Electronic Supplementary Material

Multiresidue method for determining multiclass acidic pesticides in agricultural foods by liquid chromatography–tandem mass spectrometry

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Extraction

↓ Weigh sample (cereal grains and legumes: 10.0 g; vegetables and fruits: 20.0 g)

↓ For cereal grains and legumes, add water (20 mL) and allow to stand for 30 min

↓ Add acetonitrile (50 mL) and hydrochloric acid (0.5 mol/L, 5 mL), homogenize for 1 min, and then filter with suction

↓ Add acetonitrile (20 mL) to the residue, homogenize for 1 min, and then filter with suction

↓ Combine the filtrates and adjust the volume to 100 mL with acetonitrile (A)

Salting-out

↓ Add acetonitrile (5 mL), sodium chloride (10 g), and phosphate buffer (0.1 mol/L, pH 2.1, 15 mL) to a 10-mL aliquot of (A)

↓ Shake for 5 min and then centrifuge (3000 rpm (1863 *g*), 5 min)

ODS cartridge cleanup

↓ Condition with acetonitrile (10 mL)

↓ Load the acetonitrile layer

↓ Elute with acetonitrile (5 mL)

↓ Evaporate to dryness

↓ Dissolve the residue in acetone/hexane (1:1, v/v, 2 mL) (B)

PSA cartridge cleanup

↓ Condition with acetone/hexane (1:1, v/v, 10 mL)

↓ Load (B)

↓ Wash with acetone/hexane (1:1, v/v, 8 mL)

↓ Elute with acetone/hexane/formic acid (25:25:1, v/v, 15 mL)

↓ Evaporate to dryness

↓ Dissolve the residue in methanol (cereal grains and legumes: 2 mL; vegetables and fruits: 4 mL)

LC-MS/MS analysis

Fig. S1 Sample preparation flow chart of the developed method.

Extraction

↓ Weigh sample (cereal grains and legumes: 10.0 g; vegetables and fruits: 20.0 g)

↓ For cereal grains and legumes, add water (20 mL) and allow to stand for 30 min

↓ Add acetonitrile (50 mL) and homogenize for 1 min, and then filter with suction

↓ Add acetonitrile (20 mL) to the residue, homogenize for 1 min, and then filter with suction

↓ Combine the filtrates and adjust the volume to 100 mL with acetonitrile (A)

Salting-out

↓ Add sodium chloride (10 g) and phosphate buffer (0.5 mol/L, pH 7.0, 20 mL) to a 20-mL aliquot of (A)

↓ Shake for 5 min and then centrifuge (3000 rpm (1863 *g*), 5 min)

ODS cartridge cleanup

↓ Condition with acetonitrile (10 mL)

↓ Load the acetonitrile layer

↓ Elute with acetonitrile (5 mL)

↓ Evaporate to dryness

↓ Dissolve the residue in acetonitrile/toluene (3:1, v/v, 2 mL) (B)

GCB/PSA (or GCB/aminopropyl) cartridge cleanup

↓ Condition with acetonitrile/toluene (3:1, v/v, 10 mL)

↓ Load (B)

↓ Elute with acetonitrile/toluene (3:1, v/v, 20 mL)

↓ Evaporate to dryness

↓ Dissolve the residue in methanol (4 mL)

LC-MS/MS analysis

Fig. S2 Sample preparation flow chart of the official Japanese method.

ODS cartridge cleanup is omitted for vegetables and fruits.

Table S1 Optimized parameters and retention times for LC-MS/MS analysis of the target compounds

Compound	Retention time (min)	Ionization mode	Quantifier ion					Qualifier ion				
			Precursor ion (*m/z*)	Product ion (*m/z*)	DP[a] (V)	CE[b] (eV)	CXP[c] (V)	Precursor ion (*m/z*)	Product ion (*m/z*)	DP[a] (V)	CE[b] (eV)	CXP[c] (V)
1-Naphthaleneacetic acid	14.4	ESI(-)	185.0	140.9	-28	-15	-13	–	–	–	–	–
2,4,5-T	16.2	ESI(-)	252.9	194.8	-55	-18	-15	254.9[d]	196.8	-55	-18	-15
2,4-D	14.3	ESI(-)	218.9	160.9	-52	-19	-13	220.9[d]	162.9	-52	-19	-13
2,4-DB	17.5	ESI(-)	246.9	160.9	-40	-17	-11	246.9	125.0	-40	-37	-11
4-CPA	12.1	ESI(-)	184.9	126.9	-52	-19	-13	186.9[d]	128.9	-52	-19	-13
Acifluorfen	17.1	ESI(-)	359.9	315.9	-64	-13	-25	359.9	194.9	-64	-36	-17
Azimsulfuron	15.3	ESI(-)	423.0	213.9	-76	-21	-15	423.0	135.0	-76	-42	-11
Bensulfuron methyl	15.9	ESI(+)	411.0	149.0	94	26	13	411.0	91.0	94	80	17
Bentazone	11.9	ESI(-)	238.9	131.9	-124	-40	-13	238.9	196.8	-124	-28	-17
Bispyribac sodium	16.7	ESI(+)	431.1	275.0	94	19	17	431.1	119.0	94	57	11
Bromoxynil	14.3	ESI(-)	273.7	78.8	-83	-63	-15	275.7[d]	80.8	-83	-63	-15
Chlorimuron ethyl	17.1	ESI(+)	414.9	185.9	85	25	15	414.9	82.9	85	34	15
Chlorsulfuron	13.7	ESI(+)	357.9	141.0	91	24	15	357.9	167.0	91	24	15
Clodinafop acid	16.8	ESI(-)	309.9	237.9	-77	-19	-15	309.9	217.9	-77	-29	-15
Cloransulam methyl	13.7	ESI(+)	430.0	397.9	80	19	15	430.0	369.9	80	29	15
Cyclanilide	17.9	ESI(-)	271.9	159.9	-53	-28	-15	273.9[d]	161.9	-53	-28	-15
Cyclosulfamuron	17.8	ESI(-)	420.0	238.9	-59	-21	-15	420.0	264.9	-59	-16	-17

