Preliminary Results

European Commission

EURL

European Union Reference Laboratory for Pesticide Residues in Fruits & Vegetables

EUPT-FV-18

European Proficiency Test FV-18

EUPT-Panel

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Participation

Participation

Total No. of Labs = 191	Total No. of Countries = 43
EU/EFTA Labs = 175	EU/EFTA countries = 31
Other countries Labs = 16	Other countries = 12

Participation

Member State	Laboratories
Austria	2
Belgium	7
Bulgaria	3
Croatia	7
Cyprus	2
Czech republic	3
Denmark	2
Estonia	3
Finland	2
France	8
Germany	28
Greece	9
Hungary	4
Iceland	1
Ireland	1

Member State	Laboratories
Italy	21
Latvia	1
LITHUANIA	2
Luxembourg	2
Malta	1
Norwey	1
Poland	13
Portugal	2
Romania	4
Slovakia	2
Slovenia	3
Spain	32
Sweden	2
Switzerland	3
The Netherlands	1
United Kingdom	5

Other Countries	Laboratories
China	3
Colombia	1
Costa Rica	2
Egypt	1
India	1
Indonesia	1
Israel	1
Kenya	1
Panama	1
Serbia	2
Thailand	1
Turkey	1

Target List

190 pesticides

Acephate
Acetamiprid
Acrinathrin
Aldicarb
Aldicarb Sulfone
Aldicarb Sulfoxide
Aldrin
Azinphos-methyl
Azoxystrobin
Benfuracarb
Bifenthrin
Biphenyl
Bitertanol
Boscalid
Bromopropylate
Bromuconazole
Bupirimate
Buprofezin
Cadusafos
Carbaryl
Carbendazim and benomyl
Carbofuran
Carbofuran-3-hydroxy
Carbosulfan
Chlorantraniliprole
Chlorfenapyr
Chlorfenvinphos
Chlorobenzilate
Chlorothalonil
Chlorpropham
Chlorpyrifos
Chlorpyrifos-methyl
Clofentezine
Clothianidin
Cyfluthrin
Cymoxanil
Cypermethrin
Cyproconazole
Cyprodinil
Deltamethrin
Demeton-S-methylsulfone
Diazinon
Dichlofluanid
Dichlorvos
Dicloran
Dicofol
Dieldrin
Diethofencarb
Difenoconazole
Diflubenzuron
Dimethoate
Dimethomorph
DMST
Diniconazole
Diphenylamine
Endosulfan alpha
Endosulfan beta
Endosulfan sulfate
EPN
Epoxiconazole
Ethion
Ethirimol
Ethoprophos
Etofenprox
Famoxadone
Fenamidone
Fenamiphos
Fenamiphos sulfone
Fenamiphos sulfoxide
Fenarimol
Fenazaquin
Fenbuconazole
Fenhexamid
Fenitrothion
Fenoxycarb
Fenpropathrin
Fenpropidin
Fenpropimorph
Fenpyroximate
Fenthion
Fenthion oxon
Fenthion oxon sulfone
Fenthion oxon sulfoxide
Fenthion sulfone
Fenthion sulfoxide
Fenvalerate/Esfenvalerate
Fipronil
Fludioxonil
Flufenoxuron
Fluopicolide
Fluopyram
Fluquinconazole
Flusilazole
Flutolanil
Flutriafol
Fosthiazate
Hexaconazole
Hexythiazox
Imazalil
Imidacloprid

