

# Results of EUPT-CF9 Pesticide residues in maize

Mette Erecius Poulsen and Susan Strange Herrmann

Stuttgart, 1 October 2015

$$f(x+\Delta x) = \sum_{i=0}^{\infty} \frac{(\Delta x)^i}{i!} f^{(i)}(x)$$
$$\int_a^b \Theta^{\sqrt{17}} + \Omega \int \delta e^{i\pi} =$$
$$\Theta = \{2.71828182845904523536028747135266249775724706$$
$$\infty = \text{DTU}$$
$$\chi^2 \Sigma \gg !,$$

# PTs on cereals/feed – overview

		2007 EUPT-C1 /SRM2	2008 EUPT-C2	2009 EUPT-C3 /SRM4	2010 EUPT-C4	2011 EUPT-C5 /SRM6	2012 EUPT-C6	2013 EUPT-CF7	2014 EUPT-CF8	2015 EUPT-CF9
	Test material	Wheat flour	Wheat flour	Oat flour	Rye flour	Rice flour	Barley	Feed	Wheat flour	Maize flour
	no. of participants	<b>64</b>	<b>74</b>	<b>111</b>	<b>118</b>	<b>155 (133)</b>	<b>149 (127)</b>	<b>120 (106)</b>	<b>160 (142)</b>	<b>163 (146)</b>
	No. of target pesticides	<b>37</b>	<b>55</b>	<b>60</b>	<b>72</b>	<b>104</b>	<b>107</b>	<b>116</b>	<b>116</b>	<b>117</b>
MRM	no. of <b>incurred</b> pesticides	<b>3</b>	<b>9</b>	<b>14</b>	<b>13</b>	<b>10</b>	<b>8</b>	<b>19</b>	<b>14</b>	<b>0</b>
	no. of <b>spiked</b> pesticides	<b>4</b>	<b>4</b>		<b>3</b>	<b>7</b>	<b>10</b>	<b>4</b>	<b>5</b>	<b>18</b>
SRM	no. of <b>incurred</b> pesticides	<b>2</b>	<b>2</b>	<b>2</b>	<b>2</b>					
	no. of <b>spiked</b> pesticides	<b>1</b>			<b>1</b>	<b>7</b>				
	Total no. of pesticides in test item	<b>10</b>	<b>15</b>	<b>18</b>	<b>19</b>	<b>24</b>	<b>18</b>	<b>23</b>	<b>19</b>	<b>18</b>

# Organising team at EURL

- **Mette Erecius Poulsen, Senior Chemist**
- **Anne Kruse Lykkeberg, Senior Chemist**
- **Gitte Andersen, Chemist**
- **Susan Strange Herrmann, Chemist**
- **Merete B. Ludvigsen, Chemical Technician**
- **Lisbet Pilhkjær, Chemical Technician**
- **Jens-Ole Frimann, System Developer**

# Scientific Group

## Advisory Group

**Amadeo R. Fernández-Alba**

**André de Kok**

**Antonio Valverde**

**Darinka Štajnbaher**

**Magnus Jezussek**

**Michelangelo  
Anastassiades**

**Miguel Gamón**

**Ralf Lippold**

**Dr. Sonja Masselter**

**Stewart Reynolds**

**Tuija Pihlström**

## Quality Group

**Antonio Valverde**

**Stewart Reynolds**

# Participation

Country	# labs	Country	# labs	Country	# labs
Albania	1	France	8	Norway	1
Argentina	1	Germany	23	Poland	15
Australia	1	Greece	3	Portugal	2
Austria	2	Hungary	4	Romania	6
Belgium	3	Iceland	1	Serbia	2
Brazil	2	Indonesia	3	Singapore	1
Bulgaria	3	Ireland	1	Slovakia	2
Croatia	4	Italy	22	Slovenia	3
Cyprus	2	Jamaica	1	Spain	21
Czech Republic	3	Latvia	1	Sweden	2
Denmark	1	Lithuania	1	Switzerland	1
Egypt	1	Luxembourg	1	Tanzania	2
Estonia	1	Netherlands	6	United Kingdom	3
Finland	1	New Zealand	1	<b>Total</b>	<b>163</b>

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Bulgaria	3	Ireland	1	Slovakia	2
Croatia	4	Italy	22	Slovenia	3
Cyprus	2	Jamaica	1	Spain	21
Czech Republic	3	Latvia	1	Sweden	2
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# Target Pesticide List

- 117 pesticides
- 8 new
  - Diniconazole
  - Ethirimol
  - Fluopyram
  - Isocarbophos
  - Mandipropamid
  - Metolachlor
  - Spiromesifen
  - Terbutylazine



## TARGET PESTICIDE LIST

for the EUPT-CF9 2015

(last updated: 22.01.2015)

Eight new pesticides added to the Target Pesticide List from EUPT-CF8 (2014) are marked in bold.

Two pesticides are deleted from Target Pesticide List from EUPT-CF8 (201): captan and chlorothalonil

Pesticide no.	Pesticides	MRRL (mg/kg)
1	Acephate	0.01
2	Azinphos-methyl	0.01
3	Azoxystrobin	0.01
4	Bifenthrin	0.01
5	Bixafen	0.01
6	Boscalid	0.01
7	Carbaryl	0.01
8	Carbendazim	0.01
9	Carbofuran	0.01
10	Carbofuran, 3-hydroxy	0.01
11	Carboxin	0.01
12	Chlорfenvinphos	0.01

Acephate	Endosulfan-sulfate	<b>Isocarbophos</b>	Pirimicarb-desmethyl
Azinphos-methyl	Epoxiconazole	Isoprothiolane	Pirimiphos-methyl
Azoxystrobin	Ethion	Isoproturon	Prochloraz (parent compound only)
Bifenthrin	<b>Ethirimol</b>	Kresoxim-methyl	Procymidone
Bixafen	Fenbuconazole	Lambda-cyhalothrin	Propiconazole
Boscalid	Fenhexamid	Lindane	Prothioconazole-destho
Carbaryl	Fenitrothion	Linuron	Pyraclostrobin
Carbendazim	Fenpropidin	Malaoxon	Pyrimethanil
Carbofuran	Fenpropimorph	Malathion	Quinoxifen
Carbofuran, 3-hydroxy	Fenthion	<b>Mandipropamid</b>	<b>Spiromesifen</b>
Carboxin	Fenthion-oxon	Metconazole	Spiroxamine
Chlorfenvinphos	Fenthion-oxon-sulfone	Methacrifos	Tebuconazole
Chlorpropham (parent compound only)	Fenthion-oxon-sulfoxide	Methomyl	Tebufenozide
Chlorpyrifos	Fenthion-sulfone	<b>Metolachlor</b>	<b>Terbutylazine</b>
Chlorpyrifos-methyl	Fenthion-sulfoxide	Metrafenone	Thiabendazole
Clothianidin	Fenvalerate and Esfenvalerate	Metribuzin	Thiacloprid
Cyfluthrin (sum of isomers)	Fipronil (parent compound only)	DDD- p,p'	Thiamethoxam
Cypermethrin (sum of isomers)	Flonicamid	DDE- p,p'	Thiodicarb
Cyproconazole	Fludioxonil	DDT- o,p'	Thiophanate-methyl
Cyprodinil	<b>Fluopyram</b>	DDT- p,p'	Triadimefon
Deltamethrin-cis	Fluquinconazole	Omethoate	Triadimenol
Demeton-S-methylsulfone	Flusilazole	Oxydemeton-methyl	Triazophos
Diazinon	Flutriafol	Paclobutrazol	Tricyclazole
Dichlorvos	Fluxapyroxad	Parathion	Trifloxystrobin
Difenoconazole	HCH-alpha	Penconazole	Trifluralin
Diflubenzuron	HCH-beta	Pendimethalin	Triticonazole
Dimethoate	Hexaconazole	Permethrin (sum of isomers)	Vinclozolin (parent compound only)
<b>Diniconazole</b>	Imazalil	Phenylphenol-ortho	
Endosulfan-alpha	Imidacloprid	Phos�amidon	
Endosulfan-beta	Iprodione	Pirimicarb	

# Maize – feed variety

- Grown in Denmark in 2014
- Field treated by Aarhus University, Research Department Flakkebjerg

# Pesticides in test material

Pesticide	Application on cereals in the field	Spike in laboratory	Formulation or standard
Bifenazate	x		Floramite
Cypermethrin	x		Cyperb
Epoxiconazole	x		Opera
Foramsulfuron	x		MaisTer
Iodosulfuron	x		MaisTer
Lambda-cyhalothrin	x		Karate
Pendimethalin	x		Stomp
Propiconazole	x		Bumper/Tilt
Pyraclostrobin	x		Opera
Spirotetramat	x		Movento
Tebuconazole	x		Folicur
Thifensulfuron-methyl	x		Harmony

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Pesticide	Application on cereals in the field	Spike in laboratory	Formulation or standard
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Cypermethrin	x		Cyperb
Epoxiconazole	x	x	Opera / Analytical standard
Foramsulfuron	x		MaisTer
Iodosulfuron	x		MaisTer
Lambda-cyhalothrin	x	x	Karate
Pendimethalin	x	x	Stomp / Analytical standard
Propiconazole	x	x	Bumper/Tilt
Pyraclostrobin	x		Opera
Spirotetramat	x		Movento
Tebuconazole	x		Folicur
Thifensulfuron-methyl	x		Harmony

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Pendimethalin	x	x	Stomp / Analytical standard
Propiconazole	x	x	Bumper/Tilt
Pyraclostrobin	x		Opera
Spirotetramat	x		Movento
Tebuconazole	x		Folicur
Thifensulfuron-methyl	x		Harmony
Azoxystrobin		x	Amistar
Carbendazim		x	Bavistin FL
Chlorfenvinphos		x	Analytical standard
Chlorpyrifos-methyl		x	Reldan
Clothianidin		x	Analytical standard
Fluopyram		x	Analytical standard
Isocarbophos		x	Analytical standard
Lindane		x	Analytical standard
Metolachlor		x	Analytical standard
Metribuzin		x	Analytical standard
Spiromesifen		x	Analytical standard
Terbutylazine		x	Analytical standard
Thiacloprid		x	Analytical standard
Triticonazole		x	Analytical standard

# Spike procedure

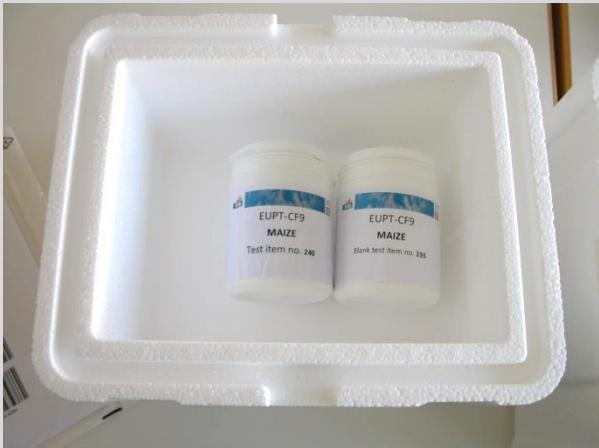






# Sample shipment

- Samples were distributed on Monday 20 April 2015 (13 April to Third Countries)
- Most samples were delivered to EU laboratories on 21 or 22 April 2015



# Homogeniety test

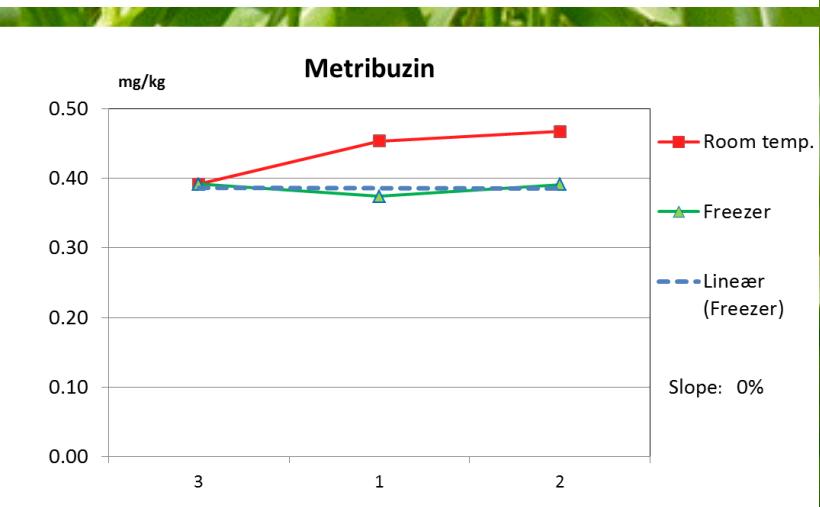
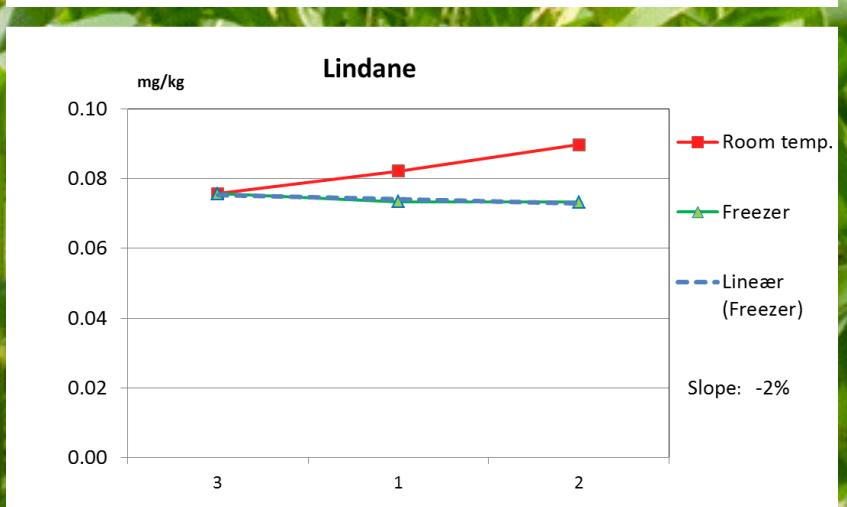
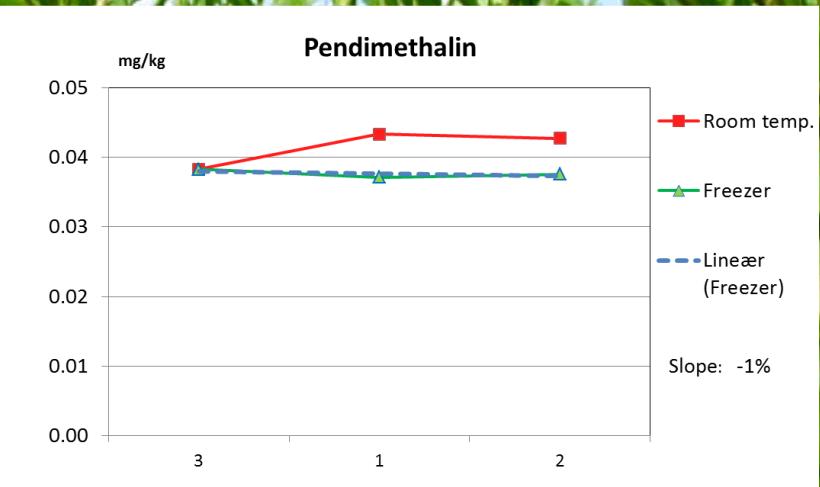
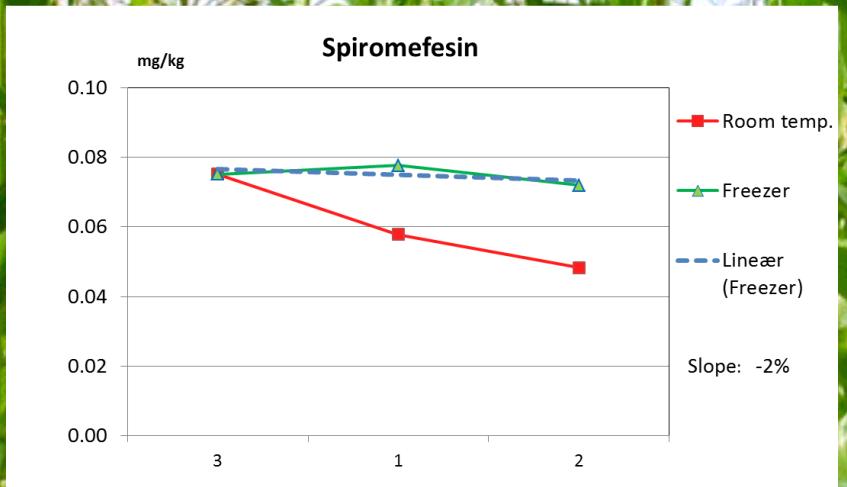
	Mean, mg/kg	$S_s^2$	c	$S_s^2 < c$
Azoxystrobin	0.050	0.00001	0.0000	Pass
Carbendazim	0.399	0.00097	0.0018	Pass
Chlorfenvinphos	0.041	0.00000	0.0001	Pass
Chlorpyrifos-methyl	0.043	0.00000	0.0000	Pass
Clothianidin	0.489	0.00116	0.0036	Pass
Epoxiconazole	0.050	0.00001	0.0000	Pass
Fluopyram	0.089	0.00005	0.0001	Pass
Isocarbofos	0.068	0.00001	0.0001	Pass
Lambda-cyhalothrin	0.100	0.00001	0.0003	Pass
Lindane	0.059	0	0.0002	Pass
Metolachlor	0.071	0.00000	0.0002	Pass
Test sample no.	0.148	0.00013	0.0003	Pass
Pendimethalin	0.043	0.00001	0.0000	Pass
Propiconazole	0.148	0.00013	0.0003	Pass
Spiromesifen	0.073	0.00000	0.0002	Pass
Terbuthylazine	0.079	0.00002	0.0001	Pass
Thiacloprid	0.086	0.00003	0.0001	Pass
Triticonazole	0.081	0.00003	0.0001	Pass

