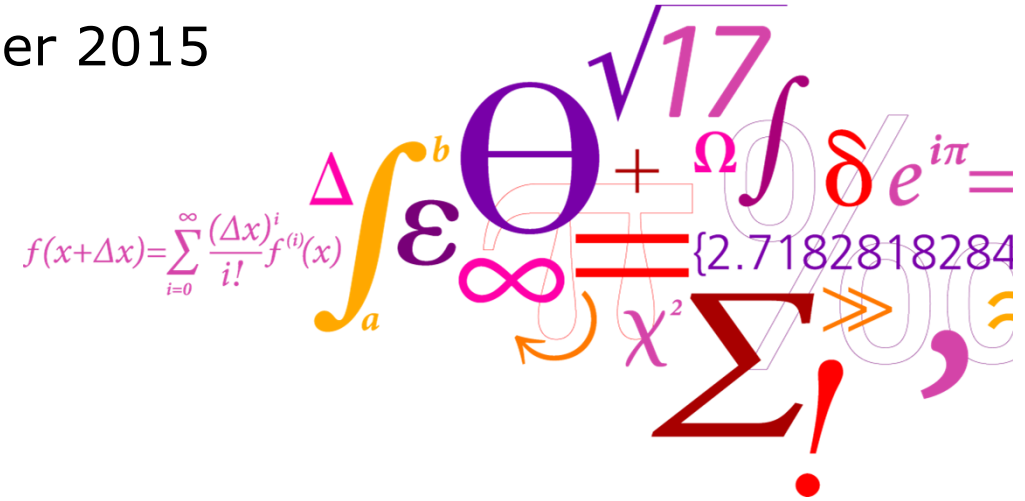
Results of EUPT-CF9
Pesticide residues in maize

Mette Erecius Poulsen

Copenhagen, 16 December 2015



DTU Food
National Food Institute

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EUPT-CF10 - Announcement

The European Union Reference Laboratory for Pesticides Residues in Cereals and Feeding stuff (EURL-CF) announces its tenth European Proficiency Test on for pesticides in cereals and feedingstuff requiring multi residue methods (MRMs), EUPT-CF10.

The aim of the test is to provide laboratories with an assessment of their analytical performance and the reliability of their data - in comparison to other laboratories. This will hopefully result in positive changes and quality improvements at each of the laboratories.

Test Items
The Test Material will be rye flour with incurred and spiked pesticides. The participants will receive approximately 125 g of a treated and approximately 125 g of a blank test material.

Activity	Dates
Announcement Calendar Target Pesticide List	December 2015
EUPT-Registration Website	11 January 2016
Deadline for registration	1 February 2016
Release of Specific Protocol	29 February 2016
Distribution of Test items	7 March 2016
Deadline for Receipt and Acceptance of Test Materials	within 24 hr on reciept
Deadline for Result Submission	11 April 2016 at 13.00 CET
Deadline for submission of additional method information for false negative results	15 April 2015
Preliminary Report (only compilation of results)	30 May 2015
Final Report	December 2015

New pesticides and voluntary compounds

- Acetamiprid
- Aldrin
- Biphenyl
- Bromuconazole
- Buprofezin
- Chlorantraniliprole
- Cymoxanil
- Dieldrin
- Famoxadone
- Flutolanil
- Methamidophos
- Pencycuron
- Propamocarb
- Tau-Fluvalinate
- Tefluthrin
- Tetraconazole
- Tetramethrin

- From DIRECTIVE 2002/32/EC

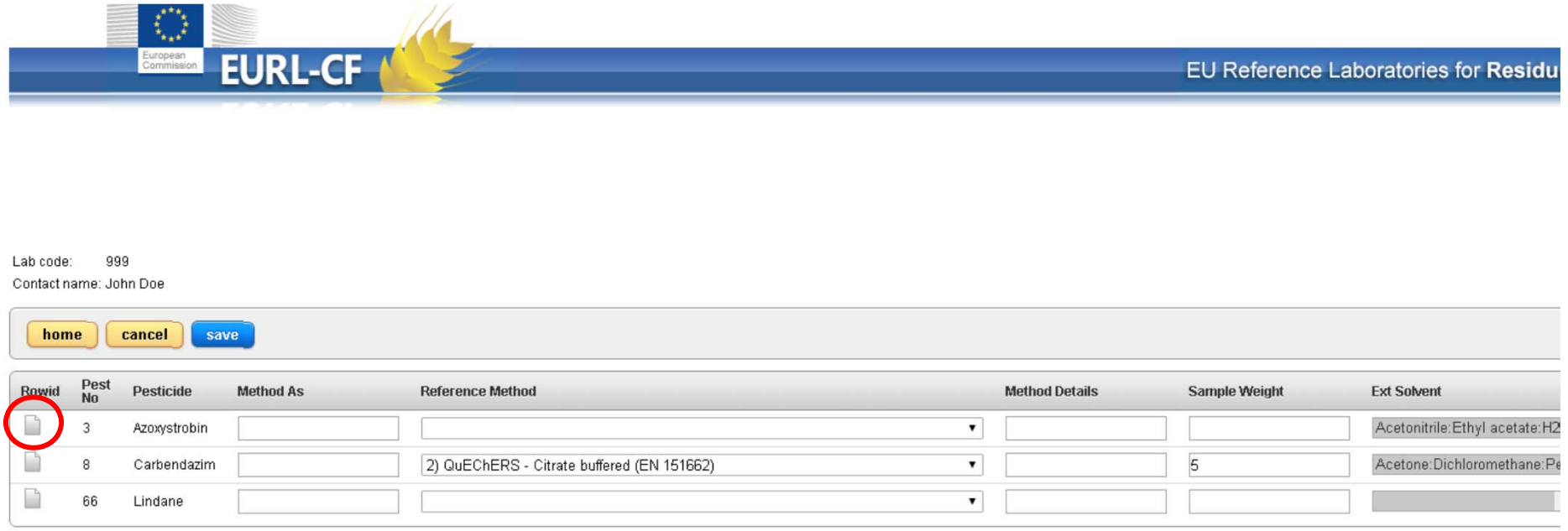
- Voluntary:
  - Chlordane, cis-
  - Chlordane, oxy
  - Chlordane, trans-
  - Endrin
  - Heptachlor
  - Heptachlorepoxid-cis
  - Heptachlorepoxid-trans

