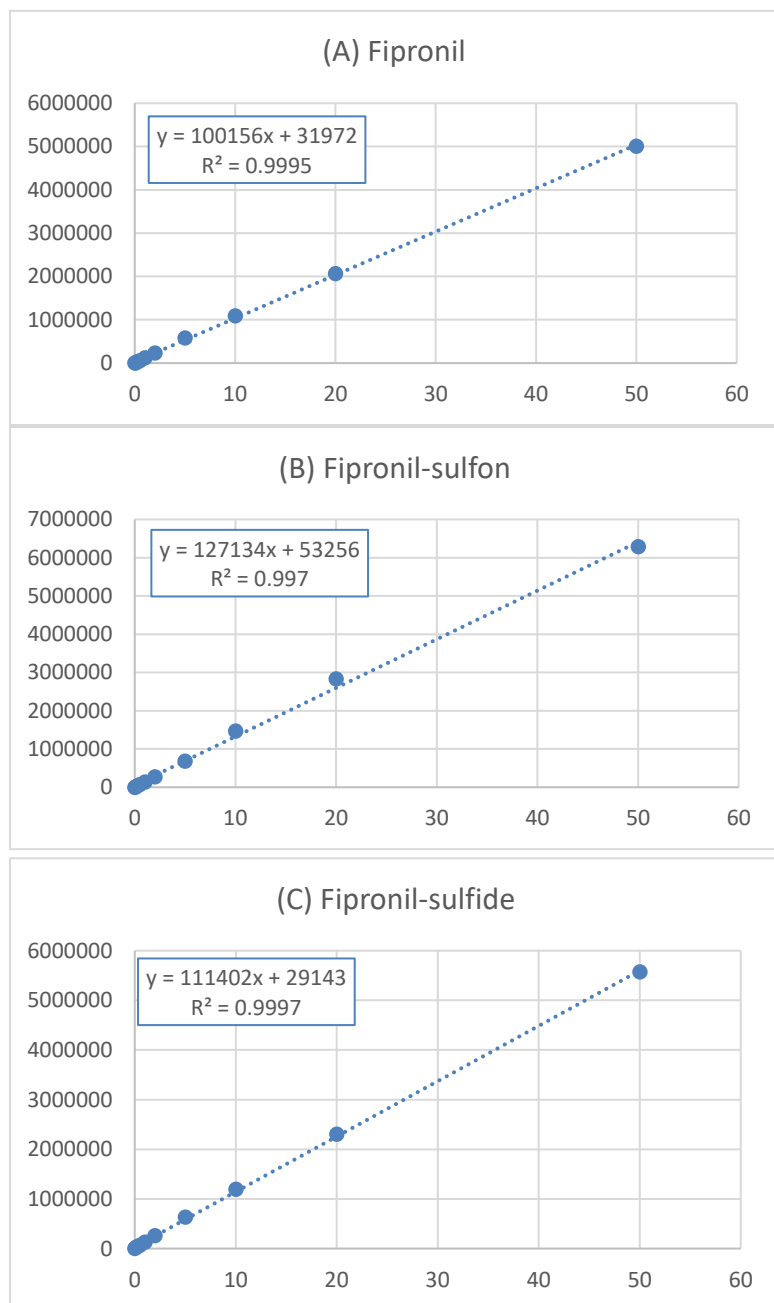
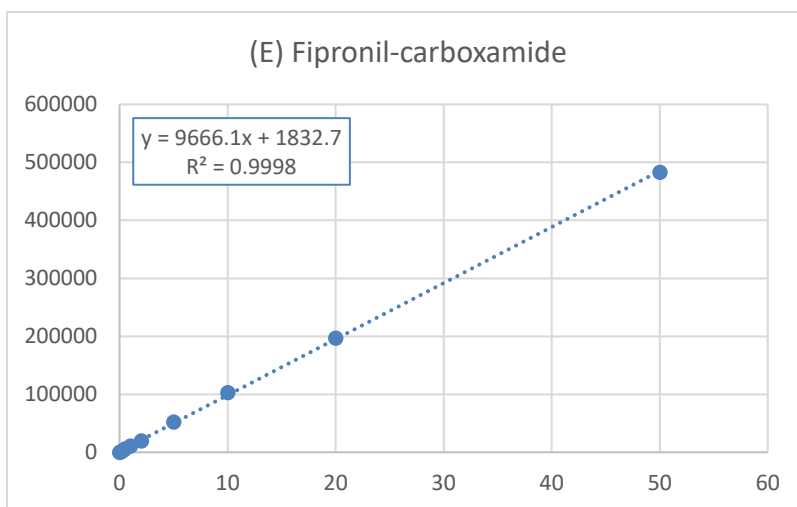
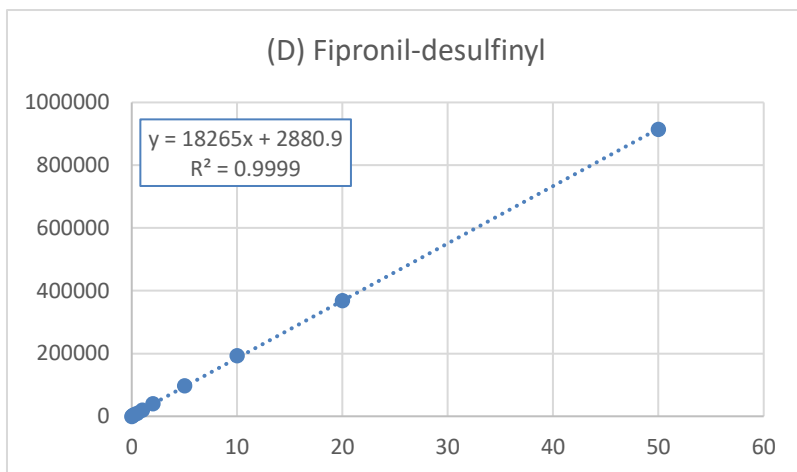


