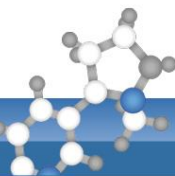




**EURL-SRM**



EU Reference Laboratories for Residues of Pesticides

Single Residue Methods

## **EU PROFICIENCY TEST**

**EUPT-SRM14, 2019**

### **Residues of Pesticides**

**Requiring Single Residue Methods**

**Test Item: Bovine Liver Homogenate**

### **Preliminary Report**

**Michelangelo Anastassiades**

**Pat Schreiter**

**10 May 2019**

## General Remark

- **Please note that all assigned values, CV\*s and z-scores presented in the following are preliminary.**  
The final report on the EUPT-SRM14 will be drafted following the joint EURL/NRL-Workshop and the evaluation of the results by the EUPT-Scientific Committee.
- **All labs are kindly asked to check their results carefully and to report any errors.**
- **Please note that only transcription errors by the organizers can be considered at this stage.**
- **Labs with questionable or unacceptable results (individual absolute z-scores > 2 ) as well as labs with false negative or false positive results are requested to give a feedback\* via e-mail by 17 June, 2019.**

*Please give feedback on each of the compound affected by poor performance and about any actions undertaken to find out the reasons.*

*If you need EURL-assistance in finding possible reasons for underperformance, please include in your feedback relevant information that may be helpful in identifying the reasons (e.g. if you suspect a degradation in the standard solution, please give details on how the standard was prepared and stored.)*

## Background

The proficiency test EUPT-SRM14 was conducted using bovine liver from calves. The test item was prepared by spiking liver homogenate with the following 16 compounds dissolved in standard solutions: 2,4-DB, avermectin B1a, bixafen metabolite, desmethyl bixafen, boscalid metabolite M510F01, bromoxynil, DDAC-C10, fenpropimorph carboxylic acid (BF-421-2), flonicamid metabolite TFNA-AM, fluopyram-benzamide (M25), MPP, glyphosate, AMPA, N-acetyl-glyphosate, haloxyfop, MCPA, and mepiquat. Glyphosate was the only one among all analytes on the Target Pesticides List that was compulsory for the laboratories to analyse (due to its inclusion in the EU-coordinated monitoring programme). Glyphosate was also present in the test material.

Following spiking, homogenisation, portioning, freezing and in case of the test items cryo-milling, portions of approx. 200 g of the test item and blank were bottled and distributed on 18 March, 2019 to the participating laboratories.

Where, according to the IATA dangerous good regulations, shipment with dry ice was allowed, the materials were sent to the participants deeply frozen in insulated boxes containing dry ice. Otherwise, the materials were precooled at -80°C for at least 3 days and sent to the participants deeply frozen in insulated boxes containing special deep frozen packs.

Using an online submission tool, the participants had the possibility to submit their results by 23 April, 2019. The requested methodology information for tentatively false negative results was to be submitted by 3 May, 2019.

10 bottles of the test item were selected randomly and tested for homogeneity. Furthermore, the stability of the test material was checked during a period covering the EUPT duration.

For the calculation of the **preliminary assigned values** only the results of the participating 57 OfLs<sup>1</sup> from EU member states and EFTA countries were considered. No classification into Category A and B based on the analytical scope of the participating laboratory will be conducted in this PT.

<sup>1</sup> These are labs performing official food controls on pesticides as well as official import controls ("669-Labs") or official organic food controls ("889-Labs")

## Results

In total 59 participating labs from EU and EFTA countries as well as 3 laboratories from third countries submitted results of at least one compound.

Within results from EU/EFTA OfLs, two results were **preliminarily judged as false positives**: Glufosinate (1x) and dichlorproprate (1x). In cases where the reported concentration of a compound not present in the Test Item was lower than the respective MRRL, the results were preliminarily not judged as false positives. This concerned BAC-C12 (3x).

13 EU/EFTA OfLs reported in 15 cases results that were **preliminarily judged as false negatives**. These concerned cases where compounds present in the test item were analysed by the labs, but not detected. For these results z-scores were calculated using the corresponding MRRL in the target pesticide list or the RL, if this was lower. The false negative results concerned Flonicamid metabolite TFNA-AM (5x), 2,4-DB (4x) as well as avermectin B1a, boscalid metabolite M510F017, bromoxynil, DDAC-C10, fenpropimorph carboxylic acid (BF-421-2) and AMPA (each 1x). In one case, the reported analysed for but not detected haloxyfop with the RL higher than the assigned value, this result was preliminarily judged as a false negative result. The final decision will be made after consulting with the Scientific Committee.