Dicamba	10.2	ESI(-)	218.9	174.7	-10	-10	-15	218.9	145.0	-10	-10	-15
Dichlorprop	16.2	ESI(-)	232.9	160.8	-49	-18	-15	232.9	124.9	-49	-38	-11
Diclosulam	14.3	ESI(+)	405.9	160.9	91	34	15	407.9[d]	162.9	91	34	15
Diflufenzopyr	13.6	ESI(+)	335.0	205.9	81	16	15	335.0	162.0	81	27	13
Ethametsulfuron methyl	14.3	ESI(+)	411.0	196.0	95	22	15	411.0	168.0	95	40	13
Ethoxysulfuron	17.2	ESI(+)	399.0	260.9	91	22	17	399.0	217.9	91	34	17
Fenoprop	17.8	ESI(-)	266.9	194.8	-49	-17	-15	266.9	158.9	-49	-40	-11
Fenoxaprop	18.4	ESI(-)	332.0	151.9	-70	-30	-15	332.0	259.9	-70	-18	-17
Flazasulfuron	15.7	ESI(+)	407.9	182.0	103	25	15	407.9	83.0	103	63	15
Florasulam	11.2	ESI(+)	359.9	129.0	109	30	11	359.9	109.0	109	79	11
Fluazifop	16.4	ESI(+)	328.0	282.0	124	26	17	328.0	254.0	124	35	17
Flucarbazone sodium	10.3	ESI(-)	394.9	112.9	-61	-43	-11	394.9	128.0	-61	-17	-11
Flucetosulfuron	16.0	ESI(+)	488.1	156.0	104	25	15	488.1	272.9	104	34	17
Flumetsulam	8.6	ESI(-)	323.9	133.0	-75	-27	-11	323.9	65.9	-75	-78	-13
Fluroxypyr	11.8	ESI(+)	254.9	208.9	61	21	15	254.9	180.9	61	30	13
Flusulfamide	19.4	ESI(-)	412.8	170.9	-112	-47	-17	412.8	348.9	-112	-38	-30
Fomesafen	16.6	ESI(-)	436.9	194.9	-82	-51	-17	436.9	221.9	-82	-45	-15
Gibberellin A_3	8.6	ESI(-)	345.0	143.0	-76	-36	-11	345.0	239.0	-76	-21	-15
Halosulfuron methyl	18.0	ESI(-)	432.9	251.8	-79	-24	-19	434.9[d]	253.8	-79	-24	-19
Haloxyfop	18.6	ESI(-)	359.9	287.8	-67	-21	-19	361.9[d]	289.8	-67	-21	-19
Imazamox	9.4	ESI(+)	306.1	261.0	106	28	17	306.1	193.0	106	35	15

Imazapic	9.8	ESI(+)	276.0	231.0	98	27	15	276.0	163.0	98	35	15
Imazapyr	7.7	ESI(+)	262.0	217.0	87	26	15	262.0	202.0	87	31	15
Imazaquin	13.0	ESI(+)	312.0	267.0	108	29	17	312.0	198.9	108	38	15
Imazethapyr	11.7	ESI(+)	290.1	245.0	91	28	17	290.1	177.0	91	36	15
Imazosulfuron	17.1	ESI(-)	410.9	229.8	-80	-22	-15	410.9	154.0	-80	-31	-11
Iodosulfuron methyl	15.8	ESI(-)	505.9	139.0	-78	-25	-13	505.9	307.8	-78	-25	-25
Ioxynil	15.5	ESI(-)	369.5	126.7	-156	-49	-13	369.5	214.8	-156	-42	-17
MCPA	14.9	ESI(-)	199.0	140.9	-52	-20	-15	201.0[d]	142.9	-52	-20	-15
MCPB	17.6	ESI(-)	226.9	140.9	-47	-19	-11	228.9[d]	142.9	-47	-19	-11
Mecoprop	16.5	ESI(-)	212.9	140.9	-55	-18	-15	214.9[d]	142.9	-55	-18	-15
Mesosulfuron methyl	14.6	ESI(-)	502.1	266.8	-80	-35	-17	502.1	346.9	-80	-21	-35
Mesotrione	9.8	ESI(-)	338.0	290.9	-62	-14	-19	338.0	211.9	-62	-43	-15
Metazosulfuron	16.7	ESI(-)	474.0	292.9	-64	-27	-19	476.0[d]	294.9	-64	-27	-19
Metosulam	13.4	ESI(+)	417.9	174.9	98	35	15	419.9[d]	176.9	98	35	15
Metsulfuron methyl	13.1	ESI(+)	381.9	166.9	85	21	13	381.9	198.9	85	30	15
Nicosulfuron	12.5	ESI(+)	411.0	181.9	98	26	13	411.0	212.9	98	23	15
Penoxsulam	14.0	ESI(+)	484.0	195.0	109	37	17	484.0	444.0	109	34	15
Primisulfuron methyl	17.1	ESI(-)	467.0	225.8	-106	-16	-15	467.0	175.9	-106	-42	-15
Propoxycarbazone	12.2	ESI(-)	397.0	112.9	-59	-40	-11	397.0	156.0	-59	-16	-15
Propyrisulfuron	17.1	ESI(+)	456.0	196.0	92	20	15	456.0	260.9	92	24	17
Prosulfuron	16.4	ESI(+)	420.0	141.0	94	25	11	420.0	167.0	94	25	15

Pyrasulfotole	10.9	ESI(-)	360.9	63.9	-84	-84	-13	360.9	78.9	-84	-17	-15
Pyrazosulfuron ethyl	17.8	ESI(+)	415.0	182.0	83	25	13	415.0	139.0	83	61	15
Pyrimisulfan	15.5	ESI(+)	420.0	369.9	80	26	30	420.0	255.0	80	37	17
Pyrithiobac sodium	15.5	ESI(+)	326.9	139.0	70	38	13	326.9	203.9	70	51	15
Quinclorac	10.7	ESI(+)	241.9	161.0	58	51	13	241.9	195.9	58	39	15
Quizalofop	18.4	ESI(-)	343.0	270.9	-62	-21	-19	345.0[d]	272.9	-62	-21	-19
Rimsulfuron	13.7	ESI(+)	432.0	182.0	98	30	15	432.0	325.0	98	21	25
Saflufenacil	15.8	ESI(+)	501.0	348.9	139	38	30	501.0	197.9	139	59	15
Sulfosulfuron	16.0	ESI(+)	471.0	210.9	73	19	15	471.0	260.9	73	26	17
Thifensulfuron methyl	12.6	ESI(+)	387.9	167.0	84	21	11	387.9	204.9	84	36	15
Triasulfuron	12.6	ESI(+)	401.9	141.0	81	27	15	401.9	167.0	81	23	15
Tribenuron methyl	15.2	ESI(+)	396.0	155.0	94	18	15	396.0	181.0	94	27	15
Triclopyr	15.7	ESI(-)	253.8	195.8	-49	-17	-15	255.8[d]	197.8	-49	-17	-15
Trifloxysulfuron	14.6	ESI(+)	438.0	182.0	91	25	13	438.0	83.0	91	67	15
Triflusulfuron methyl	17.0	ESI(+)	493.0	263.9	98	31	17	493.0	96.0	98	68	17
Warfarin	16.8	ESI(+)	309.0	162.9	83	20	15	309.0	250.9	83	27	17

[a] DP: Declustering potential

[b] CE: Collision energy

[c] CXP: Collision cell exit potential

[d] An isotopic ion was used for the precursor ion.