8 New compounds

Target List

Indoxacarb
Iprodione
Iprovalicarb
Isocarbophos
Isofenphos-methyl
Isoprothiolane
Kresoxim-methyl
Lambda-Cyhalothrin
Linuron
Lufenuron
Malaoxon
Malathion
Mandipropamid
Mepanipyrim
Metaflumizone
Metalaxyl and metalaxyl-M
Metconazole
Methamidophos
Methidathion
Methiocarb
Methiocarb sulfone
Methiocarb sulfoxide
Methomyl
Methoxyfenozide
Monocrotophos
Myclobutanyl
Omethoate
Orthophenylphenol
Oxadixyl
Oxamyl
Oxydemeton-methyl
Paclobutrazole
Paraoxon-methyl
Parathion-ethyl
Parathion-methyl
Penconazole
Pencycuron
Pendimethalin
Permethrin
Phenthoate
Phosalone
Phosmet
Phosmet oxon
Phoxim
Pirimicarb
Pirimicarb-desmethyl
Pirimiphos-methyl
Prochloraz
Procymidone
Profenofos
Propamocarb
Propargite
Propiconazole
Propyzamide
Prothioconazole
Prothiofos
Pyraclostrobin
Pyridaben
Pyrimethanil
Pyriproxyfen
Quinoxyfen
Spinosad
Spirodiclofen
Spiroxamine
Tau-Fluvalinate
Tebuconazole
Tebufenozide
Tebufenpyrad
Teflubenzuron
Tefluthrin
Terbuthylazine
Tetraconazole
Tetradifon
Thiabendazole
Thiacloprid
Thiamethoxam
Thiodicarb
Thiophanate-methyl
Tolclofos-methyl
Tolylfluanid
Triadimefon
Triadimenol
Triazophos
Trichlorfon
Trifloxystrobin
Triflumuron
Trifluralin
Triticonazole
Vinclozolin
Zoxamide

8 New compounds

Voluntary Target List

25 pesticides

Working Document SANCO/12745/2013

(Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin)

- Ametoctradin
- Benalaxyl including other mixtures of constituent isomers including benalaxyl-M (sum of isomers)
- Benzovindiflupyr
- Clomazone
- Cyazofamid
- Emamectin benzoate B1a, expressed as emamectin
- Etoxazole
- Fenpyrazamine
- Fluxapyroxad
- Heptachlor
- Heptachlor epoxide
- Isopyrazam
- Penflufen
- Penthiopyrad
- Prosulfocarb
- Pyrethrins
- Quintozene
- Pentachloro-aniline
- Rotenone
- Spirotetramat
- Spirotetramat metabolite BYI08330-enol
- Spirotetramat metabolite BYI08330-ketohydroxy
- Spirotetramat metabolite BYI08330-monohydroxy
- Spirotetramat metabolite BYI08330 enol-glucoside
- Tetramethrin

Pesticides used for the treatment

Chlorantraniliprole*	Fluopyram
Cymoxanil*	Imidacloprid
Cyazofamid**	Indoxacarb
Difenoconazole	Metalaxyl and Metalaxyl-M
Diflubenzuron	Omethoate***
Dimethoate	Penconazole
Famoxadone*	Thiacloprid
Fenpyrazamine**	Triadimenol
Total: 16	

* New in the target list

*** Not spiked

** Voluntary pesticides

Calendar

ACTIVITY	DATE
- Publishing the Target Pesticide List, Calendar and Matrix on the Web page.	9th November 2015
- Receiving Application Form from invited laboratories.	1st Dec-11th Jan 2016
- Specific Protocol published on the Web site.	25th January 2016 at the latest
- Deadline for receiving Laboratory scope: Form 0	25th Jan-5th Feb 2016
- Sample distribution.	8th February 2016
- Deadline for receiving sample acceptance: Form 1	12th February 2016
- Deadline for receiving results: Forms 2, 3 and 4	1st March 2016
- Filling in Form 5	7th-11th March 2016
- Preliminary Report: only results, no statistical treatment.	Last week of March 2016
- Final Report distributed to the Laboratories.	December 2016

EUPT-FV-18

European Proficiency Test FV-18

Spinach

Organic spinachs were grown in a greenhouse in Almería, Spain.

