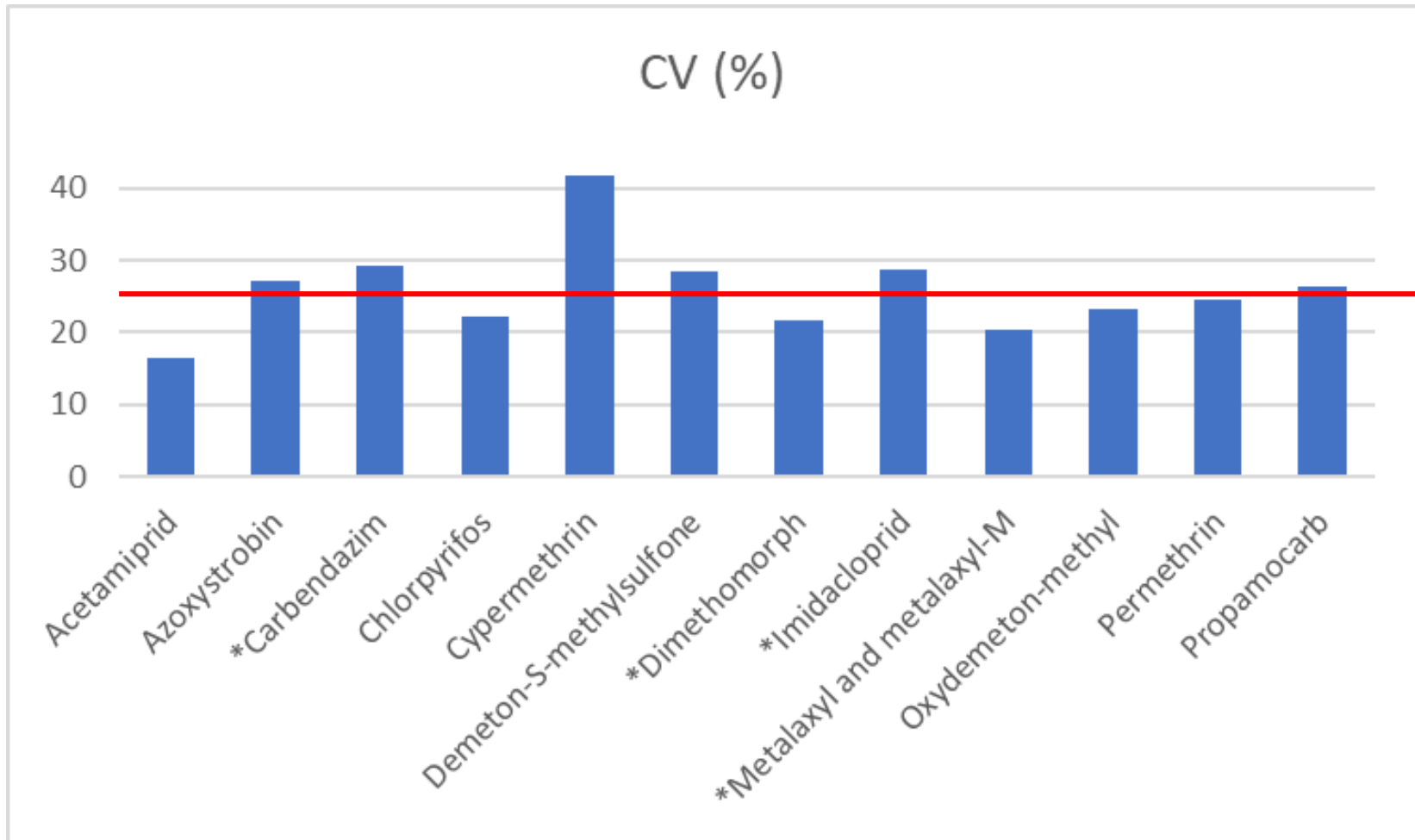
	<b>MRRL (mg/kg)</b>	<b>Robust Mean (mg/kg)</b>	<b>CV (%)</b>
Acetamiprid	0,025	0,316	16,4
Azoxystrobin	0,025	0,155	27,2
Carbendazim*	0,025	0,037	29,3
Chlorpyrifos	0,025	0,336	22,3
Cypermethrin	0,025	0,094	41,8
Demeton-S-methylsulfone	0,025	0,097	28,4
Dimethomorph*	0,025	0,070	21,7
Imidacloprid*	0,025	0,021	28,8
Metalaxyl and metalaxyl-M*	0,025	0,052	20,3
Oxydemeton-methyl	0,025	0,225	23,2
Permethrin	0,025	0,078	24,6
Propamocarb	0,025	0,093	26,4

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# Dispersion of Results



Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Labs Reporting Results (out of 36)
Acetamiprid	34	0	2	94
Azoxystrobin	33	0	3	92
Carbendazim*	30	3 Not reported	3	83
Chlorpyrifos	32	1	3	89
Cypermethrin	23	5	8	64
Demeton-S-methylsulfone	28	1	7	78
Dimethomorph*	32	0 Not reported	4	89
Imidacloprid*	19	13 Not reported	4	53
Metalaxyl and metalaxyl-M*	30	4 Not reported	2	83
Oxydemeton-methyl	27	2	7	75
Permethrin	27	6	3	75
Propamocarb	30	0	6	83