Table S2 Classifications and p*K*a values of the target compounds

Compound	Class	Chemical family	p*K*a
1-Naphthaleneacetic acid	Plant growth regulator	Naphthalene	4.2
2,4,5-T	Herbicide, plant growth regulator	Phenoxy acid	2.9
2,4-D	Herbicide, plant growth regulator	Phenoxy acid	2.7
2,4-DB	Herbicide, plant growth regulator	Phenoxy acid	4.1
4-CPA	Herbicide, plant growth regulator	Phenoxy acid	3.2
Acifluorfen	Herbicide	Diphenyl ether	3.9
Azimsulfuron	Herbicide	Sulfonylurea	3.6
Bensulfuron methyl	Herbicide	Sulfonylurea	5.2
Bentazone	Herbicide	Benzothiadiazinone	3.3
Bispyribac sodium	Herbicide	Pyrimidinyloxybenzoic acid	3.4
Bromoxynil	Herbicide	Hydroxybenzonitrile	3.9
Chlorimuron ethyl	Herbicide	Sulfonylurea	4.2
Chlorsulfuron	Herbicide	Sulfonylurea	3.4
Clodinafop acid	Herbicide	Aryloxyphenoxypropionic	3.1
Cloransulam methyl	Herbicide	Triazolopyrimidine	4.8
Cyclanilide	Plant growth regulator	Unclassified	3.5
Cyclosulfamuron	Herbicide	Sulfonylurea	5.0
Dicamba	Herbicide, plant growth regulator	Benzonic acid	1.9
Dichlorprop	Herbicide, plant growth regulator	Phenoxy acid	3.0
Diclosulam	Herbicide	Triazolopyrimidine	4.0
Diflufenzopyr	Herbicide	Semicarbazone	3.2
Ethametsulfuron methyl	Herbicide	Sulfonylurea	4.2
Ethoxysulfuron	Herbicide	Sulfonylurea	5.3
Fenoprop	Herbicide, plant growth regulator	Phenoxy acid	2.8
Fenoxaprop	Herbicide	Aryloxyphenoxypropionic acid	3.2

Flazasulfuron	Herbicide	Sulfonylurea	4.4
Florasulam	Herbicide	Triazolopyrimidine	4.5
Fluazifop	Herbicide	Aryloxyphenoxypropionic acid	3.2
Flucarbazone sodium	Herbicide	Sulfonylaminocarbonyltriazolinone	1.9
Flucetosulfuron	Herbicide	Sulfonylurea	3.5
Flumetsulam	Herbicide	Triazolopyrimidine	4.6
Fluroxypyr	Herbicide	Pyridinecarboxylic acid	2.9
Flusulfamide	Fungicide	Benzenesulfonamide	4.9
Fomesafen	Herbicide	Diphenyl ether	2.8
Gibberellin A_3	Plant growth regulator	Tetracyclic di-terpenoid	4.0
Halosulfuron methyl	Herbicide	Sulfonylurea	3.4
Haloxyfop	Herbicide	Aryloxyphenoxypropionic acid	2.9
Imazamox	Herbicide	Imidazolinone	p*K*a1 2.3; p*K*a2 3.3; p*K*a3 10.8
Imazapic	Herbicide	Imidazolinone	p*K*a1 2.0; p*K*a2 3.6; p*K*a3 11.1
Imazapyr	Herbicide	Imidazolinone	p*K*a1 1.9; p*K*a2 3.6; p*K*a3 11.0
Imazaquin	Herbicide	Imidazolinone	3.8
Imazethapyr	Herbicide	Imidazolinone	p*K*a1 2.1; p*K*a2 3.9
Imazosulfuron	Herbicide	Sulfonylurea	p*K*a1 2.2; p*K*a2 3.8; p*K*a3 9.3
Iodosulfuron methyl	Herbicide	Sulfonylurea	3.2
Ioxynil	Herbicide	Hydroxybenzonitorile	4.1
MCPA	Herbicide	Phenoxy acid	3.7
MCPB	Herbicide	Phenoxy acid	4.5
Mecoprop	Herbicide	Phenoxy acid	3.8
Mesosulfuron methyl	Herbicide	Sulfonylurea	4.4
Mesotrione	Herbicide	Aroylcyclohexanedione	3.1
Metazosulfuron	Herbicide	Sulfonylurea	3.4
Metosulam	Herbicide	Triazolopyrimidine	4.8
Metsulfuron methyl	Herbicide	Sulfonylurea	3.8
Nicosulfuron	Herbicide	Sulfonylurea	4.6
Penoxsulam	Herbicide	Triazolopyrimidine	5.1

Primisulfuron methyl	Herbicide	Sulfonylurea	3.5
Propoxycarbazone	Herbicide	Sulfonylaminocarbonyltriazolinone	2.1
Propyrisulfuron	Herbicide	Sulfonylurea	4.9
Prosulfuron	Herbicide	Sulfonylurea	3.8
Pyrasulfotole	Herbicide	Benzoylpyrazole	4.2
Pyrazosulfuron ethyl	Herbicide	Sulfonylurea	3.7
Pyrimisulfan	Herbicide	Sulfonanilide	5.4
Pyrithiobac sodium	Herbicide	Pyrimidinylthiobenzoic acid	2.3
Quinclorac	Herbicide	Quinolinecarboxylic acid	4.3
Quizalofop	Herbicide	Aryloxyphenoxypropionic acid	3.1
Rimsulfuron	Herbicide	Sulfonylurea	4.0
Saflufenacil	Herbicide	Pyrimidindione	4.4
Sulfosulfuron	Herbicide	Sulfonylurea	3.5
Thifensulfuron methyl	Herbicide	Sulfonylurea	4.0
Triasulfuron	Herbicide	Sulfonylurea	4.6
Tribenuron methyl	Herbicide	Sulfonylurea	4.7
Triclopyr	Herbicide	Pyridinecarboxylic acid	4.0
Trifloxysulfuron	Herbicide	Sulfonylurea	4.8
Triflusulfuron methyl	Herbicide	Sulfonylurea	4.4
Warfarin	Rodenticide	Hydroxycoumarin	5.9