Before harvest, the spinachs were treated with commercial formulations

Preparation of the test item

Homogeneity

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

Stability

1st Analysis - prior to the sample shipment

2nd Analysis - after the deadline for reporting results

3rd Analysis - reproducing the delivery conditions that the samples experienced during 48 hours

All the pesticides passed the homogeneity test

All the pesticides passed the stability test

Results

Assigned values

Mandatory Pesticides

	Robust Mean X* (mg/kg)
Chlorantraniliprole	3.071
Cymoxanil	0.011+
Difenoconazole	0.837
Diflubenzuron	1.121
Dimethoate	0.036
Famoxadone	1.026
Fluopyram	0.289
Imidacloprid	0.250
Indoxacarb (sum of indoxacarb and its R enantiomer)	1.436
Metalaxyl and metalaxyl-M	0.058
Omethoate	0.142
Penconazole	0.016+
Thiacloprid	0.516
Triadimenol	0.205

Voluntary Pesticides

	Robust Mean X* (mg/kg)
Cyazofamid	0.279
Fenpyrazamine	0.357

Assigned values

Voluntary Pesticides

0.011-0.100 mg/kg

	Robust Mean X* (mg/kg)
Cymoxanil	0,011
Penconazole	0,016
Dimethoate	0,036
Metalaxyl and metalaxyl-M	0,058
Omethoate	0,142
Triadimenol	0,205
Imidacloprid	0,250
Cyazofamid	0,279
Fluopyram	0,289
Fenpyrazamine	0,357
Thiacloprid	0,516
Difenoconazole	0,837
Famoxadone	1,026
Diflubenzuron	1,121
Indoxacarb (sum of indoxacarb and its R enantiomer)	1,436
Chlorantraniliprole	3,071

Assigned values

Voluntary Pesticides

0.011-0.100 mg/kg

0.100-0.500 mg/kg

	Robust Mean X* (mg/kg)
Cymoxanil	0,011
Penconazole	0,016
Dimethoate	0,036
Metalaxyl and metalaxyl-M	0,058
Omethoate	0,142
Triadimenol	0,205
Imidacloprid	0,250
Cyazofamid	0,279
Fluopyram	0,289
Fenpyrazamine	0,357
Thiacloprid	0,516
Difenoconazole	0,837
Famoxadone	1,026
Diflubenzuron	1,121
Indoxacarb (sum of indoxacarb and its R enantiomer)	1,436
Chlorantraniliprole	3,071

Assigned values

Voluntary Pesticides

0.011-0.100 mg/kg

0.100-0.500 mg/kg

> 0.500 mg/kg

	Robust Mean X* (mg/kg)
Cymoxanil	0,011
Penconazole	0,016
Dimethoate	0,036
Metalaxyl and metalaxyl-M	0,058
Omethoate	0,142
Triadimenol	0,205
Imidacloprid	0,250
Cyazofamid	0,279
Fluopyram	0,289
Fenpyrazamine	0,357
Thiacloprid	0,516
Difenoconazole	0,837
Famoxadone	1,026
Diflubenzuron	1,121
Indoxacarb (sum of indoxacarb and its R enantiomer)	1,436
Chlorantraniliprole	3,071

	MRRL (mg/kg)	Robust Mean X* (mg/kg)	CVs*(%)
Chlorantraniliprole	0.01	3.071	18.4
Cyazofamid	0.01	0.279	22.0
Difenoconazole	0.01	0.837	17.5
Diflubenzuron	0.01	1.121	18.1
Dimethoate	0.003	0.036	17.1
Famoxadone	0.01	1.026	15.3
Fenpyrazamine	0.01	0.357	14.8
Fluopyram	0.01	0.289	14.2
Imidacloprid	0.01	0.250	12.8
Indoxacarb (sum of indoxacarb and its R enantiomer)	0.01	1.436	19.1
Metalaxyl and metalaxyl-M	0.01	0.058	16.5
Omethoate	0.003	0.142	24.9
Thiacloprid	0.01	0.516	12.5
Triadimenol	0.01	0.205	16.6

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 171)
Chlorantraniliprole	118	2	51	69
Cyazofamid	89	3	79	52
Difenoconazole	155	2	14	91
Diflubenzuron	119	2	50	70
Dimethoate	162	3	6	95
Famoxadone	123	5	43	72
Fenpyrazamine	39	1	131	23
Fluopyram	111	3	57	65
Imidacloprid	146	1	24	85
Indoxacarb (sum of indoxacarb and its R enantiomer)	145	3	23	85
Metalaxyl and metalaxyl-M	156	3	12	91
Omethoate	147	3	21	86
Thiacloprid	144	1	26	84
Triadimenol	149	6	16	87