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# z-Scores

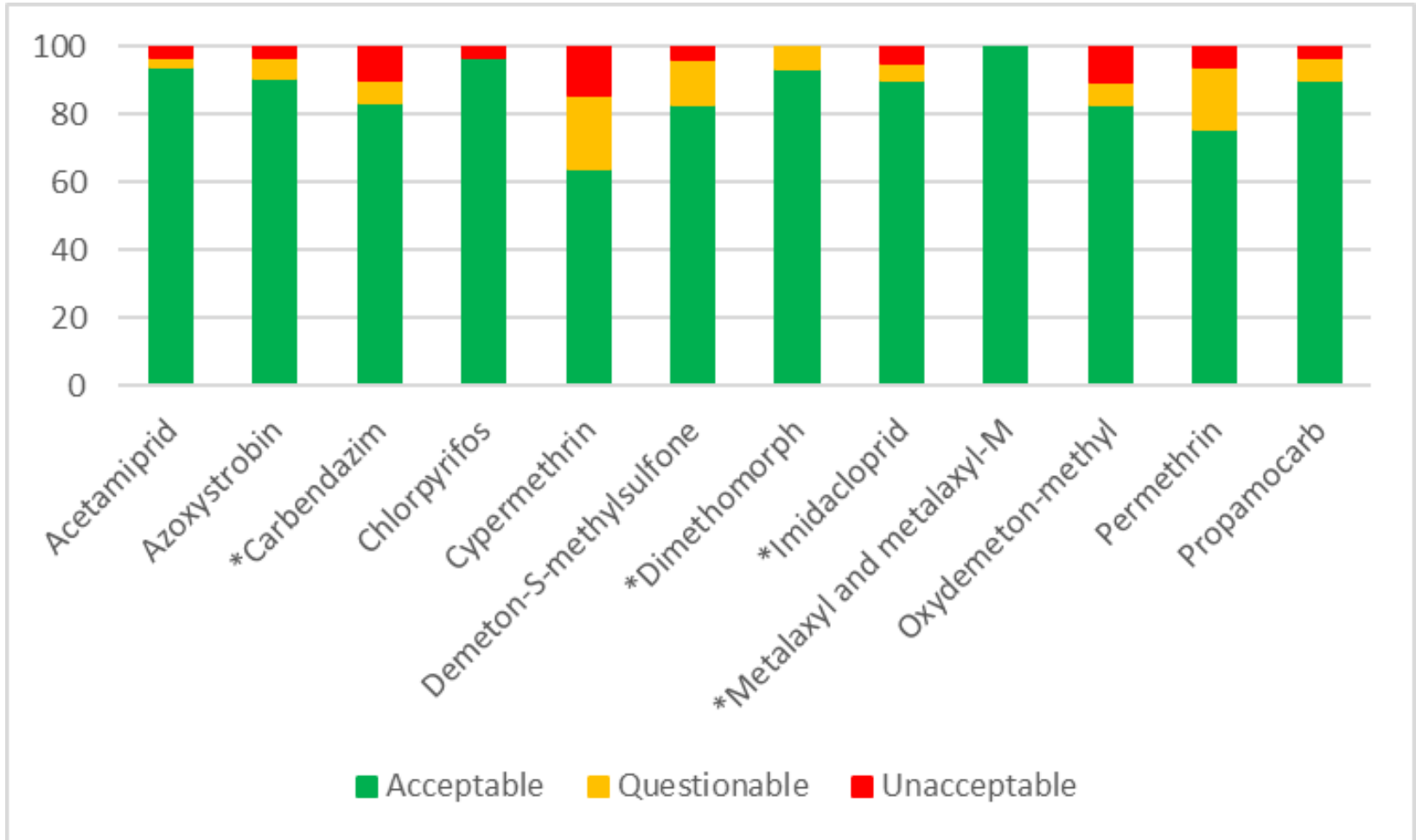
# Z Scores classification

Pesticides	Robust Mean (mg/kg)	% Acceptable z scores	% Questionable z scores	% Unacceptable z scores
Acetamiprid	0,316	94,1	2,9	2,9
Azoxystrobin	0,155	90,9	6,1	3,0
Carbendazim*	0,037	83,3	6,7	10,0
Chlorpyrifos	0,336	97,0	0,0	3,0
Cypermethrin	0,094	64,3	21,4	14,3
Demeton-S-methylsulfone	0,097	82,8	13,8	3,4
Dimethomorph*	0,070	93,8	6,3	0,0
Imidacloprid*	0,021	94,7	0,0	5,3
Metalaxyl and metalaxyl-M*	0,052	100,0	0,0	0,0
Oxydemeton-methyl	0,225	82,8	6,9	10,3
Permethrin	0,078	75,8	18,2	6,1
Propamocarb	0,093	90,0	6,7	3,3

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Azoxystrobin	0,155	90,9	6,1	3,0
Carbendazim*	0,037	83,3	6,7	10,0
Chlorpyrifos	0,336	97,0	0,0	3,0
Cypermethrin	0,094	64,3	21,4	14,3
Demeton-S-methylsulfone	0,097	82,8	13,8	3,4
Dimethomorph*	0,070	93,8	6,3	0,0
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Metalaxyl and metalaxyl-M*	0,052	100,0	0,0	0,0
Oxydemeton-methyl	0,225	82,8	6,9	10,3
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Propamocarb	0,093	90,0	6,7	3,3

## Z Scores classification

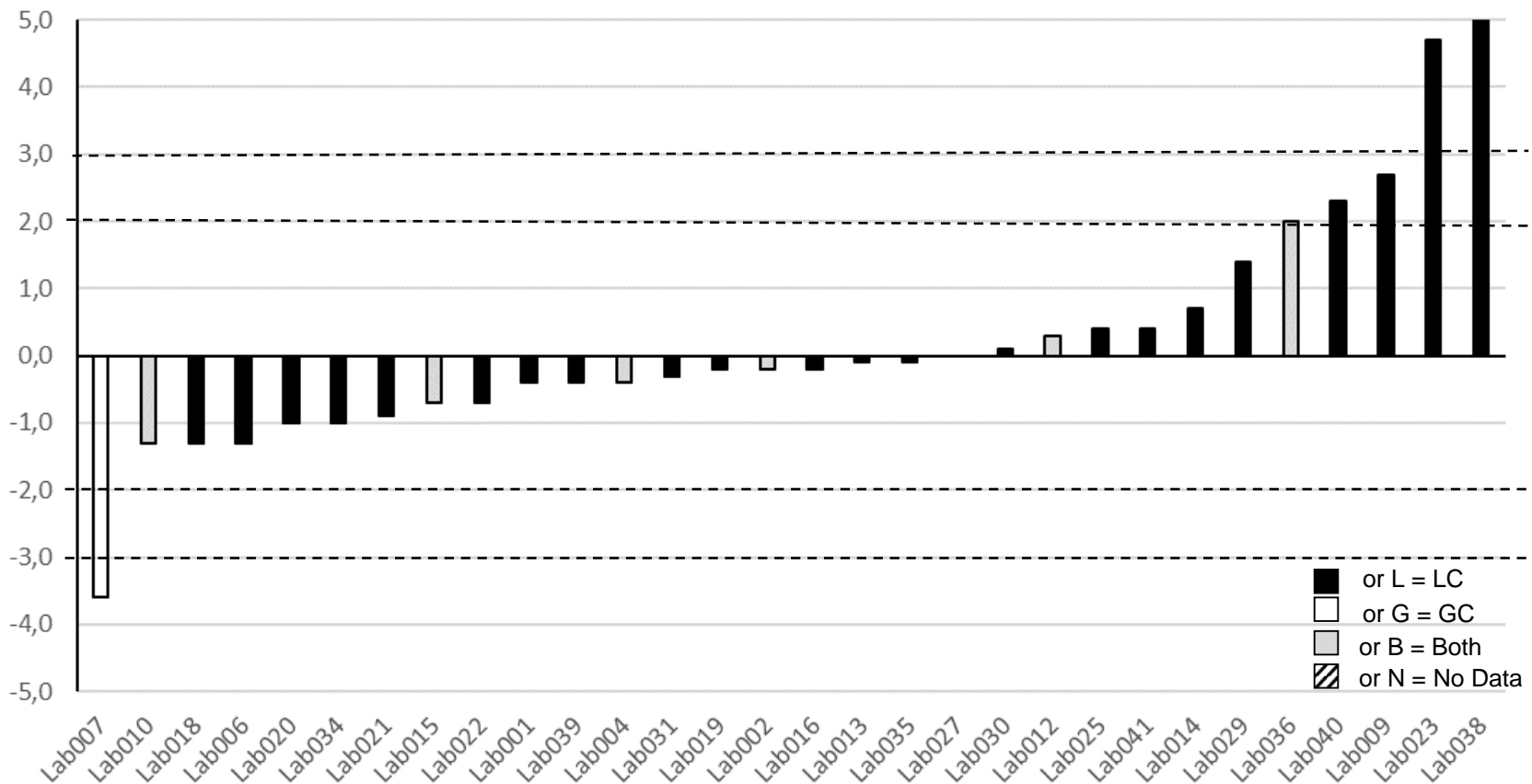




83.3 % Acceptable  
6.7 % Questionable  
10.0 % Unacceptable

Robust mean: 0.037 mg/kg  
CV: 29.3 %

### Carbendazim



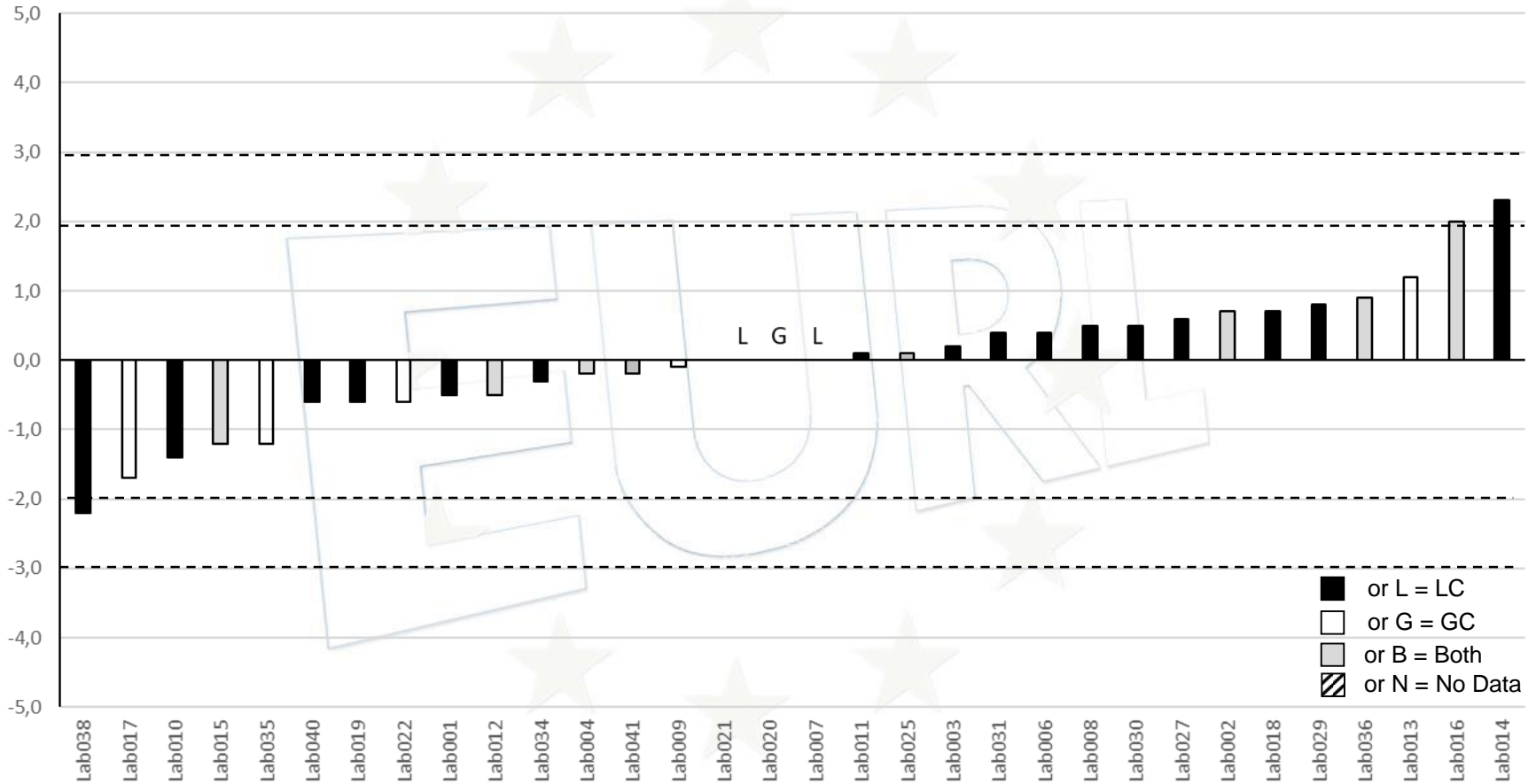
Not included in the evaluation. Information shown only for informative purposes



