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Environmental behaviors of (E) pyriminobac-methyl in agricultural soils

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Table S1

Basic physicochemical properties of the selected soils.

Soil sample	Soil type[a]	Latitude/ Longitude	Location	Texture	pH	OM[b] (%)	CEC[c] (cmol kg^{-1})	Clay (%)
S1	Phaeozem	41°36’ N/ 127°53’ E	Heilongjiang	Sandy loam	6.38	4.64	30.36	64.42
S2	Anthrosol	29°14’N/ 121°48’ E	Zhejiang	Loam	7.85	1.66	12.90	42.10
S3	Ferralsol	28°46’ N/ 115°36’ E	Jiangxi	Sandy loam	5.21	0.35	11.99	15.67
S4	Alisol	35°06’ N/ 118°21’ E	Shandong	Sandy loam	6.78	1.20	12.19	23.47
S5	Plinthosol	19°32’N/ 110°10’ E	Hainan	Silt loam	5.79	0.88	11.00	43.83

[a] Soil classification according to the World Reference Base for Soil Resources.

[b] OM: Organic matter content.

[c] CEC: Cation exchange capacity

The selective ion chromatograms of EPM in acetonitrile, paddy water, soil samples spiked at 0 and 0.1 mg kg^{-1} were shown in Fig. S2 (A-G). The results of linearity, LOD, LOQ, and matrix effect were summarized in Table S2. The calibration curve of EPM (0.005, 0.01, 0.05, 0.1, 0.5, and 1mg kg^{-1}) showed a high correlation coefficient ($R^2 > 0.994$) in all matrices. To evaluate its specificity, the method was applied to blank samples of different matrices. No interference was detected during the retention time. The LODs of EPM ranged from 0.001 to 0.013 mg kg^{-1}, while the LOQs were 0.004 – 0.049 mg kg^{-1} for all samples. All matrices had moderate matrix effects (ME < 10%), thus, the matrix standard curve could be ignored (Li et al., 2019).

Five parallel tests were conducted for each matrix spiked with EPM at three different levels (0.005, 0.01, and 0.1 mg kg^{-1}). After sample pretreatment by the optimized QuEChERS procedure, the recovery of EPM in the various matrices ranged between 90.95% and 110.12%, with RSDs of 1.3% – 9.8% for repeatability (Table S3), and with RSDs of 3.63% – 8.49% for repeatability (Table S4). Five parallel tests were conducted for the blank matrix of soil samples spiked at 0.005, 0.01 and 0.1 mg kg^{-1} of EPM, respectively, and the chromatograms were shown in Fig. S3 (A-C). Chromatograms of EPM on the five columns of one batche and on the five columns of the different batches were shown in Fig. S4 (A-B). Thus, the developed analytical method fulfills the requirements of SANTE/11813/2017 guidelines and fall within the range of 70–120 % for recovery and less than 20% for RSD (Sante, 2017).

References:

Li, W., Zhang, Y., Jia, H., Zhou, W., Li, B., and Huang, H.: Residue analysis of tetraniliprole in rice and related environmental samples by HPLC/MS, Microchemical Journal, 150, 104168, https://doi.org/10.1016/j.microc.2019.104168, 2019.

SANTE: Guidance document on analytical quality control and method validation procedures for pesticide residues analysis in food and feed, 2017.

Table S2

Linear regression equation, limit of detection (LOD), limit of quantification (LOQ), and matrix effect for EPM in various matrices

Matrices	Linear Equation	R^2	LOD (mg kg^{-1})	LOQ (mg kg^{-1})	Matrix effect (%)
Acetonitrile	y = 254868 x +2080.3	0.9999	-	-	-
Paddy water	y = 188630 x + 3059.2	0.9983	0.012	0.045	1.84
S1	y = 172440 x + 3779.2	0.9961	0.002	0.008	1.86
S2	y = 141678 x + 2501.3	0.9949	0.001	0.004	1.36
S3	y = 138732 x + 492.6	0.9985	0.011	0.042	1.01
S4	y = 1662958x + 2004.9	0.9990	0.0021	0.0069	1.52
S5	y = 152673 x + 1517.1	0.9991	0.013	0.049	1.48

