



**EU PROFICIENCY TEST
EUPPT-SRM10 - 2015**

**Residues of Pesticides
Requiring Single Residue Methods
Test Item: Corn Flour**

Preliminary Report

**Michelangelo Anastassiades
Pat Schreiter**

General Remark

Please note that all assigned values, Qn-RSDs and z-scores, presented in the following are preliminary!

The final report of the EUPT-SRM10 will be drafted towards December 2015 following thorough evaluation of the results and method information by the Organizers and EUPT-Scientific Committee.

Background

The proficiency test EUPT-SRM10 was conducted using corn flour as Test Item. To prepare the Test Item maize with incurred chlormequat and glyphosate grown in Denmark was purchased and spiked with further 12 compounds using standard solutions. 8 of the compounds (2,4-D, chlormequat, dithiocarbamates, ethephon, glyphosate, MCPA, mepiquat and propamocarb) were classified as “compulsory” and will be considered in the scope-based lab classification into Category A and B. The 5 “optional” compounds (bentazone, bromoxynil, N-acetyl glufosinate, phosphonic acid and TFNG) will not be considered in the scope-based lab classification but will still be evaluated as far as the quantitative and qualitative results are concerned (z-scores, false positives, false negatives).

Following spiking, milling and mixing ca. 350 g portions of the spiked Test Item were filled into bottles and distributed to the participants on 18 May 2015. The participants were also provided with the same amount of blank material prepared from maize found to be free of the residues of interest.

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.

The materials were sent to the participants deeply frozen (-20°C) in insulated boxes containing freezer gel packs. Using an online submission tool the participants had the possibility to submit their results as well as the requested information about the analytical methods employed by 19 June, 2014.

Results

In total 104 participating labs from EU member states and EFTA countries, 1 OfL from one EU candidate country and 5 laboratories from third countries submitted results. The assigned values were established using the robust mean of the results of those 104 official labs from EU member states and EFTA countries.

4 false positive results (3x glufosinate and 1x MPP) were submitted by 3 participating laboratories from EU member states and EFTA countries. All false positive results concerned compounds from the optional list.

6 laboratories from EU member states and EFTA countries reported in 6 cases potentially false negative results (“analyzed, but not detected”). Four of them concerned mandatory and two optional compounds present in the test material. Their 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 glyphosate (2x), dithiocarbamates, MCPA, N-acetyl glufosinate and phosphonic acid. In addition, 3 laboratories from third country or EU-candidate labs reported in 5 cases false negative results. Three of them (glyphosate (2x) and ethephon) concerned mandatory and the other two (bromoxynil (2x)) optional analytes present in the test material.

All submitted results of pesticides incurred and spiked to the test material are shown in Table 1 for compulsory compounds and Table 2 for optional compounds.

False negative results are marked in the tables with the abbreviation FN. Empty cells in the tables correspond to “not analysed” compounds.

The table also contains the results of the participating laboratories from 3rd countries and EU candidate countries. In these cases the z-scores were calculated using the assigned values derived from the results of EU and EFTA labs.

- 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 individual absolute z-scores >2 or false positive results will be requested to provide feedback about any actions undertaken to find out the reasons for questionable bad performance. This also serves in making other labs aware of potential sources of errors.

Table 1a: Results for COMPULSORY compounds in EUPT-SRM10 reported by OfLs from EU and EFTA countries (z-scores were calculated using preliminary assigned values)