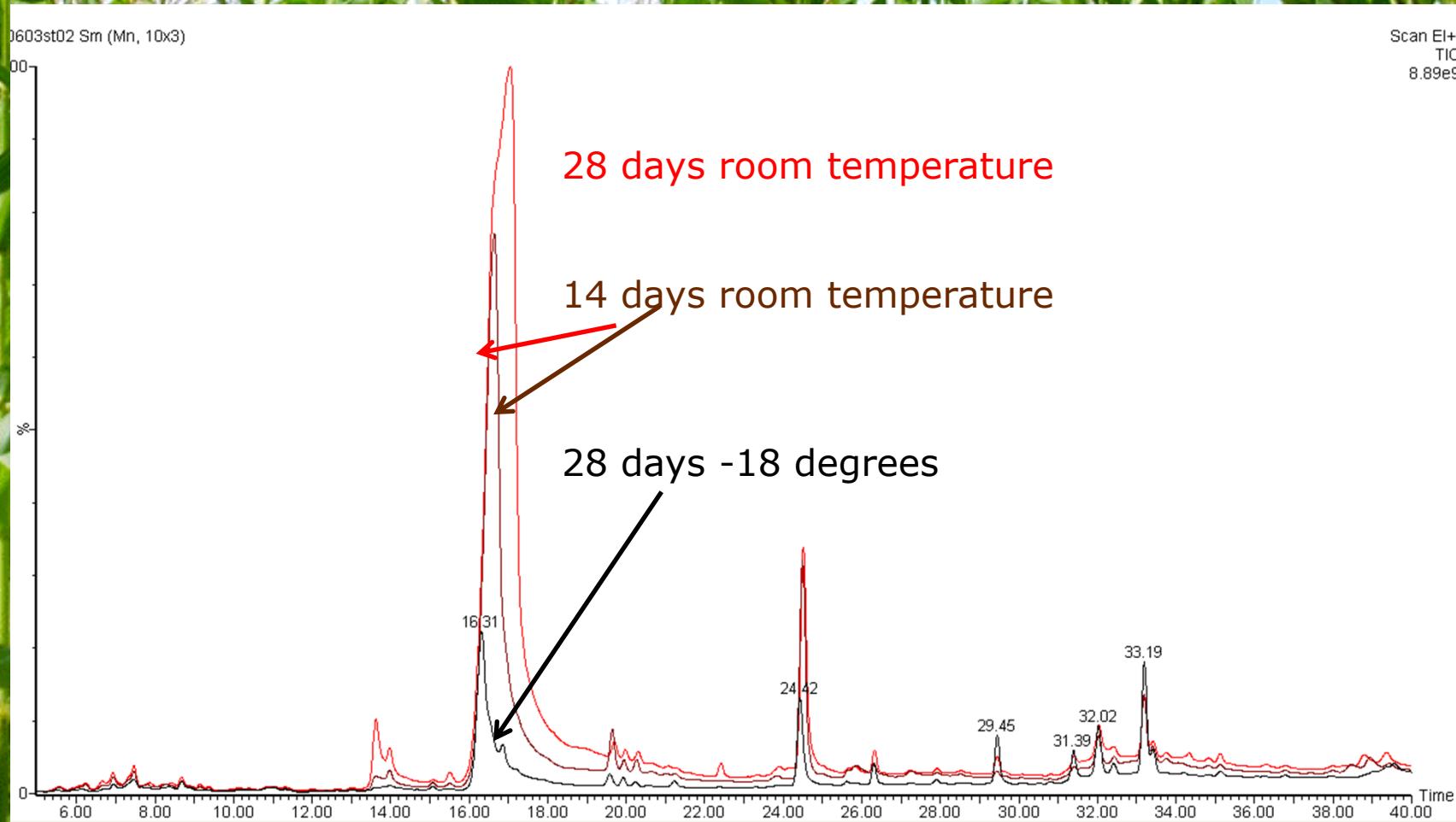
# Stability test

		Storage at -18 degrees			Storage at room temperature		
	Mean, mg/kg	x1 - yi	0.3×σ	x1 - yi   ≤ 0.3×σ		0.3×σ	x1 - yi   ≤ 0.3×σ
Azoxystrobin	0.049	0.003	0.004	Pass	0.003	0.004	Pass
Carbendazime	0.435	0.012	0.034	Pass	0.050	0.034	Fail
Chlorfenvinphos	0.050	0.002	0.004	Pass	0.003	0.004	Pass
Chlorpyrifos-methyl	0.051	0.002	0.004	Pass	0.003	0.004	Pass
Clothianidin	0.534	0.022	0.032	Pass	0.020	0.032	Pass
Epoxiconazole	0.054	0.001	0.004	Pass	0.004	0.004	Pass
Fluopyram	0.089	0.006	0.007	Pass	0.008	0.007	Fail
Isocarbofos	0.079	0.005	0.006	Pass	0.005	0.006	Pass
Lambda-cyhalothrin	0.095	0.003	0.007	Pass	0.006	0.007	Pass
Lindane	0.074	0.002	0.005	Pass	0.014	0.005	Fail
Metolachlor	0.086	0.004	0.006	Pass	0.006	0.006	Pass
Metribuzin	0.386	0.001	0.027	Pass	0.075	0.027	Fail
Pendimethalin	0.038	0.001	0.003	Pass	0.004	0.003	Fail
Propiconazole	0.147	0.003	0.011	Pass	0.002	0.011	Pass
Spiromefesin	0.075	0.003	0.005	Pass	0.027	0.005	Fail
Terbutylazine	0.088	0.004	0.006	Pass	0.004	0.006	Pass
Thiacloprid	0.094	0.005	0.007	Pass	0.003	0.007	Pass
Triticonazole	0.079	0.002	0.006	Pass	0.009	0.006	Fail

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Fluopyram	0.089	0.006	0.007	Pass	0.008	0.007	Fail
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Lambda-cyhalothrin	0.095	0.003	0.007	Pass	0.006	0.007	Pass
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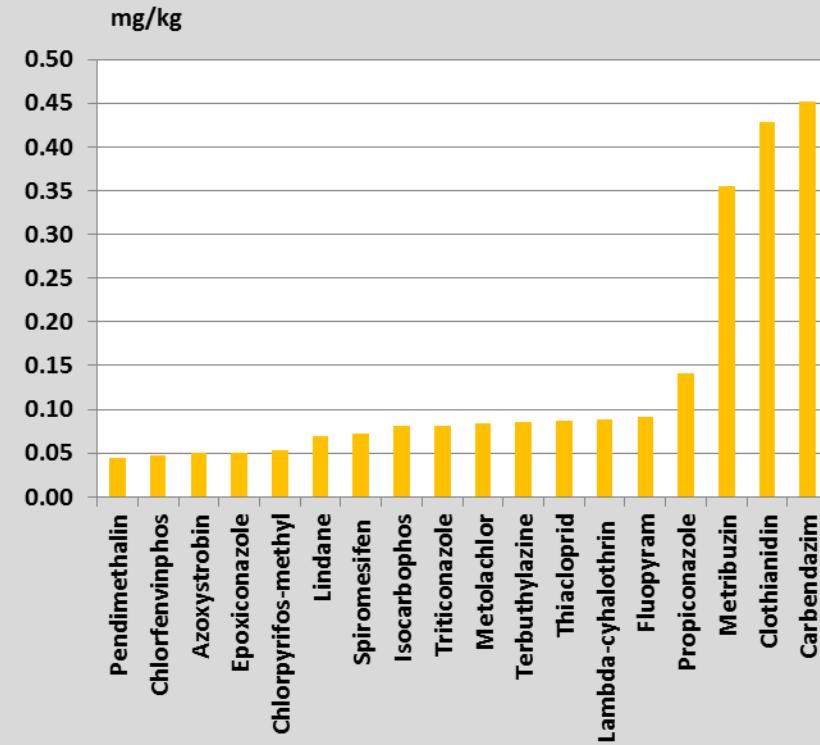
# Calculation of assigned values and uncertainty of assigned values

- Algorithm A mean of the results from EU laboratories (and EFTA)
  - Only result from participant that
    - reported to add water to the samples before extraction or
    - used mixture of solvent and water or
    - used ASE
- Outliers
  - Only obvious incorrect results
- Uncertainty of the assigned values
  - $=1.25 * ( s^* / \sqrt{N} )$
  - $s^*$  is robust standard deviation (Alg A standard deviation)
  - N is the number of participants

# Assigned values

PESTICIDES	Assigned values only water addition, mg/kg
Azoxystrobin	0.050
Carbendazim	0.452
Chlorfenvinphos	0.048
Chlorpyrifos-methyl	0.054
Clothianidin	0.429
Epoxiconazole	0.050
Fluopyram	0.092
Isocarbophos	0.081
Lambda-cyhalothrin	0.088
Lindane	0.070
Metolachlor	0.085
Metribuzin	0.355
Pendimethalin	0.044
Propiconazole	0.141
Spiromesifen	0.072
Terbutylazine	0.086
Thiacloprid	0.087
Triticonazole	0.082

## Assigned values



# Reported results and false negatives

Pesticide	No. of reported results	No. of NA	False negatives	% results
Azoxystrobin	127	15	4	89
Carbendazim	108	34	0	76
Chlорfenvinphos	124	18	2	87
Chlorpyrifos-methyl	130	12	2	92
Clothianidin	102	40	1	72
Epoxiconazole	117	25	3	82
Fluopyram	87	55	1	61
Isocarbophos	88	54	7	62
Lambda-cyhalothrin	129	13	5	91
Lindane	132	10	3	93
Metolachlor	96	46	4	68
Metribuzin	102	40	2	72
Pendimethalin	126	16	0	89
Propiconazole	126	16	2	89
Spiromesifen	88	54	3	62
Terbuthylazine	109	33	0	77
Thiacloprid	107	35	0	75
Triticonazole	107	35	4	75

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# False positive results

Lab code	Pesticide	Concentration mg/kg	Determination technique	RL, mg/kg
214	Bifenthrin	0.052	GC-MS/MS (QQQ)	
226	Thiamethoxam	0.54	LC-MS/MS QQQ	0.02
247	HCH-alpha	0.01	GC-MS/MS (QQQ)	0.01
256	Flonicamid	0.08	LC-MS/MS QQQ	0.01
283	HCH-beta	0.0657	GC-MS/MS (QQQ)	0.01
334	HCH-alpha	0.0457	GC-MSD	0.01
374	HCH-alpha	0.059	GC-MS/MS (QQQ)	0.01
374	HCH-beta	0.081	GC-MS/MS (QQQ)	0.01
406	HCH-beta	0.036	GC-MSD	0.01

< 0.01 mg/kg

Lab code	Pesticide	Concentration mg/kg	Determination technique	RL, mg/kg
382	Pirimiphos-methyl	0.0046	GC-NPD	0.003

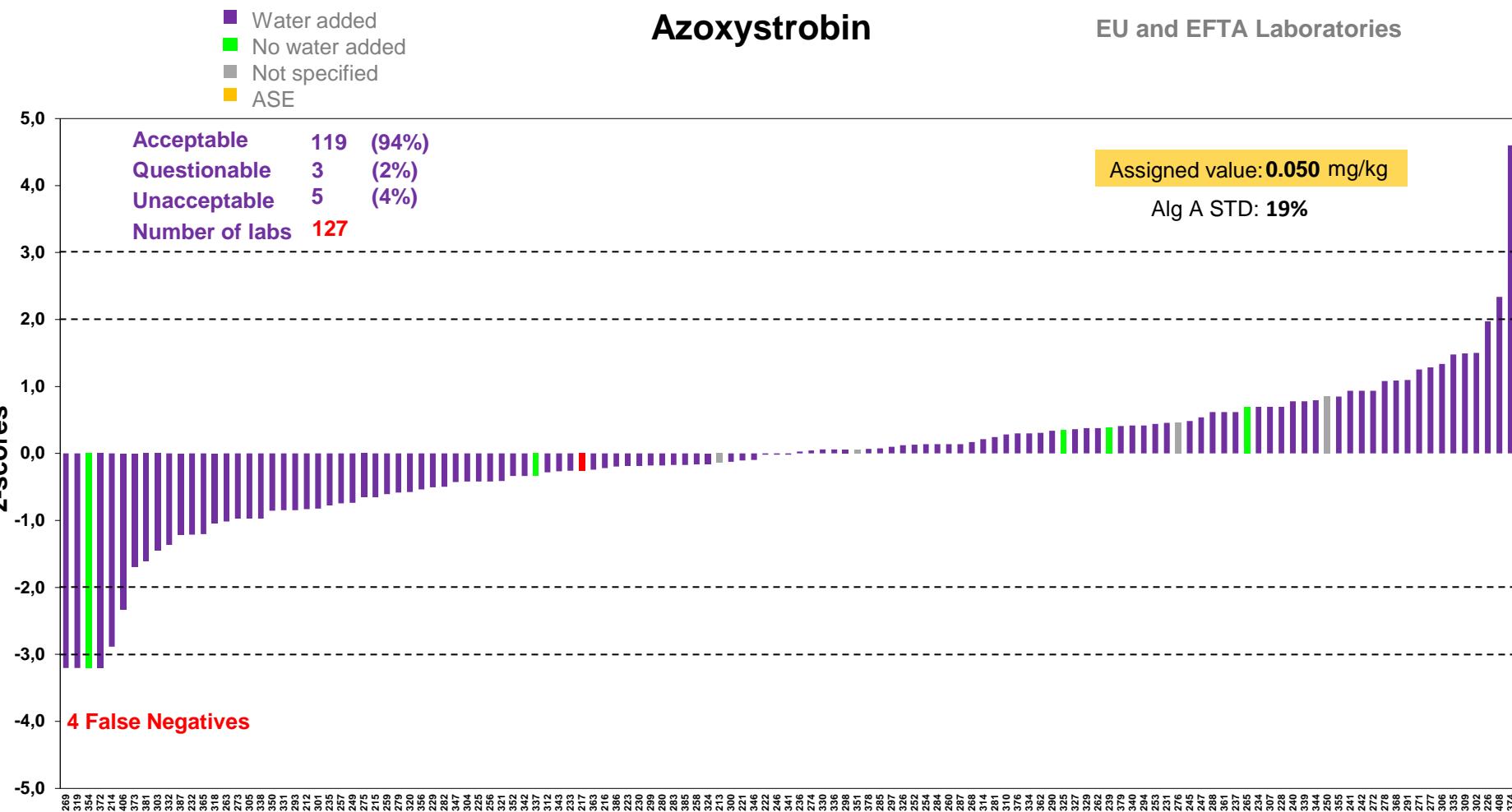
## Calculations of z-scores

- For each laboratory/pesticide combination :

$$z = (x - X) / \sigma$$

- $x$  is the result reported by the participant or the specific reporting limit of the lab for those labs not having detected the pesticide present in the test material
- $X$  is the assigned value, Alg A mean without outliers
- $\sigma$  is the target standard deviation obtained by multiplying the median by the FFP RSD of 25%