Table S3

Recovery and relative standard deviation (RSD) of EPM in various matrices spiked at levels of 0.005, 0.01, and 0.1 mg kg^{-1} (n=5)

Matrices	Spiked level	Recovery(%)					Mean recovery	RSD
	(mg kg^{-1})	1	2	3	4	5	(%)	(%)
Paddy water	0.005	93.51	107.03	91.03	90.95	104.35	97.37	7.93
	0.01	91.96	96.10	97.76	101.58	110.12	99.50	6.90
	0.1	93.93	92.88	103.45	108.15	109.67	101.62	7.72
S1	0.005	104.94	99.57	100.35	102.84	102.35	102.01	2.09
	0.01	108.01	93.85	94.10	94.22	104.79	98.99	6.93
	0.1	98.22	102.26	108.82	97.82	97.26	100.88	4.82
S2	0.005	100.73	109.29	91.89	93.56	108.38	100.77	8.02
	0.01	102.67	95.22	93.22	103.30	102.91	99.46	4.87
	0.1	93.09	95.27	109.84	90.19	95.39	96.76	7.87
S3	0.005	91.10	91.72	104.98	107.54	104.22	99.91	7.87
	0.01	92.17	109.97	108.62	91.22	97.30	99.86	8.95
	0.1	108.94	92.88	91.52	95.18	95.98	96.90	7.18
S4	0.005	103.74	103.22	101.45	99.57	104.34	102.46	1.90
	0.01	98.65	98.07	99.34	97.45	94.12	97.53	2.08
	0.1	100.20	93.41	90.42	91.05	91.07	93.23	4.35
S5	0.005	100.09	98.40	104.96	105.42	97.88	101.35	3.55
	0.01	97.21	102.68	94.47	96.10	92.12	96.52	4.09
	0.1	100.09	92.77	93.50	93.92	98.87	95.83	3.53

Table S4

Reproducibility of the rention time, precursor signal, and retention factor of EPM

	RSD (%)	
	Column-to-column reproducibility on five columns	Batch-to-batch reproducibility on six batches
Rention time	4.89	6.01
Precursor signal	7.27	8.49
Retention factor	3.63	5.87

Table S5

Results of the linear correlation analysis conducted on the Freundlich adsorption–desorption constants for EPM and the soil physicochemical properties.

Parameter	Adsorption					Desorption				
	pH	CEC (cmol kg^{-1})	Clay (%)	OM (%)	OC (%)	pH	CEC (cmol kg^{-1})	Clay (%)	OM (%)	OC (%)
Slope	0.0007	0.6078	1.1095	0.1197	0.0694	0.0558	4.7356	9.1508	0.9421	0.5465
Intercept	6.4075	10.7430	28.8710	0.7724	0.4481	6.2782	5.1749	17.5830	−0.3455	−0.2004
P	0.9880	0.0006	0.1170	0.0110	0.0110	0.8830	0.0120	0.1080	0.0240	0.0240
R^2	0.0008	0.9925	0.6144	0.9160	0.9160	0.0085	0.9107	0.6317	0.8582	0.8582

Notes: CEC: soil cation exchange capacity; OC: soil organic carbon content; OM: soil organic matter content.

Table S6

Linear correlation analysis between the half-life ($t_{1/2}$) of EPM and the soil physicochemical properties under different conditions.