93.8 % Acceptable  
 6.3 % Questionable  
 0.0 % Unacceptable

**Dimethomorph**

Robust mean: 0.070 mg/kg  
 CV: 21.7 %

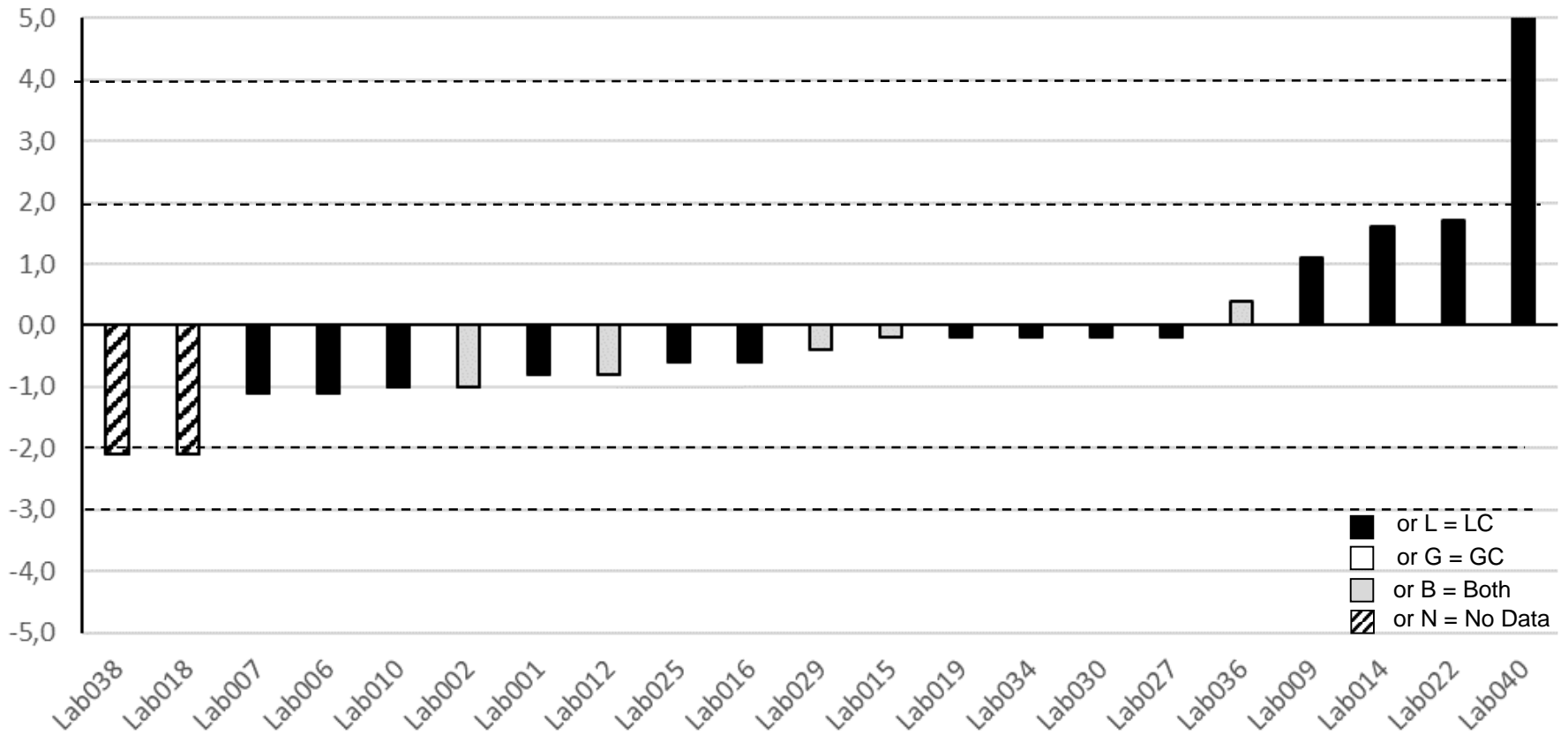


Not included in the evaluation. Information shown only for informative purposes

94.7 % Acceptable  
 0.0 % Questionable  
 5.3 % Unacceptable

Robust mean: 0.021 mg/kg  
 CV: 28.8 %

## Imidacloprid

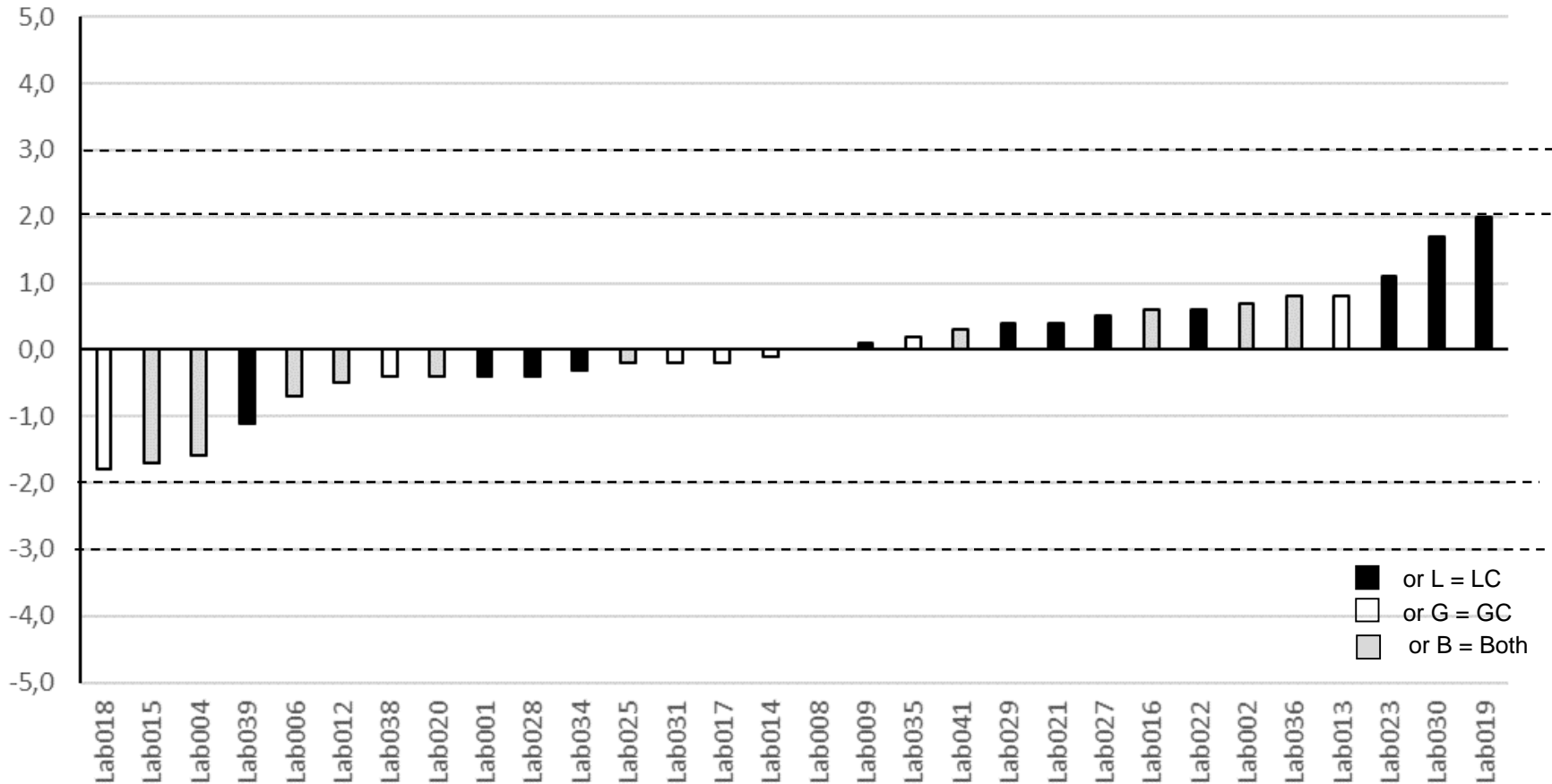


Not included in the evaluation. Information shown only for informative purposes

100 % Acceptable  
 0 % Questionable  
