



# Interlaboratory Study on Pesticide Residues in Fish

ILS F-01 (2024)

## Preliminary Report

Version 1.1

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## Document history

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# 1 GENERAL REMARKS

- Transcription errors by the organisers cannot be excluded. Therefore, all labs are kindly requested to **check their results carefully** and **to report any errors**. However, as robust statistic was applied to get the assigned values the influence of possible transcription errors is low (possible changes are in the range of  $\pm 0.1$  z-scores). Please note that only transcription errors by the organisers will be corrected.
- **Poor performance** (i.e. individual z-scores  $>3.0$  or false positive/negative results): **All NRLs and OFLs are requested to give feedback to the organiser by 25 October 2024** on any actions undertaken to find out the reasons for poor performance. For reporting, please use the excel-file attached to the e-mail with the preliminary report.

## 2 BACKGROUND INFORMATION

The test matrix of ILS F-01 was fish. PT items were prepared of salmon obtained from Gertrud-Luckner-Gewerbeschule (Freiburg, Germany) and contained eleven pesticides from the list of mandatory analytes and five voluntary pesticides (see **Table 1**). All target analytes and their concentrations were selected by EURL-AO members together with the EUPT Quality Control Group. Participating laboratories were provided with approximately 100 g portions of the prepared PT item.

**Table 1:** Overview of analytes present in the ILS F-01 PT item.

Mandatory / voluntary	Analyte	CAS number of compound spiked in lab
mandatory	Chlordane, cis-	5103-71-9
mandatory	Chlorpyrifos(-ethyl)	2921-88-2
mandatory	DDE, p,p'-	72-55-9
mandatory	Deltamethrin, cis-	52918-63-5
mandatory	Diazinon	333-41-5
mandatory	Endosulfan, beta-	33213-65-9
mandatory	Heptachlorepoxyd, cis-	1024-57-3
mandatory	Hexachlorcyclohexane (HCH), gamma-isomer (Lindane)	58-89-9
mandatory	Indoxacarb (sum of isomers)	173584-44-6
mandatory	Pendimethalin	40487-42-1
voluntary	Permethrin (sum of isomers)	52645-53-1
voluntary	Chlorate (Na-)	7775-09-9
voluntary	Didecyldimethylammoniumchlorid (DDAC-C10)	7173-51-5
voluntary	Ethoxyquin dimer, metabolite of ethoxyquin	74681-77-9
voluntary	Fluopyram	658066-35-4
voluntary	Teflubenzuron	83121-18-0

## 3 RESULTS

In total, 38 laboratories registered for participation in ILS F-01. 37 laboratories submitted results for at least one compound. For statistical evaluation only results from laboratories of EU and EFTA countries (34 laboratories) were taken into account.

### 3.1 Summary

A summary of the assigned values and robust standard deviations (RSD) is given in the following **Table 2**.

**Table 2:** Summary of reported results by all laboratories. Assigned values and robust standard deviations (RSD) are calculated based on results of labs from EU and EFTA countries (n=34), only. The percentage of reported results includes the number of acceptable, questionable, and unacceptable results reported for each compound by the total number of participating laboratories. False negatives are included in unacceptable results.