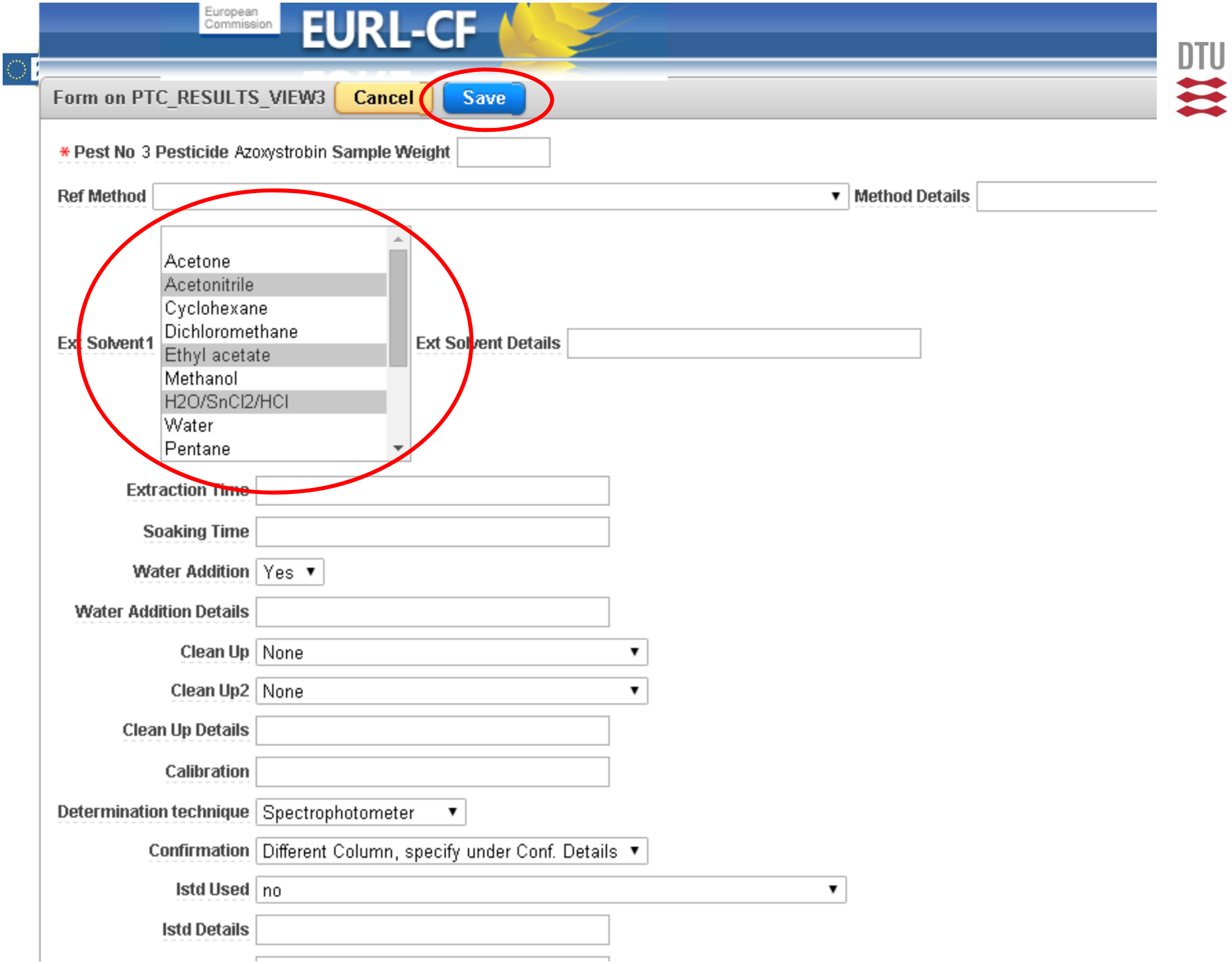
Changes in registration website

- We will include the addresses for shipment and invoice that you entered for EUPT-CF9
- Please, make sure that this is still correct

Changes in result submisstion website

- Page 3 – method information.




European Commission
EURL-CF
DTU
Form on PTC_RESULTS_VIEW3
Cancel
Save
* Pest No 3 Pesticide Azoxystrobin Sample Weight
Ref Method
Method Details
Ext Solvent1
Acetone
Acetonitrile
Cyclohexane
Dichloromethane
Ethyl acetate
Methanol
H2O/SnCl2/HCl
Water
Pentane
Ext Solvent Details
Extraction Time
Soaking Time
Water Addition
Yes
Water Addition Details
Clean Up
None
Clean Up2
None
Clean Up Details
Calibration
Determination technique
Spectrophotometer
Confirmation
Different Column, specify under Conf. Details
Istd Used
no
Istd Details

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European Commission EURL-CF EU Reference Laboratories for Residu

Lab code: 999
Contact name: John Doe

home cancel save

Rowid	Pest No	Pesticide	Method As	Reference Method	Method Details	Sample Weight	Ext Solvent
	3	Azoxystrobin					Acetonitrile:Ethyl acetate:H2
	8	Carbendazim		2) QuEChERS - Citrate buffered (EN 151662)		5	Acetone:Dichloromethane:Pe
	66	Lindane					

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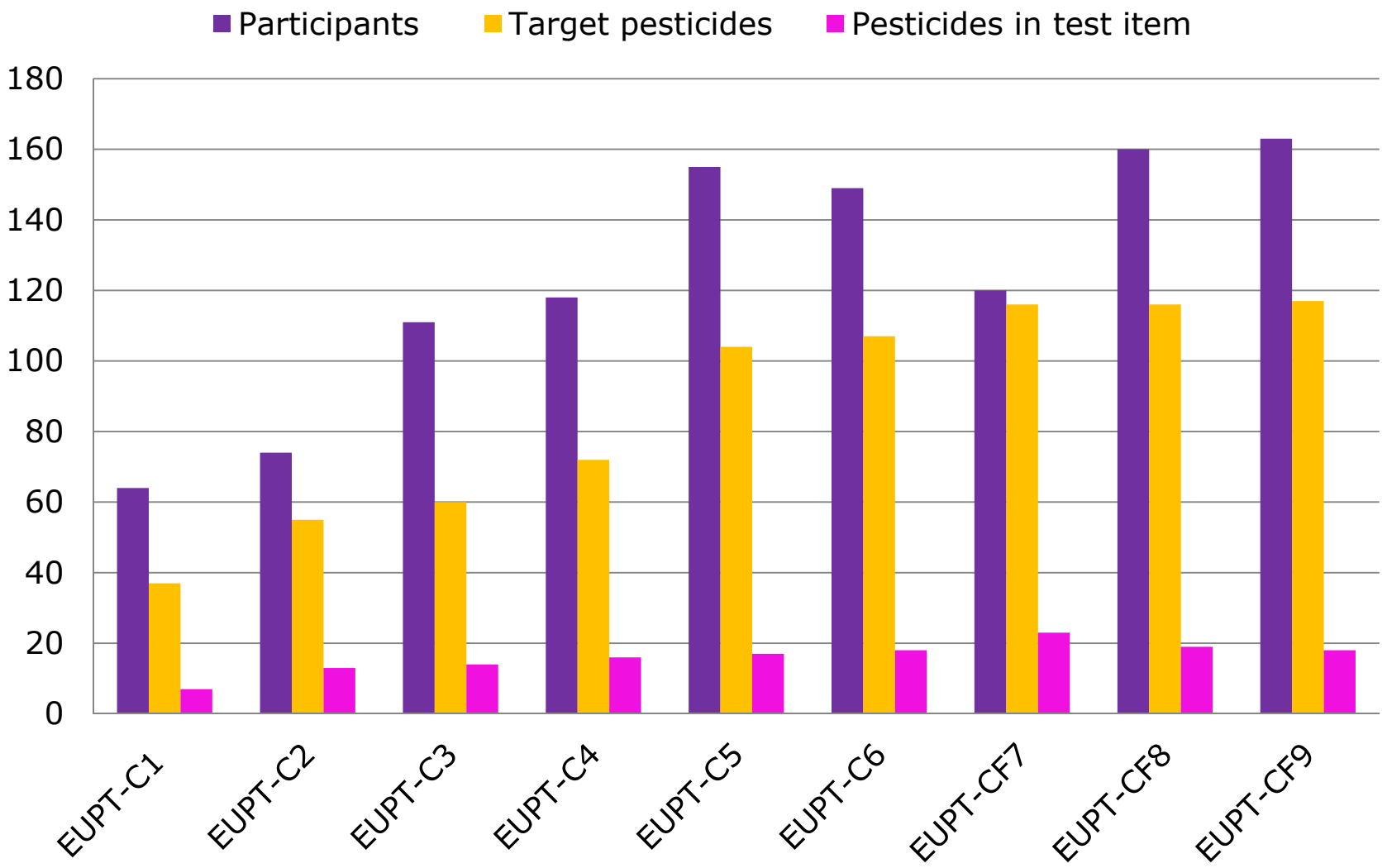
EUPT-CF9 results

Overview of PTs on cereals / feed



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Advisory Group

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Merete B. Ludwigsen,
Lisbet Pilhkjær
Jens-Ole Frimann

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	**Total**	**163**

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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
  - Terbuthylazine

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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	Chlorfenvinphos	0.01

Maize

- 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
Iodosulfurum	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

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	x	Opera / Analytical standard
Foramsulfuron	x		MaisTer
Iodosulfurum	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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Iodosulfurum	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
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
Terbuthylazine		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