 0 % Unacceptable

Robust mean: 0.052 mg/kg  
 CV: 20.3 %

### Metalaxyl and metalaxyl-M

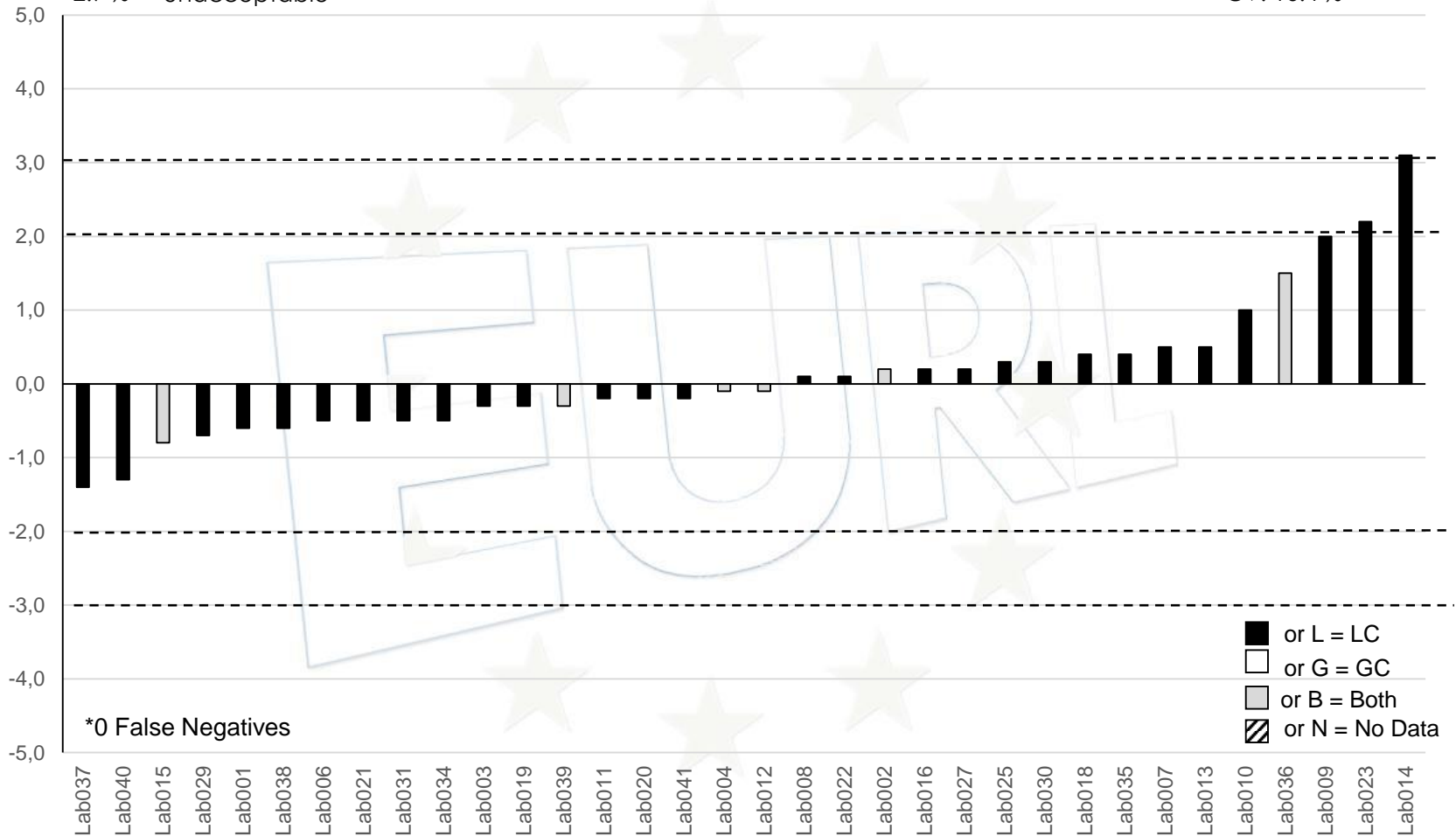


Not included in the evaluation. Information shown only for informative purposes

94.1 % Acceptable  
2.9 % Questionable  
2.9 % Unacceptable

### Acetamidiprid

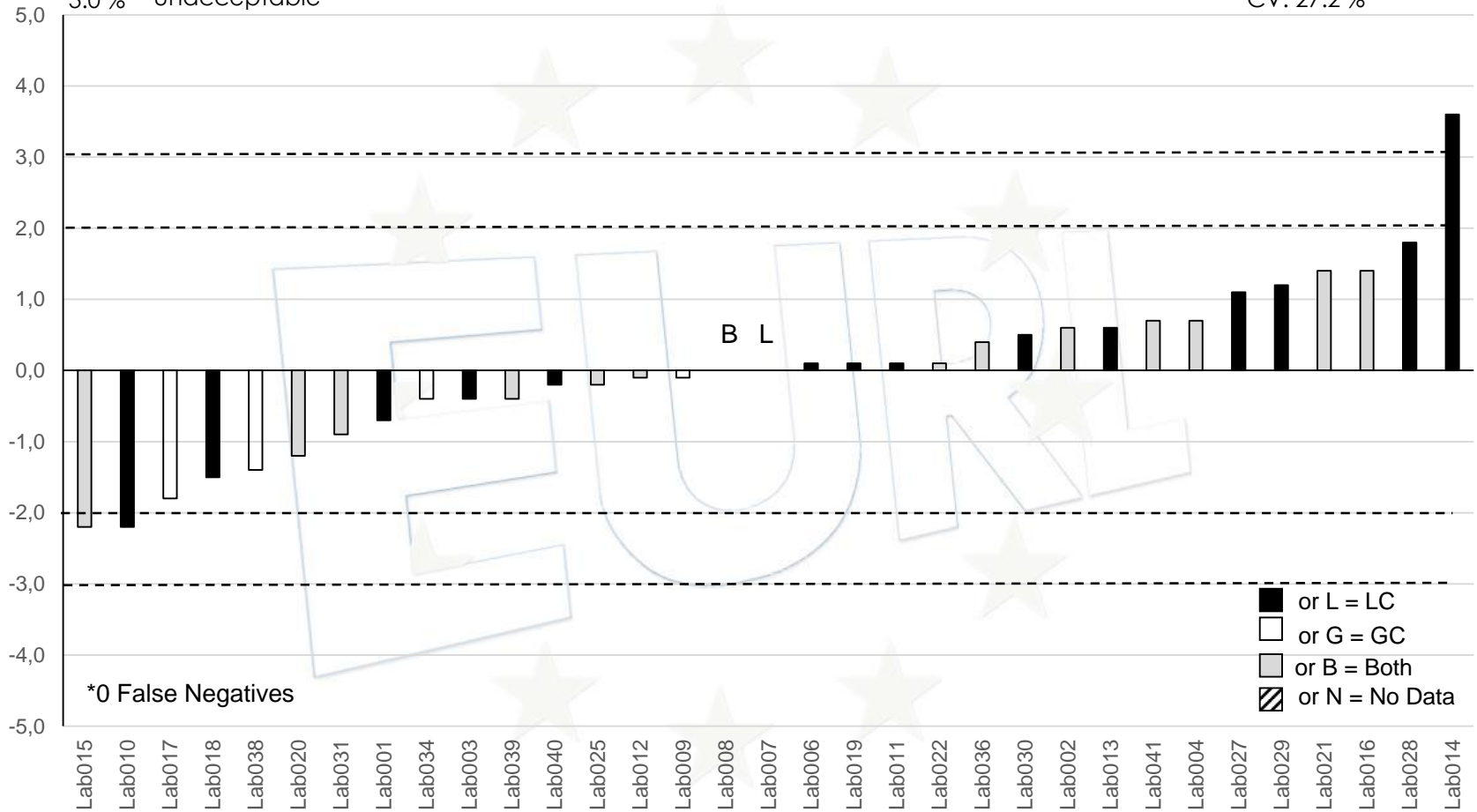
Robust mean: 0.316 mg/kg  
CV: 16.4 %



90.9 % Acceptable  
 6.1 % Questionable  
 3.0 % Unacceptable

### Azoxystrobin

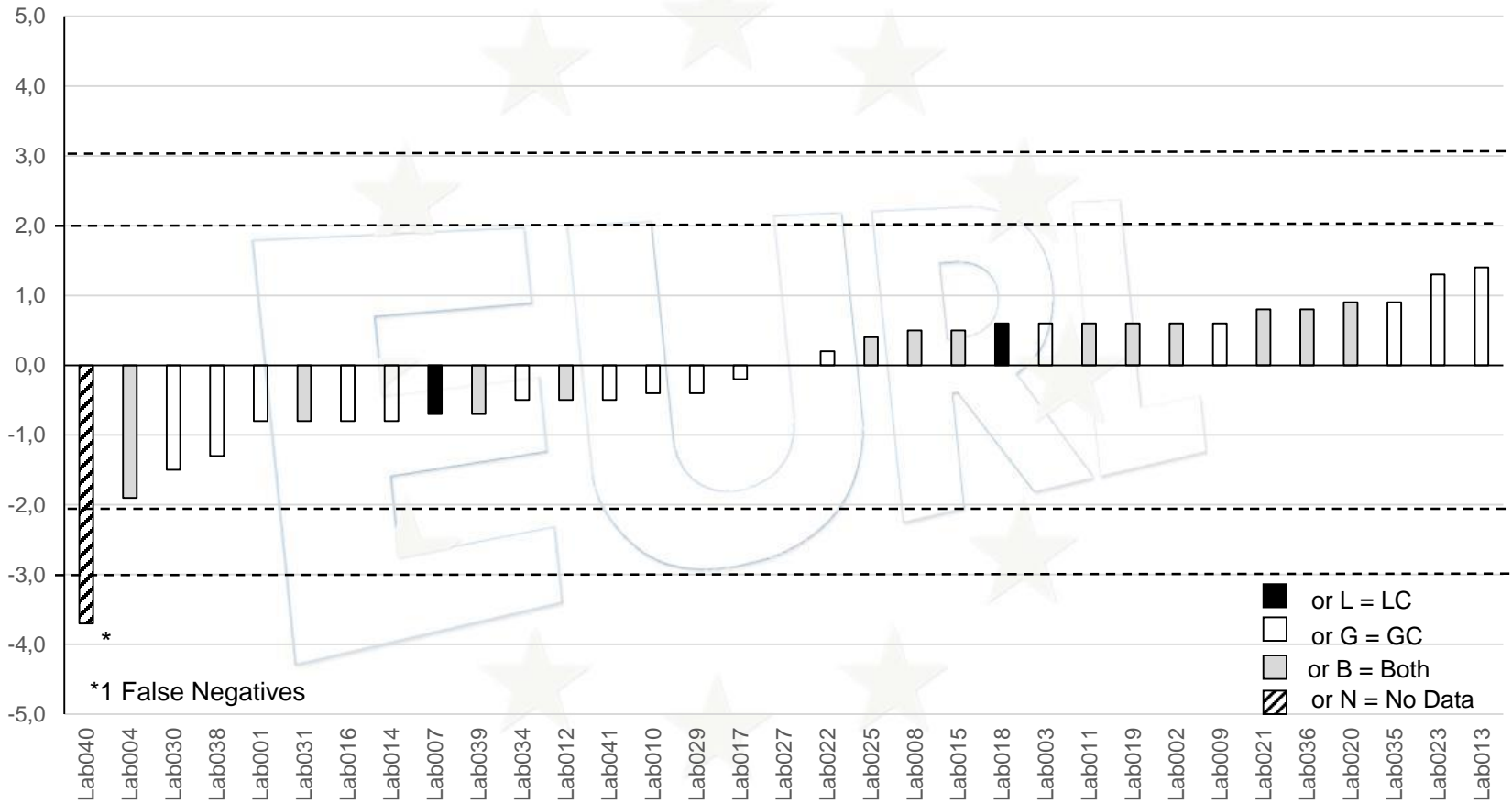
Robust mean: 0.155 mg/kg  