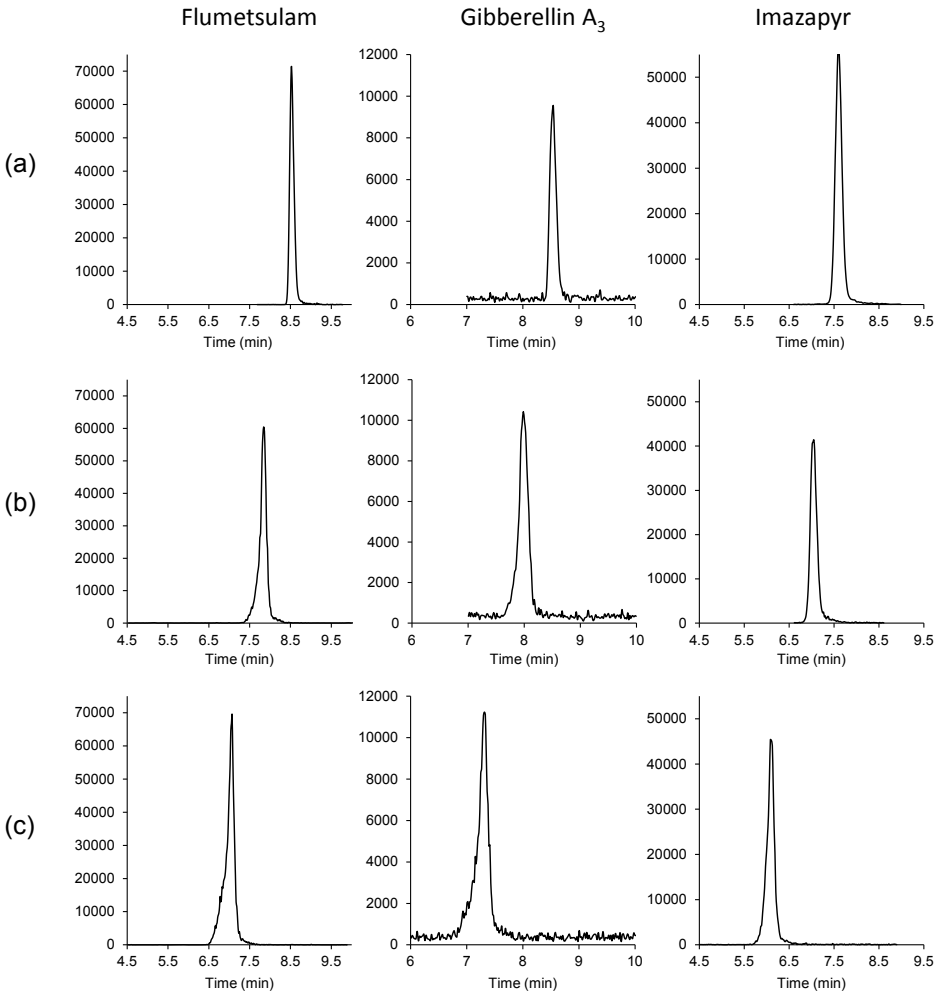

Fig. S3 Chromatograms of flumetsulam, gibberellin A3, and imazapyr using (a) Inertsil ODS-4, (b) InertSustainSwift C18, and (c) InertSustain C18 columns.

Table S3 Recoveries (%) of target compounds spiked in soybeans and extracted under various conditions at a concentration level of 0.01 mg/kg (n = 1)

	Condition 1	Condition 2	Condition 3	Condition 4	Condition 5
1-Naphthaleneacetic acid	84	99	86	88	86
2,4,5-T	74	83	73	90	86
2,4-D	75	84	79	87	85
2,4-DB	79	101	97	100	85
4-CPA	28	59	76	89	84
Acifluorfen	91	89	86	102	87
Azimsulfuron	89	98	81	87	80
Bensulfuron methyl	88	87	79	78	82
Bentazone	82	85	68	80	80
Bispyribac sodium	62	75	73	79	74
Bromoxynil	89	89	82	87	83
Chlorimuron ethyl	92	84	84	87	86
Chlorsulfuron	84	87	80	81	83
Clodinafop acid	90	94	83	91	89
Cloransulam methyl	86	88	81	76	86
Cyclanilide	85	86	81	90	84
Cyclosulfamuron	97	96	90	93	80
Dicamba	80	78	63	82	81
Dichlorprop	86	85	82	91	84
Diclosulam	92	90	81	85	81
Diflufenzopyr	79	72	69	73	62
Ethametsulfuron methyl	87	91	84	83	80
Ethoxysulfuron	96	85	88	90	88
Fenoprop	88	83	86	91	90
Fenoxaprop	83	86	74	78	57
Flazasulfuron	91	90	85	93	88
Florasulam	88	95	80	88	83
Fluazifop	101	88	85	93	88
Flucarbazone sodium	86	95	75	90	89
Flucetosulfuron	89	82	86	91	81
Flumetsulam	86	96	81	84	82
Fluroxypyr	87	85	81	85	85
Flusulfamide	86	94	83	85	81
Fomesafen	95	101	87	97	76

Gibberellin A_3	74	90	69	74	69
Halosulfuron methyl	93	90	88	90	85
Haloxyfop	83	89	79	87	79
Imazamox	70	65	69	70	64
Imazapic	69	68	71	69	58
Imazapyr	57	53	57	59	50
Imazaquin	81	73	76	77	69
Imazethapyr	78	77	75	74	67
Imazosulfuron	99	97	82	84	84
Iodosulfuron methyl	94	95	82	83	82
Ioxynil	86	89	78	82	83
MCPA	73	85	82	91	85
MCPB	86	98	89	97	84
Mecoprop	88	90	80	88	81
Mesosulfuron methyl	84	105	84	90	84
Mesotrione	79	101	87	93	83
Metazosulfuron	93	90	87	91	89
Metosulam	83	89	86	85	79
Metsulfuron methyl	88	86	84	87	77
Nicosulfuron	76	79	81	83	78
Penoxsulam	88	88	90	87	87
Primisulfuron methyl	101	94	97	98	91
Propoxycarbazone	89	96	76	89	85
Propyrisulfuron	92	85	84	91	83
Prosulfuron	112	89	90	93	87
Pyrasulfotole	81	87	78	80	75
Pyrazosulfuron ethyl	92	88	91	91	84
Pyrimisulfan	95	90	88	87	80
Pyrithiobac sodium	90	73	74	83	89
Quinclorac	83	70	69	86	83
Quizalofop	93	82	80	82	79
Rimsulfuron	83	91	81	83	81
Saflufenacil	89	89	94	95	87
Sulfosulfuron	90	86	86	89	80
Thifensulfuron methyl	90	84	87	87	83
Triasulfuron	90	94	83	81	86
Tribenuron methyl	106	100	102	98	100
Triclopyr	84	86	82	99	78

Trifloxysulfuron	92	90	87	91	84
Triflusulfuron methyl	89	84	81	83	85
Warfarin	90	82	77	77	78

The examined conditions are listed in Table 1.