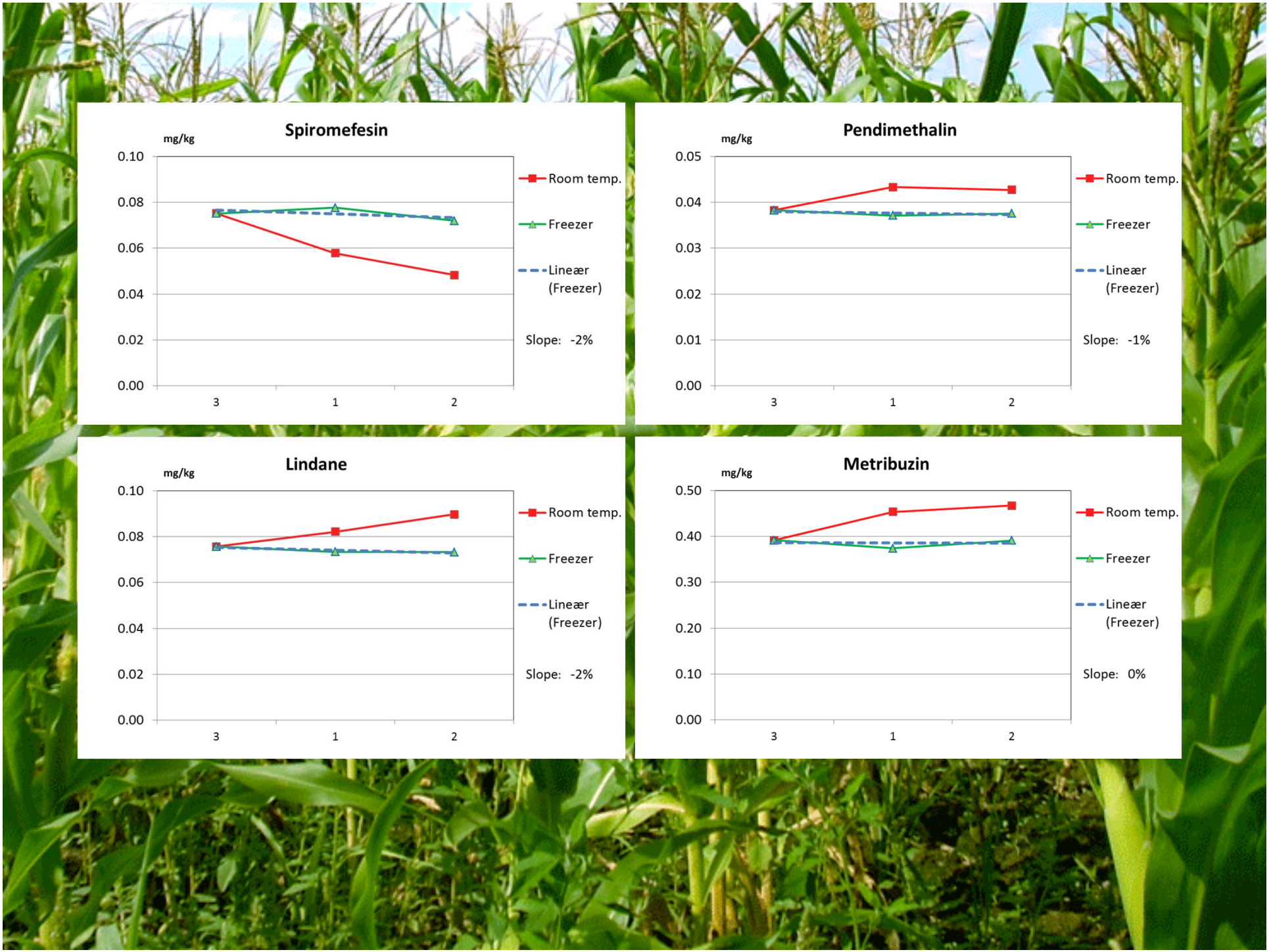
	Mean, mg/kg	S_s^2	c	$S_s^2 < c$
Azoxystrobin	0.050	0.00001	0.0000	Pass
Carbendazim	0.399	0.00097	0.0018	Pass
Chlorfenvinphos	0.041	0.00000	0.0001	Pass
Chlorpyrifos-methyl	0.043	0.00000	0.0000	Pass
Clothianidin	0.489	0.00116	0.0036	Pass
Epoxiconazole	0.050	0.00001	0.0000	Pass
Fluopyram	0.089	0.00005	0.0001	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

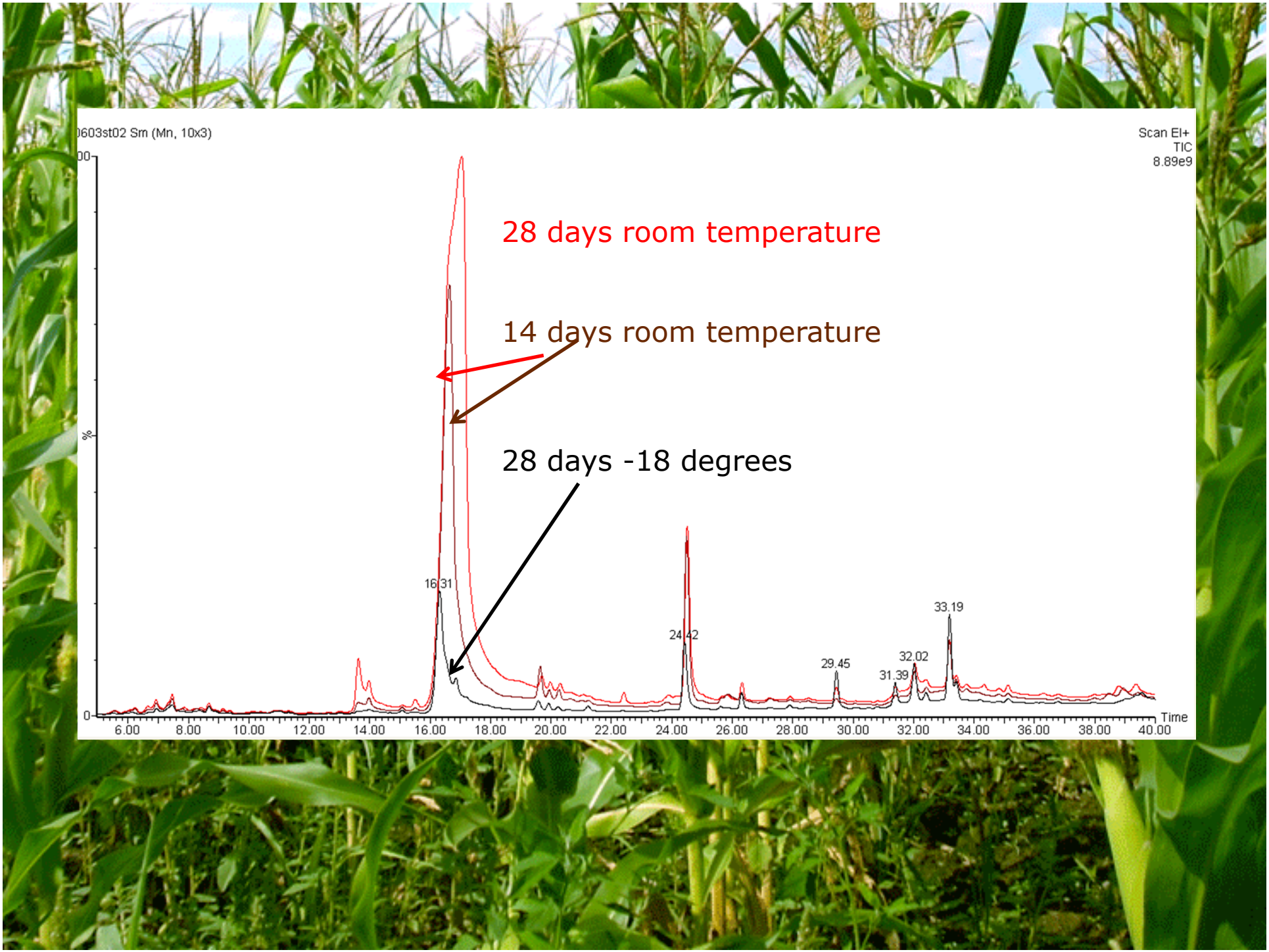
Stability test

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

Stability test

		Storage at -18 degrees			Storage at room temperature		
	Mean, mg/kg	| x1 - yi |	0.3×σ	| x1 - yi | ≤ 0.3×σ	| x1 - yi |	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
Terbuthylazine	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


Spiromefesin
mg/kg
0.10
0.08
0.06
0.04
0.02
0.00
3
1
2
Room temp.
Freezer
Lineær (Freezer)
Slope: -2%
Pendimethalin
mg/kg
0.05
0.04
0.03
0.02
0.01
0.00
3
1
2
Room temp.
Freezer
Lineær (Freezer)
Slope: -1%
Lindane
mg/kg
0.10
0.08
0.06
0.04
0.02
0.00
3
1
2
Room temp.
Freezer
Lineær (Freezer)
Slope: -2%
Metribuzin
mg/kg
0.50
0.40
0.30
0.20
0.10
0.00
3
1
2
Room temp.
Freezer
Lineær (Freezer)
Slope: 0%