Pesticide	Assigned value (mg/kg)	CV*(%)	No. of reported results	No. of acceptable results	No. of questionable results	No. of unacceptable results	No. of false negative results	No. of not analysed results	% of reported results	% of acceptable results	% of questionable results	% of unacceptable results
<b>Mandatory</b>												
Chlordane, cis-	0.044	32.2%	37	32	4	1	0	0	67%	86.5%	10.8%	2.7%
Chlorpyrifos	0.086	22.7%	36	34	2	0	0	1	69%	94.4%	5.6%	0.0%
DDE, p,p-	0.028	35.4%	37	32	3	2	0	0	69%	86.5%	8.1%	5.4%
Deltamethrin	0.111	37.1%	37	30	5	2	0	0	62%	81.1%	13.5%	5.4%
Diazinon	0.065	16.5%	35	32	1	2	1	2	72%	91.4%	2.9%	5.7%
Endosulfan, beta-	0.022	33.3%	37	30	4	3	2	0	40%	81.1%	10.8%	8.1%
Heptachlorepoxide, cis-	0.042	29.8%	37	34	2	1	0	0	69%	91.9%	5.4%	2.7%
HCH, gamma-	0.029	22.4%	37	34	1	2	0	0	72%	91.9%	2.7%	5.4%
Indoxacarb	0.126	12.8%	30	29	1	0	0	7	81%	96.7%	3.3%	0.0%
Pendimethalin	0.075	24.1%	34	32	2	0	0	3	71%	94.1%	5.9%	0.0%
Permethrin (sum)	0.057	28.6%	37	32	3	2	0	0	60%	86.5%	8.1%	5.4%
<b>Voluntary</b>												
Chlorate	0.357	22.0%	9	7	0	2	2	28	155%	77.8%	0.0%	22.2%
DDAC-C10	0.535	3.6%	11	9	1	1	1	26	289%	81.8%	9.1%	9.1%
Ethoxyquin, C-N-Dimer	-	-	2	-	-	-	-	35	-	-	-	-
Fluopyram	0.111	12.0%	24	22	2	0	0	13	89%	91.7%	8.3%	0.0%
Teflubenzuron	0.149	30.6%	21	18	1	2	0	16	79%	85.7%	4.8%	9.5%

## 3.2 False positives

Seven laboratories (including non-EU countries) reported in total nine tentative false positive results (**Table 3**). *Results below the MRRL will not be considered as false positive in the final report.*

**Table 3:** Laboratories that reported results for pesticides that were not present in the treated PT item (false positives). RL = reporting level, MRRL = minimum required reporting level

Lab code	Pesticide	Concentration submitted by participants (mg/kg)	RL (mg/kg)	MRRL (mg/kg)	Determination technique
8	Endosulfan, alpha-	0.0063	0.005	0.005	GC-MSMS (QQQ)
8	Oxychlorane	0.0056	0.01	0.005	GC-MSMS (QQQ)
9	Benzalkonium Chloride (C8,C10,C12,C14,C16,C18)	0.01	0.01	0.010	LC-MSMS (QQQ)
10	Endosulfan, alpha-	0.007	0.005	0.005	GC-MSMS (QQQ)
15	Benzalkonium Chloride (C8,C10,C12,C14,C16,C18)	0.0105	0.01	0.010	LC-Q-Orbitrap
21	Benzalkonium Chloride (C8,C10,C12,C14,C16,C18)	0.011	0.01	0.010	LC-MSMS
24	Benzalkonium Chloride (C8,C10,C12,C14,C16,C18)	0.0325	0.01	0.010	LC-MSMS (QQQ)
24	Oxychlorane	0.00532	0.005	0.005	GC-MSMS (QQQ)
39	Endosulfan, alpha-	0.01	0.01	0.005	GC-MSMS (QQQ)

## 3.3 False negatives

Six laboratories (including non-EU countries) reported in total six tentative false negative (FN) results (**Table 4**).



**Table 4:** Laboratories that did not report results for pesticides that were present in the treated PT item (false negatives (FN)). Voluntary analytes are marked with an asterisk (\*).

Lab code	Chlordane, cis-	Chlorpyrifos	DDE, p,p-	Deltamethrin	Diazinon	Endosulfan, beta-	Heptachlorepoxide, cis-	HCH, gamma-	Indoxacarb	Pendimethalin	Permethrin (sum)	Chlorate*	DDAC-C10*	Ethoxyquin, C-N-Dimer*	Fluopyram*	Teflubenzuron*
9												FN				
19						FN										
20													FN			
21												FN				
28						FN										
32					FN											

### 3.4 Individual submitted concentrations and assigned values, robust standard deviations and z-scores

Individual submitted concentrations by participants (including non-EU countries), assigned values, robust standard deviations and z-scores for the mandatory pesticides cis-Chlordane, Chlorpyrifos, p,p-DDE, Deltamethrin, Diazinon, beta-Endosulfan, cis-Heptachlorepoxide, gamma-HCH, Indoxacarb and Pendimethalin are given in the following **Table 5**.