 CV: 27.2 %



97.0 % Acceptable  
 0.0 % Questionable  
 3.0 % Unacceptable

Robust mean: 0.336 mg/kg  
 CV: 22.3 %

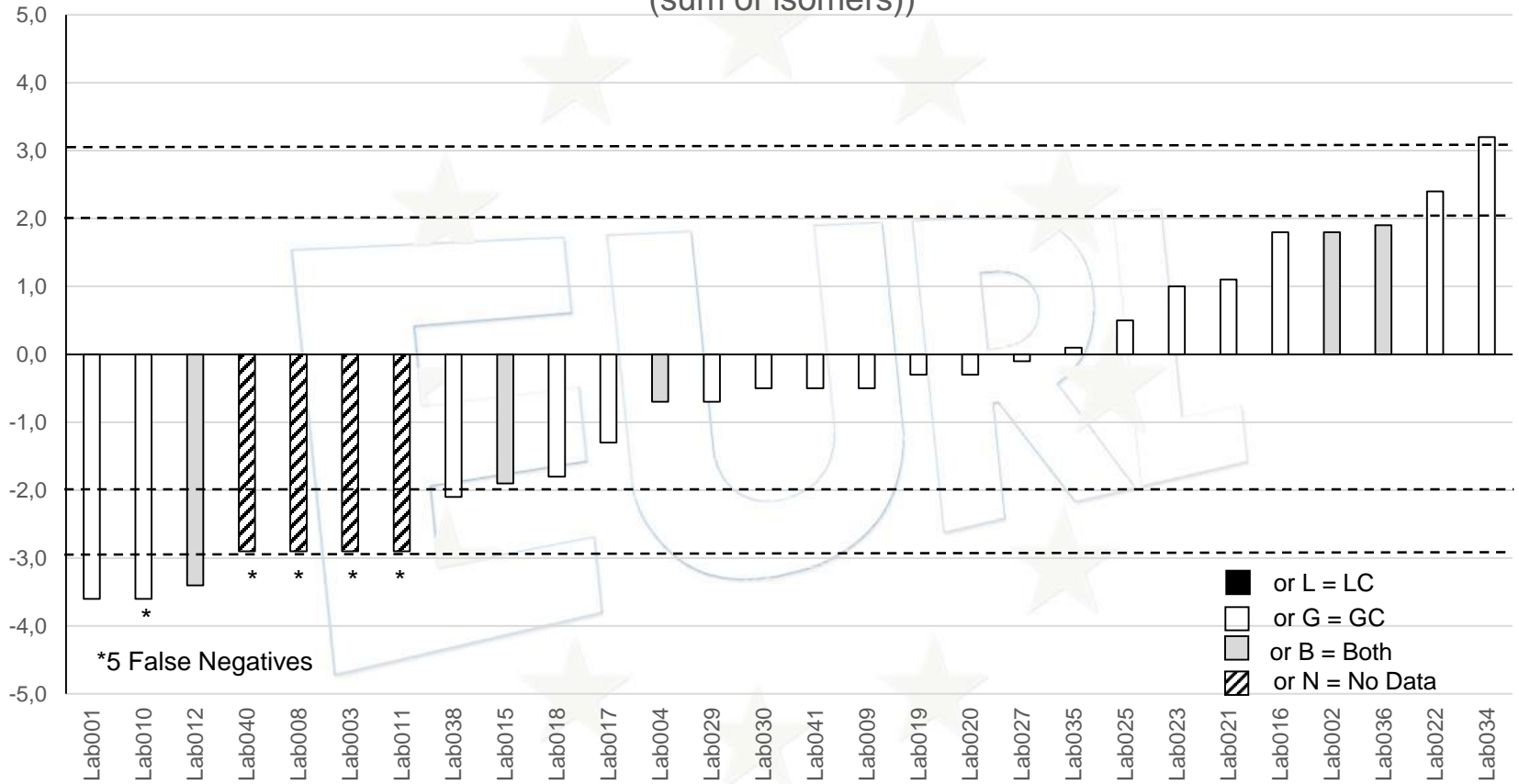
### Chlorpyrifos



64.3 % Acceptable  
21.4 % Questionable  
14.3 % Unacceptable

### Cypermethrin (cypermethrin incl. other mixtures of constituent isomers (sum of isomers))

Robust mean: 0.094 mg/kg  
CV: 41.8 %

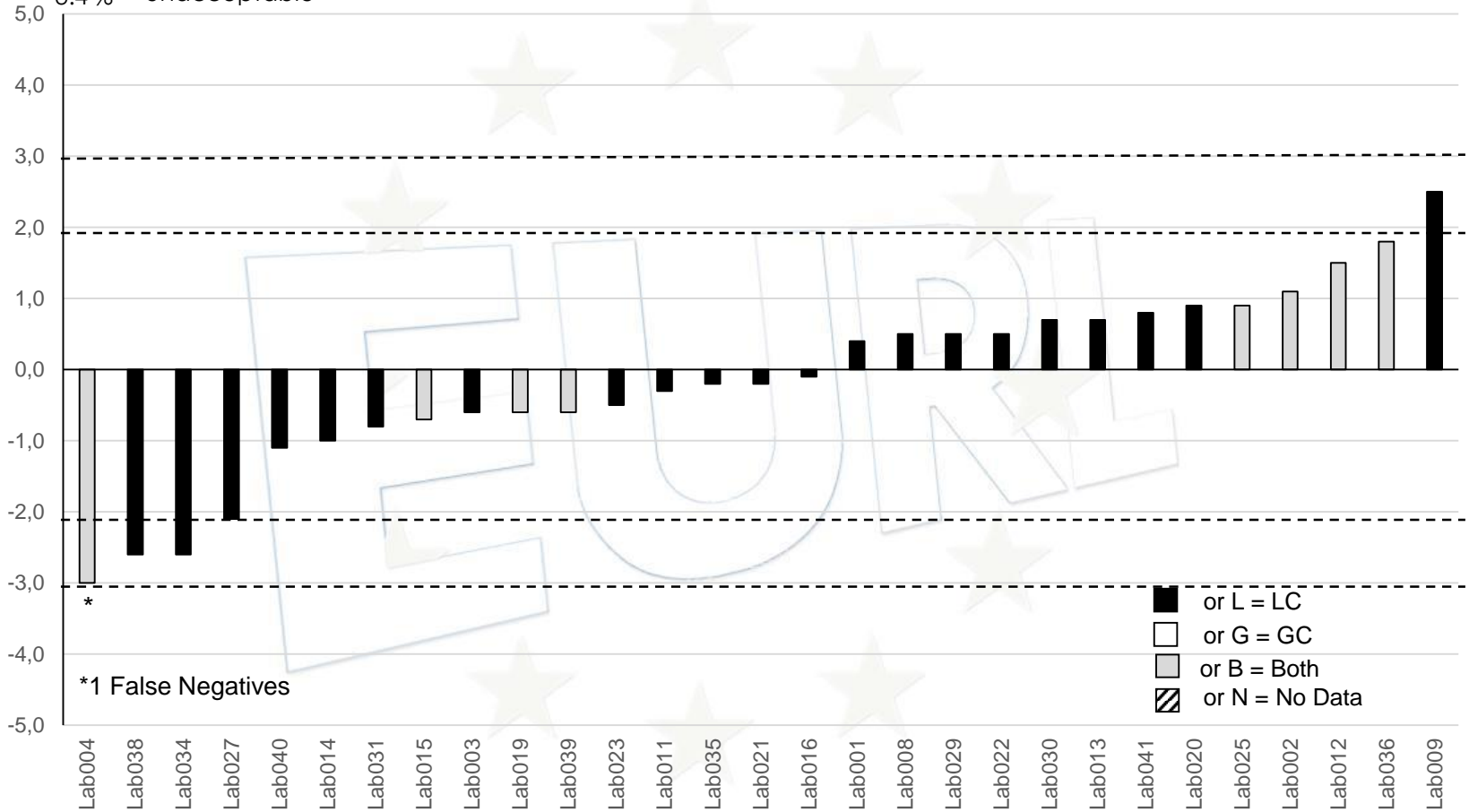




82.8 % Acceptable  
 13.8 % Questionable  
 3.4 % Unacceptable

### Demeton-S-methylsulfone

Robust mean: 0.097 mg/kg  
