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**Poulsen, Mette Erecius; Fromberg, Arvid**

*Publication date:*  
2012

*Document Version*  
Publisher's PDF, also known as Version of record

[Link back to DTU Orbit](#)

*Citation (APA):*  
Poulsen, M. E., & Fromberg, A. (2012). Pesticide residues in feeding stuff for laying hens – results from samples collected in 20 EU member states. Poster session presented at 9th European Pesticide Residue Workshop , Vienna, Austria.

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# Pesticide residues in feeding stuff for laying hens – results from samples collected in 20 EU member states

Mette Erecius Poulsen and Arvid Fromberg

DTU National Food Institute, Moerkhoej Bygade 19, DK-2860 Soeborg, Denmark; e-mail : mpou@food.dtu.dk

**Introduction:** The EURL-AO and EURL-CF<sup>1</sup> has initiated a study on ‘Pesticide residues in feed for laying hens and pesticide residues in eggs’. The aim of the project is to detect pesticide residues in the eggs when known pesticides residues are present in the feed provided for the laying hens. Included in the project was a survey on pesticide residues in the feeding stuff for laying hens in EU. The study will enable the estimation of the risk for consumers in relation to pesticide residues in eggs. To perform a survey to cover feeds in EU the NRL-CFs were asked to collect 2 samples of feeds for laying hens from local suppliers. 20 member states shipped 42 samples for analysis at the EURL-CF. The samples were mixed feeds with cereals and/or fatty matrices of plant origin, like soya/rape, vitamins and minerals.

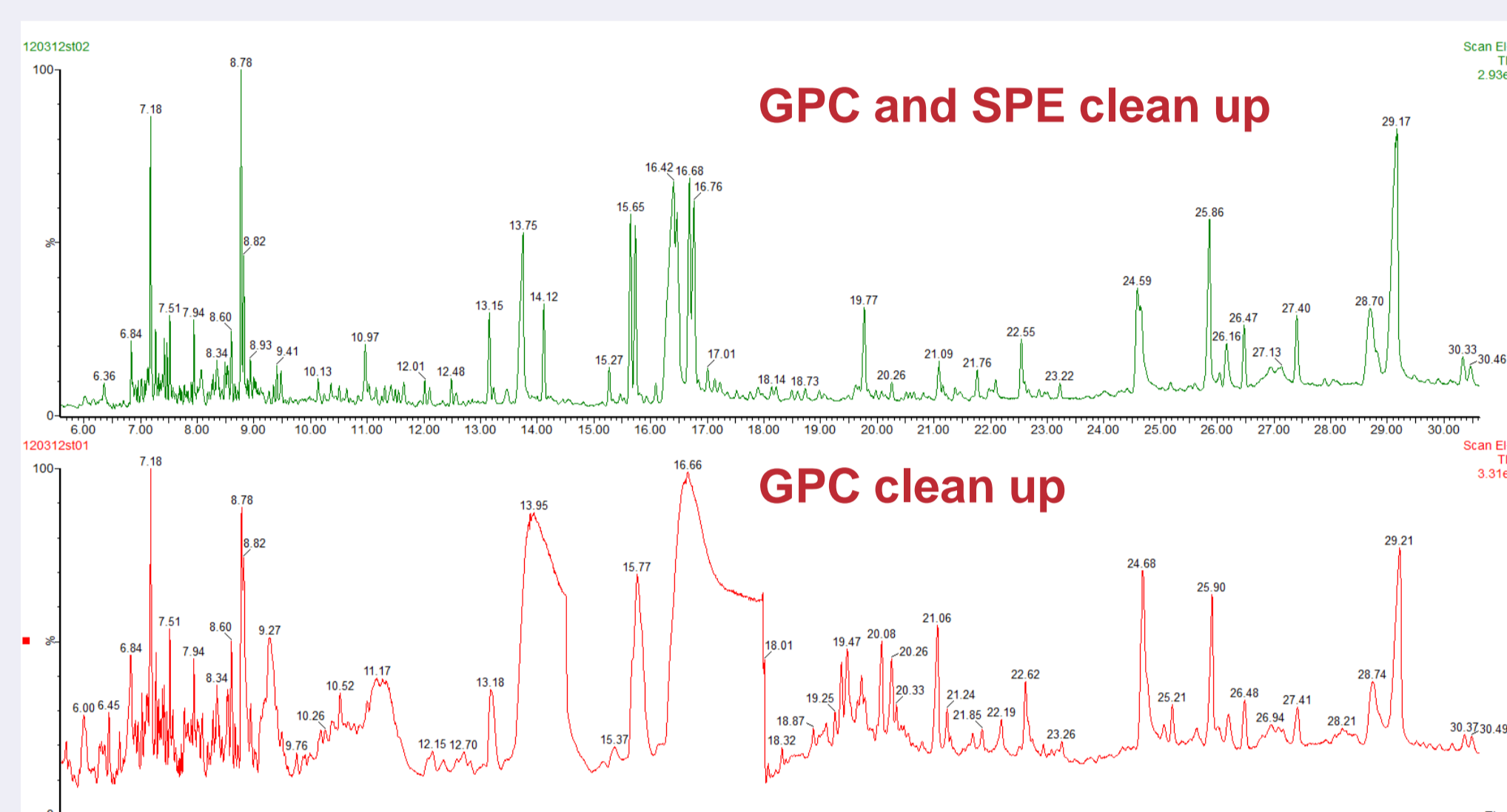


Figure 1. Chromatogramme of feed extract after GPC clean up and GPC and SPE clean-up