- $|z| \leq 2$                       Acceptable
- $2 < |z| < 3$                       Questionable
- $|z| \geq 3$                       Unacceptable



- █ Water added
- █ No water added
- █ Not specified
- █ ASE

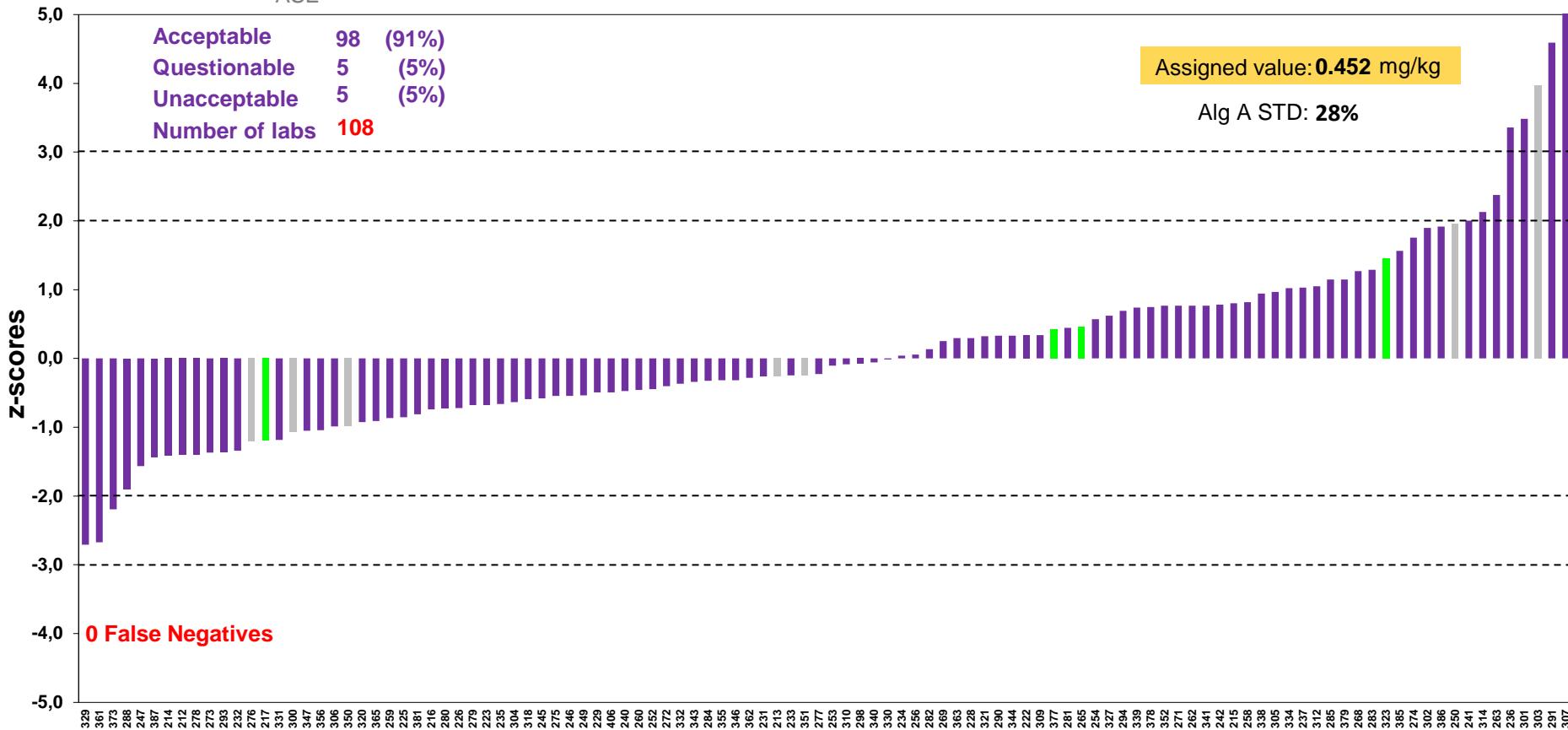
## Carbendazim

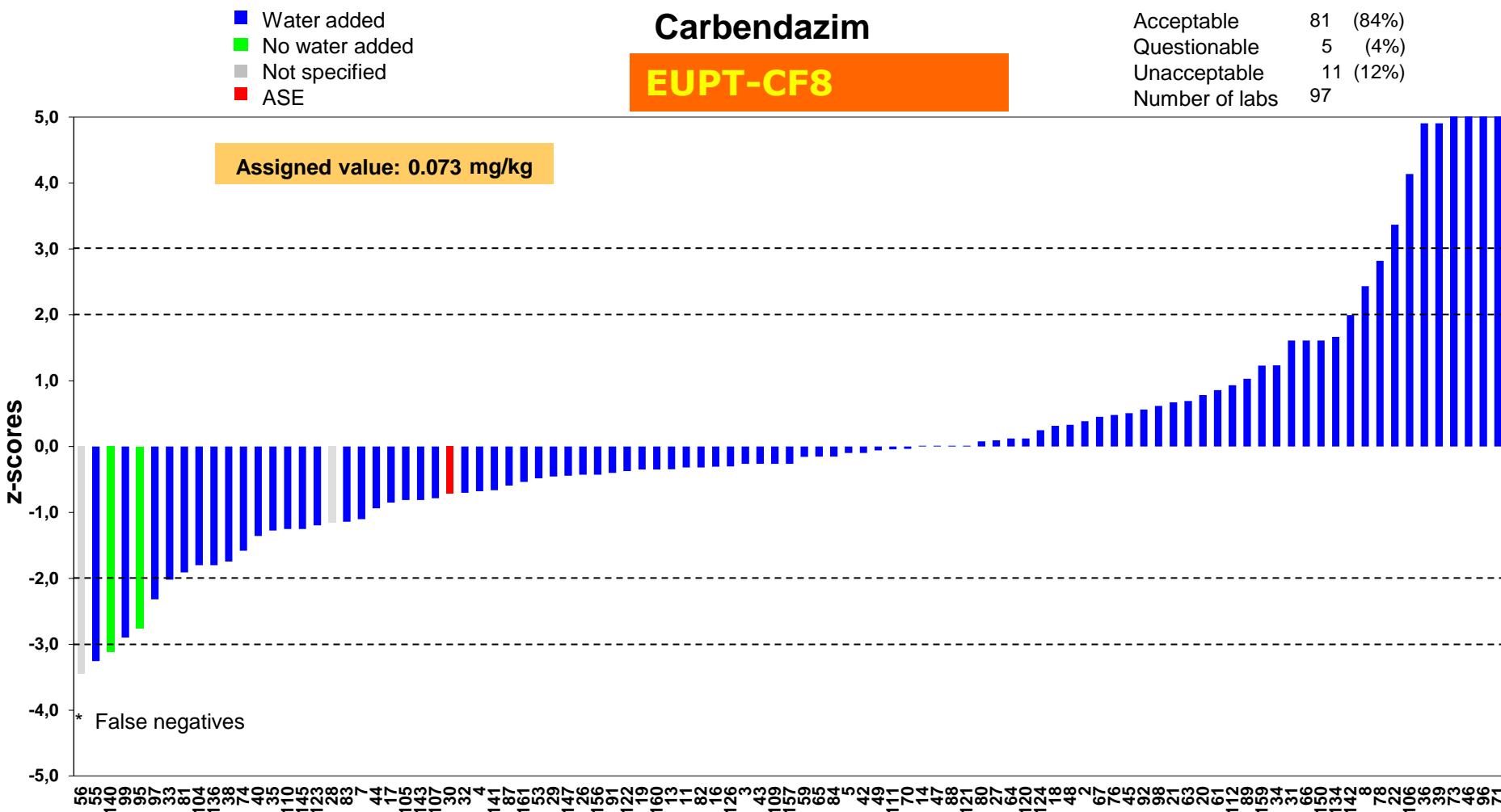
EU and EFTA Laboratories

**Acceptable** 98 (91%)  
**Questionable** 5 (5%)  
**Unacceptable** 5 (5%)  
**Number of labs** 108

Assigned value: **0.452 mg/kg**

Alg A STD: **28%**





- Water added
- No water added
- Not specified
- ASE

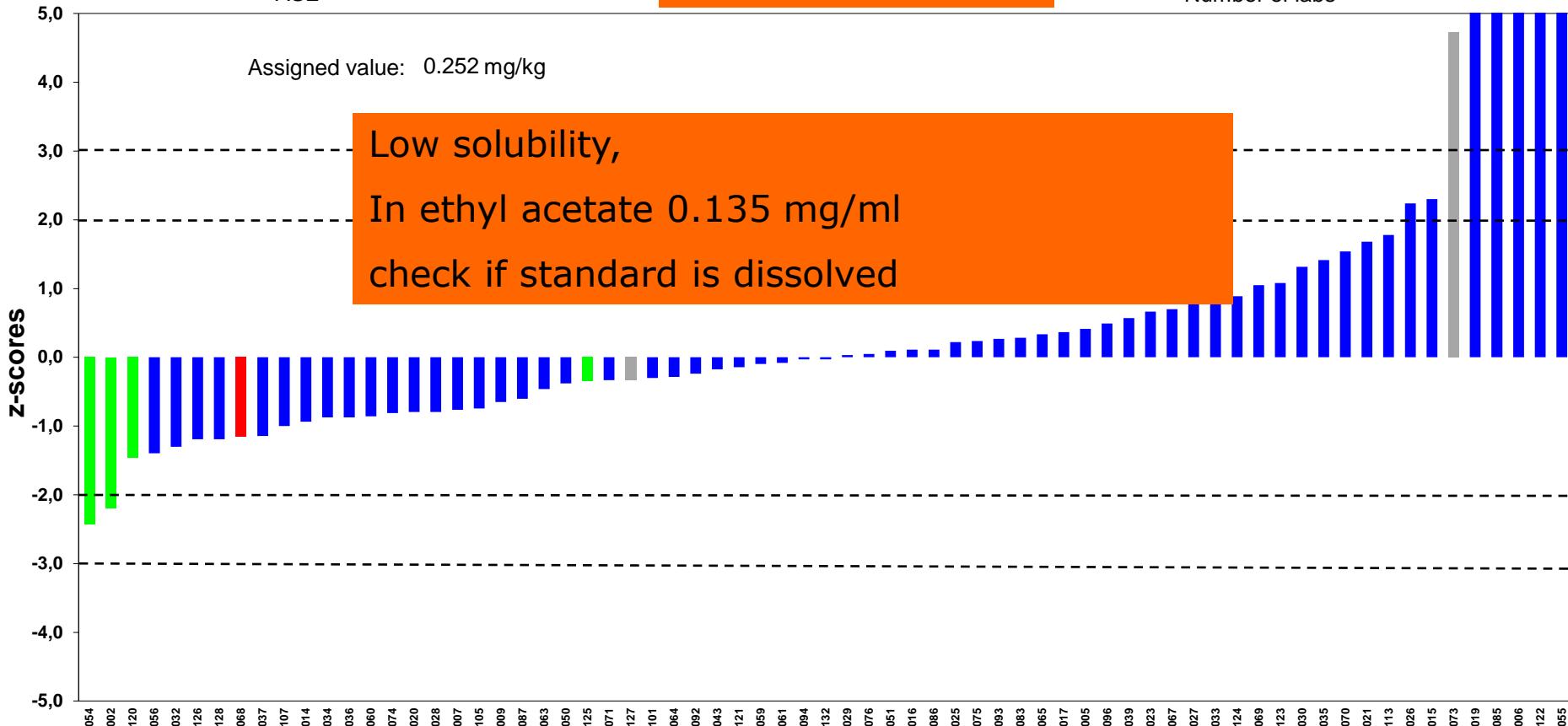
## Carbendazim

### EUPT-CF7

Acceptable	59
Questionable	4
Unacceptable	6
Number of labs	69

Assigned value: 0.252 mg/kg