 CV: 28.4 %



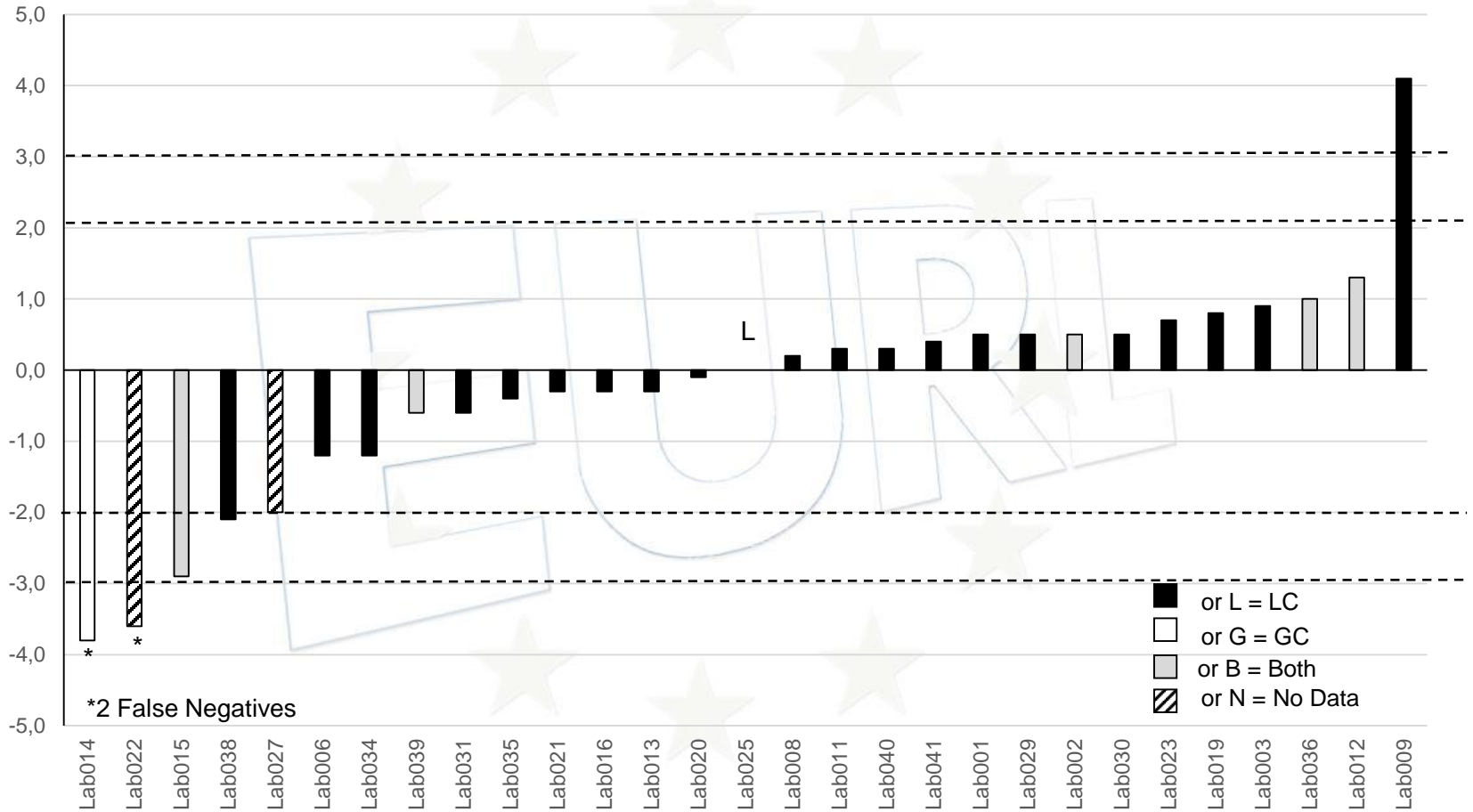
\*1 False Negatives

- or L = LC
- or G = GC
- ▒ or B = Both
- ▨ or N = No Data

82.8 % Acceptable  
 6.9 % Questionable  
 10.3 % Unacceptable

### Oxydemeton-methyl

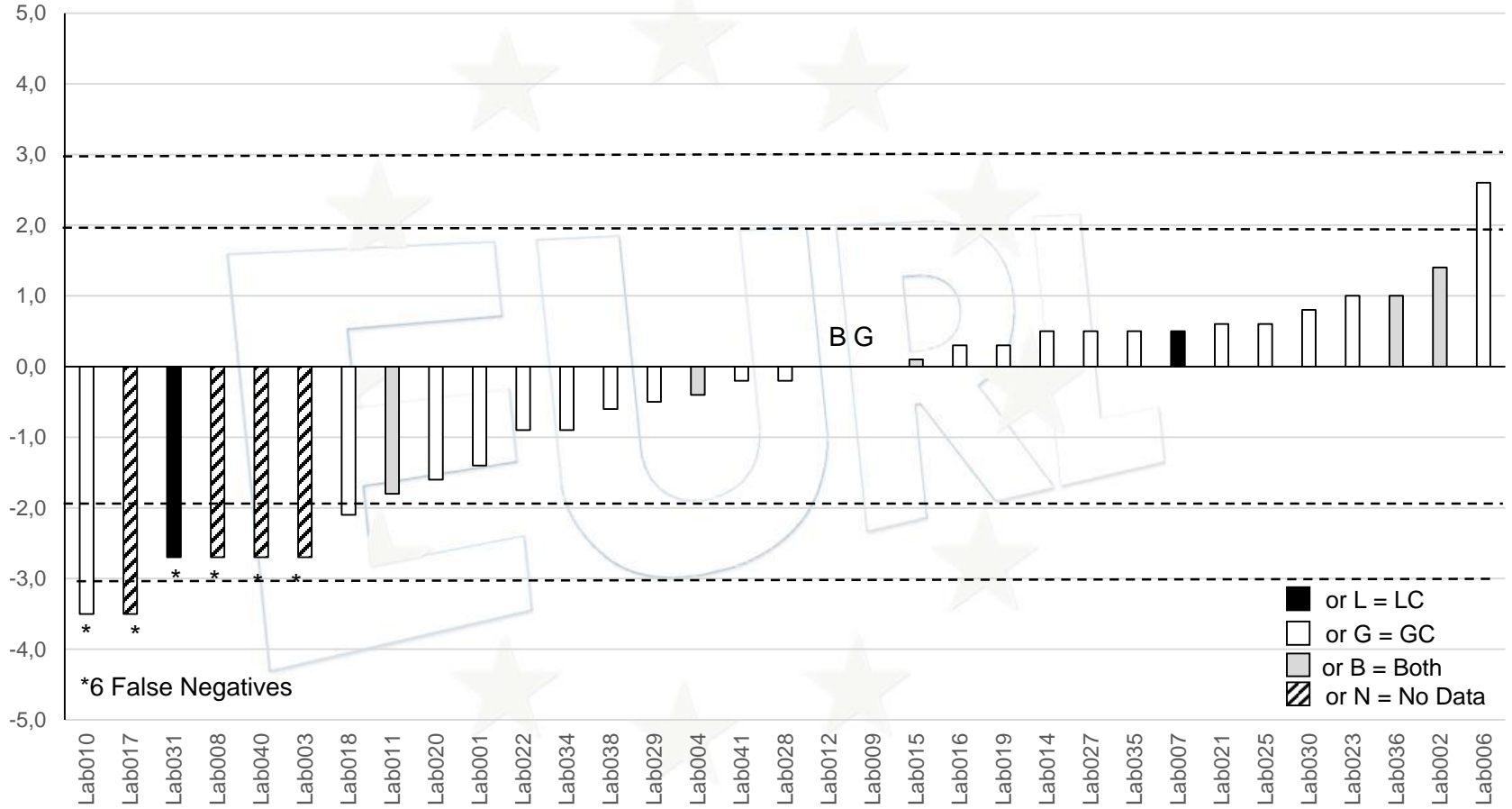
Robust mean: 0.225 mg/kg  
 CV: 23.2 %

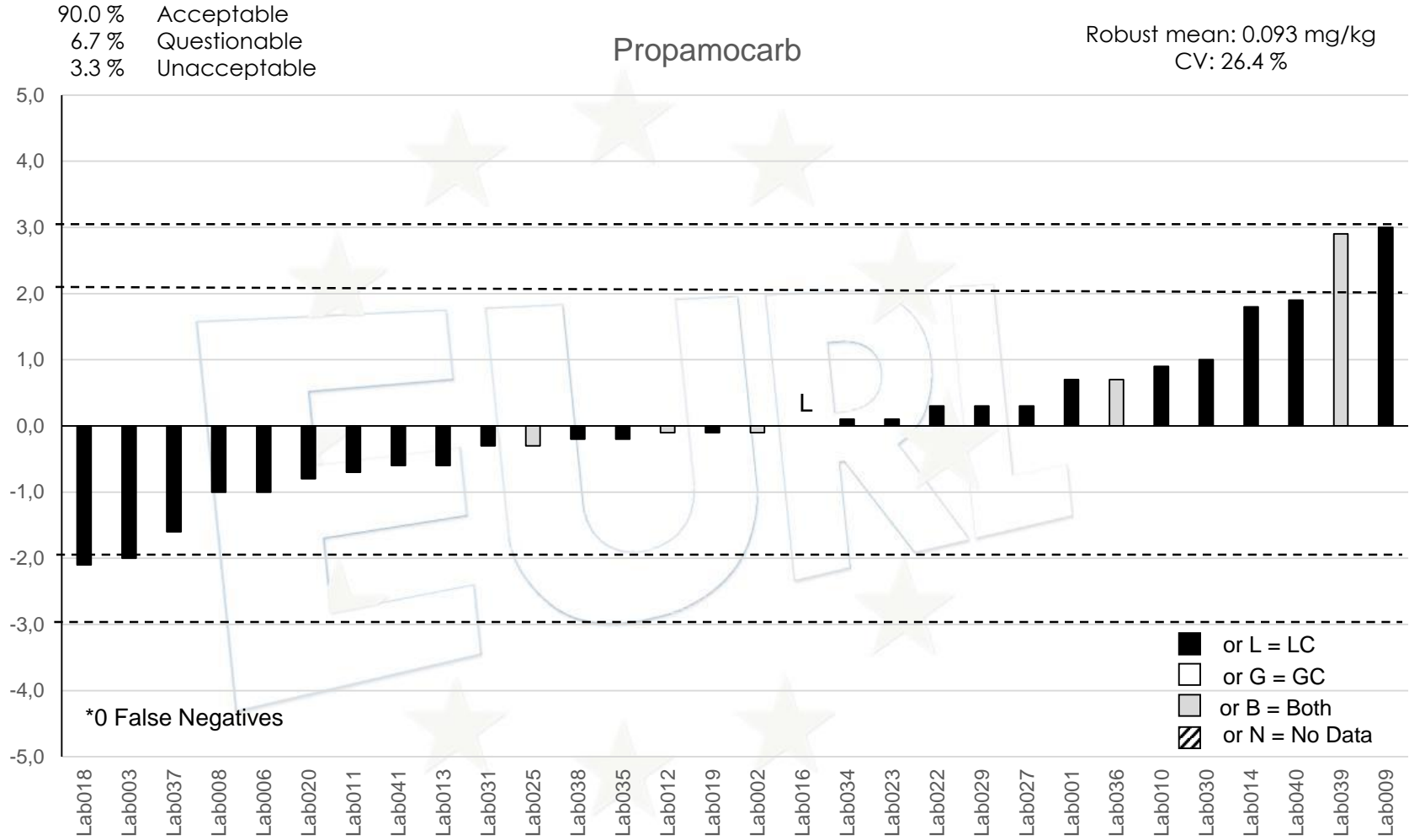


75.8 % Acceptable  
 18.2 % Questionable  
 6.1 % Unacceptable

Robust mean: 0.078 mg/kg  
 CV: 24.6 %

### Permethrin (sum of isomers)







# Combined z-Scores

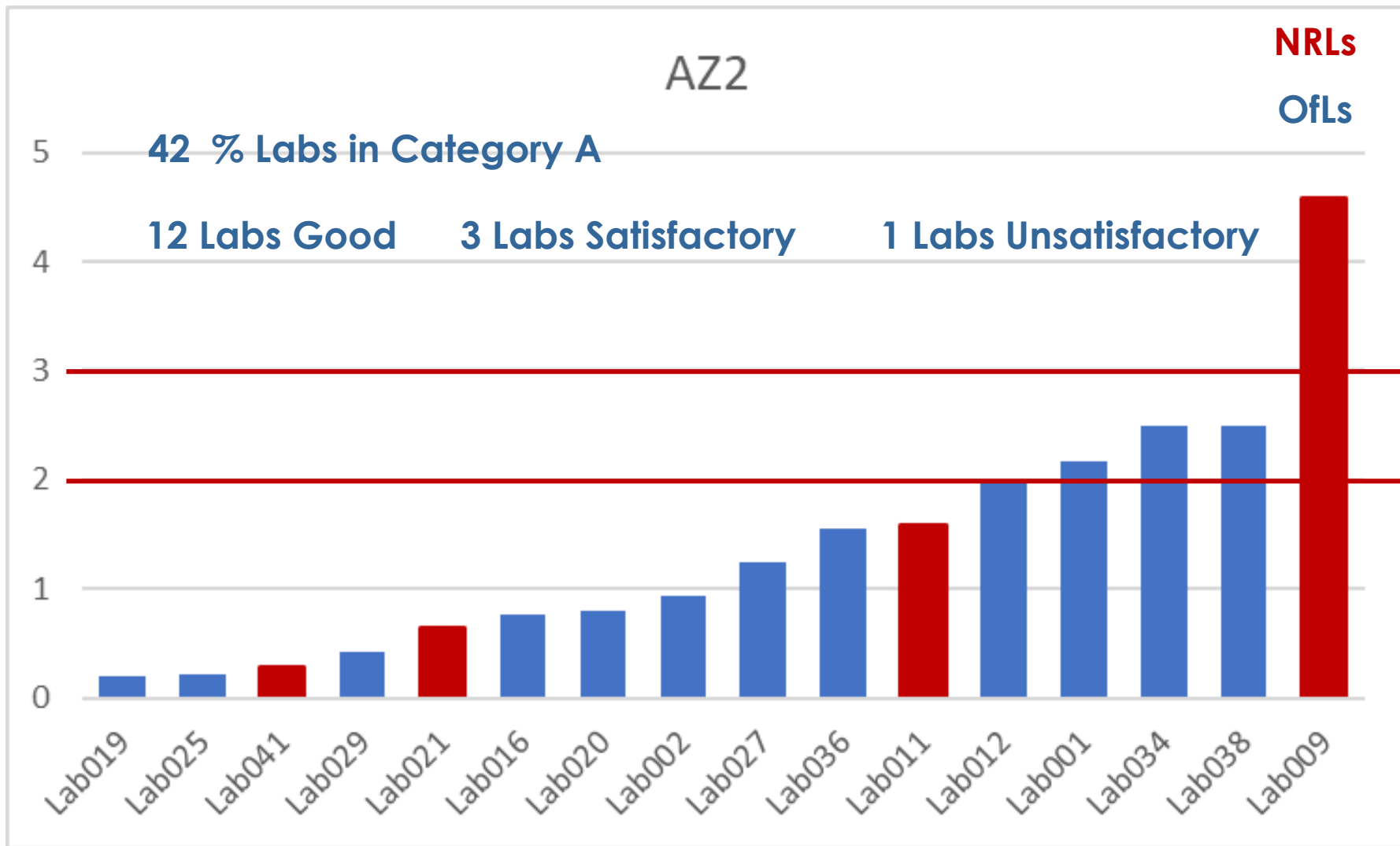