Pesticides	No. of Reported Results	No. of False Negative Results	No. of Not Analysed Results	Percentage of Labs Reporting Results (out of 171)
Chlorantraniliprole	118	2	51	69
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Famoxadone	123	5	43	72
Fenpyrazamine	39	1	131	23
Fluopyram	111	3	57	65
Imidacloprid	146	1	24	85
Indoxacarb (sum of indoxacarb and its R enantiomer)	145	3	23	85
Metalaxyl and metalaxyl-M	156	3	12	91
Omethoate	147	3	21	86
Thiacloprid	144	1	26	84
Triadimenol	149	6	16	87

z-Scores

Z Scores classification

Acceptable **Questionable** **Unacceptable**

Pesticides	Robust Mean (mg/kg)	% Acceptable z-scores	% Questionable z-scores	% Unacceptable z-scores
Chlorantraniliprole	3.1	93.3	1.7	5.0
Difenoconazole	0.84	95.5	1.3	3.2
Cyazofamid	0.28	88.0	0.0	12.0
Diflubenzuron	1.1	88.3	2.5	9.2
Dimethoate	0.04	95.2	3.0	1.8
Famoxadone	1.0	90.6	2.4	7.1
Fenpyrazamine	0.36	95.0	2.5	2.5
Fluopyram	0.29	93.9	3.5	2.6
Imidacloprid	0.25	95.2	4.1	0.7
Indoxacarb	1.4	92.6	3.4	4.1
Metalaxyl	0.06	93.7	1.3	5.0
Omethoate	0.14	87.3	7.3	5.3
Thiacloprid	0.52	97.9	1.4	0.7
Triadimenol	0.21	92.9	1.9	5.2

Chlorantraniliprole

EU/EFTA Laboratories

Difenoconazole

EU/EFTA Laboratories

Diflubenzuron

EU/EFTA Laboratories

Dimethoate

EU/EFTA Laboratories

Famoxadone

EU/EFTA Laboratories

Fluopyram

EU/EFTA Laboratories

Imidacloprid

EU/EFTA Laboratories

Indoxacarb

EU/EFTA Laboratories

Metalaxyl and Metalaxyl-M

Omethoate

EU/EFTA Laboratories

Thiacloprid

EU/EFTA Laboratories

Triadimenol

EU/EFTA Laboratories

Voluntary Componds

Cyazofamid

EU/EFTA Laboratories

Fenpyrazamine

EU/EFTA Laboratories

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.**

AZ² - Graphical Representation

Preliminary False Positives
(Pending evaluation by the Scientific Committee)

10 different laboratories from EU/EFTA countries reported **12** pesticides as false positives

Preliminary False Positives

(Pending evaluation by the Scientific Committee)

EU/EFTA Laboratories

Laboratory Code	Pesticide	Concentration (mg/kg)	Determination Technique	RL (mg/Kg)	MRRL (mg/Kg)
Lab164	Chlorpropham	0.513	GC-MS/MS	0.01	0.01
Lab083	Demeton-S-methylsulfone	0.043	LC-MS/MS	0.025	0.006
Lab070	Dicofol (sum of p, p' and o,p' isomers)	0.013	GC-MS/MS	0.01	0.01
Lab031	Diphenylamine	0.01	GC-MS/MS	0.01	0.01
Lab177	Endosulfan alpha	0.01	GC-ECD	0.01	0.01
Lab177	Endosulfan sulfate	0.04		0.01	0.01
Lab031	Flutolanil	0.05	GC-MS/MS	0.01	0.01
Lab047	Flutolanil	0.035	GC-MS	0.02	0.01
Lab077	Flutolanil	0.161	GC-MS/MS	0.01	0.01
Lab162	Flutolanil	0.107	GC-MS/MS	0.01	0.01
Lab042	Orthophenylphenol	0.126	GC-MS	0.01	0.01
Lab034	Triadimefon	0.21	GC-MS/MS	0.05	0.01