Low solubility,  
In ethyl acetate 0.135 mg/ml  
check if standard is dissolved



- █ Water added
- █ No water added
- █ Not specified
- █ ASE

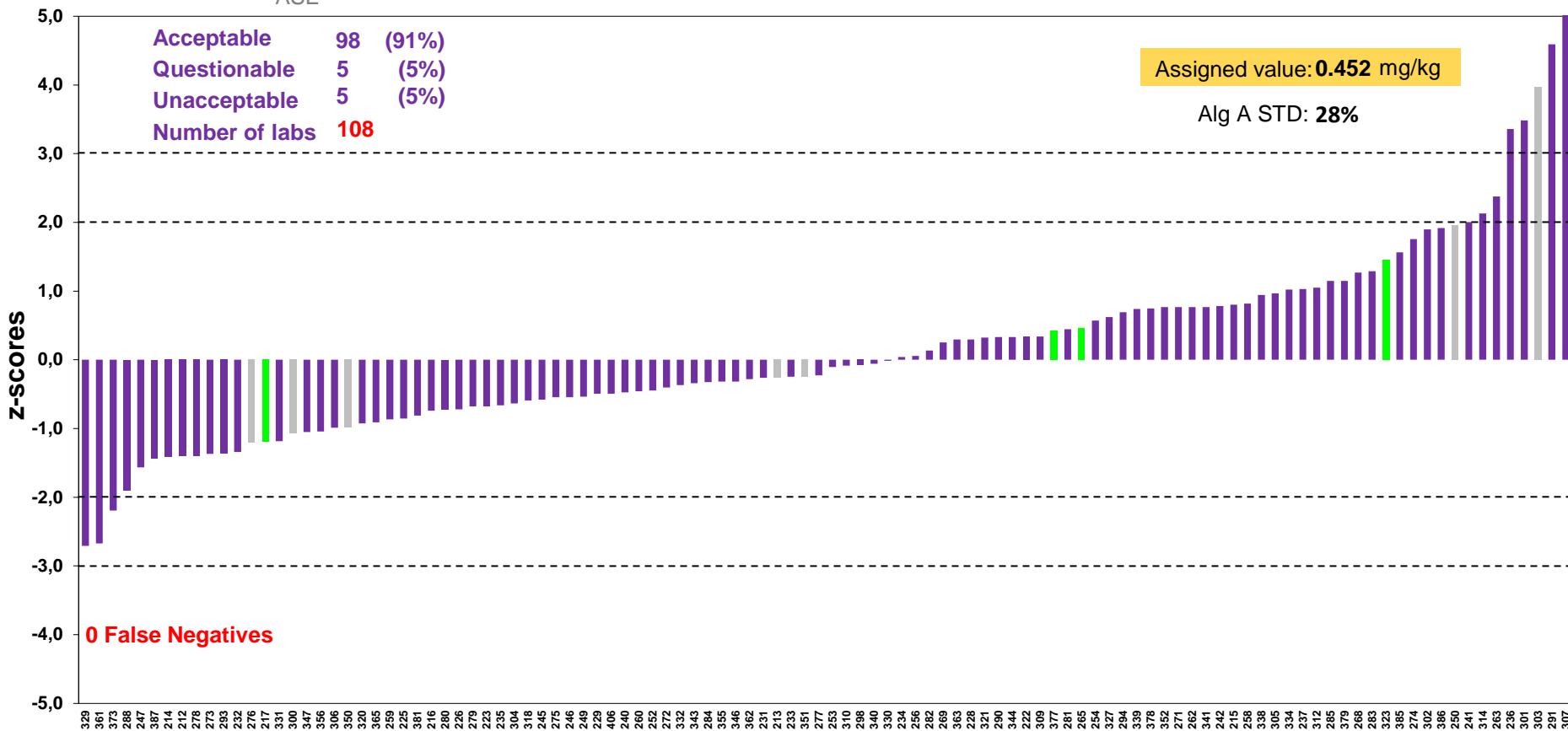
## Carbendazim

EU and EFTA Laboratories

**Acceptable** 98 (91%)  
**Questionable** 5 (5%)  
**Unacceptable** 5 (5%)  
**Number of labs** 108

Assigned value: **0.452 mg/kg**

Alg A STD: **28%**



- Water added
- No water added
- Not specified
- ASE

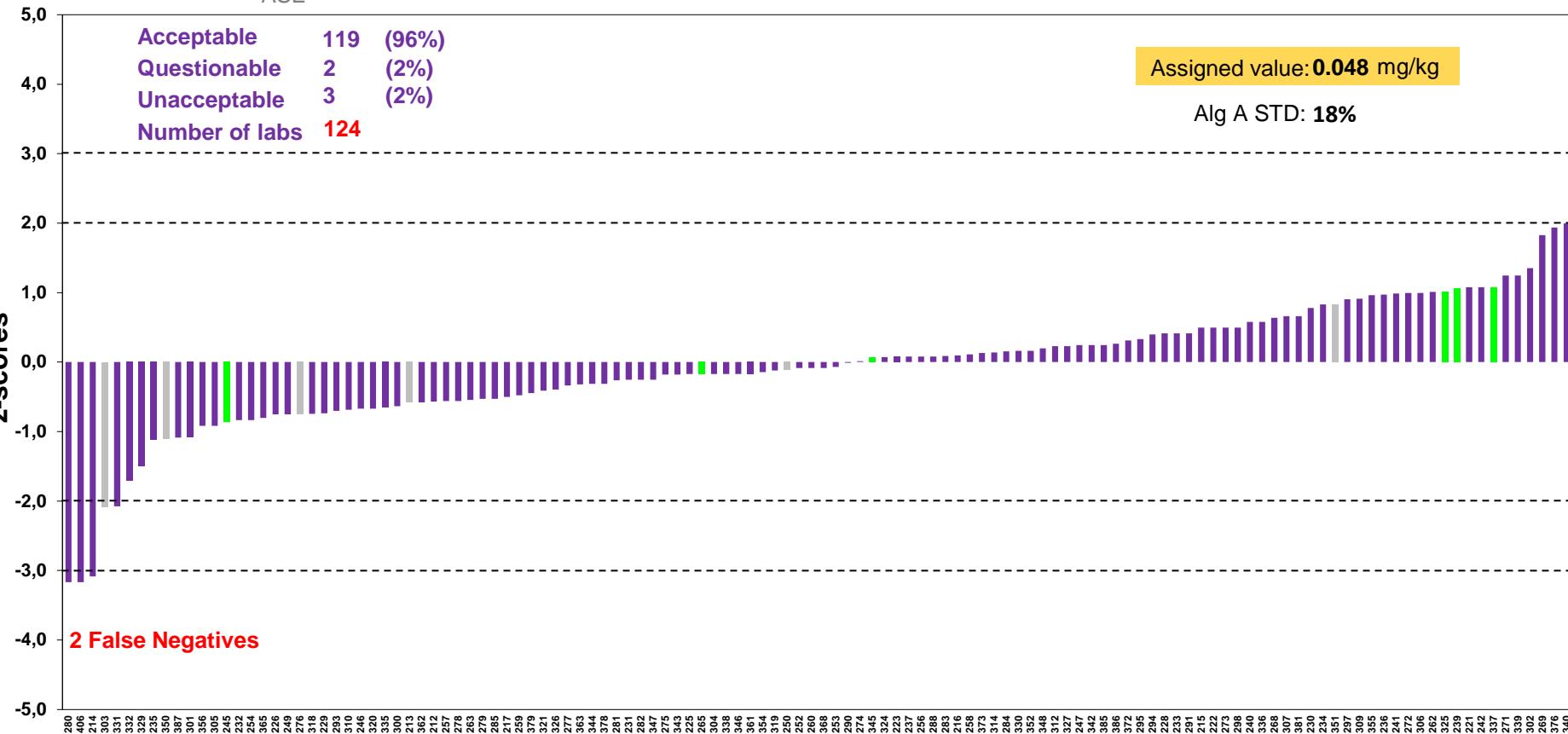
## Chlorfenvinphos

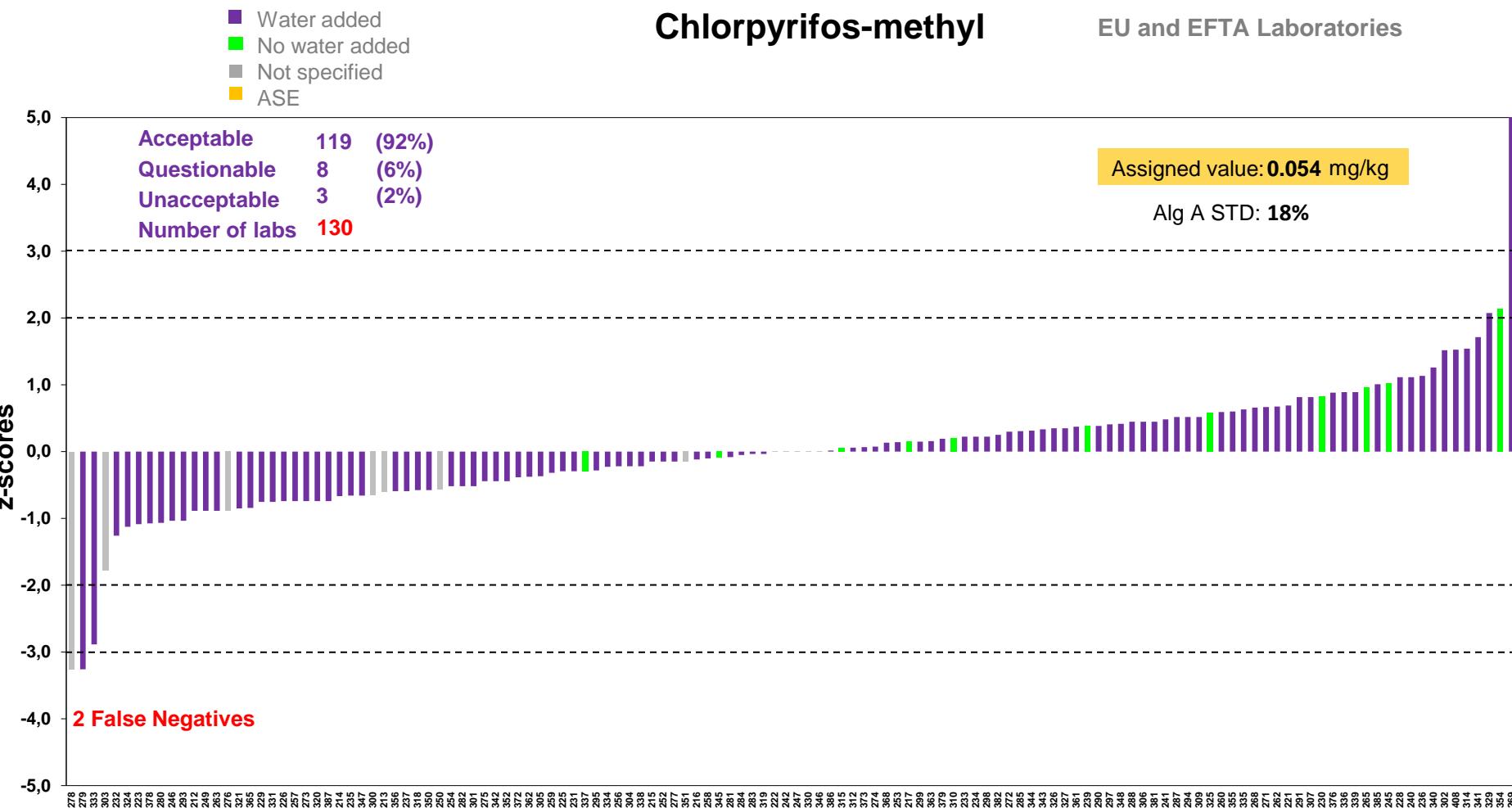
EU and EFTA Laboratories

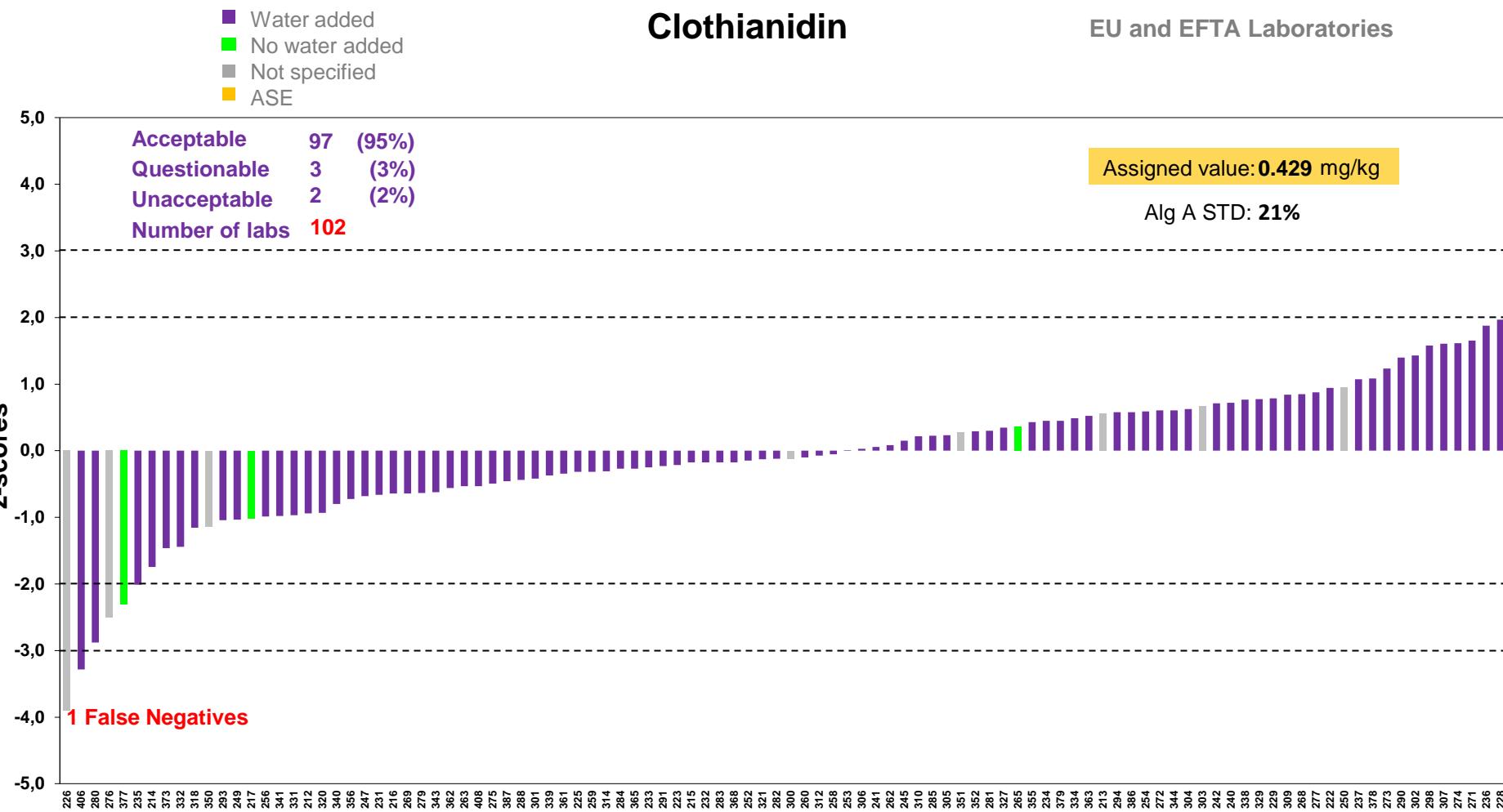
**Acceptable** 119 (96%)  
**Questionable** 2 (2%)  
**Unacceptable** 3 (2%)  
**Number of labs** 124