PRELIMINARY Assigned Value [mg/kg]	2,4-D (free acid)	Chlormequat	Dithiocarbamates	Etephon	Glyphosate	MCPA	Mepiquat	Propamocarb
	0.092	0.167	0.559	0.162	0.568	0.081	0.114	0.067
Robust-RSD	18.2 %	18.2 %	36.9 %	30.8 %	22.8 %	18.9 %	18.5 %	23.3 %
No. Results	82	75	86	61	64	80	76	87
Labor No.	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score
1	0.095	0.1	0.208	1.0	0.590	0.2		
2	0.082	-0.4	0.155	-0.3	0.635	0.5	0.151	-0.3
3	0.098	0.3	0.137	-0.7	0.698	1.0	0.148	-0.3
4	0.127	1.5	0.159	-0.2	0.950	2.8	0.215	1.3
5	0.095	0.1	0.200	0.8	0.320	-1.7	0.130	-0.8
6	0.098	0.3	0.136	-0.8	0.458	-0.7	0.157	-0.1
7					0.532	-0.2		
8	0.090	-0.1	0.226	1.4	0.450	-0.8	0.025	-3.4
9	0.092	0.0	0.167	0.0	0.774	1.5		
10	0.106	0.6	0.212	1.1	0.462	-0.7	0.185	0.6
11	0.096	0.2	0.159	-0.2	0.547	-0.1	0.162	0.0
12	0.098	0.3	0.168	0.0	0.392	-1.2	0.169	0.2
13							FN	-3.6
14			0.152	-0.4			0.162	0.0
15	0.0923	0.0	0.158	-0.2	0.435	-0.9	0.193	0.8
16	0.065	-1.2	0.136	-0.8	0.832	2.0	0.139	-0.6
18	0.068	-1.0	0.154	-0.3				
19	0.110	0.8	0.130	-0.9	0.65	0.6		
20	0.0767	-0.7	0.172	0.1	0.500	-0.4	0.178	0.4
21	0.088	-0.2	0.171	0.1	0.883	2.3	0.159	-0.1
22	0.103	0.5			0.704	1.0	0.193	0.8
23	0.118	1.1	0.171	0.1	0.210	-2.5	0.158	-0.1
24	0.0131	-3.4						
25	0.106	0.6	0.214	1.1	0.648	0.6	0.139	-0.6
26	0.092	0.0	0.183	0.4	0.325	-1.7	0.388	5.6
27	0.111	0.8	0.170	0.1	0.270	-2.1	0.153	-0.2

Table 1a: Results for COMPULSORY compounds in EUPT-SRM10 reported by OfLs from EU and EFTA countries (z-scores were calculated using preliminary assigned values)

PRELIMINARY Assigned Value [mg/kg]	2,4-D (free acid)	Chlormequat	Dithiocarbamates	Etephon	Glyphosate	MCPA	Mepiquat	Propamocarb
	0.092	0.167	0.559	0.162	0.568	0.081	0.114	0.067
Robust-RSD	18.2 %	18.2 %	36.9 %	30.8 %	22.8 %	18.9 %	18.5 %	23.3 %
No. Results	82	75	86	61	64	80	76	87
Labor No.	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score
28	0.078	-0.6			0.498	-0.4	0.164	0.1
29	0.094	0.1	0.154	-0.3	0.558	0.0	0.218	1.4
30	0.105	0.6	0.179	0.3	0.228	-2.4	0.141	-0.5
31	0.062	-1.3	0.280	2.7	0.460	-0.7	0.047	-2.8
32	0.082	-0.4	0.240	1.7	0.860	2.2		0.340
33	0.135	1.9	0.154	-0.3	0.484	-0.5	0.21	1.2
34	0.026	-2.9	0.120	-1.1	0.688	0.9	0.137	-0.6
35	0.0706	-0.9	0.161	-0.2	0.568	0.1	0.240	1.9
36	0.109	0.7	0.0804	-2.1			0.411	6.2
37	0.095	0.1			0.280	-2.0		
38	0.083	-0.4	0.162	-0.1	0.696	1.0	0.224	1.5
40	0.092	0.0	0.134	-0.8	0.541	-0.1	0.128	-0.8
41					0.334	-1.6		
43	0.081	-0.5	0.155	-0.3	0.870	2.2	0.150	-0.3
44	0.100	0.3	0.198	0.7	0.393	-1.2	0.351	4.7
45	0.0986	0.3	0.150	-0.4	0.661	0.7	0.148	-0.3
46	0.108	0.7	0.178	0.3	0.496	-0.5	0.168	0.2
47	0.123	1.3	0.168	0.0	0.544	-0.1	0.155	-0.2
48					0.881	2.3		
49			1.350	28.3	0.530	-0.2		
50					0.465	-0.7		
51	0.088	-0.2	0.169	0.0	0.706	1.1	0.185	0.6
52							0.602	0.2
53	0.100	0.3			0.920	2.6		
54	0.092	0.0	0.153	-0.3	0.432	-0.9		0.469
55	0.092	0.0	0.258	2.2	0.568	0.1	0.270	2.7