Individual submitted concentrations by participants (including non-EU countries), assigned values, robust standard deviations and z-scores for the mandatory pesticide Permethrin (sum) and the voluntary pesticides Chlorate, DDAC-C10, Ethoxyquin (C-N-Dimer), Fluopyram and Teflubenzuron are given in **Table 6**.

**Table 5:** Results reported for the mandatory pesticides cis-Chlordane, Chlorpyrifos, p,p-DDE, Deltamethrin, Diazinon, beta-Endosulfan, cis-Heptachlorepoide, gamma-HCH, Indoxacarb and Pendimethalin in mg/kg, the preliminary z-scores using a fit-for-purpose relative standard deviation of 25% (FFP-RSD 25%), the MRRLs in mg/kg and the assigned values and robust relative standard deviations (RSD) in mg/kg.

Lab code	Chlordane, cis-	z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		
MRRL	0.005	Chlorpyrifos		DDE, p,p-		Deltamethrin		Diazinon		Endosulfan, beta-		Heptachlorepoide, cis-		HCH, gamma-		Indoxacarb		Pendimethalin		
Assign. value	0.044	0.010		0.005		0.010		0.010		0.005		0.005		0.005		0.010		0.010		
Robust RSD	0.014	0.086		0.028		0.111		0.065		0.022		0.042		0.029		0.126		0.075		
Number of results	33	0.020		0.010		0.041		0.011		0.007		0.012		0.006		0.016		0.018		
		32		33		33		32		33		33		33		27		31		
2	0.033	-1.0	0.06	-1.03	0.02	-1.53	0.07	-1.37	0.06	-0.56	0.02	-0.92	0.03	-0.83	0.03	-0.23	0.12	-0.29	0.05	-1.27
3	0.049	0.5	0.088	0.1	0.034	1.0	0.117	0.2	0.058	-0.5	0.024	0.3	0.045	0.3	0.031	0.3	0.150	0.8	0.073	-0.1
4	0.050	0.6	0.105	0.9	0.035	1.1	0.335	5.0	0.080	0.9	0.026	0.7	0.048	0.6	0.031	0.3	0.121	-0.2	0.107	1.7
5	0.033	-1.0	0.079	-0.3	0.015	-1.8	0.112	0.0	0.060	-0.3	0.019	-0.6	0.035	-0.6	0.026	-0.4	0.119	-0.2	0.065	-0.5
6	0.035	-0.8	0.077	-0.4	0.027	-0.1	0.110	0.0	0.060	-0.3	0.021	-0.2	0.042	0.0	0.026	-0.4	na	na	0.066	-0.5
7	0.008	-3.3	na	na	0.005	-3.3	0.080	-1.1	na	na	0.003	-3.5	0.007	-3.3	0.006	-3.1	na	na	na	na
8	0.063	1.7	0.106	0.9	0.038	1.6	0.156	1.6	0.073	0.5	0.028	1.0	0.053	1.0	0.036	1.0	0.137	0.3	0.091	0.9
9	0.051	0.7	0.090	0.2	0.034	0.9	0.079	-1.1	0.070	0.3	0.020	-0.4	0.040	-0.2	0.020	-1.2	0.133	0.2	0.053	-1.2
10	0.019	-2.3	0.057	-1.4	0.022	-0.8	0.063	-1.7	0.062	-0.2	0.016	-1.1	0.030	-1.1	0.028	-0.1	na	na	0.062	-0.7
11	0.060	1.5	0.076	-0.5	0.033	0.8	0.125	0.5	0.062	-0.2	0.027	0.9	0.062	1.9	0.038	1.3	0.101	-0.8	0.066	-0.5
12	0.050	0.6	0.095	0.4	0.033	0.8	0.145	1.2	0.067	0.1	0.019	-0.6	0.045	0.3	0.029	0.1	0.122	-0.1	0.098	1.3