Results preliminarily judged as false negatives are marked in the tables with the abbreviation FN. Empty cells in the tables correspond to “not analysed” compounds.

Please note that the final judgement on false positive and false negative results will be done in the final report following consultation with the EUPT-Scientific Committee.

All submitted results on pesticides incurred and spiked to the test material are shown in Table 1a and Table 1b. The preliminary false positive results are shown in Table 2.

Since the robust standard deviation of N-acetyl-glyphosate is very high and the number of the numerical results for fenpropimorph carboxylic acid (BF-421-2) was not sufficient for a reliable statistical evaluation, the z-scores calculated for these two compounds are currently considered uncertain and displayed in italic letters. Following a detailed evaluation of the method information and other aspects, the EUPT-Scientific Committee will decide whether an alternative assigned value and z-scores based on it may be calculated.

The tables also contain the results of the participating laboratories from 3<sup>rd</sup> countries. In all these cases the z-scores were calculated using the same assigned values as for the EU and EFTA labs.

Tab 1a: Results for 1 compulsory and 7 out of 15 optional compounds in EUPT-SRM14 reported (z-scores were calculated using preliminary assigned values)

Pesticides	Glyphosate		2,4-DB		Avermectin B1a		Bixafen Met. Desmethyl bixafen		Boscalid Met. M510F017		Bromoxynil		DDAC-C10		Fenpropimorph car- boxylic acid (BF-421-2)	
MRRL [mg/kg]	0.1		0.01		0.01		0.01		0.01		0.01		0.03		0.01	
Assigned Value [mg/kg]	0.534		0.061		0.057		0.05		0.081		0.058		0.178		0.089	
CV*	23.9%		19.0%		29.8%		20.0%		13.1%		15.8%		20.2%		12.1%	
No. of numerical results / FN (EU/EFTA)	42 / 0		34 / 2		37 / 1		20 / 0		17 / 1		33 / 1		29 / 1		10 / 1	
Lab Code	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score
950	0.548	0.1	0.0813	1.4	0.0593	0.2	0.0495	-0.1	0.0754	-0.3	0.0864	1.9	0.175	-0.1		
952																
956	0.428	-0.8	FN	-3.3			0.0560	0.5	0.0902	0.5	0.0619	0.2				
992							0.0510	0.1	0.0760	-0.2					0.0810	-0.4
1022							0.034	-1.3	FN	-3.5						
1024																
1072	0.465	-0.5	0.065	0.3	0.049	-0.5					0.060	0.1	0.216	0.9		
1090	0.527	-0.1	0.066	0.3	0.030	-1.9	0.055	0.4	0.088	0.4	0.063	0.3	0.209	0.7	0.104	0.7
1092	0.688	1.2	0.0358	-1.6	0.0419	-1.0							FN	-3.3		
1150			0.027	-2.2									0.021	-3.5		
1206	0.511	-0.2	0.068	0.5	0.058	0.1	0.046	-0.3	0.072	-0.4	0.058	0.0	0.182	0.1	0.081	-0.4
1214	0.47	-0.5	0.065	0.3	0.064	0.5					0.056	-0.2	144	3235.8		
1218	0.570	0.3	0.052	-0.6	0.046	-0.8					0.064	0.4	0.175	-0.1		
1224	0.620	0.6			0.026	-2.2										
1228	0.416	-0.9														
1240			0.0560	-0.3	0.0354	-1.5										
1244	0.353	-1.4	0.0437	-1.1	0.0829	1.8					0.0728	1.0				
1246																
1248	0.572	0.3														
1250	0.498	-0.3			0.0565	0.0										
1266	1.104	4.3	0.064	0.2	0.061	0.3	0.055	0.4	0.084	0.2	0.065	0.5	0.174	-0.1	0.090	0.0
1270	0.928	3.0	0.0621	0.1	0.0456	-0.8					0.0479	-0.7				
1274	0.550	0.1			0.0703	1.0							0.139	-0.9		
1276	0.431	-0.8	0.0653	0.3	0.0832	1.9					FN	-3.3	0.221	1.0		
1278	0.551	0.1			0.059	0.2										
1290			FN	-3.3												
1292	0.854	2.4	0.0479	-0.8			0.0543	0.3	0.0810	0.0	0.0438	-1.0	0.248	1.6		
1298			0.0756	1.0	0.0644	0.5					0.0580	0.0				
1300	0.529	0.0	0.064	0.2	0.079	1.6					0.059	0.0	0.174	-0.1		
1302			0.101	2.7			0.080	2.4	0.085	0.2			0.207	0.7		
1306	2.33	13.5									0.0537	-0.3	0.0864	-2.1		
1310	0.321	-1.6	0.046	-1.0	0.098	2.9					0.057	-0.1				