# Average of Squared z-Scores

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

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

## Category A

Laboratories that were able to analyse at least **90% of the compulsory pesticides in the target pesticides list**, that detected and quantified at least **90 % of the pesticides present in the Test Item** and reported **no false positives**.





## False Positives

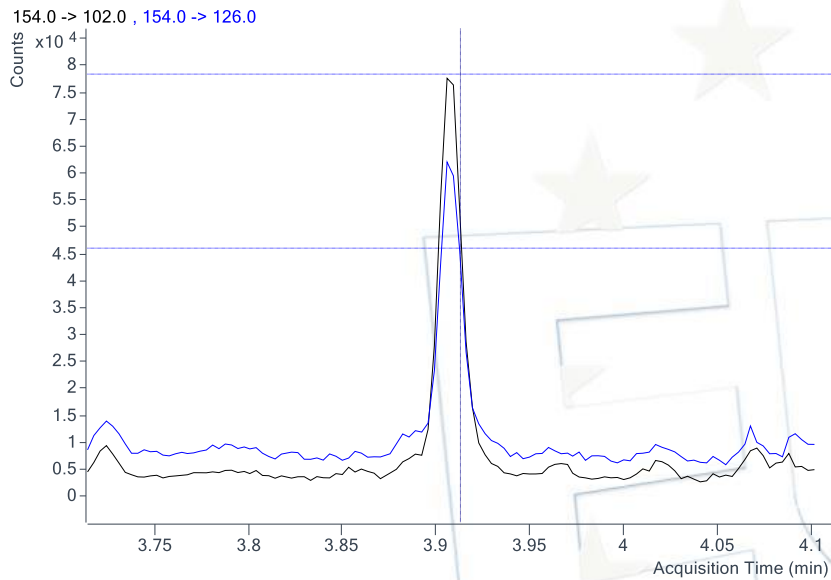