Preliminary False Positives

(Pending evaluation by the Scientific Committee)

GC-ECD/-MS or GC-MS/MS

- Chlorpropham
- Dicofol (sum of p, p' and o,p' isomers)
- Diphenylamine
- Endosulfan alpha
- Endosulfan sulfate
- Flutolanil
- Orthophenylphenol
- Triadimefon

LC-MS/MS

- Demeton-S-methylsulfone

Chlorantraniliprole Fluopyram Cymoxanil
Imidacloprid Cyazofamid Indoxacarb
Difenoconazole Metalaxyl Diflubenzuron
Omethoate Dimethoate Penconazole
Famoxadone Thiacloprid Fenpyrazamine
Triadimenol

Fenamiphos sulfone
Fenamiphos sulfoxide
Tetraconazole
Thiabendazole
Spiromesifen
…

Concentrations below MRRL

EUROPEAN COMMISSION PROFICIENCY TEST FOR PESTICIDE RESIDUES IN FRUITS AND VEGETABLES

SCREENING METHODS 08

RESULTS

Preparation of the test item

ACTIVITY	DATE
Publishing the Calendar and Matrix on the Web page.	9th December 2015
Receiving Application Form from invited laboratories.	1st December 2015 - 11th January 2016
Specific Protocol published on the Web site.	25th January 2016 at the latest
Sample distribution.	8th February 2016
Deadline for receiving results: Fill in "Results Page"	**72 hours after receiving the sample**
Preliminary Report: only results, no statistical treatment.	Last week of March 2016
Final Report distributed to the Laboratories.	December 2016

Participation

EUPT-FV-SM08- Participants		83 Participants	
COUNTRY	**No.**	**COUNTRY**	**No.**
Austria	1	India	1
Belgium	3	Ireland	1
Bulgary	1	Italy	9
China	3	Kenia	1
Croatia	1	Latvia	1
Cyprus	1	Norway	1
Czech Republic	3	Romania	1
Denmark	1	Serbia	1
Estonia	1	Slovenia	1
Finland	1	Spain	16
France	6	Sweden	2
Germany	15	Switzerland	1
Greece	1	The Netherlands	3
Hungary	3	UK	3

Participation

24 EU/EFTA Countries

- Austria
- Belgium
- Bulgary
- Croatia
- Cyprus
- Czech Republic
- Denmark
- Estonia
- Finland
- France
- Germany
- Greece
- Hungary
- Italy
- Ireland
- Latvia
- Netherlands
- Norway
- Romania
- Serbia
- Slovenia
- Spain
- Sweden
- Switzerland
- United Kingdom

4 Non EU/EFTA Countries

- China
- India
- Kenya
- Serbia

Pesticides used for the treatment

Benalaxyl
Chlozolinate
Clomazone
Cyazofamid
Fenpyrazamine
Heptachlor
Isopyrazam
Phenthoate
Prosulfocarb
Prothiofos
Pyrethrin
Quintozene
Rotenone
Tetramethrin
Triticonazole

Pesticides used for the treatment

Pesticides	Robust mean (mg/kg)	CV (%)
Benalaxyl	0.052	20.3
Chlozolinate	0.047	42.4
Clomazone	0.075	25.0
Cyazofamid	0.070	33.3
Fenpyrazamine	0.049	17.7
Heptachlor	0.061	38.1
Isopyrazam	0.074	23.2
Phenthoate	0.068	24.0
Prosulfocarb	0.051	22.4
Prothiofos	0.076	24.7
Pyrethrin	0.222	
Quintozene	0.063	34.0
Rotenone	0.073	22.5
Tetramethrin	0.068	30.4
Triticonazole	0.055	20.7