Lab code	Chlordane, cis-		Chlorpyrifos		DDE, p,p-		Deltamethrin		Diazinon		Endosulfan, beta-		Heptachlorepoide, cis-		HCH, gamma-		Indoxacarb		Pendimethalin	
		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)
MRRL	0.005		0.010		0.005		0.010		0.010		0.005		0.005		0.005		0.010		0.010	
Assign. value	0.044		0.086		0.028		0.111		0.065		0.022		0.042		0.029		0.126		0.075	
Robust RSD	0.014		0.020		0.010		0.041		0.011		0.007		0.012		0.006		0.016		0.018	
Number of results	33		32		33		33		32		33		33		33		27		31	
<b>13</b>	0.033	-1.0	0.064	-1.0	0.028	0.0	0.094	-0.6	0.055	-0.6	0.016	-1.1	0.030	-1.1	0.029	0.0	0.107	-0.6	0.056	-1.0
<b>14</b>	0.047	0.3	0.097	0.5	0.032	0.6	0.121	0.4	0.071	0.4	0.023	0.2	0.039	-0.2	0.030	0.2	0.132	0.2	0.078	0.2
<b>15</b>	0.037	-0.7	0.096	0.4	0.030	0.3	0.152	1.5	0.040	-1.5	0.023	0.2	0.036	-0.5	0.018	-1.5	0.128	0.1	0.062	-0.7
<b>16</b>	0.030	-1.3	0.078	-0.4	0.020	-1.1	0.083	-1.0	0.063	-0.1	0.020	-0.4	0.038	-0.4	0.025	-0.5	0.115	-0.4	0.068	-0.4
<b>17</b>	0.044	0.0	0.093	0.3	0.024	-0.5	0.129	0.7	0.062	-0.2	0.019	-0.7	0.044	0.2	0.029	0.0	0.125	0.0	0.081	0.3
<b>18</b>	0.045	0.1	0.081	-0.2	0.023	-0.7	0.121	0.4	0.053	-0.7	0.016	-1.2	0.036	-0.6	0.037	1.2	0.101	-0.8	0.068	-0.4
<b>19</b>	0.072	2.6	0.095	0.4	0.033	0.7	0.117	0.2	0.075	0.6	nd	-4.0	0.071	2.8	0.048	2.7	0.156	0.9	0.074	0.0
<b>20</b>	0.040	-0.3	0.083	-0.1	0.026	-0.2	0.012	-3.6	0.069	0.2	0.016	-1.1	0.040	-0.2	0.023	-0.8	0.118	-0.3	0.089	0.8
<b>21</b>	0.047	0.3	0.097	0.5	0.028	0.1	0.090	-0.8	0.068	0.2	0.022	0.0	0.044	0.2	0.027	-0.2	0.130	0.1	0.081	0.3
<b>22</b>	0.053	0.8	0.100	0.6	0.035	1.1	0.152	1.5	na	na	0.029	1.3	0.057	1.5	0.032	0.4	na	na	na	na
<b>23</b>	0.057	1.2	0.103	0.8	0.049	3.1	0.174	2.3	0.076	0.7	0.034	2.1	0.060	1.7	0.038	1.3	0.174	1.5	0.090	0.8
<b>24</b>	0.063	1.7	0.101	0.7	0.038	1.5	0.137	0.9	0.064	-0.1	0.025	0.5	0.055	1.3	0.036	1.0	0.126	0.0	0.080	0.3
<b>25</b>	0.044	0.1	0.083	-0.2	0.035	1.1	0.121	0.4	0.043	-1.3	0.030	1.4	0.052	0.9	0.026	-0.4	0.109	-0.5	0.077	0.1
<b>26</b>	0.038	-0.5	0.080	-0.3	0.024	-0.6	0.083	-1.0	0.069	0.2	0.017	-0.9	0.043	0.1	0.030	0.2	0.138	0.4	0.069	-0.3
<b>27</b>	0.030	-1.3	0.104	0.8	0.019	-1.2	0.109	-0.1	0.067	0.1	0.023	0.2	0.025	-1.6	0.024	-0.6	0.131	0.2	0.097	1.2