Parameter	Aerobic				Anaerobic				Sterilized			
	pH	CEC (cmol kg^{-1})	Clay (%)	OM (%)	pH	CEC (cmol kg^{-1})	Clay (%)	OM (%)	pH	CEC (cmol kg^{-1})	Clay (%)	OM (%)
Slope	−0.0593	−0.9124	−2.0192	−0.2032	−0.0775	−1.0042	−2.1455	−0.2276	−0.4809	−0.7979	−4.6080	−0.3591
Intercept	9.3711	61.3570	138.9700	11.9190	10.4450	68.0520	149.7700	13.6160	36.6770	65.9150	327.9800	24.3530
P	0.4160	0.0400	0.0650	0.0050	0.3450	0.0640	0.1130	0.0140	0.0170	0.7590	0.4200	0.4830
R^2	0.2276	0.8022	0.7299	0.9478	0.2939	0.7343	0.6226	0.8983	0.8850	0.0363	0.2248	0.1750

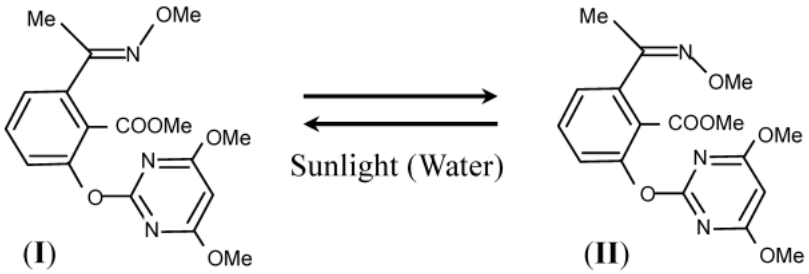


Fig. S1 Chemical structure of EPM(I) and ZPM(II).