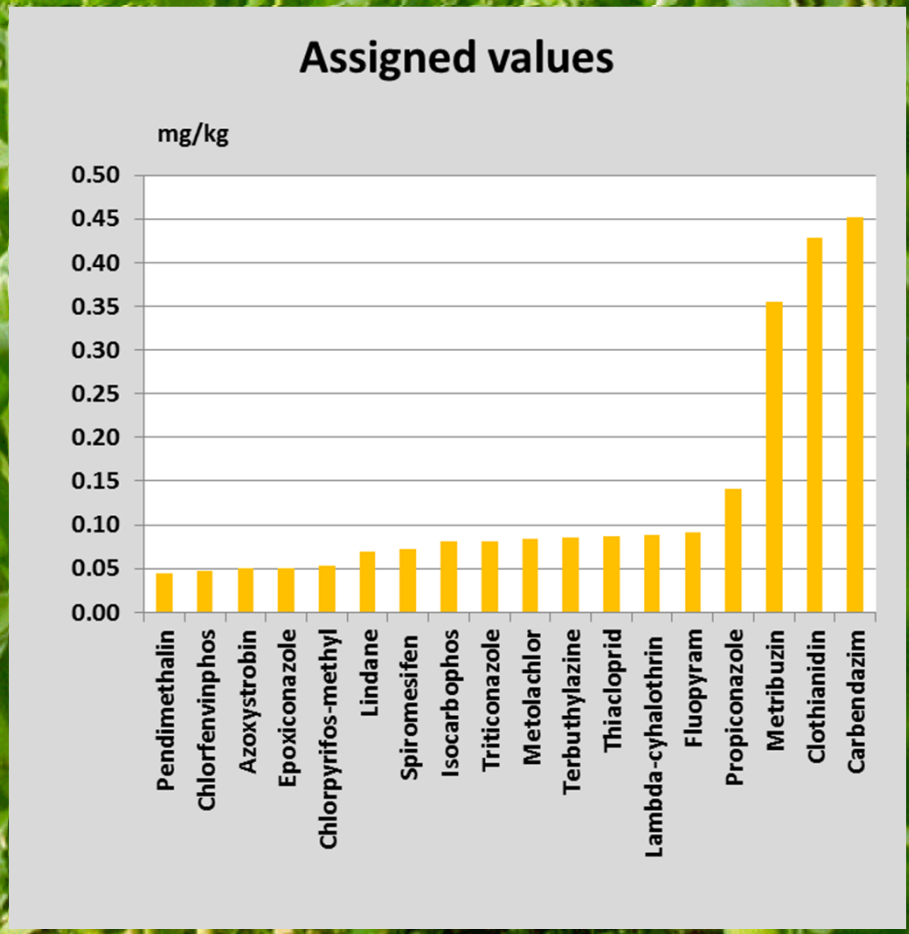

0603st02 Sm (Mn, 10x3)
Scan EI+
TIC
8.89e9
28 days room temperature
14 days room temperature
28 days -18 degrees
16.31
24.42
29.45
31.39
32.02
33.19
%
0
Time
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00
24.00
26.00
28.00
30.00
32.00
34.00
36.00
38.00
40.00

Calculation of assigned values and uncertainty of assigned values

- Algoritm A mean of the results from EU laboratories (and EFTA)
  - Only result from participant that
    - reported to add water to the samples before extration or
    - used mixture of solvent and water or
    - used ASE
- Outliers
    - Only obvious incorrect results
- Uncertainty
    - $u = 1.25 * (s^* / \sqrt{n})$
    - 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
Terbuthylazine	0.086
Thiacloprid	0.087
Triticonazole	0.082



Reported results and false negatives

Pesticide	No. of reported results	No. of NA	False negatives	% results
Azoxystrobin	137	19	3	88
Carbendazim	116	40	0	74
Chlorfenvinphos	135	21	2	87
Chlorpyrifos-methyl	142	14	1	91
Clothianidin	108	48	1	69
Epoxiconazole	124	32	2	79
Fluopyram	92	64	1	59
Isocarbophos	92	64	8	59
Lambda-cyhalothrin	139	17	4	89
Lindane	143	13	2	92
Metolachlor	103	53	5	66
Metribuzin	113	43	2	72
Pendimethalin	135	21	0	87
Propiconazole	135	21	1	87
Spiromesifen	94	62	4	60
Terbuthylazine	115	41	0	74
Thiacloprid	116	40	0	74
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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
349	Carbofuran, 3-hydroxy	0.011	LC-MS/MS QQQ	0.01
367	Chlorpyrifos	0.0117	GC- (μ) ECD	0.005
367	Cypermethrin (sum of isomers)	0.0155	GC- (μ) ECD	0.005
367	Endosulfan-beta	0.01	GC- (μ) ECD	0.005
369	Chlorpyrifos	0.022	LC-MS/MS QQQ	0.05
369	Tebuconazole	0.021	LC-MS/MS QQQ	0.01
374	HCH-alpha	0.059	GC-MS/MS (QQQ)	0.01
374	HCH-beta	0.081	GC-MS/MS (QQQ)	0.01
384	HCH-beta	0.0344	GC-MSD	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
367	Endosulfan-alpha	0.0019	GC- (μ) ECD	0.005
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
  - σ is the target standard deviation obtained by multiplying the median by the FFP RSD of 25%

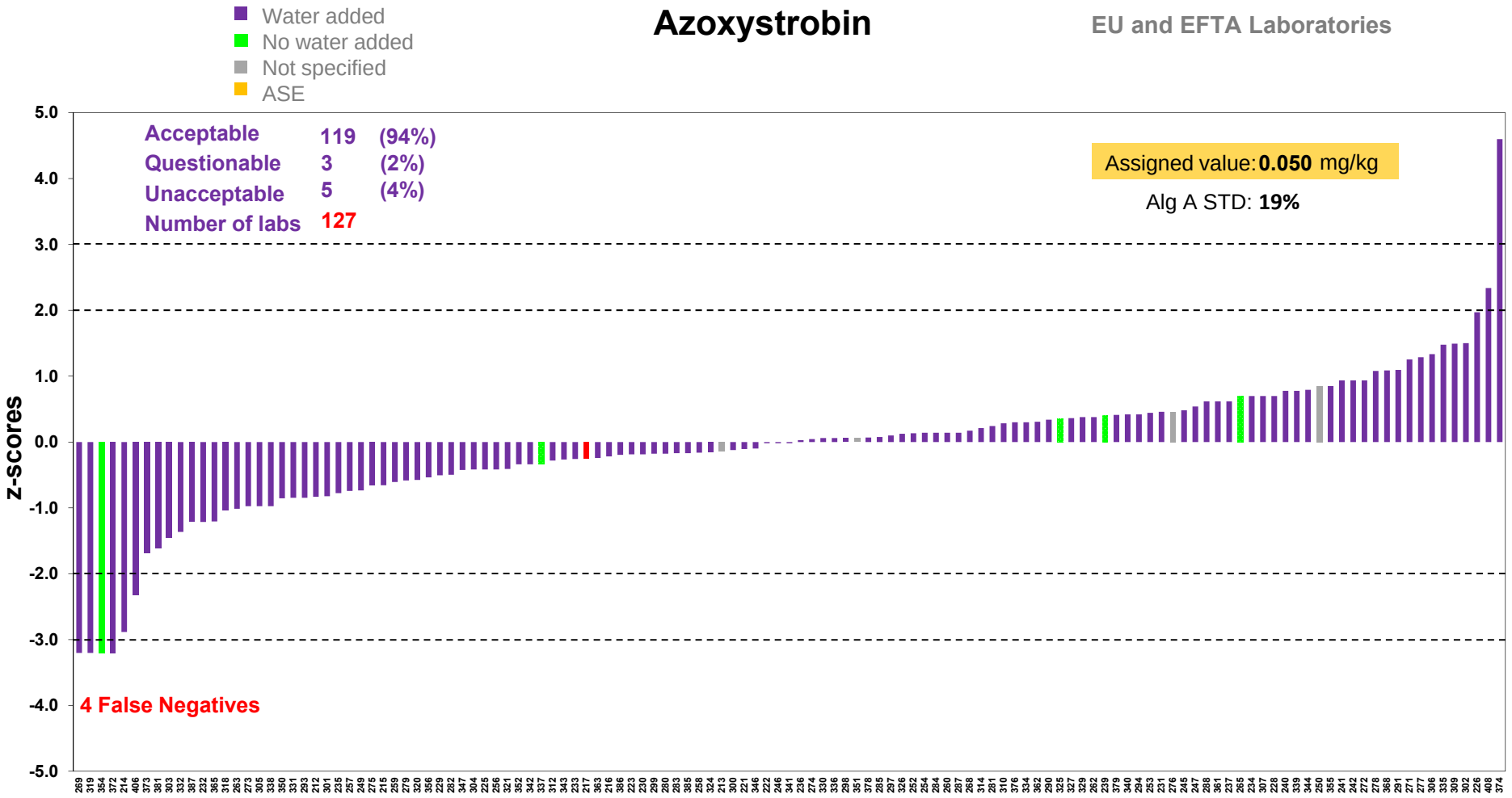
- $|z| \leq 2$ Acceptable
- $2 < |z| < 3$ Questionable
- $|z| \geq 3$ Unacceptable