**2** different laboratories from EU/EFTA countries reported **1** pesticide as false positive



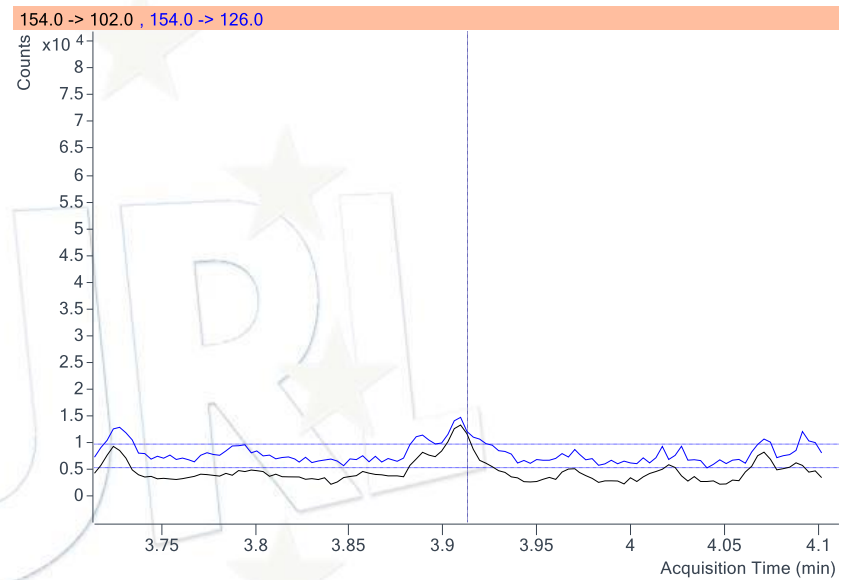
## False Positives

Laboratory Code	Pesticide	Concentration (mg/kg)	Determination Technique	RL (mg/Kg)	MRRL (mg/Kg)
Lab010	Biphenyl	0.535	GC-MS/MS (QQQ)	0.01	0.025
Lab030	Biphenyl	0.099	GC-MS/MS (QQQ) CI neg	0.01	0.025

# False Positives



Standard Biphenyl  
20 ppb



Test item EUPT-SC02  
(Sample 15)

**GC-MS/MS (QqQ)**

**Thank You  
for Your Attention**



**EURL** EUROPEAN  
UNION  
REFERENCE  
LABORATORY