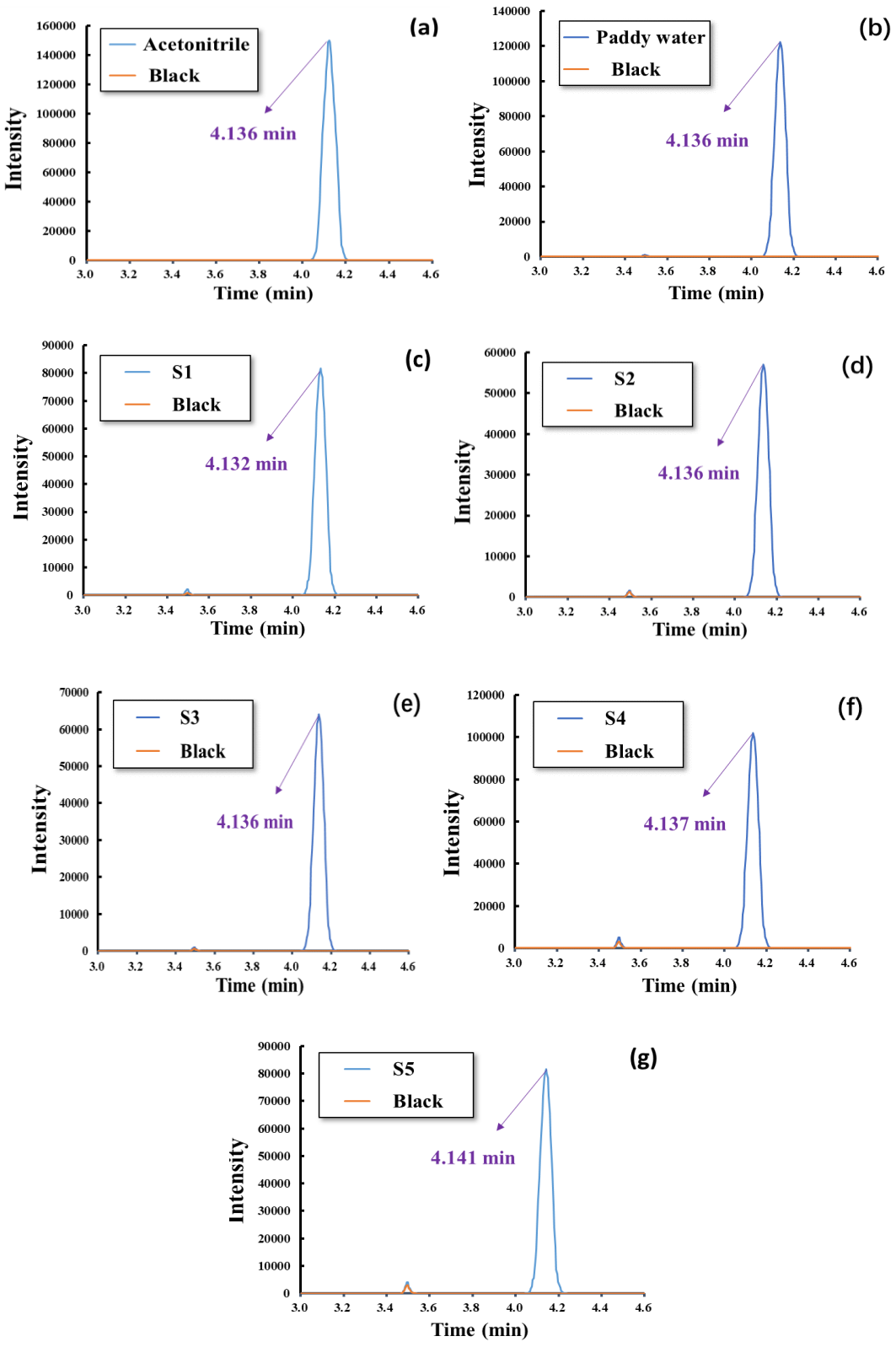
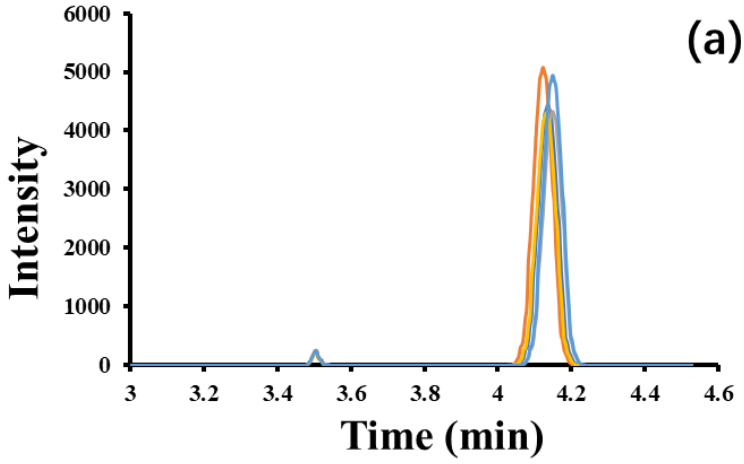


Fig. S2 The selective ion chromatograms of EPM spiked at 0 and 0.1 mg kg^{-1}(A: acetonitrile; B: paddy water; C: S1; D: S2; E: S3; F: S4; G: S5) (S1 to S5 are defined in Table 1)



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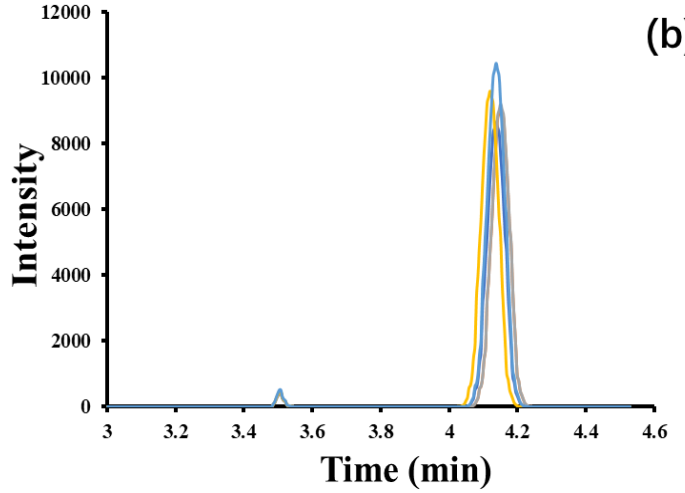