Assigned value: **0.048 mg/kg**

Alg A STD: **18%**







- Water added
- No water added
- Not specified
- ASE

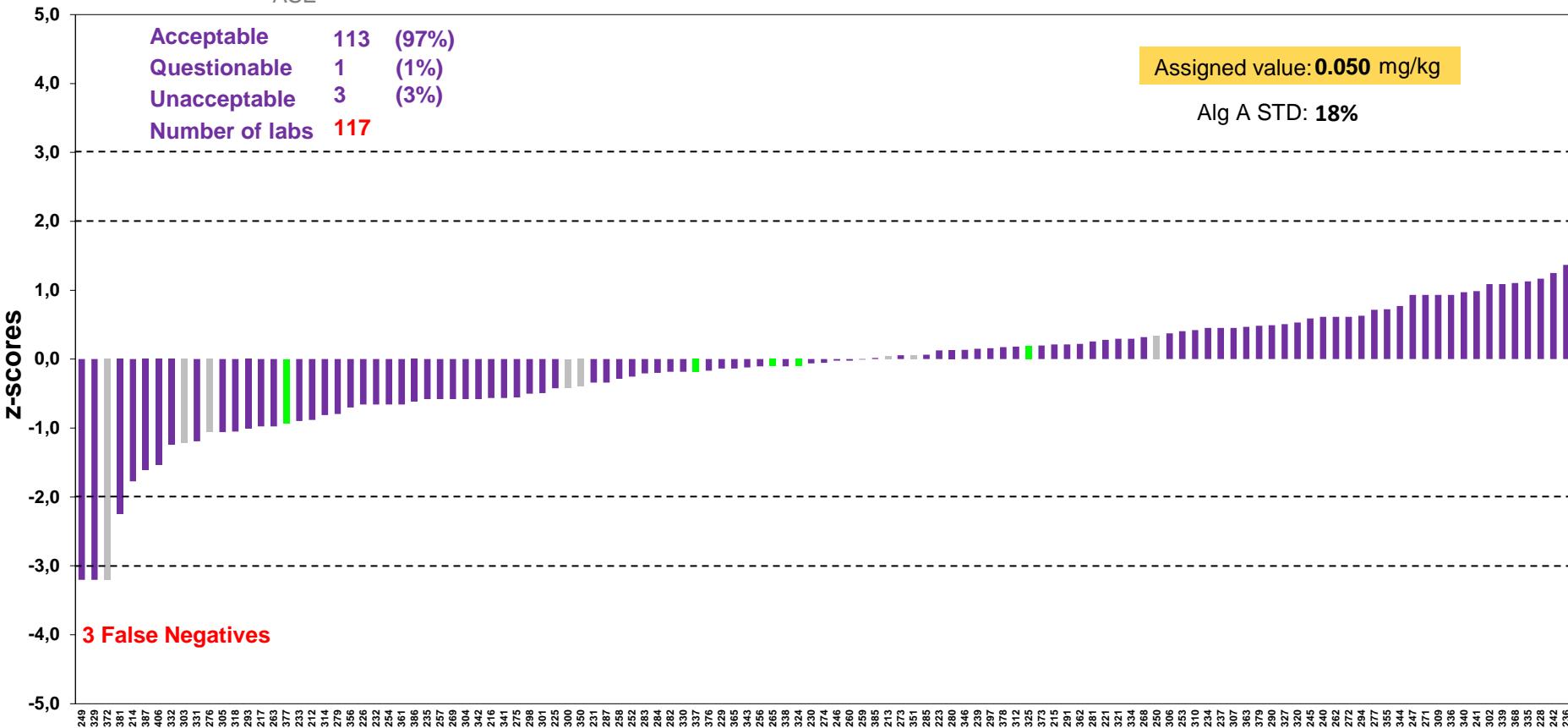
## Epoxiconazole

EU and EFTA Laboratories

**Acceptable** 113 (97%)  
**Questionable** 1 (1%)  
**Unacceptable** 3 (3%)  
**Number of labs** 117

Assigned value: **0.050 mg/kg**

Alg A STD: **18%**



- Water added
- No water added
- Not specified
- ASE

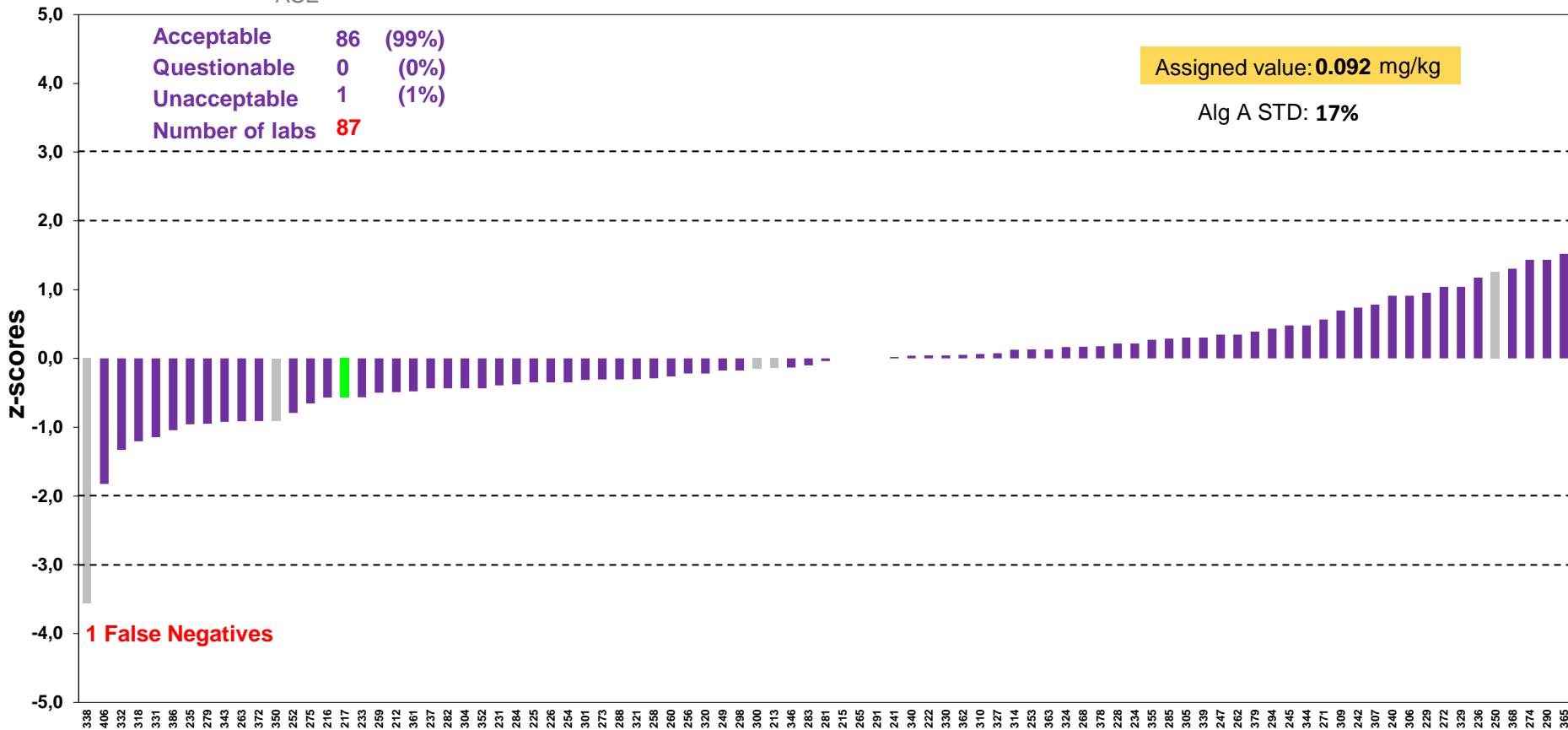
## Fluopyram

EU and EFTA Laboratories

Acceptable 86 (99%)  
Questionable 0 (0%)  
Unacceptable 1 (1%)  
Number of labs 87

Assigned value: 0.092 mg/kg

Alg A STD: 17%



- Water added
- No water added
- Not specified
- ASE

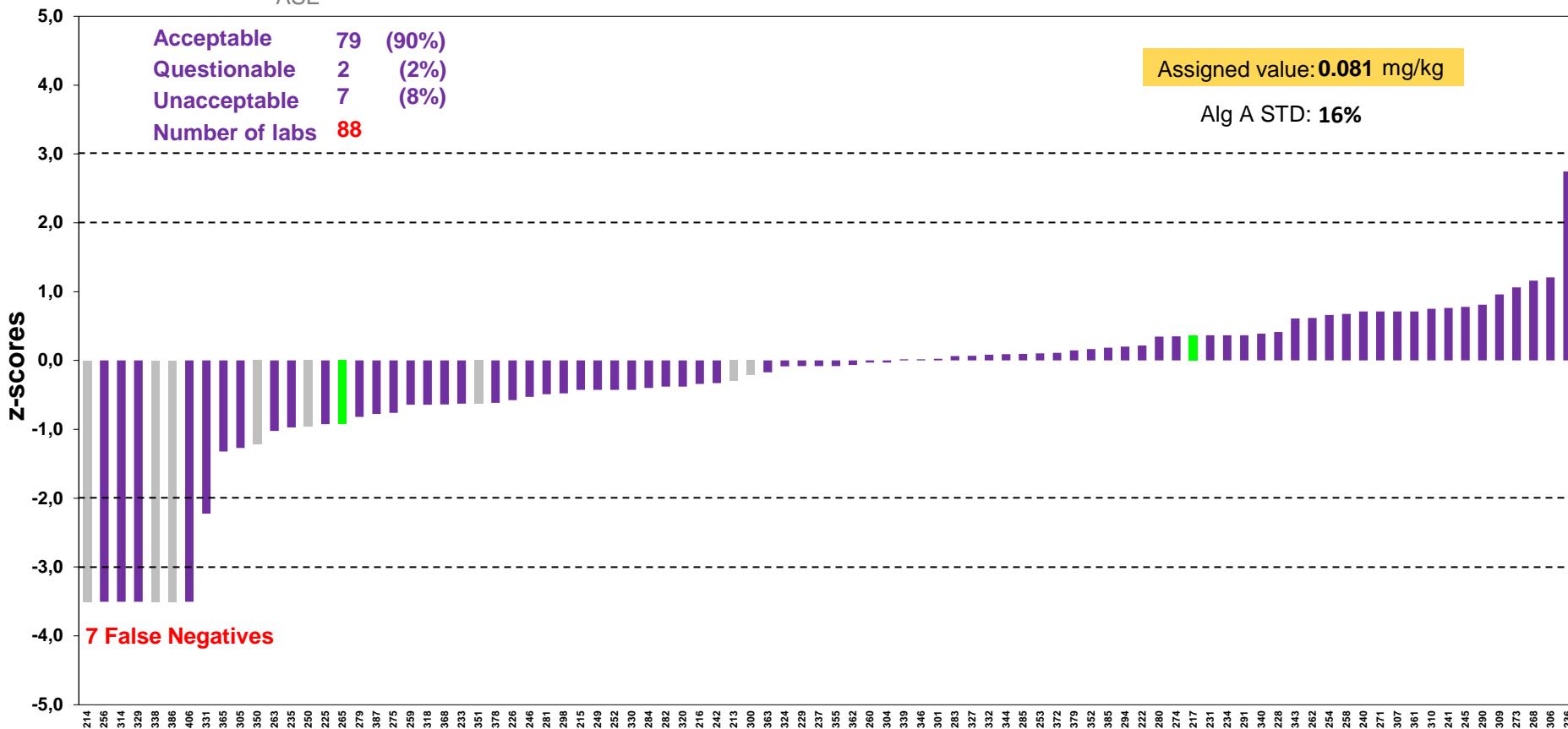
## Isocarbophos

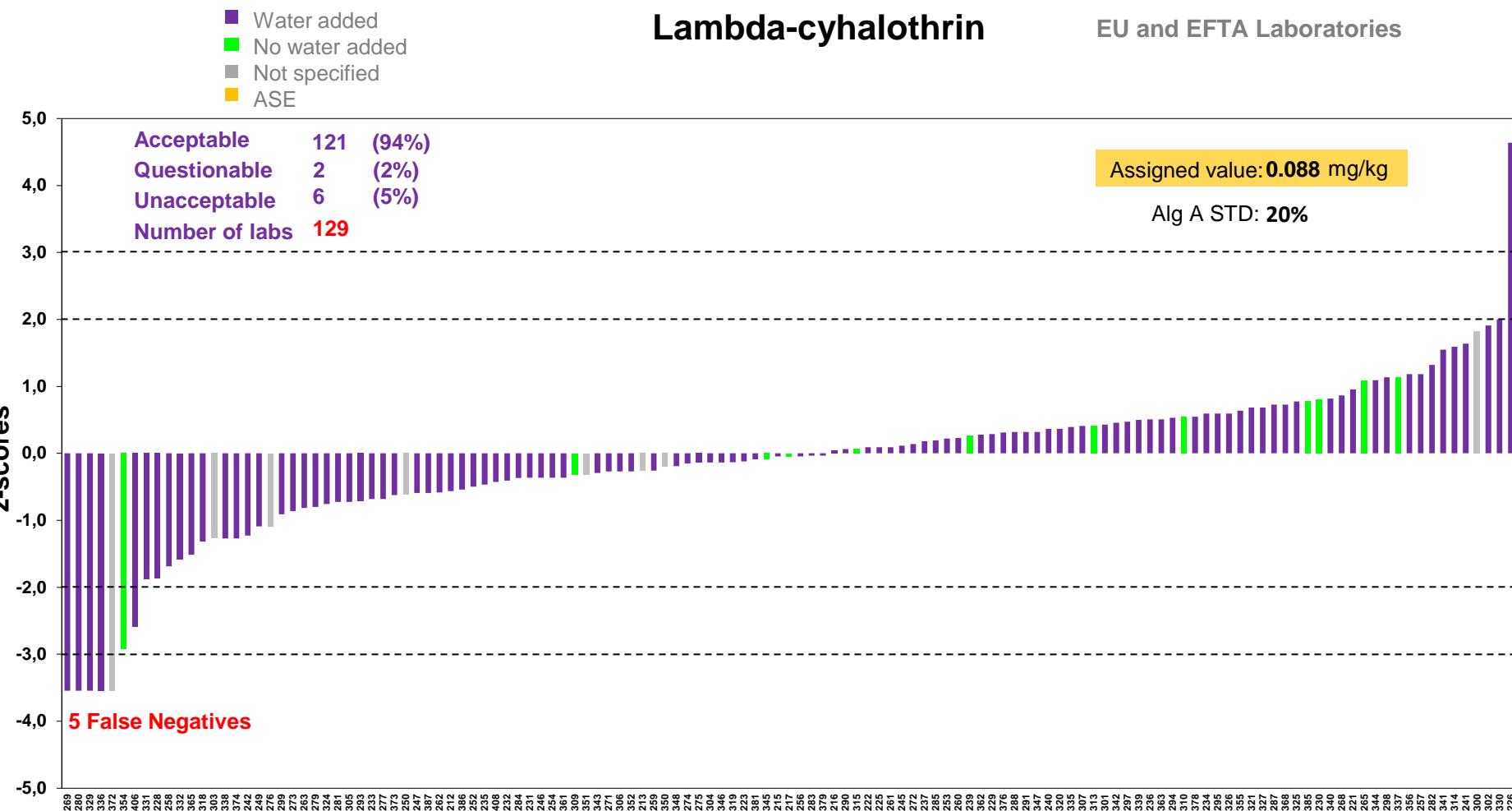
EU and EFTA Laboratories

<b>Acceptable</b>	79	(90%)
<b>Questionable</b>	2	(2%)
<b>Unacceptable</b>	7	(8%)
<b>Number of labs</b>	<b>88</b>	