Lab code	Chlordane, cis-		Chlorpyrifos		DDE, p,p-		Deltamethrin		Diazinon		Endosulfan, beta-		Heptachlorepoide, cis-		HCH, gamma-		Indoxacarb		Pendimethalin	
		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)		z-score (FFP-RSD 25%)
MRRL	0.005		0.010		0.005		0.010		0.010		0.005		0.005		0.005		0.010		0.010	
Assign. value	0.044		0.086		0.028		0.111		0.065		0.022		0.042		0.029		0.126		0.075	
Robust RSD	0.014		0.020		0.010		0.041		0.011		0.007		0.012		0.006		0.016		0.018	
Number of results	33		32		33		33		32		33		33		33		27		31	
<b>28</b>	0.041	-0.3	0.116	1.4	0.024	-0.5	0.152	1.5	0.080	0.9	nd	-4.0	0.046	0.4	0.032	0.5	0.149	0.7	0.095	1.1
<b>29</b>	0.060	1.5	0.112	1.2	0.041	1.9	0.114	0.1	0.078	0.8	0.031	1.6	0.051	0.9	0.039	1.5	0.154	0.9	0.093	1.0
<b>31</b>	0.028	-1.4	0.038	-2.2	0.019	-1.2	0.036	-2.7	0.100	2.1	0.013	-1.6	0.022	-1.9	0.032	0.5	0.042	-2.7	0.033	-2.2
<b>32</b>	0.047	0.3	0.057	-1.4	0.022	-0.8	0.076	-1.3	nd	-4.0	0.011	-1.9	0.042	0.0	0.023	-0.9	na	na	na	na
<b>33</b>	0.016	-2.5	0.029	-2.7	0.009	-2.7	0.033	-2.8	0.017	-3.0	0.006	-2.9	0.012	-2.8	0.006	-3.2	na	na	0.024	-2.7
<b>34</b>	0.037	-0.6	0.057	-1.4	0.018	-1.4	0.042	-2.5	0.068	0.1	0.015	-1.3	0.021	-2.0	0.025	-0.6	0.132	0.2	0.040	-1.9
<b>35</b>	0.043	-0.1	0.082	-0.2	0.022	-0.8	0.087	-0.9	0.063	-0.1	0.034	2.2	0.039	-0.3	0.036	1.0	0.133	0.2	0.078	0.2
<b>36</b>	0.067	2.1	0.108	1.0	0.044	2.4	0.132	0.8	0.050	-0.9	0.030	1.4	0.056	1.4	0.031	0.3	0.066	-1.9	0.086	0.6
<b>37</b>	0.052	0.7	0.087	0.0	0.032	0.6	0.122	0.4	0.089	1.5	0.037	2.7	0.036	-0.5	0.028	-0.1	0.119	-0.2	0.088	0.7
<b>38</b>	0.044	0.0	0.066	-0.9	0.030	0.4	0.178	2.4	0.054	-0.7	0.023	0.2	0.040	-0.2	0.028	-0.1	na	na	0.061	-0.7
<b>39</b>	0.030	-1.3	0.080	-0.3	0.010	-2.5	0.090	-0.8	0.070	0.3	0.020	-0.4	0.030	-1.1	0.030	0.2	0.110	-0.5	0.060	-0.8

nd: not detected; na: not analysed

Table 6: Results reported for the mandatory pesticide Permethrin (sum), and the voluntary pesticides Chlorate, DDAC-C 10, Ethoxyquin (C-N-Dimer), Fluopyram, Teflubenzuron in mg/kg, the preliminary z-scores using a fit-for-purpose relative standard deviation of 25% (FFP-RSD 25%), the MRRLs in mg/kg and the assigned values and robust relative standard deviations (RSD) in mg/kg.