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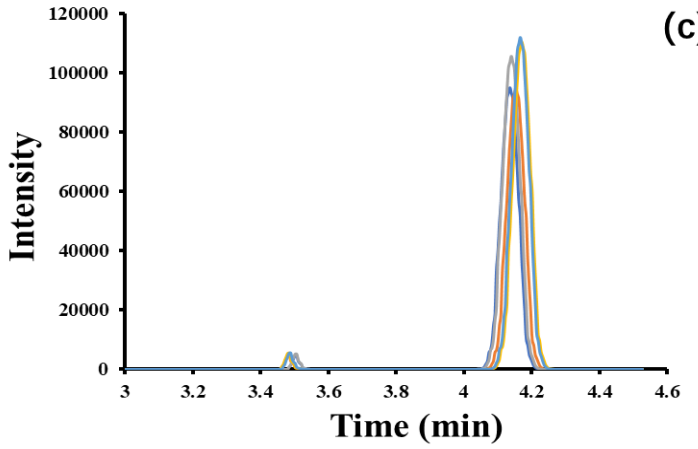

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135 **Fig. S3** Recovery of EPM in S1 sample spiked at levels of 0.005(a), 0.01(b), and 0.1(c) mg kg^{-1} (n=5)

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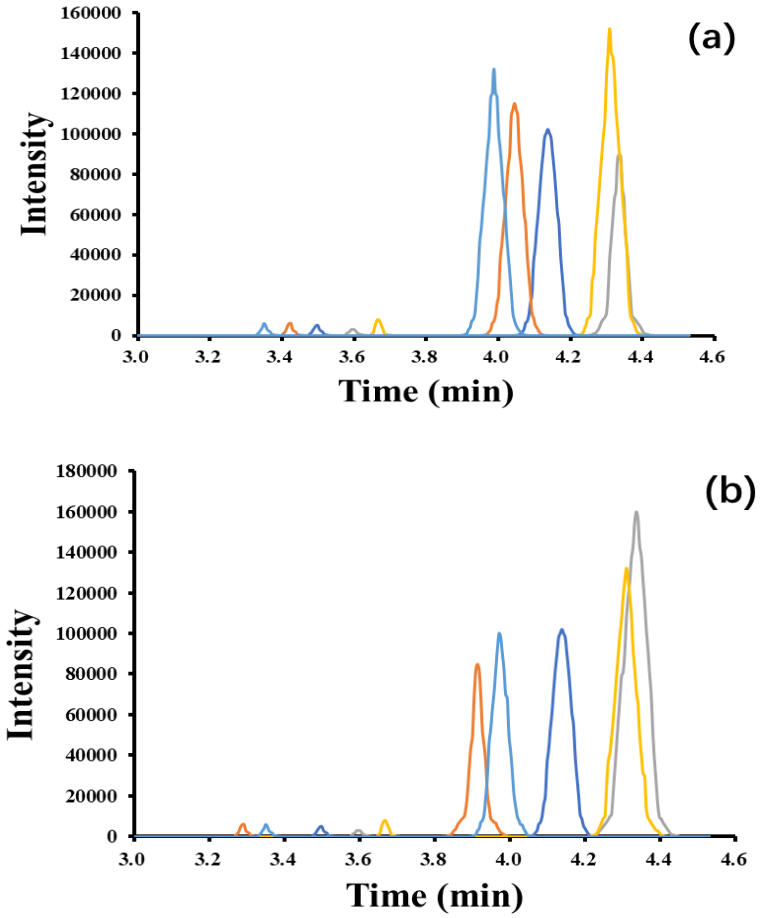

Fig. S4 Chromatograms of EPM in S1 sample on the five columns of the first batche (A) and on the five columns of the different batches (B) samples spiked at 0.1 mg kg^{-1}