Figure S1 Calibration curves of (A) fipronil, (B), fipronil-sulfon, (C) fipronil-sulfide, (D) fipronil-desulfinitil, and (E) fipronil-carboxamide. Recovery is determined by adding 0.4 and 10 ng/g of the quantified components to blank material and quantification is performed by bracketed calibration upon a reference solution of 5 ng/ml. The resulted concentration of the quantified components is assessed against the theoretical concentration. Quantitation parameters are given in the Table below.





Quantitation parameters

Active substance/metabolite	Recovery	RSD _r	N=	Range (ng/g)	Guidance value
Fipronil	76-84%	3.9%	6	0.4-10	70-120%
Fipronil-sulfon	74-91%	8.5%	6	0.4-10	70-120%
Fipronil-sulfide	77-95%	9.5%	4	0.4-10	70-120%
Fipronil-desulfinyl	78-91%	7.2%	4	0.4-10	70-120%
Fipronil-carboxamide	77-96%	7.8%	4	0.4-10	70-120%

Additional information.

The obtained recoveries are well within the guidance values (70-120%) as set in SANTE/11813/2017, "Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed". When the values are within the guidance values no correction has to be applied to the sample results, therefore no correction was applied.

Identification parameters

Active substance/metabolite	Retention time (min)	Retention time range (min)	Guidance value (min)	Ion ratio	Ion ratio range	Guidance value
Fipronil	5.16	5.16-5.17	±0.1	20%	17-20%	±30%
Fipronil-sulfon	5.27	5.27-5.27	±0.1	88%	80-105%	±30%
Fipronil-sulfide	5.21	5.20-5.21	±0.1	92%	82-119%	±30%
Fipronil-desulfinyl	5.10	5.08-5.18	±0.1	28%	21-28%	±30%
Fipronil-carboxamide	4.58	4.57-4.59	±0.1	104%	78-111%	±30%