Table S4 Recoveries (%) of target compounds following salting out using various solutions in the absence of a matrix

□	Solution (a)	Solution (b)	Solution (c)	Solution (d)	Solution (e)
1-Naphthaleneacetic acid	75	95	101	104	102
2,4,5-T	40	106	92	101	101
2,4-D	23	103	93	105	104
2,4-DB	98	93	95	102	101
4-CPA	10	104	90	101	102
Acifluorfen	96	108	98	105	103
Azimsulfuron	95	104	106	117	99
Bensulfuron methyl	100	100	107	100	98
Bentazone	102	104	106	109	94
Bispyribac sodium	84	96	98	93	91
Bromoxynil	102	84	88	97	95
Chlorimuron ethyl	99	99	108	100	99
Chlorsulfuron	95	97	90	95	94
Clodinafop acid	68	105	97	106	98
Cloransulam methyl	105	104	104	100	98
Cyclanilide	95	110	94	114	107
Cyclosulfamuron	98	104	101	107	96
Dicamba	30	100	94	92	97
Dichlorprop	56	102	96	106	99
Diclosulam	107	101	105	107	97
Diflufenzopyr	63	72	43	86	92
Ethametsulfuron methyl	92	95	107	107	94
Ethoxysulfuron	109	98	106	100	92
Fenoprop	79	99	93	108	98
Fenoxaprop	68	54	0	15	19
Flazasulfuron	87	97	104	100	96
Florasulam	106	102	105	102	96
Fluazifop	73	105	98	105	91
Flucarbazone sodium	95	104	104	109	98
Flucetosulfuron	96	97	105	100	96
Flumetsulam	91	98	104	108	96
Fluroxypyr	19	97	98	104	97
Flusulfamide	108	104	106	109	98
Fomesafen	101	104	103	110	97
Gibberellin A_3	5	60	51	93	93

Halosulfuron methyl	105	101	104	108	97
Haloxyfop	76	103	98	108	100
Imazamox	9	76	60	107	95
Imazapic	14	74	59	102	91
Imazapyr	4	67	46	106	83
Imazaquin	37	86	73	103	94
Imazethapyr	31	87	71	102	97
Imazosulfuron	99	103	106	111	101
Iodosulfuron methyl	105	105	95	108	98
Ioxynil	101	103	100	99	101
MCPA	27	104	92	104	104
MCPB	97	108	93	98	98
Mecoprop	59	99	94	93	95
Mesosulfuron methyl	88	101	108	113	101
Mesotrione	67	87	75	110	107
Metazosulfuron	101	94	106	104	97
Metosulam	108	106	108	91	92
Metsulfuron methyl	84	96	91	101	103
Nicosulfuron	29	94	99	96	103
Penoxsulam	102	99	86	101	98
Primisulfuron methyl	106	103	104	102	96
Propoxycarbazone	93	106	106	105	99
Propyrisulfuron	103	104	107	102	101
Prosulfuron	106	103	95	103	90
Pyrasulfotole	73	70	21	100	98
Pyrazosulfuron ethyl	102	103	106	107	99
Pyrimisulfan	108	98	105	104	97
Pyrithiobac sodium	83	105	99	103	89
Quinclorac	11	99	98	115	96
Quizalofop	63	103	95	100	103
Rimsulfuron	70	95	95	95	97
Saflufenacil	106	100	109	108	98
Sulfosulfuron	100	110	107	99	102
Thifensulfuron methyl	75	91	91	97	95
Triasulfuron	102	94	90	100	98
Tribenuron methyl	99	88	90	90	89
Triclopyr	43	106	94	95	103
Trifloxysulfuron	93	107	104	87	89
Triflusulfuron methyl	105	108	106	101	100

Warfarin	99	98	84	91	91

The mixed standard solution (1 μg/mL, 100 μL (corresponding to 0.1 μg)) was used for this experiment. See ***Optimizing the salting-out procedure*** in the ***Material and methods***.

The following aqueous solutions were used for salting out: (a) phosphate buffer (0.5 mol/L, pH 7), (b) 0.01 mol/L hydrochloric acid, (c) 0.1 mol/L hydrochloric acid, (d) phosphate buffer (0.5 mol/L, pH 2.1), and (e) phosphate buffer (0.1 mol/L, pH 2.1).

Table S5 pH values of aqueous solutions obtained after salting out (solution (e)) of various food extracts

□	pH
Brown rice	1.4
Soybeans	1.6
Peanuts	1.4
Cabbage	1.4
Spinach	1.5
Eggplant	1.6
Potato	1.5
Orange	1.6
Apple	1.3

Table S6 Recoveries (%) of target compounds from the ODS cartridge in the absence of a matrix

□	Loading	Elution (acetonitrile)		Total
□	(acetonitrile layer)	0–5 mL	5–10 mL	
1-Naphthaleneacetic acid	90	12	0	102
2,4,5-T	105	13	0	118
2,4-D	100	12	0	112
2,4-DB	91	14	0	104
4-CPA	97	12	0	108
Acifluorfen	99	12	0	111
Azimsulfuron	101	11	0	112
Bensulfuron methyl	88	10	0	98
Bentazone	97	13	0	110
Bispyribac sodium	84	10	0	94
Bromoxynil	66	13	0	79
Chlorimuron ethyl	88	11	0	99
Chlorsulfuron	81	10	0	91
Clodinafop acid	96	12	0	108
Cloransulam methyl	92	11	0	103
Cyclanilide	100	14	0	114
Cyclosulfamuron	93	12	0	105
Dicamba	86	10	0	96
Dichlorprop	89	12	0	102
Diclosulam	91	11	0	101
Diflufenzopyr	83	9	0	92
Ethametsulfuron methyl	91	10	0	101
Ethoxysulfuron	87	11	0	98
Fenoprop	87	13	0	100
Fenoxaprop	36	12	0	47
Flazasulfuron	81	10	0	92
Florasulam	94	11	0	105
Fluazifop	96	10	0	106
Flucarbazone sodium	94	11	0	104
Flucetosulfuron	85	9	0	94
Flumetsulam	96	10	0	106
Fluroxypyr	98	10	0	108
Flusulfamide	98	16	0	114
Fomesafen	94	11	0	105
Gibberellin A_3	53	1	0	54
Halosulfuron methyl	93	11	0	104