Table 1a: Results for COMPULSORY compounds in EUPT-SRM10 reported by OfLs from EU and EFTA countries (z-scores were calculated using preliminary assigned values)

PRELIMINARY Assigned Value [mg/kg]	2,4-D (free acid)	Chlormequat	Dithiocarbamates	Etephon	Glyphosate	MCPA	Mepiquat	Propamocarb
	0.092	0.167	0.559	0.162	0.568	0.081	0.114	0.067
Robust-RSD	18.2 %	18.2 %	36.9 %	30.8 %	22.8 %	18.9 %	18.5 %	23.3 %
No. Results	82	75	86	61	64	80	76	87
Labor No.	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score
56	0.0854	-0.3	0.156	-0.3	0.451	-0.8		
57	0.092	0.0	0.117	-1.2	0.452	-0.8	0.221	1.5
58	0.049	-1.9	0.194	0.6	0.328	-1.7	0.146	-0.4
59	0.072	-0.9	0.163	-0.1	1.064	3.6		
60	0.0829	-0.4	0.166	0.0			0.0677	-2.3
61	0.090	-0.1	0.150	-0.4	0.700	1.0	0.150	-0.3
62								
63	0.130	1.6	0.194	0.6	1.03	3.4	0.150	-0.3
64	0.083	-0.4	0.168	0.0	1.03	3.4	0.196	0.8
65	0.0735	-0.8	0.249	1.9			0.215	1.3
66	0.100	0.3	0.185	0.4				
67	0.1106	0.8	0.1542	-0.3	0.894	2.4	0.1238	-0.9
68					0.630	0.5		
70	0.0906	-0.1	0.167	0.0	0.589	0.2	0.152	-0.2
71	0.133	1.8						
72	0.0982	0.3			0.653	0.7	0.146	-0.4
73	0.0849	-0.3			0.599	0.3		
74	0.076	-0.7	0.170	0.1	0.910	2.5	0.241	2.0
75	0.104	0.5	0.170	0.1	0.459	-0.7	0.082	-2.0
76	0.080	-0.5			0.890	2.4		
77	0.086	-0.3	0.245	1.9			0.059	-2.5
78	0.058	-1.5	0.100	-1.6	0.810	1.8	0.083	-1.9
79	0.055	-1.6						
80	0.105	0.6	0.197	0.7				
81	0.084	-0.4	0.171	0.1	0.616	0.4	0.137	-0.6
82	0.112	0.9	0.167	0.0	0.526	-0.2	0.200	0.9

Table 1a: Results for COMPULSORY compounds in EUPT-SRM10 reported by OfLs from EU and EFTA countries (z-scores were calculated using preliminary assigned values)