Lab code	Permethrin (sum)	z-score (FFP-RSD 25%)	Chlorate	z-score (FFP-RSD 25%)	DDAC-C10	z-score (FFP-RSD 25%)	Ethoxyquin, C-N-Dimer	z-score (FFP-RSD 25%)	Fluopyram	z-score (FFP-RSD 25%)	Teflubenzuron	z-score (FFP-RSD 25%)		
MRRL	0.010		0.010		0.010		0.010		0.010		0.010		0.010	0.010
Assign. value	0.057		0.357		0.535		-		0.111		0.149			
Robust RSD	0.016		0.079		0.019		-		0.013		0.046			
Number of results	33		8		9		2		22		18			
<b>2</b>	0.05	-0.69	na	na	na	na	na	na	0.10	-0.26	0.12	-0.71		
<b>3</b>	0.086	2.0	0.289	-0.8	0.277	-1.9	na	na	0.107	-0.2	0.105	-1.2		
<b>4</b>	0.099	3.0	na	na	na	na	na	na	0.167	2.0	na	na		
<b>5</b>	0.050	-0.5	na	na	na	na	na	na	0.109	-0.1	0.141	-0.2		
<b>6</b>	0.055	-0.1	na	na	na	na	na	na	na	na	na	na		
<b>7</b>	0.009	-3.3	na	na	na	na	na	na	na	na	na	na		
<b>8</b>	0.088	2.2	0.336	-0.2	na	na	na	na	0.118	0.2	0.073	-2.0		
<b>9</b>	0.065	0.6	nd	-4.0	0.475	-0.5	na	na	0.090	-0.8	0.117	-0.9		
<b>10</b>	0.041	-1.1	na	na	na	na	na	na	na	na	na	na		
<b>11</b>	0.077	1.4	na	na	na	na	na	na	0.116	0.2	0.308	4.2		
<b>12</b>	0.065	0.6	na	na	na	na	na	na	na	na	na	na		

Lab code	Permethrin (sum)	z-score (FFP-RSD 25%)	Chlorate	z-score (FFP-RSD 25%)	DDAC-C10	z-score (FFP-RSD 25%)	Ethoxyquin, C-N-Dimer	z-score (FFP-RSD 25%)	Fluopyram	z-score (FFP-RSD 25%)	Teflubenzuron	z-score (FFP-RSD 25%)
MRRL	0.010		0.010		0.010		0.010		0.010		0.010	
Assign. value	0.057		0.357		0.535		-		0.111		0.149	
Robust RSD	0.016		0.079		0.019		-		0.013		0.046	
Number of results	33		8		9		2		22		18	
<b>13</b>	0.042	-1.1	na	na	na	na	na	na	na	na	0.116	-0.9
<b>14</b>	0.052	-0.3	0.461	1.2	0.452	-0.6	na	na	0.121	0.4	0.163	0.4
<b>15</b>	0.052	-0.3	0.420	0.7	0.441	-0.7	na	na	0.095	-0.6	0.209	1.6
<b>16</b>	0.036	-1.5	0.322	-0.4	na	na	na	na	0.095	-0.6	0.127	-0.6
<b>17</b>	0.057	0.0	na	na	na	na	na	na	0.108	-0.1	na	na
<b>18</b>	0.059	0.2	na	na	0.415	-0.9	na	na	na	na	na	na
<b>19</b>	0.040	-1.2	na	na	0.588	0.4	na	na	0.122	0.4	0.163	0.4
<b>20</b>	0.061	0.3	na	na	nd	-4.0	na	na	0.116	0.2	0.130	-0.5
<b>21</b>	0.063	0.4	nd	-4.0	0.900	2.7	0.540	-	0.110	0.0	0.140	-0.3
<b>22</b>	0.091	2.4	na	na	na	na	na	na	na	na	0.140	-0.3
<b>23</b>	0.080	1.7	na	na	na	na	na	na	0.120	0.3	na	na
<b>24</b>	0.062	0.3	0.328	-0.3	0.595	0.4	0.183	-	0.105	-0.2	0.178	0.8
<b>25</b>	0.072	1.1	0.275	-0.9	na	na	na	na	na	na	na	na
<b>26</b>	0.054	-0.2	na	na	na	na	na	na	0.129	0.6	na	na
<b>27</b>	0.058	0.1	na	na	0.512	-0.2	na	na	0.112	0.0	0.135	-0.4