Haloxyfop	92	12	0	104
Imazamox	109	11	0	120
Imazapic	102	11	0	112
Imazapyr	101	11	0	113
Imazaquin	96	11	0	107
Imazethapyr	99	11	0	110
Imazosulfuron	90	11	0	101
Iodosulfuron methyl	90	11	0	101
Ioxynil	80	14	0	94
MCPA	94	12	0	106
MCPB	87	13	0	99
Mecoprop	81	12	0	93
Mesosulfuron methyl	91	10	0	101
Mesotrione	97	11	0	108
Metazosulfuron	90	11	0	101
Metosulam	77	11	0	88
Metsulfuron methyl	87	11	0	98
Nicosulfuron	83	10	0	93
Penoxsulam	85	10	0	96
Primisulfuron methyl	93	10	0	104
Propoxycarbazone	93	11	0	104
Propyrisulfuron	91	12	0	103
Prosulfuron	83	11	0	94
Pyrasulfotole	97	10	0	107
Pyrazosulfuron ethyl	90	13	0	103
Pyrimisulfan	94	12	0	106
Pyrithiobac sodium	98	12	0	109
Quinclorac	103	12	0	115
Quizalofop	92	13	0	105
Rimsulfuron	71	9	0	81
Saflufenacil	92	11	0	103
Sulfosulfuron	85	10	0	95
Thifensulfuron methyl	84	10	0	94
Triasulfuron	87	12	0	98
Tribenuron methyl	73	11	0	84
Triclopyr	95	12	0	107
Trifloxysulfuron	77	10	0	87
Triflusulfuron methyl	89	12	0	101
Warfarin	87	11	0	98

The mixed standard solution (1 μg/mL, 100 μL (corresponding to 0.1 μg)) was dissolved in the acetonitrile layer obtained by salting out (solution (e)), then loaded onto the ODS cartridge, and eluted with acetonitrile.

Table S7 Recoveries (%) of the target compounds from the PSA cartridge in the absence of a matrix

	Loading & washing	Elution			
	Acetone/hexane (1:1, v/v)	Acetone/hexane/formic acid (25:25:1, v/v/v)			
	0–10 mL	0–5 mL	5–10 mL	10–15 mL	Total
1-Naphthaleneacetic acid	0	105	0	0	105
2,4,5-T	0	88	2	0	89
2,4-D	0	84	3	0	86
2,4-DB	0	94	0	0	95
4-CPA	0	76	4	0	80
Acifluorfen	0	1	106	1	107
Azimsulfuron	0	105	0	0	105
Bensulfuron methyl	0	92	0	0	93
Bentazone	0	102	0	0	103
Bispyribac sodium	0	94	1	0	95
Bromoxynil	0	98	1	0	99
Chlorimuron ethyl	0	90	0	0	90
Chlorsulfuron	0	100	0	0	100
Clodinafop acid	0	98	1	0	98
Cloransulam methyl	0	98	0	0	98
Cyclanilide	0	101	1	0	102
Cyclosulfamuron	0	106	0	0	107
Dicamba	0	39	63	0	102
Dichlorprop	0	99	1	0	100
Diclosulam	0	99	0	0	99
Diflufenzopyr	0	92	1	0	93
Ethametsulfuron methyl	0	99	0	0	99
Ethoxysulfuron	0	78	0	0	79
Fenoprop	0	97	1	0	98
Fenoxaprop	0	88	1	0	89
Flazasulfuron	0	93	0	0	93
Florasulam	0	98	0	0	98
Fluazifop	0	94	0	0	95

Flucarbazone sodium	0	0	85	4	89
Flucetosulfuron	0	90	0	0	90
Flumetsulam	0	101	1	0	101
Fluroxypyr	0	82	7	0	90
Flusulfamide	0	108	0	0	109
Fomesafen	0	107	0	0	107
Gibberellin A_3	0	22	64	0	85
Halosulfuron methyl	0	110	0	0	110
Haloxyfop	0	100	0	0	101
Imazamox	0	95	0	0	95
Imazapic	0	96	0	0	97
Imazapyr	0	89	1	0	89
Imazaquin	0	98	0	0	98
Imazethapyr	0	96	0	0	96
Imazosulfuron	0	108	0	0	109
Iodosulfuron methyl	0	103	0	0	103
Ioxynil	0	101	1	0	101
MCPA	0	87	1	0	88
MCPB	0	96	0	0	97
Mecoprop	0	99	0	0	99
Mesosulfuron methyl	0	103	3	0	105
Mesotrione	0	80	0	0	81
Metazosulfuron	0	110	0	0	110
Metosulam	0	98	1	0	98
Metsulfuron methyl	0	100	0	0	100
Nicosulfuron	0	21	53	0	74
Penoxsulam	0	91	0	0	91
Primisulfuron methyl	0	109	0	0	109
Propoxycarbazone	0	85	5	0	90
Propyrisulfuron	0	97	0	0	97
Prosulfuron	0	100	0	0	100
Pyrasulfotole	2	1	80	1	82
Pyrazosulfuron ethyl	0	99	0	0	99
Pyrimisulfan	0	97	0	0	97
Pyrithiobac sodium	0	88	14	0	102

Quinclorac	0	0	85	2	87
Quizalofop	0	97	1	0	98
Rimsulfuron	0	87	0	0	87
Saflufenacil	0	100	0	0	100
Sulfosulfuron	0	86	0	0	87
Thifensulfuron methyl	0	106	0	0	107
Triasulfuron	0	98	0	0	98
Tribenuron methyl	0	97	0	0	98
Triclopyr	0	97	1	0	98
Trifloxysulfuron	0	98	1	0	98
Triflusulfuron methyl	0	94	0	0	94
Warfarin	0	80	10	2	92

The mixed standard solution (1 μg/mL, 100 μL (corresponding to 0.1 μg)) was evaporated to dryness under a stream of nitrogen and dissolved in acetone/hexane (1:1, v/v, 2 mL). The resultant solution was loaded on the PSA cartridge; the cartridge was washed with acetone/hexane (1:1, v/v, 8 mL) and then eluted using acetone/hexane/formic acid (25:25:1, v/v/v).