PRELIMINARY Assigned Value [mg/kg]	2,4-D (free acid)	Chlormequat	Dithiocarbamates	Etephon	Glyphosate	MCPA	Mepiquat	Propamocarb								
	0.092	0.167	0.559	0.162	0.568	0.081	0.114	0.067								
Robust-RSD	18.2 %	18.2 %	36.9 %	30.8 %	22.8 %	18.9 %	18.5 %	23.3 %								
No. Results	82	75	86	61	64	80	76	87								
Labor No.	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score								
83	0.086	-0.3	0.158	-0.2			0.078	-0.2	0.110	-0.1	0.085	1.1				
84	0.087	-0.2	0.163	-0.1	0.762	1.5	0.212	1.2	0.652	0.6	0.079	-0.1	0.106	-0.3	0.055	-0.7
85	0.076	-0.7	0.065	-2.4	1.250	4.9	0.156	-0.1			0.070	-0.5	0.026	-3.1	0.067	0.0
86					0.617	0.4										
87	0.095	0.1	0.172	0.1	0.563	0.0	0.133	-0.7			0.086	0.2	0.126	0.4	0.077	0.6
88					0.547	-0.1										
89	0.104	0.5	0.197	0.7	0.520	-0.3	0.194	0.8	0.442	-0.9	0.057	-1.2	0.100	-0.5	0.052	-0.9
90	0.0875	-0.2	0.165	-0.1	0.423	-1.0					0.0805	0.0	0.0881	-0.9	0.0414	-1.5
91	0.0826	-0.4	0.183	0.4	0.665	0.8	0.187	0.6	0.242	-2.3	0.0752	-0.3	0.110	-0.1	0.0756	0.5
93					0.450	-0.8										
94	0.111	0.8	0.166	0.0	0.292	-1.9	0.133	-0.7	0.472	-0.7	0.0963	0.7	0.130	0.6	0.0583	-0.5
95			0.180	0.3	FN	-3.6	0.070	-2.3	0.536	-0.2			0.214	3.5	0.039	-1.7
97	0.060	-1.4	0.090	-1.8							0.053	-1.4	0.065	-1.7	0.025	-2.5
98	0.102	0.4	0.172	0.1	0.509	-0.4					0.087	0.3	0.095	-0.7	0.056	-0.6
99					0.590	0.2										
100	0.14365	2.2	0.6009	10.4	0.05622	-3.6	0.23061	1.7	0.84408	1.9	0.11445	1.6	0.48479	13.1	0.08399	1.0
102					0.550	-0.1										
103	0.093	0.0			0.550	-0.1	0.124	-0.9			0.087	0.3			0.080	0.8
104					0.508	-0.4										
105															0.058	-0.5
106	0.110	0.8	0.210	1.0	0.630	0.5			0.670	0.7	0.079	-0.1	0.110	-0.1	0.069	0.1
107	0.082	-0.4	0.0614	-2.5	0.510	-0.4					0.0792	-0.1	0.104	-0.3	0.0571	-0.6
108					0.470	-0.6										
109					0.196	-2.6									0.060	-0.4
111			0.162	-0.1					0.597	0.2			0.090	-0.8	0.060	-0.4
112					0.470	-0.6										

Table 1b: Results for COMPULSORY compounds in EUPT-SRM10 reported by EU candidate and 3rd countries (z-scores were calculated using preliminary assigned values)

<u>PRELIMINARY Assigned Value [mg/kg]</u>	2,4-D (free acid)	Chlormequat	Dithiocarbamates	Ethephon	Glyphosate	MCPA	Mepiquat	Propamocarb
Qn-RSD	18.2 %	18.2 %	36.9 %	30.8 %	22.8 %	18.9 %	18.5 %	23.3 %
No. Results	82	75	86	61	64	80	76	87
Labor No.	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score
113	0.125	1.4	0.192	0.6	0.650	0.6	0.083	-1.9
114	0.028	-2.8			0.840	2.0		0.58
115	0.030	-2.7	0.020	-3.5	0.660	0.7	FN	-3.5
116	0.110	0.8	0.120	-1.1	0.880	2.3	0.280	2.9
117	0.104	0.5			0.392	-1.2	0.114	-1.2
118	0.0853	-0.3	0.00094	-4.0	0.721	1.2	0.0032	-3.9
							FN	-3.6
							0.0667	-0.7
							0.0154	-3.5
							0.0884	1.3

Table 2a: Results for OPTIONAL compounds in EUPT-SRM10 reported by OfLs from EU and EFTA countries (z-scores were calculated using preliminary assigned values)