**Tab 1a: Results for 1 compulsory and 7 out of 15 optional compounds in EUPT-SRM14 reported (z-scores were calculated using preliminary assigned values)**

Pesticides	Glyphosate		2,4-DB		Avermectin B1a		Bixafen Met. Desmethyl bixafen		Boscalid Met. M510F017		Bromoxynil		DDAC-C10		Fenpropimorph carboxylic acid (BF-421-2)	
MRL [mg/kg]	0.1		0.01		0.01		0.01		0.01		0.01		0.03		0.01	
Assigned Value [mg/kg]	0.534		0.061		0.057		0.05		0.081		0.058		0.178		0.089	
CV*	23.9%		19.0%		29.8%		20.0%		13.1%		15.8%		20.2%		12.1%	
No. of numerical results / FN (EU/EFTA)	42 / 0		34 / 2		37 / 1		20 / 0		17 / 1		33 / 1		29 / 1		10 / 1	
Lab Code	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score
1312	0.770	1.8	0.061	0.0	0.126	4.9	0.052	0.1	0.075	-0.3	0.056	-0.2	0.159	-0.4	0.079	-0.5
1318	0.558	0.2			FN	-3.3					0.056	-0.2	0.202	0.5		
1320	0.483	-0.4	0.064	0.2	0.056	-0.1	0.043	-0.6	0.060	-1.0	0.042	-1.1	0.190	0.3	0.082	-0.3
1322			0.0588	-0.1	0.0551	-0.1					0.0625	0.3				
1324	0.806	2.0			0.058	0.1					0.060	0.1				
1330	0.654	0.9	0.0721	0.7	0.0675	0.8					0.0453	-0.9	0.204	0.6		
1332	0.403	-1.0	0.0600	0.0	0.0558	-0.1	0.0512	0.1			0.0662	0.5	0.199	0.5		
1336	0.619	0.6														
1338	0.463	-0.5	0.0750	0.9			0.132	6.5	0.0890	0.4	0.0900	2.2			0.112	1.0
1340							0.030	-1.6					0.144	-0.8	FN	-3.6
1342	0.683	1.1	0.057	-0.2	0.071	1.0										
1346					0.0382	-1.3										
1350	0.60	0.5														
1352	0.513	-0.2	0.0485	-0.8	0.0483	-0.6					0.0396	-1.3				
1354	0.0618	-3.5	0.0524	-0.5	0.0519	-0.3					0.0546	-0.3				
1356	0.305	-1.7									0.0580	0.0	0.0969	-1.8		
1358	0.508	-0.2	0.0562	-0.3	0.0428	-1.0	0.0309	-1.5	0.0460	-1.7	0.0608	0.2	0.151	-0.6		
1364							0.0443	-0.5	0.0849	0.2			0.178	0.0		
1366	0.57	0.3											0.17	-0.2		
1368	0.562	0.2	0.0691	0.6	0.0596	0.2					0.0614	0.2	0.178	0.0		
1370					0.036	-1.5										
1392			0.0526	-0.5	0.0553	-0.1					0.0436	-1.0				
1394	0.520	-0.1	0.067	0.4	0.072	1.1	0.054	0.3	0.096	0.8	0.065	0.5	0.156	-0.5	0.095	0.3
1396																
1398	0.399	-1.0	0.065	0.3	0.066	0.7	0.069	1.5	0.094	0.7	0.064	0.4	0.272	2.1	0.089	0.0
1400	0.502	-0.2	0.050	-0.7	0.032	-1.7	0.050	0.0	0.079	-0.1	0.050	-0.6	0.146	-0.7		
1402	0.466	-0.5	0.073	0.8	0.053	-0.3	0.058	0.6	0.073	-0.4	0.068	0.7	0.194	0.4	0.087	-0.1
978 (3 <sup>rd</sup> Country)					0.0520	-0.3										
1404 (3 <sup>rd</sup> Country)											0.0596	0.1				
1406 (3 <sup>rd</sup> Country)	1.8	9.5	0.05	-0.7	0.026	-2.2					0.015	-3.0	FN	-3.3		

