

Supplementary Materials

Dissipation Dynamics and Residue of Four Herbicides in Paddy Fields using HPLC-MS/MS and GC-MS

Table S1. Final residues of four herbicides in husked rice sample.

Herbicides	Dosage (g a.i.ha ⁻¹)	Sprayed Times	Husked Rice Residue (mg·kg ⁻¹)										
			BB-01	BB-02	BS-01	BS-02	HC-01	HC-02	JJ-01	JJ-02	TN-01	TN-02	
Pyrazosulfuron-ethyl	22.5	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	45	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Bensulfuron-methyl	26.2	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	52.4	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Acetochlor	52.5	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	105	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Butachlor	112.4	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	224.8	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

ND: <0.01 mg/kg (pyrazosulfuron-ethyl, bensulfuron-methyl and acetochlor); ND: <0.05 mg/kg (butachlor).

Table S2. Final residues of four herbicides in rice hull sample.

Herbicides	Dosage (g a.i.ha ⁻¹)	Sprayed Times	Rice Hull Residue (mg·kg ⁻¹)										
			BB-01	BB-02	BS-01	BS-02	HC-01	HC-02	JJ-01	JJ-02	TN-01	TN-02	
Pyrazosulfuron-ethyl	22.5	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	45	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Bensulfuron-methyl	26.2	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	52.4	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Acetochlor	52.5	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	105	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Butachlor	112.4	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	224.8	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

ND: <0.01 mg/kg (pyrazosulfuron-ethyl, bensulfuron-methyl and acetochlor); ND: <0.05 mg/kg (butachlor).

Table S3. Comparison of QuEchERS-HPLC-MS with other analytical methods for determination of sulfonylurea herbicides.

Method	Analyte	LOD (ng)	Sample Volume (mL)	Average Recovery (%)	RSD (%)	Ref.
SPE-LC-DAD ^a	pyrazosulfuron-ethyl	300	10	79.2–92.1	9.9–14.8	[1]
	bensulfuron-methyl	300	10	71.4–93.8	7.7–12.9	
SPE-LC-MS ^a	pyrazosulfuron-ethyl	5	500	92.5–98.5	0.4–1.6	[2]
	bensulfuron-methyl	5	500	99.0–104.9	0.6–1.6	
SPE-LC-MS ^a	pyrazosulfuron-ethyl	56–320	5	74.5–114.6	1.4–17.3	[3]
	bensulfuron-methyl	120–630	5	61.1–110.2	2.5–19.4	
QuEchERS -HPLC-MS	pyrazosulfuron-ethyl	0.1–0.25	5	81–102.8	1.5–6.0	This method
	bensulfuron-methyl	0.1–0.25	5	78.9–102.7	3.1–14.9	

^a:C₁₈.**Table S4.** Comparison of QuEchERS-GC-MS with other analytical methods for determination of amide herbicides.

Method	Analyte	LOD (ng)	Sample Volume (mL)	Average Recovery (%)	RSD (%)	Ref.
LLE-GC-MS	acetochlor	2 × 10 ⁴	10	84.5–93.5	2.20–5.82	[4]
	butachlor	2 × 10 ⁴	10	66.6–89.5	2.74–7.35	
SPME-GC-MS	acetochlor	1.2	10	90	6.9	[5]
	butachlor	2.7	10	79	8.1	
DLPME-GC-MS	acetochlor	40	5	80.3–108.8	2.9	[6]
	butachlor	3	5	85.2–108.5	1.6	
QuEchERS -GC-MS	acetochlor	0.04–1	5	87.4–99.5	0.9–9.7	This method
	butachlor	0.2–1	5	86.3–108	2.2–9.2	

References

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