PRELIMINARY Assigned Value [mg/kg]	Bentazone	Bromoxynil	N-Acetyl Glufosinate	Phosphonic acid	TFNG			
	0.098 18.5 %	0.125 17.0 %	0.319 11.8 %	0.584 27.3 %	0.168 18.6 %			
No. Results	69	65	16	25	30			
Labor No.	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score
1			0.155	1.0				
2	0.100	0.1	0.114	-0.4	0.329	0.1		
3	0.104	0.3	0.125	0.0			0.174	0.2
4	0.0917	-0.2	0.133	0.2	0.337	0.2	0.615	0.2
5	0.120	0.9	0.147	0.7			0.971	2.6
6	0.094	-0.2	0.139	0.4	0.305	-0.2	0.598	0.1
7								
8	0.082	-0.6	0.111	-0.5			0.553	-0.2
9			0.137	0.4				
10	0.100	0.1	0.133	0.2			0.617	0.2
11	0.099	0.1	0.121	-0.1				
12	0.118	0.8	0.130	0.2	0.279	-0.5		0.132
13								
14				0.278	-0.5	FN	-3.7	
15	0.0958	-0.1	0.123	-0.1	0.321	0.0		
16	0.071	-1.1						
18	0.073	-1.0	0.083	-1.3	0.350	0.4		
19	0.100	0.1	0.150	0.8				
20	0.075	-0.9	0.0805	-1.4	0.318	0.0	0.585	0.0
21	0.110	0.5	0.127	0.1	0.335	0.2	0.559	-0.2
22			0.129	0.1				
23			0.146	0.7				
24	0.0338	-2.6	0.0579	-2.2				
25	0.103	0.2	0.151	0.8	0.306	-0.2	0.334	-1.7
26	0.101	0.1	0.147	0.7			0.174	0.2
27	0.160	2.5	0.148	0.7			0.606	0.1
							0.186	0.4

Table 2a: Results for OPTIONAL compounds in EUPT-SRM10 reported by OfLs from EU and EFTA countries (z-scores were calculated using preliminary assigned values)

PRELIMINARY Assigned Value [mg/kg]	Bentazone	Bromoxynil	N-Acetyl Glufosinate	Phosphonic acid	TFNG				
	0.098 18.5 %	0.125 17.0 %	0.319 11.8 %	0.584 27.3 %	0.168 18.6 %				
No. Results	69	65	16	25	30				
Labor No.	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	
28	0.100	0.1	0.146	0.7					
29			0.130	0.2			0.165	-0.1	
30	0.084	-0.6	0.100	-0.8		0.591	0.0	0.140	-0.7
31	0.070	-1.1	0.140	0.5					
32	0.101	0.1	0.105	-0.6					
33	0.126	1.2	0.154	0.9		0.774	1.3	0.180	0.3
34	0.049	-2.0	0.039	-2.8			0.029	-3.3	
35									
36	0.0754	-0.9	0.0636	-2.0					
37									
38	0.092	-0.2							
40	0.093	-0.2	0.118	-0.2					
41									
43	0.109	0.5	0.132	0.2					
44	0.115	0.7							
45	0.107	0.4	0.127	0.1			0.162	-0.1	
46	0.112	0.6	0.127	0.1		0.550	-0.2		
47									
48									
49									
50									
51	0.105	0.3	0.127	0.1	0.416	1.2		0.164	-0.1
52									
53	0.100	0.1	0.118	-0.2		0.469	-0.8	0.212	1.1
54	0.105	0.3							
55	0.102	0.2	0.138	0.4				0.180	0.3

Table 2a: Results for OPTIONAL compounds in EUPT-SRM10 reported by OfLs from EU and EFTA countries (z-scores were calculated using preliminary assigned values)