**Tab 1b: Results for the other 8 out of 15 optional compounds in EUPT-SRM14 reported (z-scores were calculated using preliminary assigned values)**

Pesticides	Flonicamid Met. TFNA-AM		Fluopyram-benzamide (M25)		MPP		AMPA		N-Acetyl-glyphosate		Haloxypop		MCPA		Mepiquat	
MRRL [mg/kg]	0.01		0.02		0.05		0.1		0.1		0.01		0.01		0.01	
Assigned Value [mg/kg]	0.072		0.101		0.309		0.751		0.591		0.036		0.046		0.051	
CV*	22.9%		12.4%		20.5%		19.5%		43.3%		22.1%		25.8%		17.4%	
No. of numerical results / FN (EU/EFTA)	18 / 5		22 / 1		0		20 / 1		15 / 0		40 / *		44 / 0		45 / 0	
Lab Code	Conc [mg/kg]	Conc [mg/kg]	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score
950	0.0498	-1.2	0.105	0.2	0.314	0.1	0.569	-1.0	0.354	-1.6	0.0454	1.0	0.0513	0.5	0.0455	-0.4
952																
956	FN	-3.4	0.0835	-0.7	0.231	-1.0	0.855	0.6	0.0438	-3.7	0.0382	0.2	0.0435	-0.2	0.0334	-1.4
992			0.104	0.1	0.320	0.1										
1022																
1024																
1072											0.039	0.3	0.052	0.5	0.075	1.9
1090	0.081	0.5	0.102	0.0	0.302	-0.1	0.707	-0.2	0.693	0.7	0.044	0.8	0.049	0.3	0.053	0.2
1092					0.651	4.4	FN	-3.5	0.874	1.9	0.0120	-2.7	0.0367	-0.8	0.0480	-0.2
1150													0.032	-1.2	0.055	0.3
1206	0.069	-0.2	0.100	0.0	0.303	-0.1	0.705	-0.2	0.625	0.2	0.038	0.2	0.050	0.3	0.050	-0.1
1214					0.27	-0.5	0.69	-0.3			0.046	1.1	0.065	1.6	0.052	0.1
1218	FN	-3.4					0.795	0.2			0.040	0.4	0.049	0.3	0.055	0.3
1224							1.200	2.4							0.052	0.1
1228	0.074	0.1			0.313	0.1	0.303	-2.4			0.057	2.3	0.078	2.8	0.054	0.2
1240	0.0342	-2.1									0.0392	0.3	0.0377	-0.7		
1244			0.101	0.0	0.350	0.5	0.791	0.2	0.517	-0.5	0.0318	-0.5	0.0430	-0.3	0.0427	-0.6
1246																
1248																
1250			0.0919	-0.4									0.0317	-1.2	0.0588	0.6
1266	0.079	0.4	0.108	0.3	0.291	-0.2	0.679	-0.4	0.665	0.5	0.042	0.6	0.056	0.9	0.042	-0.7
1270			0.0925	-0.3							0.036	0.0	0.0454	-0.1	0.042	-0.7
1274											0.0342	-0.2	0.0356	-0.9	0.0543	0.3
1276					0.229	-1.0	0.781	0.2	0.354	-1.6	0.0391	0.3	0.0467	0.1	0.0726	1.7
1278							0.857	0.6			0.038	0.2	0.050	0.3	0.049	-0.2
1290											0.02	-1.8	0.02	-2.3	0.05	-0.1
1292	0.0716	0.0	0.102	0.0			1.00	1.3			0.0321	-0.5	0.0286	-1.5	0.0631	1.0
1298											0.0384	0.2	0.0467	0.1	0.055	0.3
1300							0.730	-0.1			0.048	1.3	0.071	2.2	0.048	-0.2
1302	FN	-3.4	0.099	-0.1									0.078	2.8	0.052	0.1
1306											0.0238	-1.4			0.0479	-0.2
1310											0.024	-1.4	0.038	-0.7	0.033	-1.4

**Tab 1b: Results for the other 8 out of 15 optional compounds in EUPT-SRM14 reported (z-scores were calculated using preliminary assigned values)**