Assigned value: **0.081 mg/kg**

Alg A STD: **16%**





- Water added
- No water added
- Not specified
- ASE

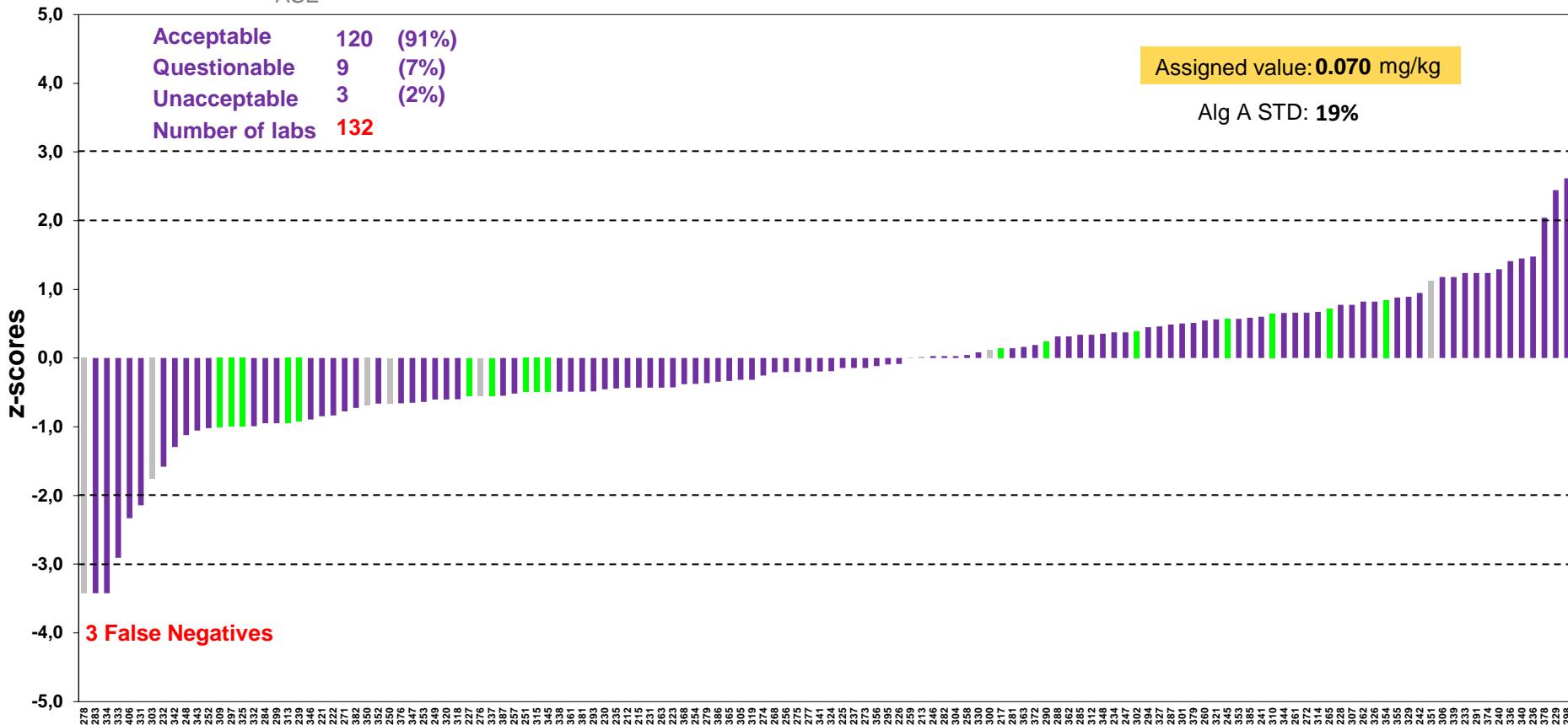
## Lindane

EU and EFTA Laboratories

**Acceptable** 120 (91%)  
**Questionable** 9 (7%)  
**Unacceptable** 3 (2%)  
**Number of labs** 132

Assigned value: **0.070 mg/kg**

Alg A STD: **19%**



- Water added
- No water added
- Not specified
- ASE

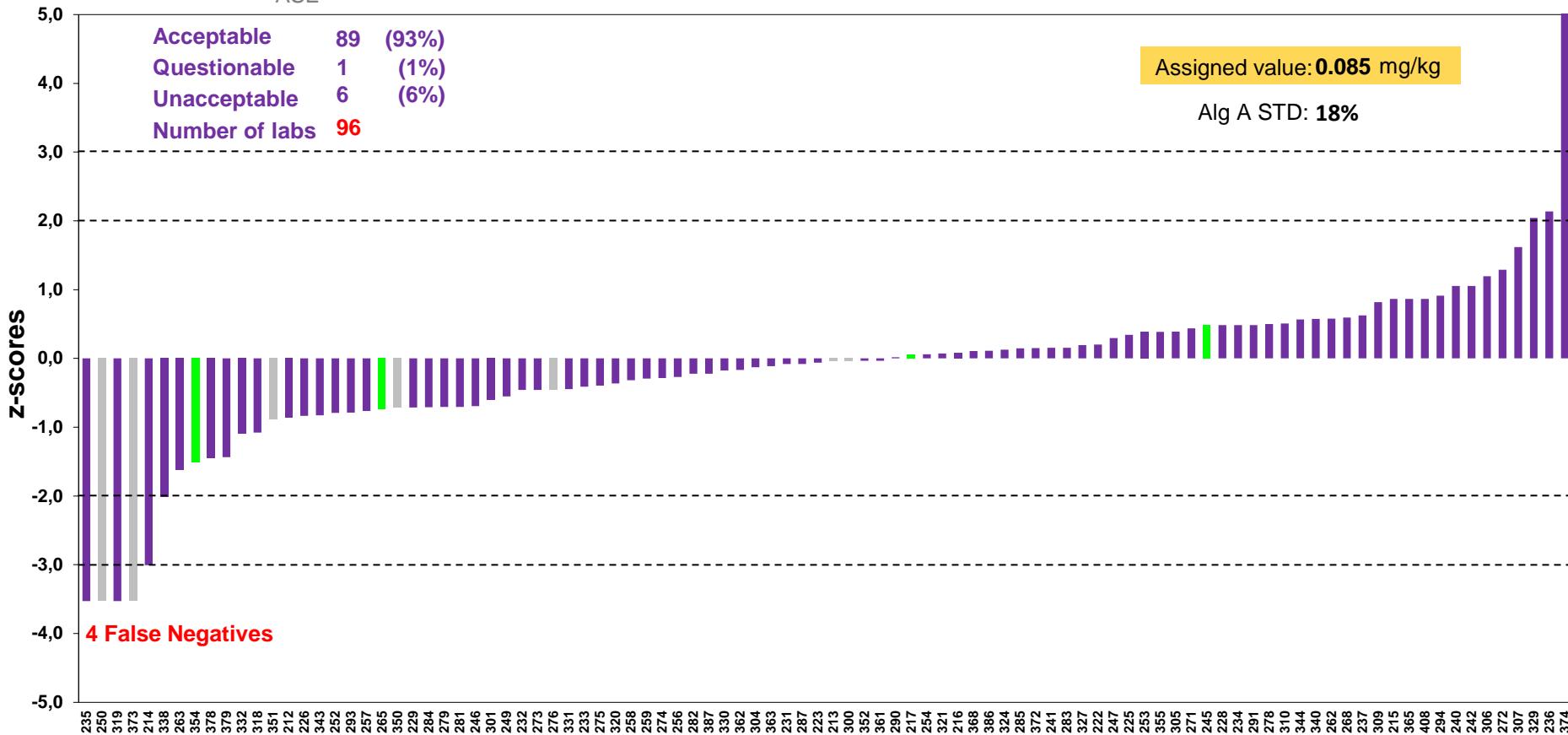
## Metolachlor

EU and EFTA Laboratories

**Acceptable** 89 (93%)  
**Questionable** 1 (1%)  
**Unacceptable** 6 (6%)  
**Number of labs** 96

Assigned value: **0.085 mg/kg**

Alg A STD: **18%**



- █ Water added
- █ No water added
- █ Not specified
- █ ASE

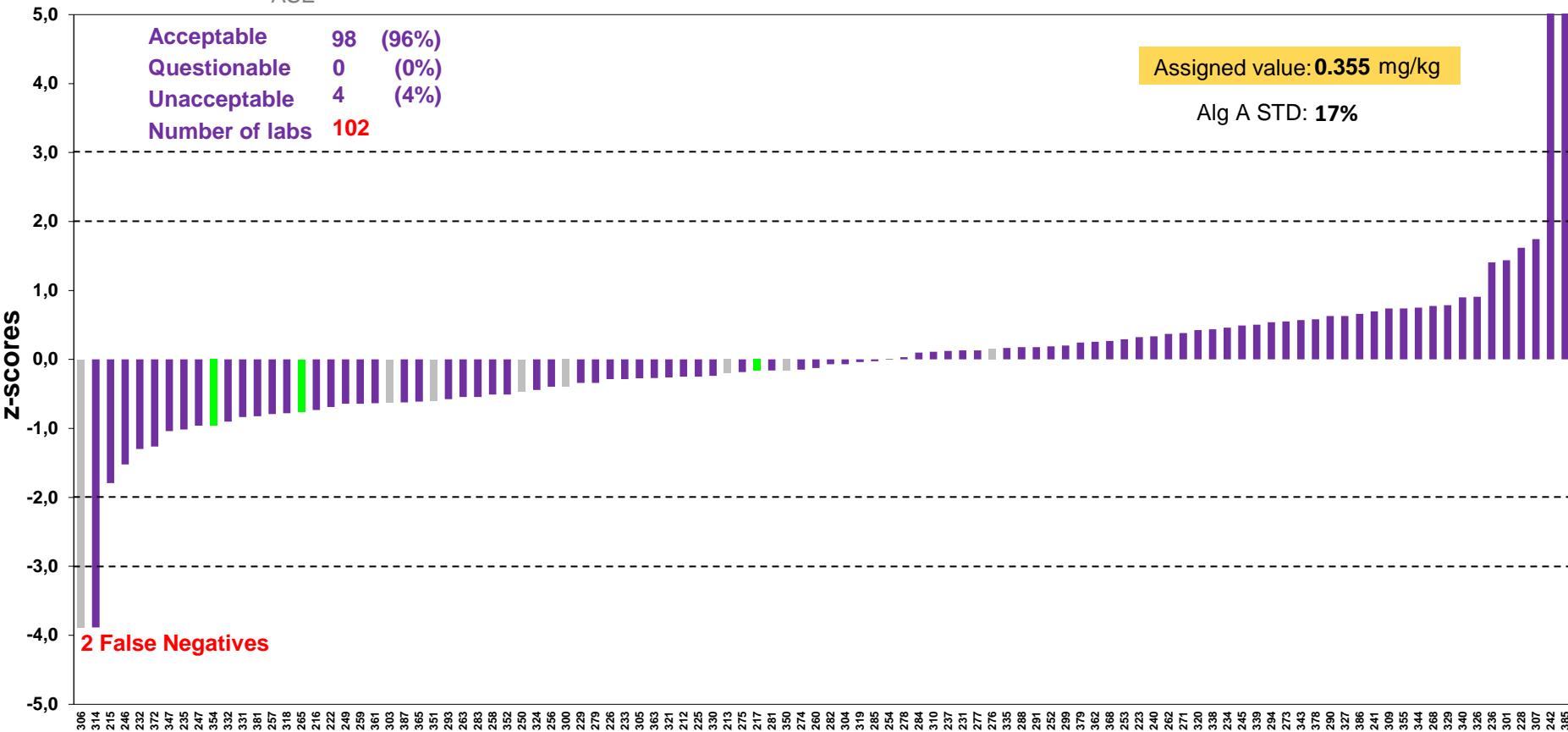
## Metribuzin

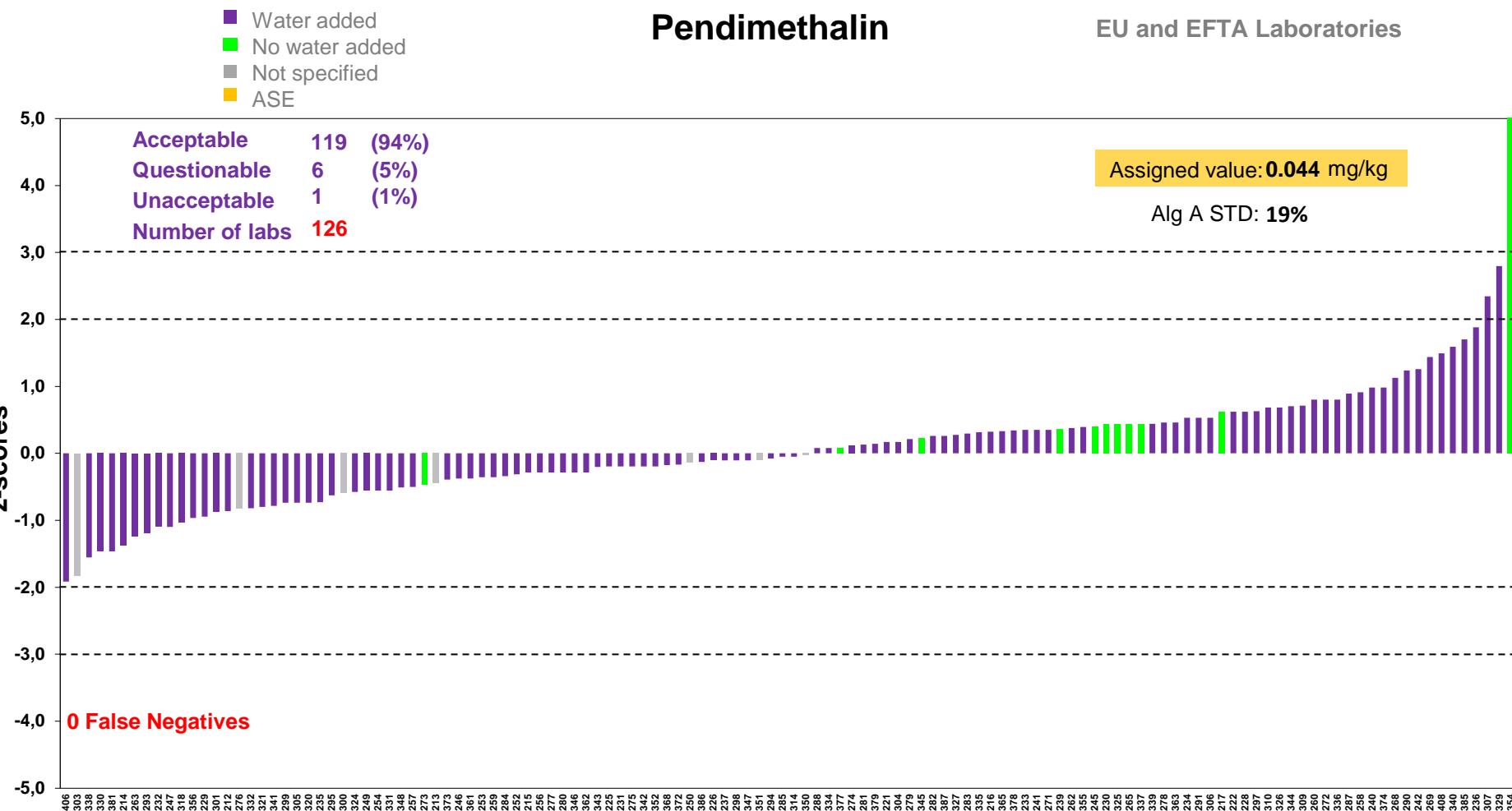
EU and EFTA Laboratories

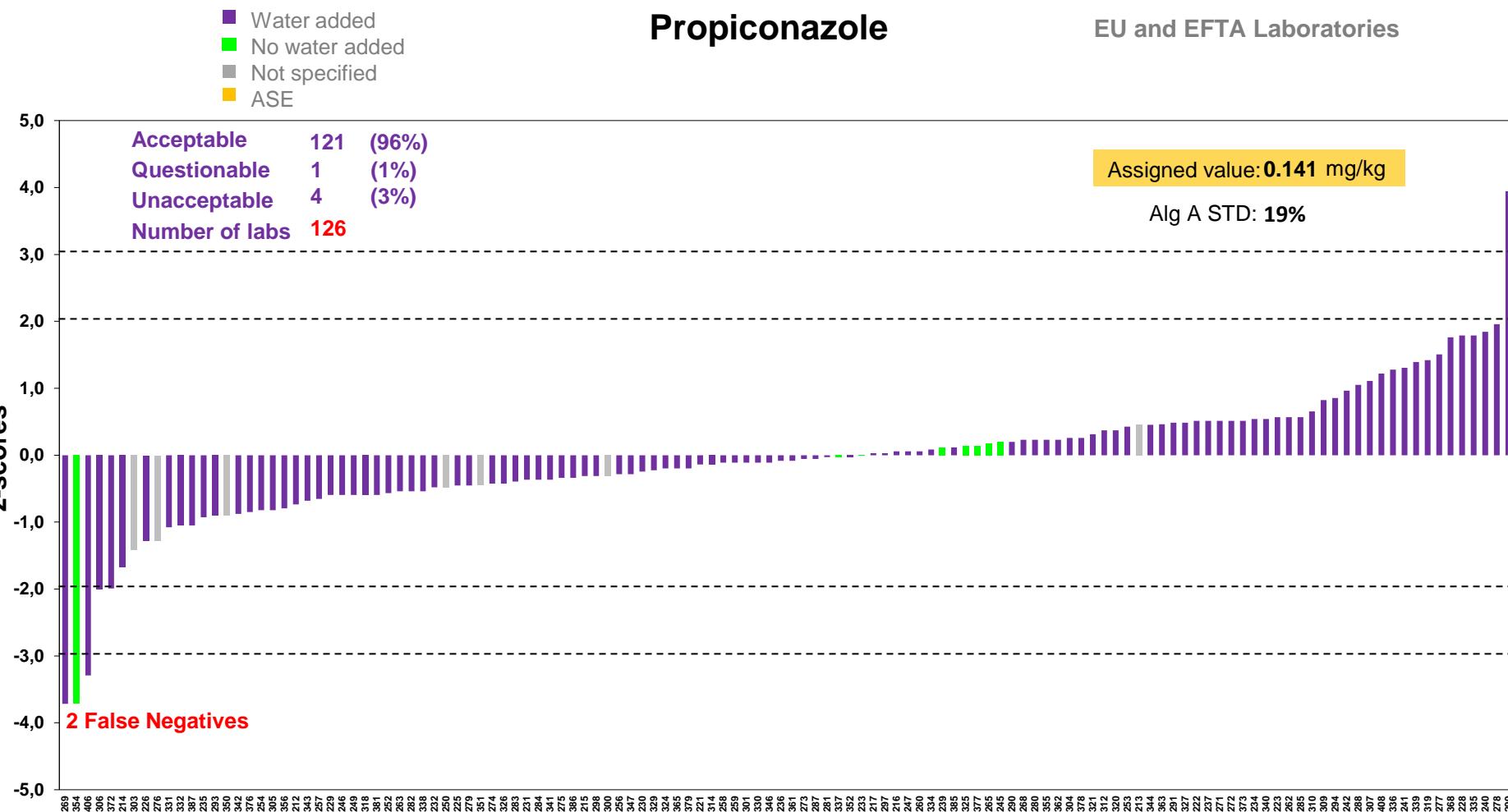
**Acceptable** 98 (96%)  
**Questionable** 0 (0%)  
**Unacceptable** 4 (4%)  
**Number of labs** 102