PRELIMINARY Assigned Value [mg/kg]	Bentazone	Bromoxynil	N-Acetyl Glufosinate	Phosphonic acid	TFNG					
	0.098 18.5 %	0.125 17.0 %	0.319 11.8 %	0.584 27.3 %	0.168 18.6 %					
No. Results	69	65	16	25	30					
Labor No.	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score		
56	0.0947	-0.1	0.120	-0.2						
57	0.116	0.7	0.116	-0.3			0.276	-2.1		
58	0.068	-1.2	0.091	-1.1	0.356	0.5	0.592	0.1	0.014	-3.7
59										
60	0.134	1.5	0.173	1.5	FN	-3.7	0.226	-2.5		
61	0.097	0.0	0.100	-0.8	0.35	0.4	0.560	-0.2	0.200	0.8
62										
63	0.140	1.7	0.130	0.2			1.106	3.6		
64	0.038	-2.4	0.062	-2.0						
65	0.0898	-0.3								
66	0.0893	-0.3	0.126	0.0						
67	0.0834	-0.6	0.1161	-0.3				0.160	-0.2	
68										
70	0.0806	-0.7	0.124	0.0			0.672	0.6		
71	0.083	-0.6								
72	0.105	0.3	0.135	0.3				0.165	-0.1	
73	0.0989	0.0	0.116	-0.3				0.140	-0.7	
74	0.103	0.2	0.146	0.7	0.260	-0.7				
75	0.120	0.9	0.120	-0.2						
76										
77	0.125	1.1	0.128	0.1				0.160	-0.2	
78	0.092	-0.2	0.099	-0.8			0.760	1.2		
79	0.096	-0.1								
80	0.103	0.2	0.113	-0.4						
81			0.120	-0.2	0.282	-0.5	0.717	0.9	0.134	-0.8
82	0.116	0.7	0.153	0.9			0.566	-0.1	0.210	1.0

Table 2a: Results for OPTIONAL compounds in EUPT-SRM10 reported by OfLs from EU and EFTA countries (z-scores were calculated using preliminary assigned values)

PRELIMINARY Assigned Value [mg/kg]	Bentazone	Bromoxynil	N-Acetyl Glufosinate	Phosphonic acid	TFNG				
	0.098 18.5 %	0.125 17.0 %	0.319 11.8 %	0.584 27.3 %	0.168 18.6 %				
No. Results	69	65	16	25	30				
Labor No.	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	
83	0.102	0.2	0.132	0.2					
84	0.093	-0.2	0.122	-0.1					
85	0.044	-2.2	0.080	-1.4					
86									
87	0.090	-0.3	0.108	-0.6		0.534	-0.3	0.102	-1.6
88									
89	0.094	-0.2	0.115	-0.3					
90									
91	0.0921	-0.2	0.124	0.0			0.188	0.5	
93									
94									
95	0.106	0.3							
97									
98	0.068	-1.2							
99									
100	0.17459	3.1	0.20976	2.7		0.35534	-1.6	0.19125	0.6
102									
103	0.081	-0.7	0.104	-0.7			0.156	-0.3	
104									
105									
106	0.110	0.5	0.14	0.5					
107	0.101	0.1							
108									
109									
111									
112									

Table 2a: Results for OPTIONAL compounds in EUPT-SRM10 reported by EU candidate and 3rd countries (z-scores were calculated using preliminary assigned values)

PRELIMINARY Assigned Value [mg/kg]	Bentazone	Bromoxynil	N-Acetyl Glufosinate	Phosphonic acid	TFNG			
	0.098	0.125	0.319	0.584	0.168			
Robust-RSD	18.5 %	17.0 %	11.8 %	27.3 %	18.6 %			
No. Results	69	65	16	25	30			
Labor No.	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score	Conc. [mg/kg]	z-score		
113	0.116	0.7	FN	-3.7			0.134	-0.8
114	0.029	-2.8	0.043	-2.6				
115	0.020	-3.2	FN	-3.7				
116	0.090	-0.3						
117	0.097	0.0	0.108	-0.6				
118	0.0308	-2.7	0.0521	-2.3				