Lab code	Permethrin (sum)	z-score (FFP-RSD 25%)	Chlorate	z-score (FFP-RSD 25%)	DDAC-C10	z-score (FFP-RSD 25%)	Ethoxyquin, C-N-Dimer	z-score (FFP-RSD 25%)	Fluopyram	z-score (FFP-RSD 25%)	Teflubenzuron	z-score (FFP-RSD 25%)		
MRRL	0.010		0.010		0.010		0.010		0.010		0.010		0.010	0.010
Assign. value	0.057		0.357		0.535		-		0.111		0.149			
Robust RSD	0.016		0.079		0.019		-		0.013		0.046			
Number of results	33	8	9	2	22	18								
28	0.053	-0.3	na	na	na	na	na	na	0.120	0.3	na	na		
29	0.070	0.9	na	na	na	na	na	na	0.124	0.5	0.199	1.3		
31	0.031	-1.8	na	na	na	na	na	na	0.046	-2.3	0.041	-2.9		
32	0.046	-0.7	na	na	na	na	na	na	na	na	na	na		
33	0.022	-2.4	na	na	na	na	na	na	na	na	na	na		
34	0.066	0.7	na	na	0.547	0.1	na	na	0.110	0.0	na	na		
35	0.061	0.3	na	na	na	na	na	na	na	na	na	na		
36	0.039	-1.3	na	na	na	na	na	na	0.054	-2.1	0.088	-1.6		
37	0.057	0.0	na	na	na	na	na	na	na	na	0.152	0.1		
38	0.060	0.2	na	na	na	na	na	na	na	na	na	na		
39	0.040	-1.2	na	na	na	na	na	na	0.100	-0.4	0.340	5.0		