Table S8 Matrix effect values of the target compounds

□	Brown rice	Soybeans	Peanuts	Spinach	Cabbage	Eggplant	Potatoes	Apples	Oranges
1-Naphthaleneacetic acid	0.93	0.94	0.89	0.99	0.97	1.01	1.00	0.99	0.91
2,4,5-T	0.90	0.97	0.94	0.89	0.96	0.99	0.92	1.09	1.01
2,4-D	0.91	0.97	0.98	0.92	0.96	1.00	0.97	0.98	0.98
2,4-DB	0.91	0.97	1.02	0.86	0.87	1.03	0.99	0.93	0.88
4-CPA	0.94	0.96	1.00	0.92	0.98	0.94	1.01	0.98	1.00
Acifluorfen	0.84	0.95	0.96	0.94	0.92	1.00	0.95	1.00	0.94
Azimsulfuron	0.89	0.92	0.88	0.89	0.89	1.00	0.91	0.94	0.92
Bensulfuron methyl	0.90	1.04	1.02	0.94	0.95	1.03	1.12	1.04	1.05
Bentazone	0.93	0.99	0.95	0.90	0.95	1.04	1.00	0.98	0.98
Bispyribac sodium	0.89	0.95	1.00	0.96	0.91	0.98	0.87	1.06	1.04
Bromoxynil	0.93	1.00	0.99	0.94	0.98	1.02	1.03	0.95	0.98
Chlorimuron ethyl	0.88	0.95	0.96	0.89	0.88	0.99	0.97	0.94	0.89
Chlorsulfuron	0.94	0.66	0.96	0.87	0.94	0.99	1.05	0.91	0.97
Clodinafop acid	0.82	0.94	0.92	0.98	0.93	0.91	0.80	0.96	0.94
Cloransulam methyl	0.99	0.59	0.98	0.93	1.01	1.02	1.06	0.98	1.02
Cyclanilide	0.94	0.95	0.98	0.94	0.96	1.03	0.99	0.99	0.98
Cyclosulfamuron	0.84	0.86	0.89	0.92	0.87	0.95	0.92	0.92	0.90
Dicamba	1.03	0.96	1.00	0.92	0.99	0.96	0.99	1.05	0.97
Dichlorprop	0.93	0.96	0.96	1.00	0.98	0.98	1.01	0.97	0.99

Diclosulam	0.86	0.88	0.90	0.91	0.90	0.93	0.95	0.99	0.83
Diflufenzopyr	0.89	0.71	0.95	0.83	0.93	0.97	0.97	0.95	0.92
Ethametsulfuron methyl	0.94	0.95	0.96	0.97	0.91	1.02	1.02	1.04	1.01
Ethoxysulfuron	0.90	0.94	0.98	0.87	0.91	1.02	1.04	0.93	0.95
Fenoprop	0.90	0.97	1.02	0.92	0.96	0.97	1.00	0.98	1.00
Fenoxaprop	0.83	0.87	0.91	0.86	0.95	0.94	0.95	0.95	0.93
Flazasulfuron	0.86	0.94	0.96	0.83	0.93	0.96	1.00	0.94	0.91
Florasulam	0.94	0.85	0.98	0.89	0.91	1.02	0.96	0.95	0.94
Fluazifop	0.80	0.89	0.92	0.96	0.94	0.97	0.91	0.99	0.93
Flucarbazone sodium	0.93	0.94	0.96	0.91	0.99	1.01	0.97	1.02	1.00
Flucetosulfuron	0.99	1.13	1.16	1.17	1.10	0.99	1.10	1.17	1.17
Flumetsulam	0.95	0.92	0.77	0.92	0.95	1.01	1.00	1.01	1.00
Fluroxypyr	0.87	0.85	0.85	0.86	0.91	0.94	0.93	0.95	0.89
Flusulfamide	0.94	0.99	0.98	0.94	0.95	1.02	1.04	0.95	0.98
Fomesafen	0.83	0.94	0.89	0.96	0.99	1.02	1.00	0.99	0.98
Gibberellin A_3	0.98	0.92	0.86	0.86	0.98	1.00	1.01	1.00	0.98
Halosulfuron methyl	0.84	0.94	0.98	0.85	0.89	1.00	0.98	0.92	0.95
Haloxyfop	0.83	0.95	0.95	0.92	0.93	0.91	0.94	1.00	0.98
Imazamox	0.93	0.99	1.02	0.86	0.92	1.05	0.98	1.00	1.01
Imazapic	0.96	0.97	1.00	0.87	0.96	1.05	1.01	1.02	0.99
Imazapyr	1.00	0.95	0.99	0.81	0.95	1.05	1.00	1.01	0.98
Imazaquin	0.96	0.98	1.00	0.94	0.99	1.03	1.06	0.99	1.00
Imazethapyr	0.95	0.96	0.99	0.88	0.95	1.05	1.03	1.01	1.00

Imazosulfuron	0.85	0.86	0.93	0.90	0.94	0.99	0.92	0.89	0.92
Iodosulfuron methyl	0.83	0.89	0.92	0.88	0.93	0.93	0.83	1.01	0.92
Ioxynil	0.94	1.01	0.98	0.93	1.01	1.00	1.00	0.97	1.00
MCPA	0.91	1.00	0.97	0.95	0.98	0.99	0.99	0.97	1.01
MCPB	0.88	0.95	0.92	0.95	0.97	0.96	0.93	1.00	0.93
Mecoprop	0.86	0.93	0.90	0.96	0.97	0.96	1.00	0.98	0.99
Mesosulfuron methyl	0.85	0.89	0.94	0.92	0.93	0.99	1.01	0.97	0.89
Mesotrione	0.91	0.89	0.91	0.94	0.96	0.98	1.00	0.96	0.93
Metazosulfuron	0.87	0.89	0.92	0.88	0.91	0.97	0.76	0.91	0.91
Metosulam	0.96	0.91	1.02	0.98	0.96	1.04	1.03	1.00	0.99
Metsulfuron methyl	0.90	0.99	1.01	0.92	0.97	1.01	0.96	0.99	0.96
Nicosulfuron	0.94	0.74	0.97	0.83	0.91	1.01	1.01	0.96	0.98
Penoxsulam	0.96	0.85	1.00	0.91	1.07	1.00	1.00	1.01	1.02
Primisulfuron methyl	0.82	0.87	0.94	0.83	0.87	1.02	0.96	0.88	0.81
Propoxycarbazone	0.90	0.76	0.98	0.93	0.95	1.00	0.96	0.98	0.95
Propyrisulfuron	0.87	0.99	0.94	0.88	0.91	1.00	0.95	0.98	0.98
Prosulfuron	0.80	0.81	0.85	0.83	0.85	0.96	0.89	0.91	0.84
Pyrasulfotole	0.92	0.96	1.01	0.90	0.88	1.02	1.00	0.94	0.96
Pyrazosulfuron ethyl	0.86	0.92	0.95	0.90	0.91	0.96	0.97	1.01	0.92
Pyrimisulfan	0.92	1.00	0.97	0.90	0.98	1.01	1.05	0.98	0.99
Pyrithiobac sodium	0.96	0.99	0.99	0.89	0.96	1.02	0.97	0.99	0.96
Quinclorac	0.93	0.95	0.98	0.93	0.92	1.00	0.99	0.98	0.96
Quizalofop	0.86	0.91	0.93	0.90	0.92	0.91	0.95	0.95	0.99