EURL EUROPEAN UNION REFERENCE LABORATORY PESTICIDE RESIDUES IN CEREALS & FEEDING STUFF

DTU

Azoxystrobin

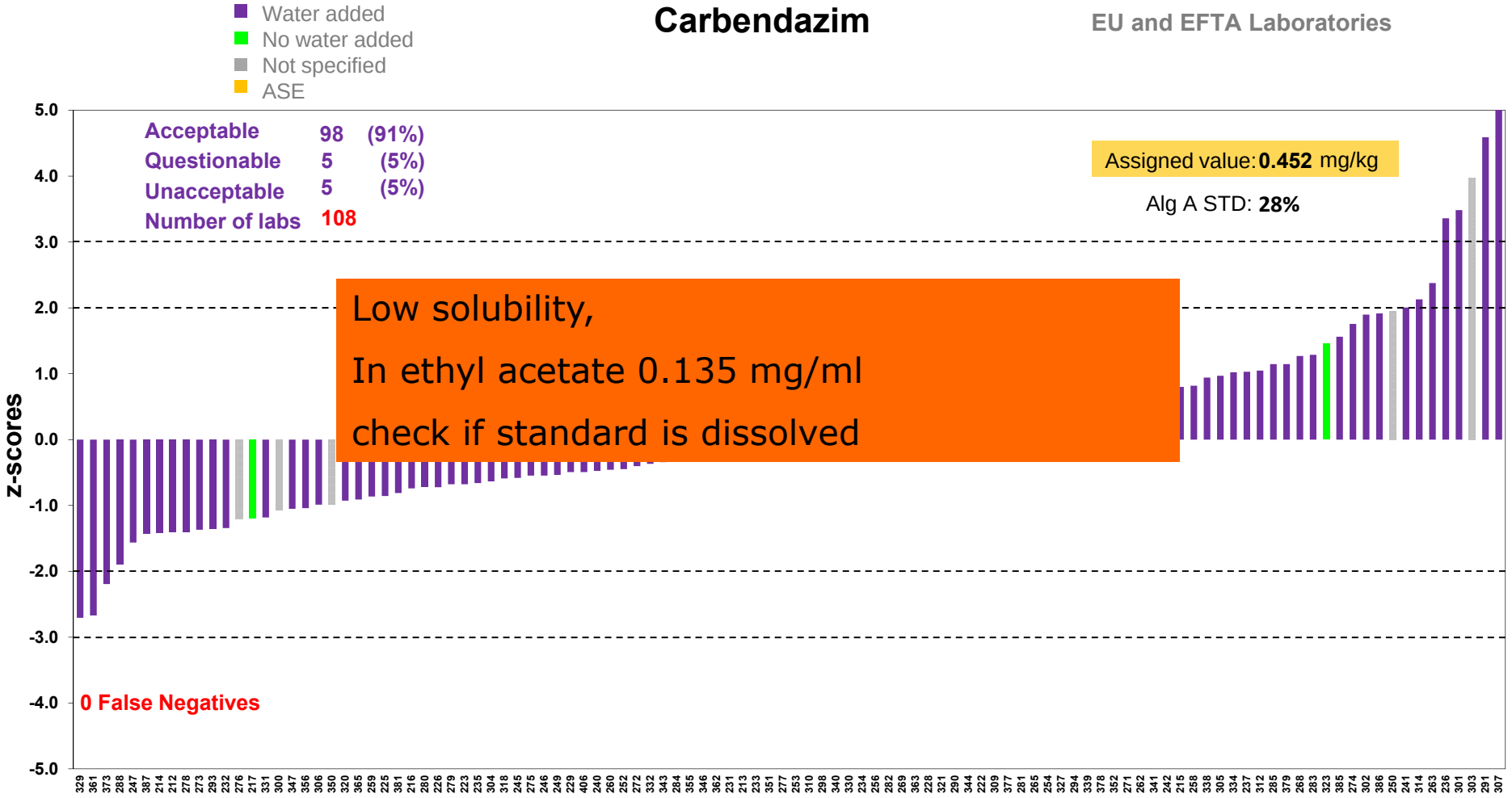
EU and EFTA Laboratories



National Food Institute, Technical University of Denmark

Carbendazim

EU and EFTA Laboratories

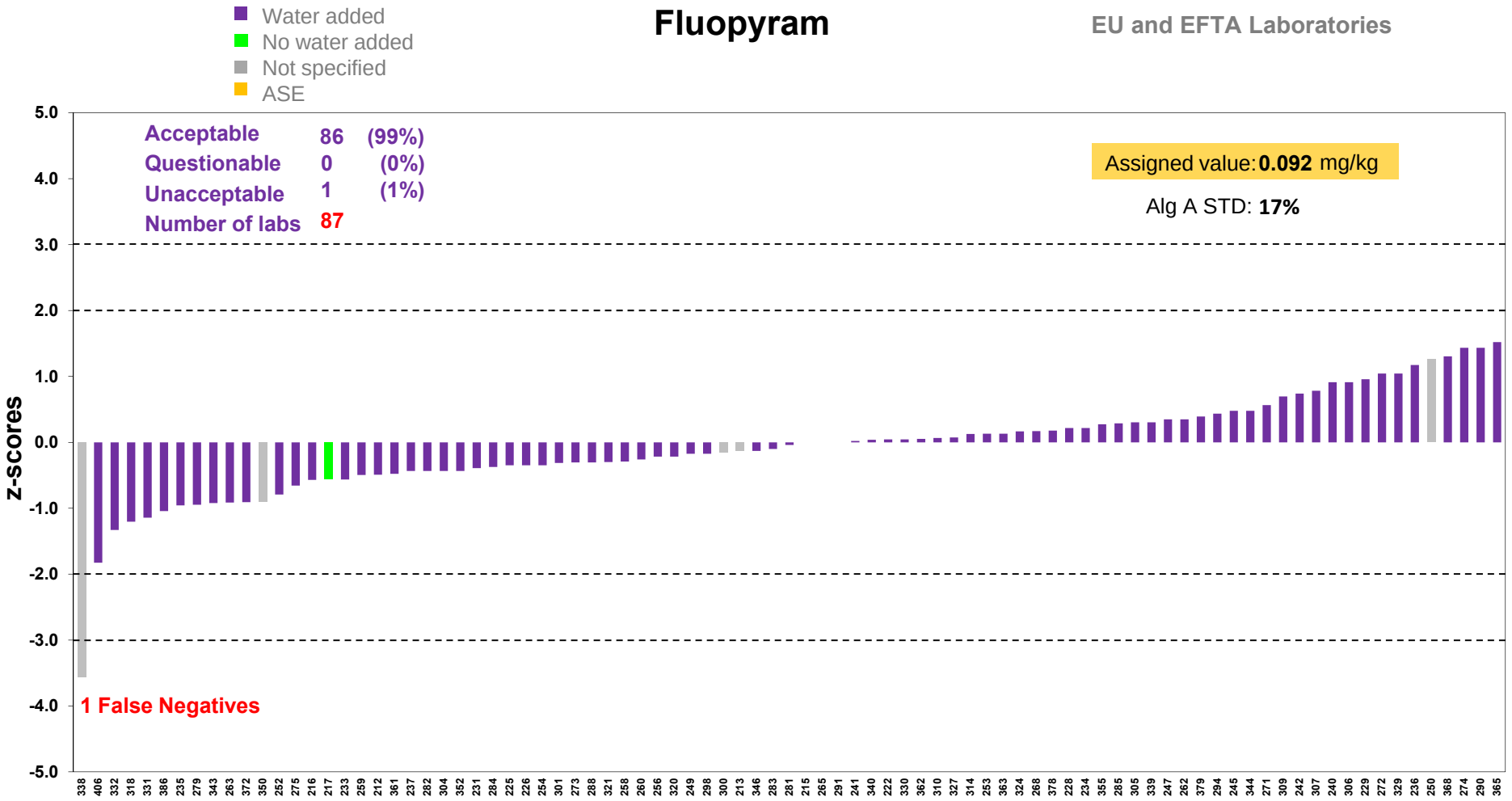


EURL EUROPEAN UNION REFERENCE LABORATORY PESTICIDE RESIDUES IN CEREALS & FEEDING STUFF

DTU

Fluopyram

EU and EFTA Laboratories

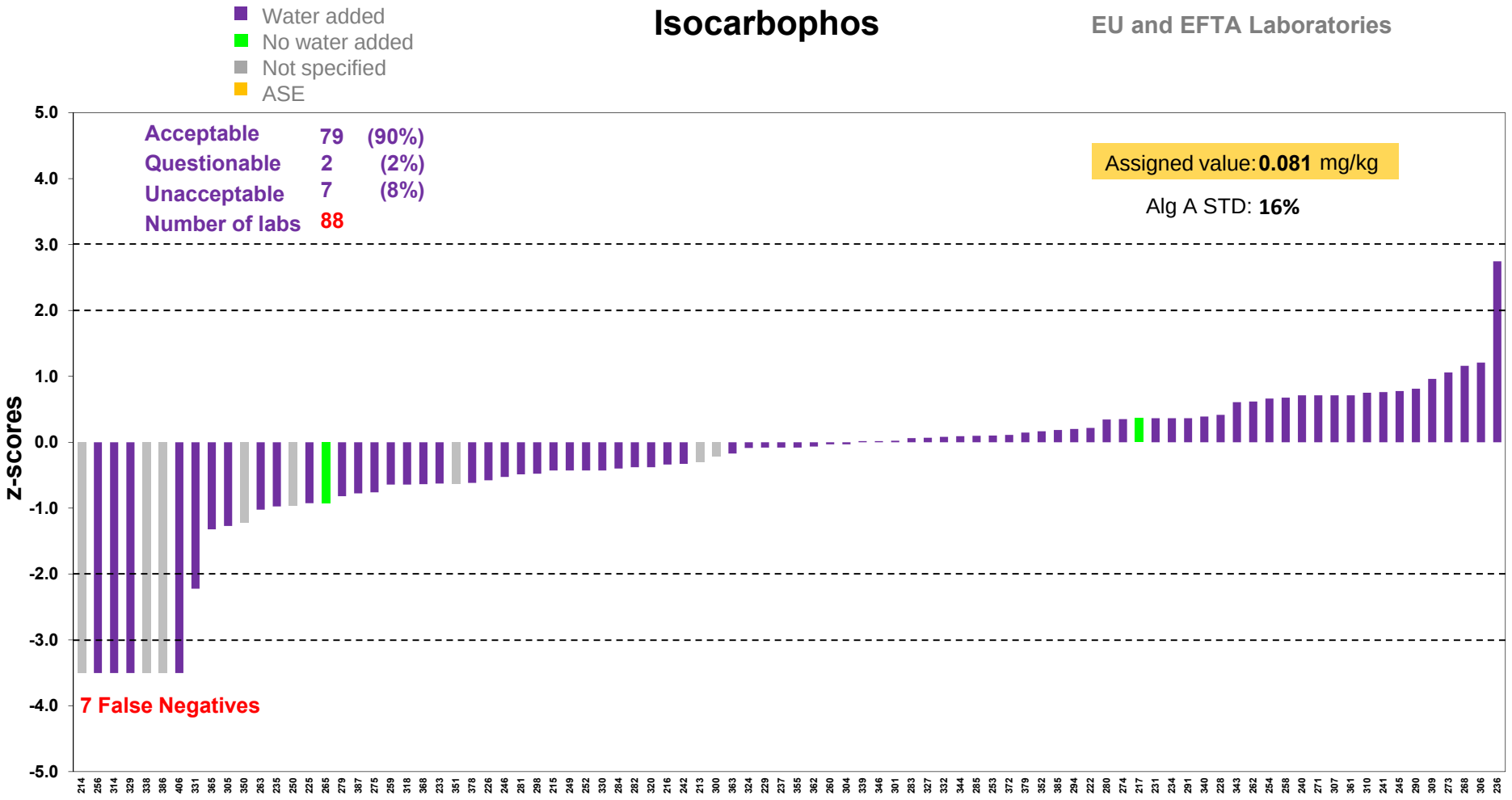