Pesticides	Flonicamid Met. TFNA-AM		Fluopyram- benzamide (M25)		MPP		AMPA		<i>N-Acetyl- glyphosate</i>		Haloxypop		MCPA		Mepiquat	
MRRL [mg/kg]	0.01		0.02		0.05		0.1		0.1		0.01		0.01		0.01	
Assigned Value [mg/kg]	0.072		0.101		0.309		0.751		0.591		0.036		0.046		0.051	
CV*	22.9%		12.4%		20.5%		19.5%		43.3%		22.1%		25.8%		17.4%	
No. of numerical results / FN (EU/EFTA)	18 / 5		22 / 1		0		20 / 1		15 / 0		40 / *		44 / 0		45 / 0	
Lab Code	Conc [mg/kg]	Conc [mg/kg]	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score	Conc [mg/kg]	z-Score
1312	0.071	-0.1	0.094	-0.3	0.680	4.8					0.042	0.6	0.047	0.1	0.141	7.1
1318							0.790	0.2			0.038	0.2	0.053	0.6	0.032	-1.5
1320	0.096	1.3	0.120	0.8	0.309	0.0	0.532	-1.2	1.889	8.8	0.032	-0.5	0.039	-0.6	0.032	-1.5
1322	0.0576	-0.8									0.0423	0.6	0.0460	0.0		
1324					0.288	-0.3	0.898	0.8			FN*	-2.9	0.054	0.7	0.051	0.0
1330					0.268	-0.5					0.0387	0.3	0.0395	-0.6	0.0504	0.0
1332	0.0690	-0.2	0.0953	-0.2							0.0390	0.3	0.0534	0.6	0.0421	-0.7
1336							0.58	-0.9								
1338	0.0825	0.6	0.116	0.6	0.376	0.9	0.949	1.1	0.487	-0.7	0.0356	-0.1	0.0625	1.4	0.0472	-0.3
1340			0.101	0.0							0.022	-1.6	0.031	-1.3	0.042	-0.7
1342	FN	-3.4									0.044	0.8	0.059	1.1	0.076	2.0
1346																
1350							0.84	0.5							0.058	0.6
1352									0.496	-0.6	0.0309	-0.6	0.0361	-0.9	0.0542	0.3
1354	0.0666	-0.3	0.0766	-1.0							0.0288	-0.8	0.0405	-0.5	0.0633	1.0
1356	0.0340	-2.1					0.659	-0.5			0.0257	-1.2	0.013	-2.9	0.0505	0.0
1358			0.0532	-1.9			0.599	-0.8	2.01	9.6	0.0212	-1.7	0.0487	0.2	0.0560	0.4
1364			0.0934	-0.3												
1366					0.32	0.1	0.87	0.6	0.46	-0.9						
1368	FN	-3.4			0.237	-0.9	0.833	0.4	0.491	-0.7	0.0418	0.6	0.0484	0.2	0.0586	0.6
1370																
1392											0.0322	-0.5	0.0416	-0.4		
1394	0.094	1.2	0.114	0.5			0.637	-0.6			0.040	0.4	0.054	0.7	0.052	0.1
1396															0.052	0.1
1398	0.087	0.8	0.114	0.5	0.490	2.3							0.056	0.9	0.046	-0.4
1400	0.072	0.0	FN	-3.2			0.753	0.0			0.039	0.3	0.030	-1.4	0.033	-1.4
1402	0.082	0.6	0.117	0.6	0.245	-0.8	0.697	-0.3	0.689	0.7	0.042	0.6	0.053	0.6	0.056	0.4
978 (3 <sup>rd</sup> Country)																
1404 (3 <sup>rd</sup> Country)	0.0778	0.3									0.0385	0.2	0.0499	0.3		
1406 (3 <sup>rd</sup> Country)	0.197	6.9	0.291	7.6			3.1	12.5			0.0055	-3.4	FN	-3.1	0.045	-0.5

FN\* (analysed, not detected, lab's RL &gt; Assigned value)

Lab Code	Analyte	MRRL [mg/kg]	RL [mg/kg]	Reported results [mg/kg] (cp. to MRRL)	Preliminary Judgement
1072	BAC C-12	0.03	0.03	0.018 (< RL and < MRRL)	NO FP
1214	BAC C-12	0.03	0.005	0.005 (<MRRL, but = RL)	NO FP?
1332	BAC C-12	0.03	0.03	0.0102 (< RL and < MRRL)	NO FP
1338	Dichlorprop	0.01	0.01	0.0770	FP
1356	Glufosinate	0.1	0.1	0.135	FP
1406 (3 <sup>rd</sup> Country)	BAC C-12	0.03	0.03	0.16	FP
1406 (3 <sup>rd</sup> Country)	Glufosinate	0.1	0.1	0.59	FP