Assigned value: **0.355 mg/kg**

Alg A STD: **17%**







- Water added
- No water added
- Not specified
- ASE

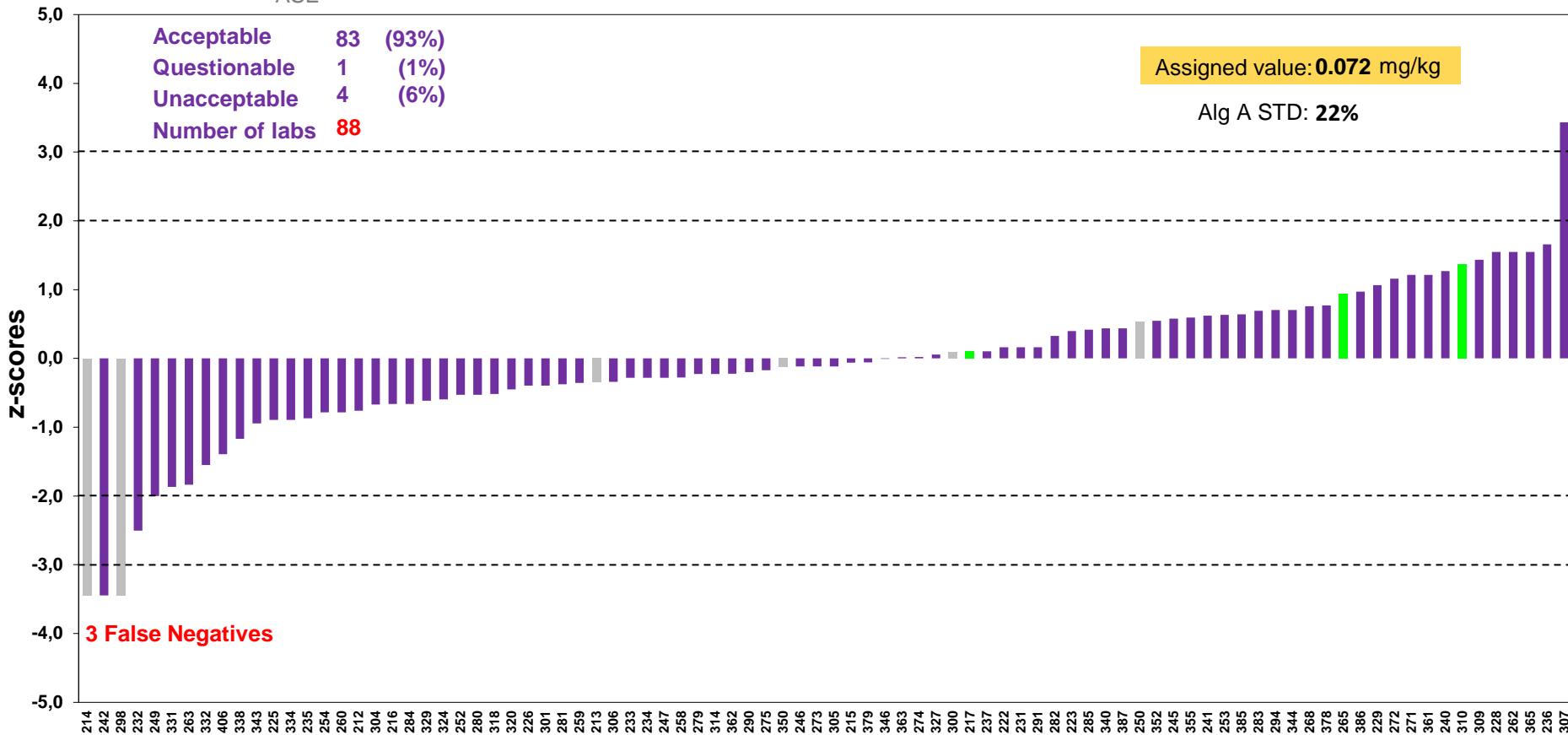
## Spiromesifen

EU and EFTA Laboratories

**Acceptable** 83 (93%)  
**Questionable** 1 (1%)  
**Unacceptable** 4 (6%)  
**Number of labs** 88

Assigned value: **0.072 mg/kg**

Alg A STD: **22%**



- Water added
- No water added
- Not specified
- ASE

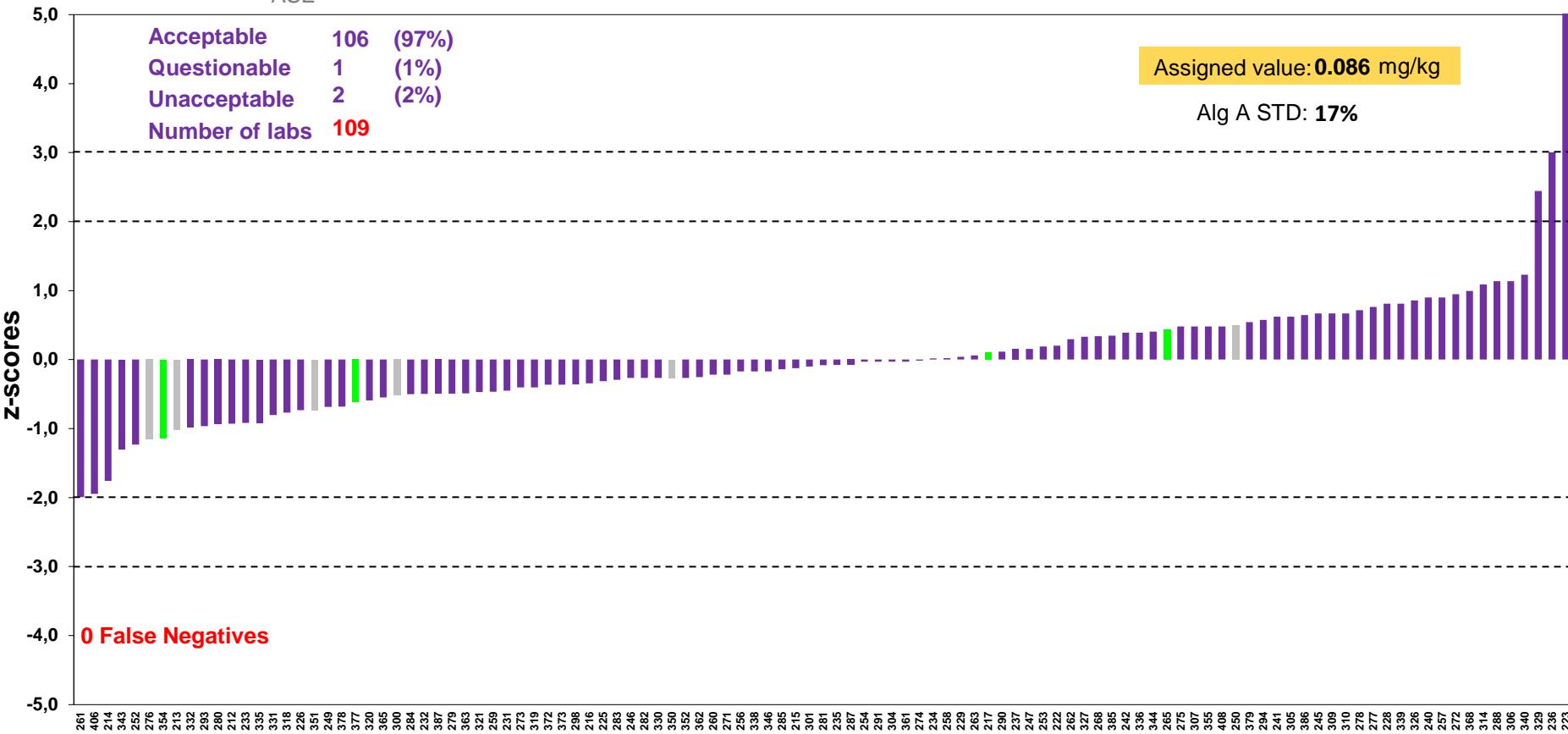
## Terbuthylazine

EU and EFTA Laboratories

**Acceptable**      106 (97%)  
**Questionable**    1 (1%)  
**Unacceptable**   2 (2%)  
**Number of labs**   109

Assigned value: **0.086 mg/kg**

Alg A STD: **17%**



- Water added
- No water added
- Not specified
- ASE

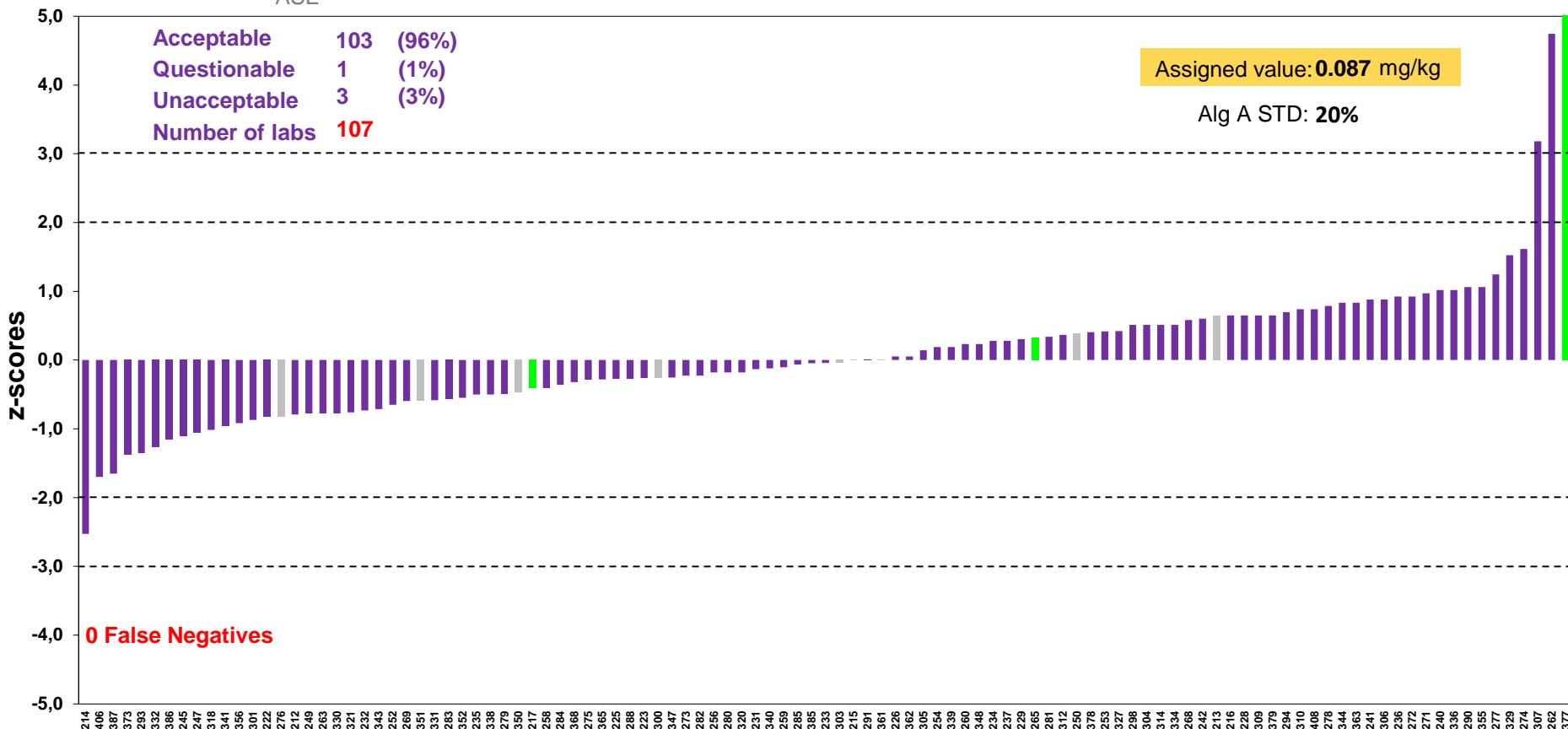
## Thiacloprid

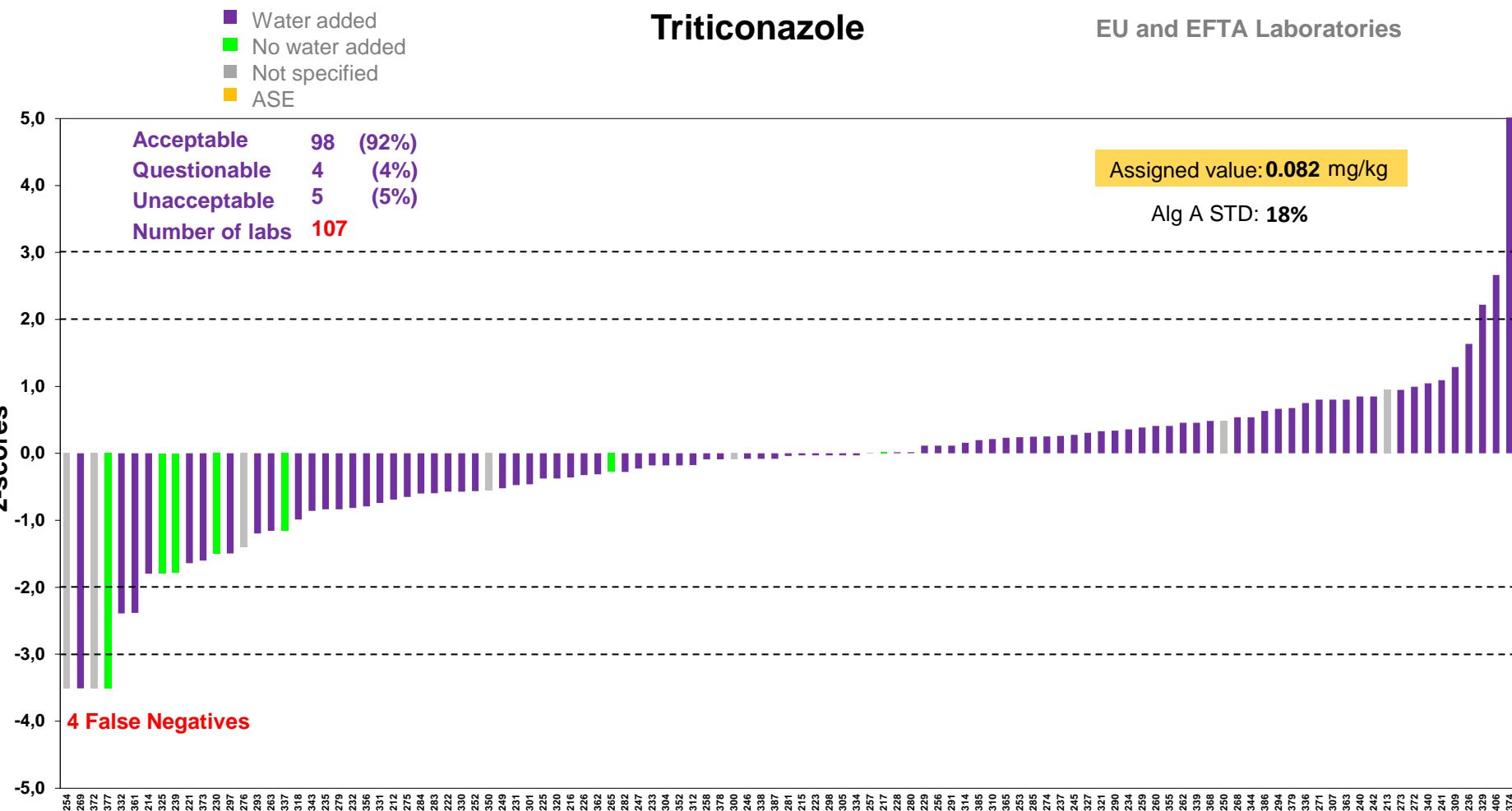
EU and EFTA Laboratories

**Acceptable** 103 (96%)  
**Questionable** 1 (1%)  
**Unacceptable** 3 (3%)  
**Number of labs** 107

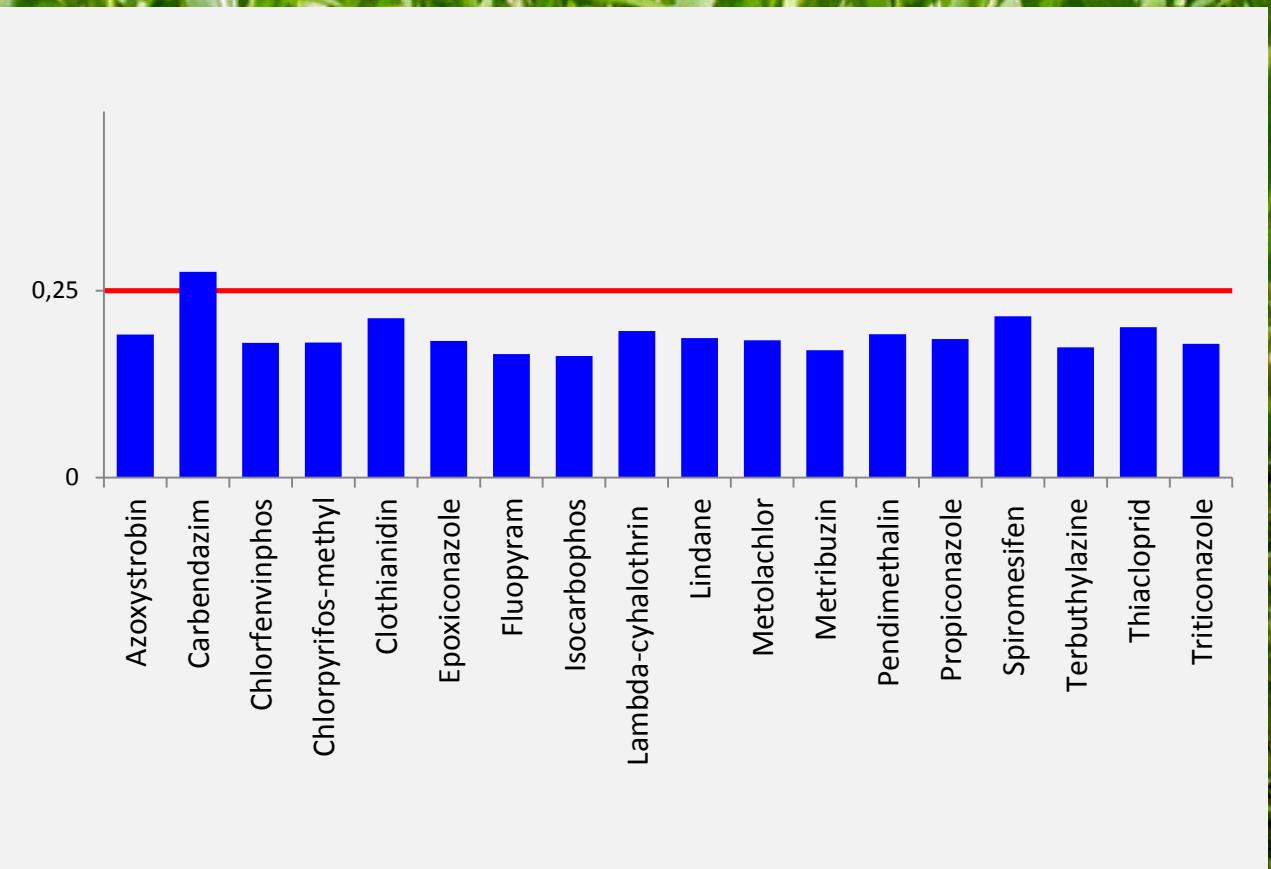
Assigned value: **0.087 mg/kg**

Alg A STD: **20%**



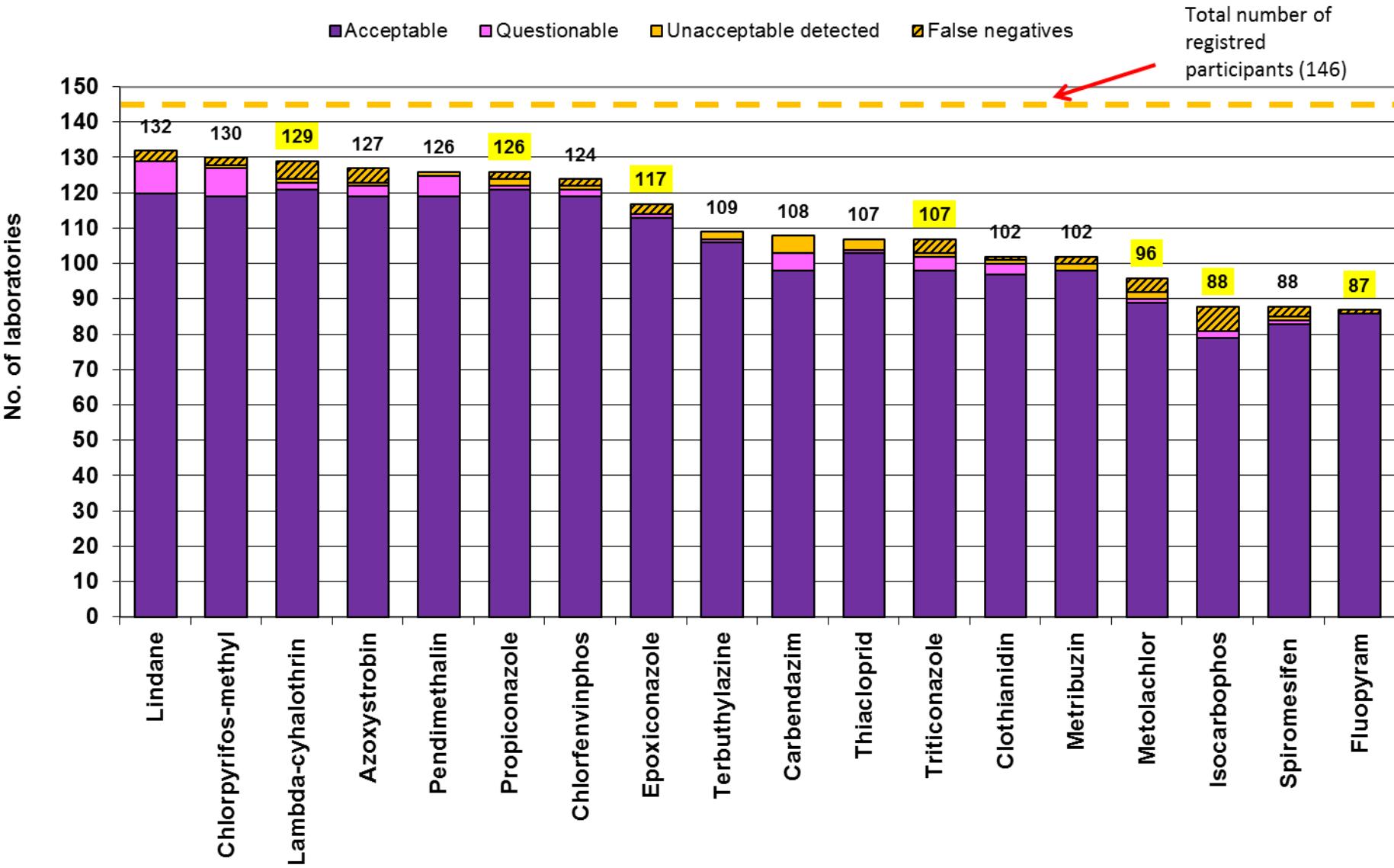


# Alg. A RSD



# Acceptable, questionable and unacceptable results

	No. of reported results	Assigned values mg/kg	Acceptable %	Questionable %	Unacceptable %	False negatives %
Azoxystrobin	127	0.050	94	2	4	3
Carbendazim	108	0.452	91	5	5	0
Chlorfenvinphos	124	0.048	96	2	2	2