EURL EUROPEAN UNION REFERENCE LABORATORY PESTICIDE RESIDUES IN CEREALS & FEEDING STUFF

DTU

Isocarbophos

EU and EFTA Laboratories

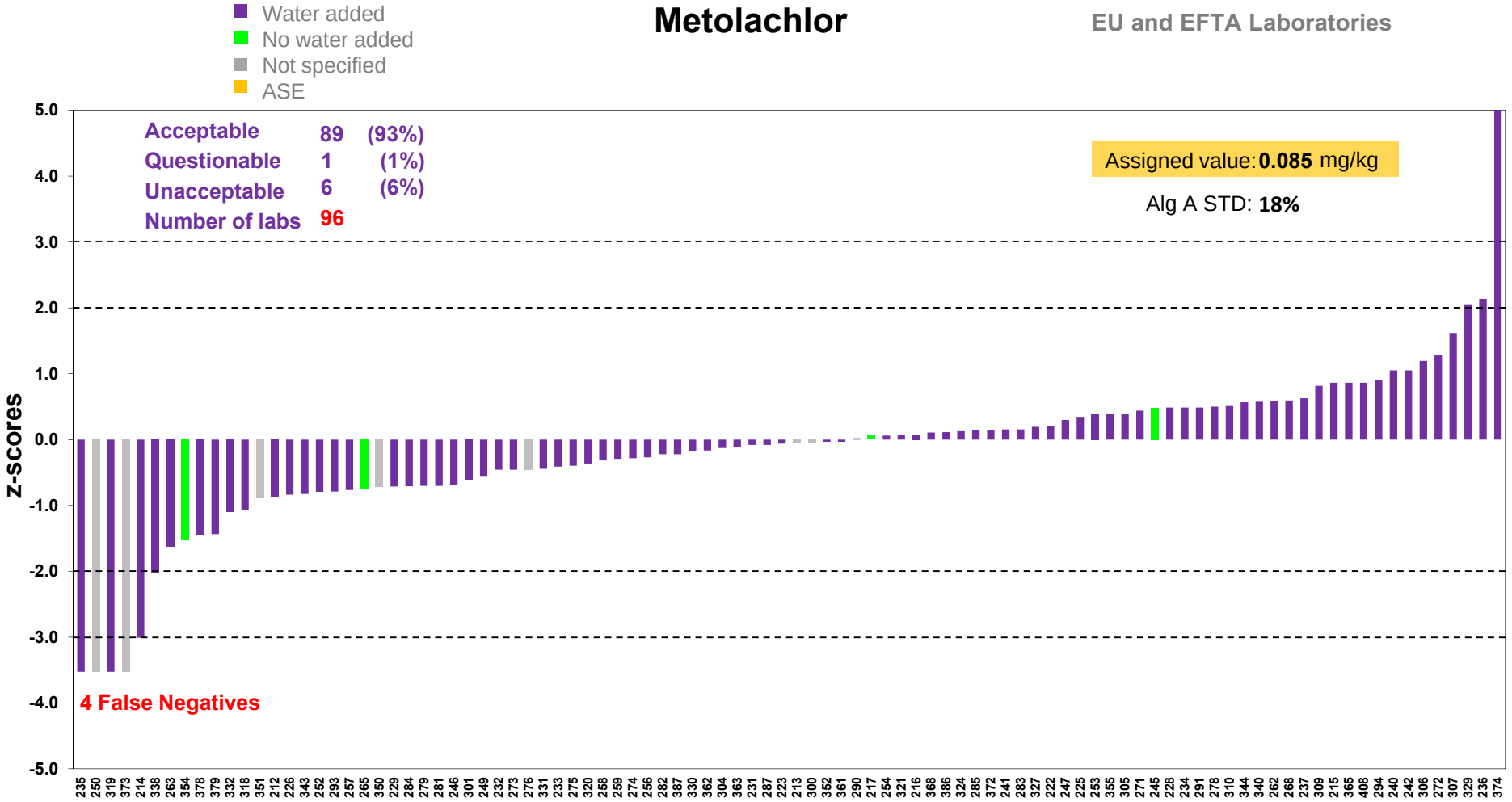


EURL EUROPEAN UNION REFERENCE LABORATORY PESTICIDE RESIDUES IN CEREALS & FEEDING STUFF

DTU

Metolachlor

EU and EFTA Laboratories



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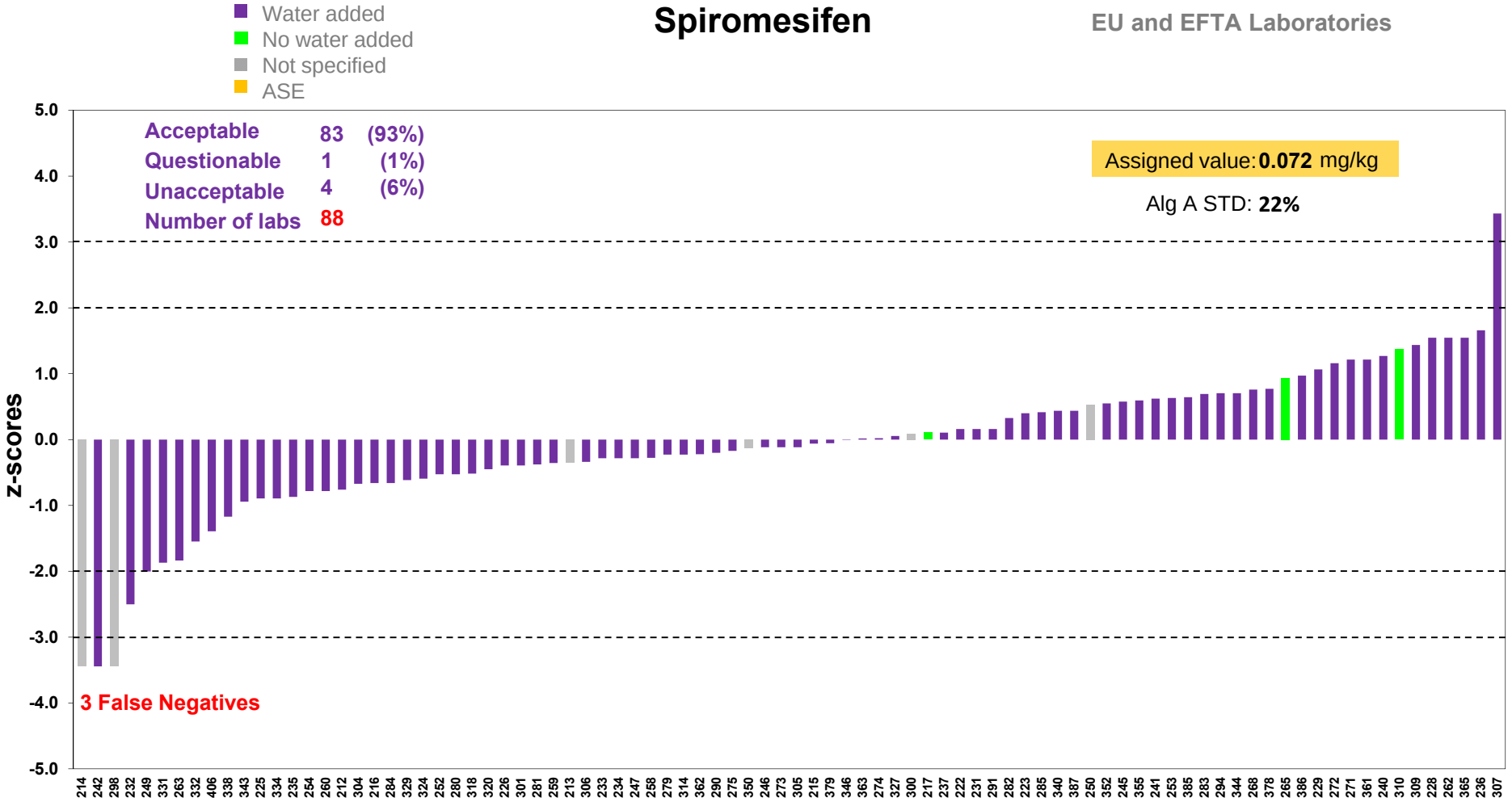
EURL EUROPEAN UNION REFERENCE LABORATORY PESTICIDE RESIDUES IN CEREALS & FEEDING STUFF