**Analytical method:** The homogenised sample was extracted by a Dionex ASE-300 ASE with acetone:hexane. The extract was concentrated into ethyl-acetat:cyclohexane and cleaned-up using Gilson ASPEC with a SX-3 GPC. Additional clean-up was performed after concentration into ethyl acetate, using NH<sub>2</sub> SPE columns with dichloromethane as eluent. The extract was concentrated and analysed by GC/MS/MS and LC/MS/MS. The SPE clean-up step caused some decrease in recoveries but without SPE clean-up the GC chromatography deteriorated significantly for a part of the eluting peaks, resulting in bad peak shape and shift in retention time, see Figure 1.

Table 1. Pesticide residues in 42 feed for laying hens samples collected in 20 EU member states

Country	2-Phenylphenol	Boscalid	Chlorpyrifos	Chlorpyrifos-methyl	Cypermethrin	Deltamethrin	Diflufenican	Endosulfan-alpha	Isoprothiolane	Malathion	Pririmphos-methyl	Procymidone
1				0.055							0.652	
1					0.096						0.105	
2						0.050				0.836	0.102	
2	0.018					0.046				0.159	0.101	
3	0.016											
3	0.025			0.012				0.121				
3				0.018								
3			0.015									
4	0.074											
4	0.043											
4		0.014									0.012	0.014
5				0.014	0.013	0.057						
5				0.019	0.042						0.013	
6	0.061										0.102	
6	0.010							0.012				
7												
7												
8												
8												
9						0.057			0.010		0.038	
9	0.014					0.028					0.012	
10											0.014	
10	0.016				0.014						0.012	
11	0.015											
11	0.014											
12												
12												
13												
13	0.009					0.045						
14												
14						0.013						
15											0.022	
16	0.010			0.012	0.015						0.461	
16												
17												
17				0.057							0.275	
18	0.107											
19	0.012			0.060							1.035	
19	0.015										0.037	
20	0.022			0.018	0.049	0.015					0.116	
20				0.010							0.059	
No of residues	17	1	3	9	6	4	3	2	1	2	18	1
Minimum	0.009	0.014	0.010	0.012	0.013	0.028	0.013	0.012	0.010	0.159	0.012	0.014
Maximum	0.107	0.014	0.018	0.060	0.096	0.057	0.050	0.121	0.010	0.836	1.03	0.014

**Validation:** A validation was performed according to SANCO/12495/2011 on a mixture of feed samples, which were composed by different kind of cereals, oils, vitamins and minerals. Five parallel extractions at two concentration levels (0.01 and 0.1 mg/kg) were analysed. Recoveries between 70-120% were obtained for 90 GC/MS/MS and 48 LC/MS/MS compounds. Additionally, 25 compounds resulted in recoveries between 60-70% or 120-140% and 47 compounds below 50%. The LOQs obtained for most the validated compounds were between 0.01-0.02 mg/kg. In parallel with the analysis of the feed samples, EUPT-C6 and EUPT test materials were analysed twice. All validated pesticide residues were detected and z-scores were between ±2.0 (median -1.0).

**Results:** Pesticides residues were found in 31 of the 42 samples. In total 67 residues was detected at levels between 0.009 and 1.03 mg/kg, see Table 1. Twelve different pesticides were detected, mainly organo phosphorous pesticides (48 %). The most frequently found pesticides were pirimiphos-methyl (0.012-1.03 mg/kg) and 2-phenyl-phenol (0.009-0.107 mg/kg), both pesticides used post-harvest, the latter probably used for packing materials. The feed with the highest residues, pirimiphos-methyl, 1.03 mg/kg contained 62 % oat, wheat and maize for which MRL are 5 mg/kg. If the pirimiphos-methyl originated from the cereals in the feed, the MRL were not exceeded.

**Conclusions:** Validation of a method for mixed feeding stuff was performed successfully for 146 compounds. Analysis of 42 samples from 20 EU member states resulted in 67 findings of 12 different pesticides in 31 of the feed samples. None of the sample exceeded the MRLs, if the residues originated from the constituent with the highest MRL.

<sup>1</sup> EURL-CF: EU Reference Laboratory for pesticide Residues in Cereals and Feeding stuff . DTU National Food Institute, Moerkhoej Bygade 19, DK-2860 Soeborg, Denmark  
e-mail : [crccereals@food.dtu.dk](mailto:crccereals@food.dtu.dk), [www.crl-pesticides.eu](http://www.crl-pesticides.eu)