Chlorpyrifos-methyl	130	0.054	92	6	2	2
Clothianidin	102	0.429	95	3	2	1
Epoxiconazole	117	0.050	97	1	3	3
Fluopyram	87	0.092	99	0	1	1
Isocarbophos	88	0.081	90	2	8	8
Lambda-cyhalothrin	129	0.088	94	2	5	4
Lindane	132	0.070	91	7	2	2
Metolachlor	96	0.085	93	1	6	4
Metribuzin	102	0.355	96	0	4	2
Pendimethalin	126	0.044	94	5	1	0
Propiconazole	126	0.141	96	1	3	2
Spiromesifen	88	0.072	94	1	5	3
Terbutylazine	109	0.086	97	1	2	0
Thiacloprid	107	0.087	96	1	3	0
Triticonazole	107	0.082	92	4	5	4

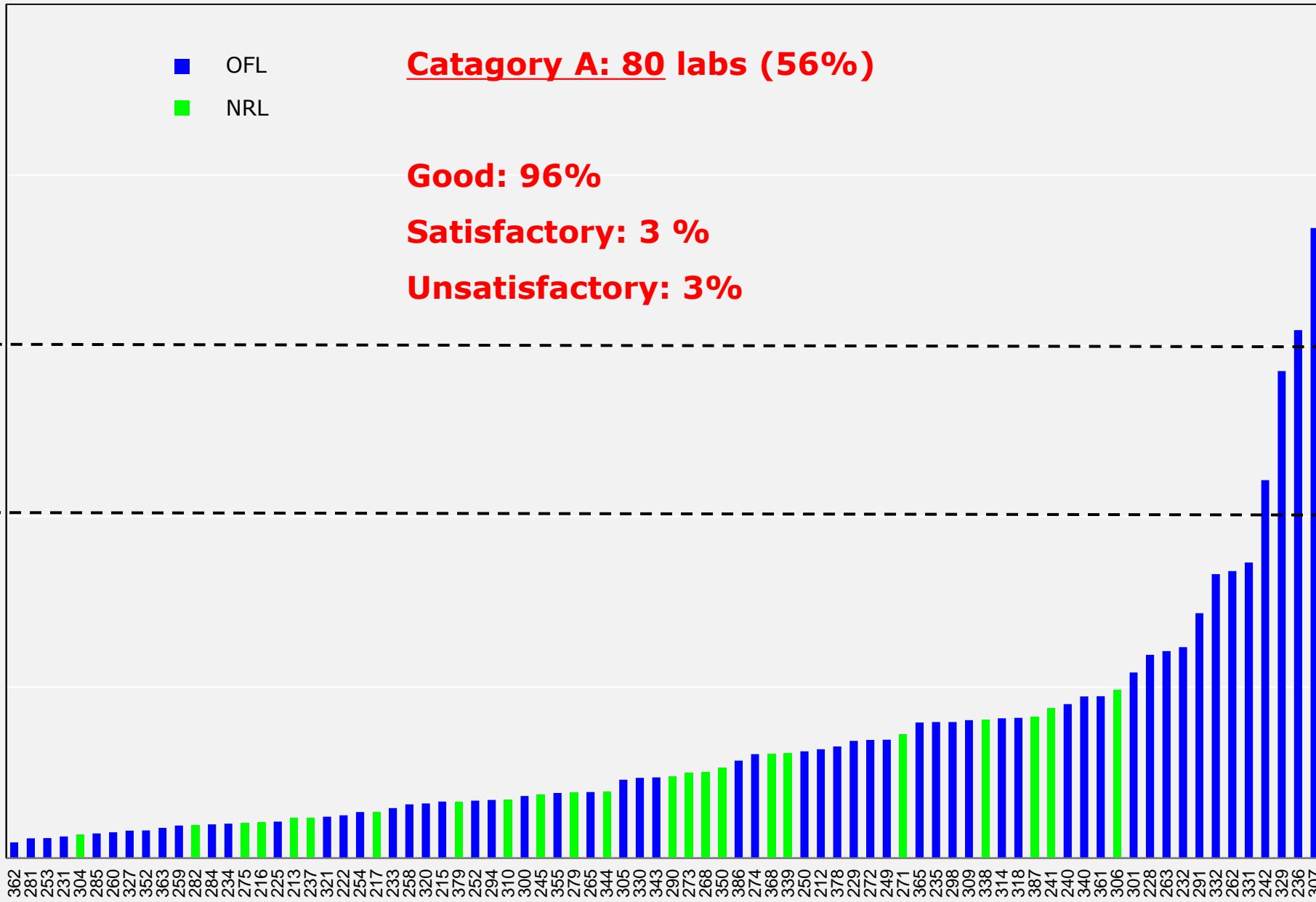


# Category A laboratories and combined z-scores

- To be classified as Category A laboratory the labs had to report at least 90% of the pesticide present in the test material ( $\geq 17$  pesticide residues) and report no false positive.
- Evaluation of the overall performance, the Average of the Squared z-Score (AZ2) are calculated:

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

- $z \leq 2$  Good
- $2 < z < 3$  Satisfactory
- $z \geq 3$  Unsatisfactory



# EUPT-CF10

EUPT	Tentative date	Matrix
FV-18	February 2016	Spinach
CF-10	March 2016	Rye
SRM-11	April 2016	Spinach
AO-11	April-May 2016	Pork fat

A photograph of a lush, green cornfield. The plants are tall with long, narrow leaves and small ears of corn visible at the tops. The sky above is a clear, pale blue.

**Thank you for your attention**