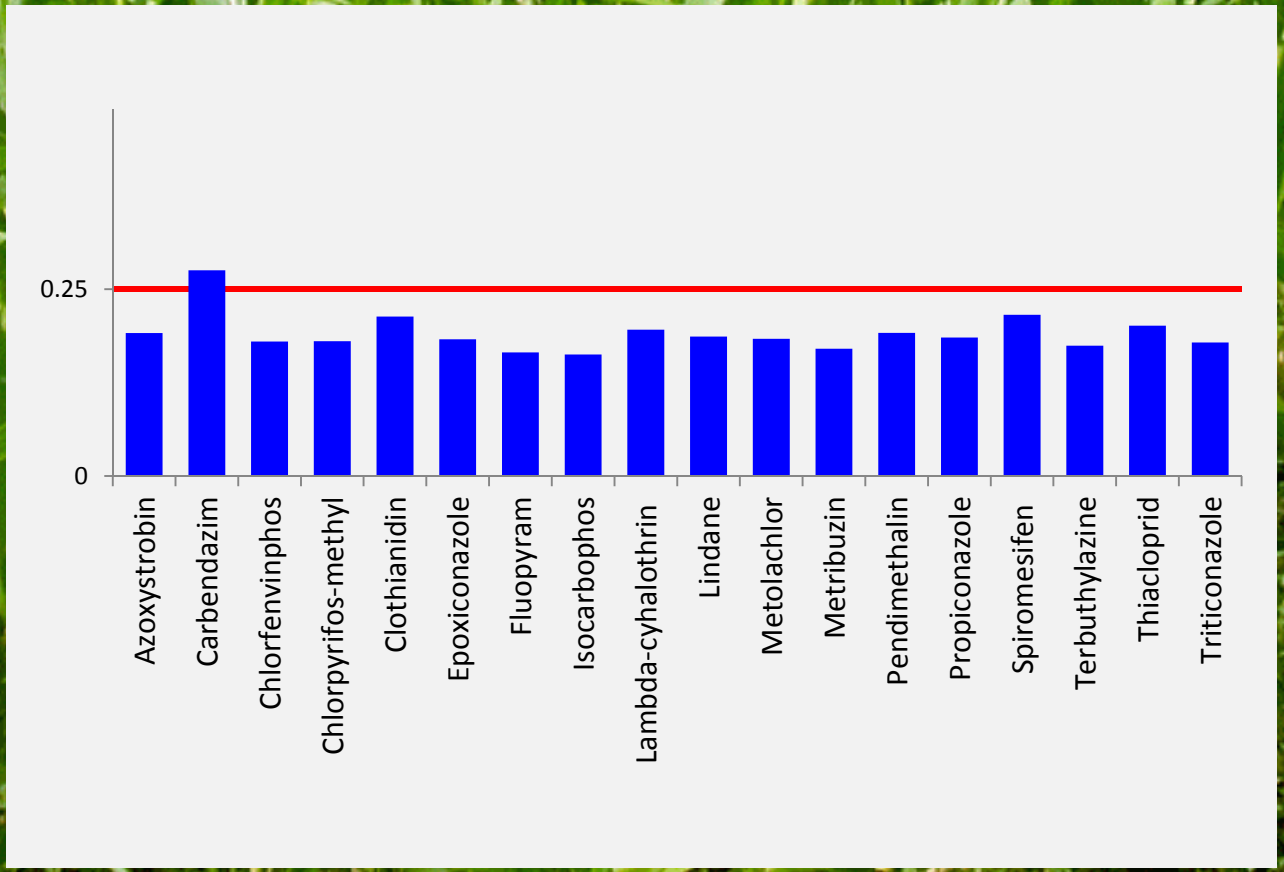
DTU

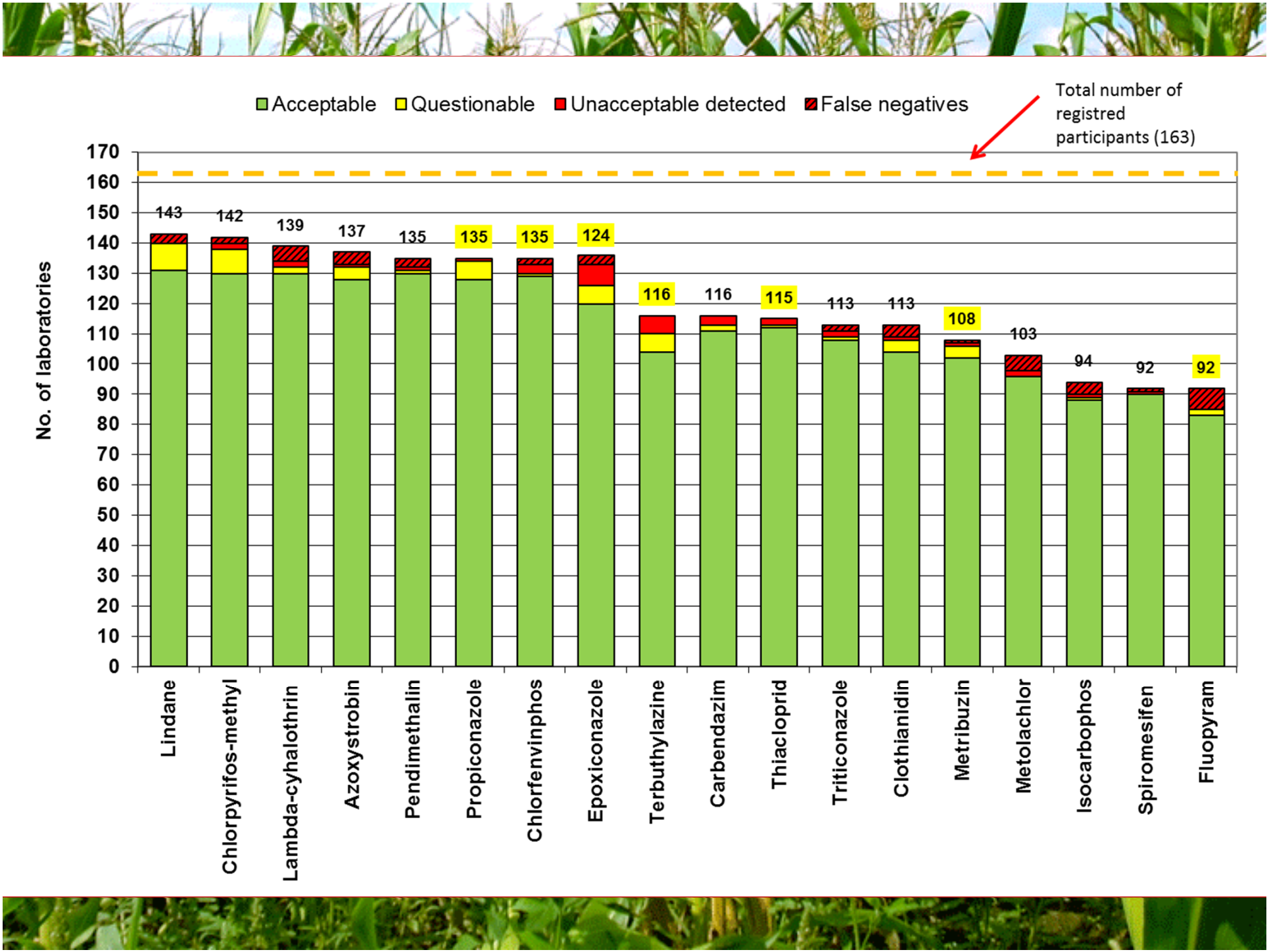
Spiromesifen

EU and EFTA Laboratories



Alg. A RSD


0.25
0
Azoxystrobin
Carbendazim
Chlorfenvinphos
Chlorpyrifos-methyl
Clothianidin
Epoxiconazole
Fluopyram
Isocarbophos
Lambda-cyhalothrin
Lindane
Metolachlor
Metribuzin
Pendimethalin
Propiconazole
Spiromesifen
Terbuthylazine
Thiacloprid
Triticonazole


Acceptable
Questionable
Unacceptable detected
False negatives
Total number of registred participants (163)
No. of laboratories
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
143
142
139
137
135
135
135
124
116
116
115
113
113
108
103
94
92
92
Lindane
Chlorpyrifos-methyl
Lambda-cyhalothrin
Azoxystrobin
Pendimethalin
Propiconazole
Chlorfenvinphos
Epoxiconazole
Terbuthylazine
Carbendazim
Thiacloprid
Triticonazole
Clothianidin
Metribuzin
Metolachlor
Isocarbophos
Spiromesifen
Fluopyram

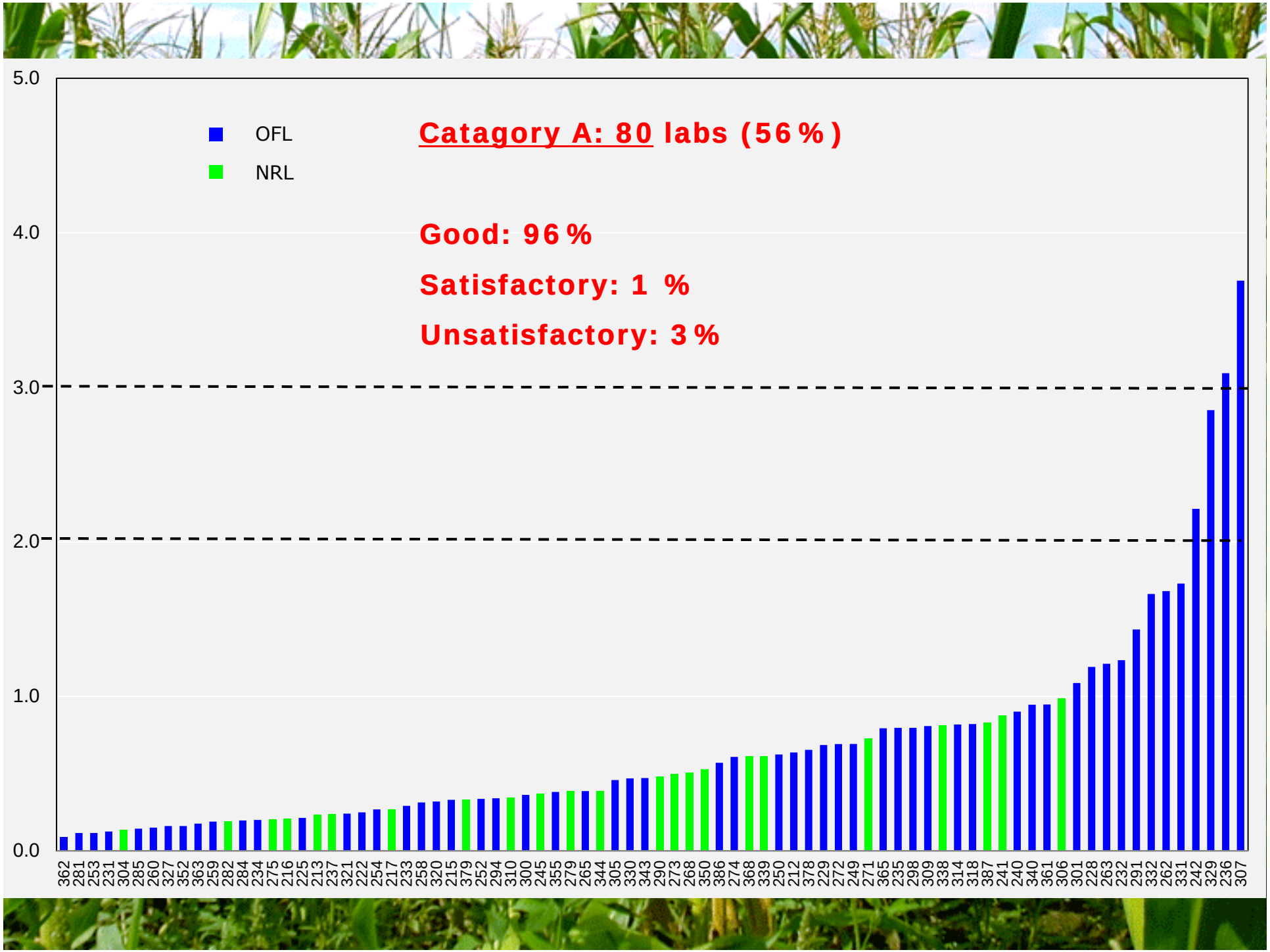
Catagory A laboratories and combined z-scores

- To be classified as Category A laboratory the labs had to report at least 90% of the pesticide present in the test material (≥16 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
- $2 < z \leq 3$ Satisfactory
- $z > 3$ Unsatisfactory


5.0
4.0
3.0
2.0
1.0
0.0
OFL
NRL
Catagory A: 80 labs (56%)
Good: 96%
Satisfactory: 1 %
Unsatisfactory: 3%
362 281 253 231 304 285 260 327 352 363 259 282 284 234 275 216 225 213 237 321 222 254 217 233 258 320 215 379 252 294 310 300 245 355 279 265 344 305 330 343 290 273 268 350 386 274 368 339 250 212 378 229 272 249 271 365 235 298 309 338 314 318 387 241 240 340 361 306 301 228 263 232 291 332 262 331 242 329 236 307

EUPT-CF10

EUPT	Tentative date	Matrix
FV-18	8 February 2016	Spinach
CF-10	7 March 2016	Rye
SRM-11	April 2016	Spinach
AO-11	April-May 2016	Pork fat


QUESTIONS


Merry Christmas and Happy New year