RESULTS

Results

15 Evaluated Pesticides
83 Laboratories

15 Pesticides= 2+2+11

	Benalaxyl	Chlozolinate	Clomazone	Cyazofamid	Fenpyrazamine	Heptachlor	Isopyrazam	Phenthoate	Prosulfocarb	Prothiofos	Pyrethrin	Quintozene	Rotenone	Tetramethrin	Triticonazole
Total Number of Reported Pesticides	78	53	71	62	28	71	33	76	71	71	53	60	64	71	69
% of Reported Pesticides	94	64	86	75	34	86	40	92	86	86	64	72	77	86	83

Results

SM08 % of Reported Pesticides

(83 Laboratories Reported Data)

Results

SM08 % of Reported Pesticides
(15 Pesticides 83 Laboratories)
84%
67%
36%
100
90
80
70
60
50
40
30
20
10
0

Results

Pesticides reported by 3 or more laboratories:

- Azoxystrobin
- Difenoconazole
- Endosulfan sulfate
- Fenamiphos sulfone
- Fenamiphos sulfoxide
- Hexachlorobenzene
- Indoxacarb
- Metconazole
- Tecnazene
- Tetraconazole
- Thiabendazole

Azoxystrobin (LC-MS/MS)

Standard in spinach at 0.01 mg/kg

EUPT-SM Sample

Ratio SRM2/SRM1: 13.4
Conc < 0.01 mg/kg

Difenoconazole (LC-MS/MS)

Standard in spinach at 0.01 mg/kg

EUPT-SM Sample

Ratio SRM2/SRM1: 29.2
Conc < 0.01 mg/kg

Fenamiphos sulfone (LC-MS/MS)

Standard in spinach at 0.01 mg/kg

EUPT-SM Sample

Ratio SRM2/SRM1: 58.6
Conc < 0.01 mg/kg

Fenamiphos sulfoxide (LC-MS/MS)

Standard in spinach at 0.01 mg/kg

EUPT-SM Sample

Ratio SRM2/SRM1: 37.2
Conc < 0.01 mg/kg

Indoxacarb (LC-MS/MS)

Standard in spinach at 0.01 mg/kg

EUPT-SM Sample

Ratio SRM2/SRM1: 104.4
Conc < 0.01 mg/kg

Tecnazene (GC-MS/MS)

Standard in spinach at 0.01 mg/kg

EUPT-SM Sample

Ratio SRM2/SRM1: 35.6
Conc < 0.01 mg/kg

Tetraconazole (LC-MS/MS)

Standard in spinach at 0.01 mg/kg

EUPT-SM Sample

Ratio SRM2/SRM1: 66.1
Conc < 0.01 mg/kg

Thiabendazole (LC-MS/MS)

Standard in spinach at 0.01 mg/kg

EUPT-SM Sample

Ratio SRM2/SRM1: 71.8
Conc < 0.01 mg/kg

Analytical Techniques

Analytical Techniques

| LAB. CODE | Lab001 | Lab002 | Lab003 | Lab004 | Lab005 | Lab006 | Lab007 | Lab008 | Lab011 | Lab012 | Lab013 | Lab014 | Lab015 | Lab016 | Lab018 | Lab019 | Lab020 | Lab021 | Lab022 | Lab023 | Lab024 | Lab025 | Lab026 | Lab027 | Lab029 | Lab030 | Lab031 | Lab032 | Lab033 | Lab034 | Lab035 | Lab036 | Lab037 | Lab038 | Lab039 | Lab040 | Lab041 | Lab042 | Lab043 | Lab044 | Lab045 | Lab046 | Lab047 | Lab048 | Lab049 | Lab050 | Lab051 | Lab052 | Lab053 | Lab054 | Lab056 | Lab057 | Lab059 | Lab060 | Lab061 | Lab062 | Lab063 | Lab064 | Lab065 | Lab066 | Lab067 | Lab068 | Lab069 | Lab070 | Lab071 | Lab072 | Lab073 | Lab074 | Lab075 | Lab076 | Lab077 | Lab078 | Lab079 | Lab080 | Lab081 | Lab082 | Lab083 | Lab084 | Lab085 | Lab086 | Lab087 | Lab089 | Lab090 |
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Thank You
for Your Attention

EURL EUROPEAN UNION REFERENCE LABORATORY