nd: not detected; na: not analysed

## Appendix 1 - Target pesticide list

**Table A-1:** List of mandatory analytes and minimum required reporting levels (MRRL).

Analyte	MRRL (mg/kg)
Aldrin	0.005
Azinphos-ethyl	0.010
Azinphos-methyl	0.010
Bifenthrin (sum of isomers)	0.010
Chlordane, cis-	0.005
Chlordane, trans-	0.005
Chlorfenvinphos	0.010
Chlorpyrifos(-ethyl)	0.010
Chlorpyrifos-methyl	0.010
Cyfluthrin (sum of isomers)	0.010
Cyhalothrin, Lambda- (sum of isomers)	0.010
Cypermethrin (sum of isomers)	0.010
DDD, p,p'- (TDE)	0.005
DDE, p,p'-	0.005
DDT, o,p'-	0.005
DDT, p,p'-	0.005
Deltamethrin (cis-isomer)	0.010
Diazinon	0.010
Dieldrin	0.005
Endosulfan sulfate	0.005
Endosulfan, alpha-	0.005
Endosulfan, beta-	0.005
Endrin	0.005
Famoxadone (MACP mandatory)	0.010
Fenvalerate/Esfenvalerate (sum of RR, SS, RS & SR isomers)	0.010
Fipronil	0.005
Fipronil sulfone	0.005
Heptachlor	0.005
Heptachlorepoxid, Cis-	0.005
Heptachlorepoxid, trans-	0.005
Hexachlorcyclohexane (HCH), alpha-isomer	0.005
Hexachlorcyclohexane (HCH), beta-isomer	0.005
Hexachlorcyclohexane (HCH), gamma-isomer (Lindane)	0.005
Hexachlorobenzene (HCB)	0.005
Indoxacarb (sum of isomers)	0.010
Malathion (parent only)	0.010
Methidathion	0.010
Methoxychlor, 4,4'-	0.010
Nitrofen	0.005
Oxychlordane	0.005
Parathion(-ethyl)	0.010
Parathion-methyl (parent only)	0.010



Analyte	MRRL (mg/kg)
Pendimethalin	0.010
Permethrin (sum of isomers)	0.010
Phosmet (parent only)	0.010
Phoxim	0.010
Pirimiphos-methyl	0.010
Profenofos	0.010
Pyrazophos	0.010
Quintozene (parent only)	0.005
Resmethrin (sum of isomers)	0.010
Tecnazene	0.005
Vinclozolin (parent only)	0.005

**Table A-2:** List of voluntary analytes and minimum required reporting levels (MRRL).

Analyte	MRRL (mg/kg)
Benzalkonium Chloride (C8,C10,C12,C14,C16,C18)	0.01
Benzovindiflupyr	0.01
Bixafen (parent only)	0.01
Bixafen-desmethyl	0.01
Boscalid (parent only)	0.01
Carbendazim (Carbendazim only)	0.01
Chlorate	0.01
Chlorpropham (parent only)	0.01
Cyproconazole	0.01
Didecyldimethylammoniumchlorid (DDAC-C8,C10,C12)	0.01
Diflubenzuron (parent only)	0.01
Emamectin	0.01
Epoconazole	0.01
Ethoxyquin	0.01
Ethoxyquin dimer, metabolite of ethoxyquin	0.01
Ethoxyquin quinone imine (EQI)	0.01
Etofenprox	0.01
Fenpropidin (parent only)	0.01
Fenpropimorph (parent only)	0.01
Fenpyrazamine	0.01
Fluopyram	0.01
Fluopyram-benzamide	0.01
Fluquinconazole	0.01
Flusilazole (parent only)	0.01
Hexaflumoron	0.01
Imidacloprid	0.01
Lufenuron	0.01
Mefentrifluconazole	0.01
Metaflumizone (sum of isomers)	0.01
Penflufen (sum of isomers)	0.01
Penthiopyrad	0.01

Analyte	MRRL (mg/kg)
Prochloraz (parent only)	0.01
BTS 44595 (Prochloraz metabolite)	0.01
BTS 44596 (Prochloraz metabolite)	0.01
Prothioconazole-desthio	0.01
Spinosad <sup>(1)</sup>	0.01
Spinosyn A <sup>(2)</sup>	0.01
Spinosyn D <sup>(2)</sup>	0.01
Spiroxamine (parent only, sum of isomers)	0.01
Sulfoxaflor (sum of isomers)	0.01
tau-Fluvalinate (sum of isomers)	0.01
Tebuconazole	0.01
Tebuconazole, Hydroxy- (free phenol only)	0.01
Teflubenzuron	0.01
Tetraconazole	0.01
Thiacloprid	0.01
Thiophanate methyl	0.01

<sup>(1)</sup> Results for Spinosad should be reported either if individual standards for Spinosyn A and D or a mixture of Spinosyn A and D are used for quantification.

<sup>(2)</sup> Results for Spinosyn A or D should be reported, if the individual standards were used for quantification.