Rimsulfuron	0.94	0.77	0.91	0.89	0.93	1.00	1.00	0.98	0.90
Saflufenacil	0.91	0.97	0.87	0.92	0.84	0.98	0.90	1.02	1.07
Sulfosulfuron	0.87	0.94	0.91	0.87	0.87	0.98	0.98	1.08	0.93
Thifensulfuron methyl	0.95	0.83	1.00	0.97	0.88	1.01	1.00	1.01	0.95
Triasulfuron	0.95	0.63	0.97	0.90	0.95	0.98	1.05	0.92	0.96
Tribenuron methyl	0.69	0.70	0.71	0.62	0.62	0.88	0.92	0.66	0.60
Triclopyr	0.97	0.98	0.97	0.92	0.95	0.97	0.88	0.99	1.04
Trifloxysulfuron	0.91	0.95	0.94	0.94	0.90	1.03	1.00	0.98	0.99
Triflusulfuron methyl	0.88	0.92	0.93	0.90	0.95	0.97	0.97	0.97	0.95
Warfarin	0.87	0.99	0.95	0.93	0.96	1.05	0.81	0.97	0.97

Table S9 Target compound trueness and precision at a concentration level of 0.01 mg/kg (n = 5)

	Brown rice		Soybeans		Peanuts		Spinach		Cabbage		Eggplant		Potatoes		Apples		Oranges	
	Trueness (%)	RSD (%)	Trueness (%)	RSD (%)	Trueness (%)	RSD (%)	Trueness (%)	RSD (%)	Trueness (%)	RSD (%)	Trueness (%)	RSD (%)	Trueness (%)	RSD (%)	Trueness (%)	RSD (%)	Trueness (%)	RSD (%)
1-Naphthaleneacetic acid	87	8	93	5	82	3	87	8	95	5	89	8	90	5	90	5	83	6
2,4,5-T	91	4	80	2	82	4	84	5	88	4	84	6	85	5	91	7	89	3
2,4-D	88	6	82	3	82	3	88	5	88	3	88	5	93	3	91	4	88	4
2,4-DB	88	10	98	5	92	3	83	10	78	8	90	7	88	10	90	7	82	3
4-CPA	86	4	57	5	48	6	85	5	86	2	96	7	92	3	87	5	87	2
Acifluorfen	86	7	85	4	85	3	85	7	85	4	86	5	89	7	88	4	87	6
Azimsulfuron	84	3	90	2	87	2	84	6	80	3	86	7	92	5	88	5	85	6
Bensulfuron methyl	89	3	91	3	99	3	86	5	100	3	94	6	100	6	100	6	93	3
Bentazone	89	4	84	5	86	3	84	5	85	3	86	4	90	4	76	6	88	3
Bispyribac sodium	76	5	72	7	82	3	82	4	80	3	80	10	74	5	80	5	82	6
Bromoxynil	86	4	89	6	83	4	85	5	85	4	86	5	90	4	87	5	84	4
Chlorimuron ethyl	83	4	80	3	91	3	85	3	88	4	92	2	88	3	98	9	89	2
Chlorsulfuron	85	4	58	3	86	2	80	2	86	3	89	5	93	3	88	5	84	2
Clodinafop acid	83	5	88	7	88	5	87	7	87	2	84	9	84	7	88	5	88	2
Cloransulam methyl	91	2	52	4	91	3	84	5	93	5	90	5	94	3	92	7	90	2

Cyclanilide	85	4	82	3	78	4	85	3	83	4	87	7	89	4	86	6	85	3
Cyclosulfamuron	78	6	83	9	95	3	85	6	86	2	77	8	89	4	96	9	87	3
Dicamba	90	3	75	7	74	3	85	8	84	2	88	6	87	6	89	4	90	4
Dichlorprop	82	5	81	5	83	3	86	4	88	3	86	8	96	4	93	6	86	4
Diclosulam	86	4	79	3	84	3	82	3	82	2	88	4	90	5	88	7	74	2
Diflufenzopyr	82	6	51	6	75	2	77	4	83	3	78	6	82	7	87	7	74	3
Ethametsulfuron methyl	86	5	87	3	90	1	87	5	89	5	91	2	95	5	94	6	86	3
Ethoxysulfuron	87	4	80	3	85	3	86	4	88	7	93	3	94	6	93	9	88	4
Fenoprop	87	8	81	3	81	6	86	8	87	2	85	7	98	5	92	6	90	6
Fenoxaprop	70	7	74	3	82	3	77	6	69	3	63	7	74	5	68	11	61	5
Flazasulfuron	85	3	84	2	90	2	83	3	85	4	91	4	90	5	91	6	84	3
Florasulam	85	4	81	3	85	2	81	2	81	3	77	8	84	3	75	6	83	3
Fluazifop	81	6	78	3	85	5	86	6	82	4	84	5	81	2	89	8	83	2
Flucarbazone sodium	91	3	89	3	86	3	86	6	88	2	90	5	91	3	90	5	88	2
Flucetosulfuron	100	7	93	4	107	3	92	4	105	5	91	4	95	5	96	4	111	2
Flumetsulam	85	4	88	3	74	1	84	5	83	1	75	9	84	3	76	5	84	2
Fluroxypyr	77	5	73	3	78	3	79	4	78	5	83	5	88	6	89	5	80	4
Flusulfamide	88	4	93	3	81	4	89	4	86	3	86	8	93	3	85	7	85	3
Fomesafen	88	3	95	8	92	4	83	7	86	3	85	8	85	5	93	5	86	3
Gibberellin A_3	82	12	82	3	76	2	82	6	79	2	83	6	79	7	79	4	73	3
Halosulfuron	85	3	85	5	88	2	86	5	84	3	84	8	94	5	92	6	85	3

methyl																		
Haloxyfop	81	5	85	3	88	3	86	7	82	2	81	8	87	5	90	7	85	4
Imazamox	77	3	65	8	75	3	75	6	74	4	75	9	76	7	74	7	71	5
Imazapic	77	2	66	5	75	3	72	6	74	5	73	7	76	6	72	6	74	4
Imazapyr	67	4	51	7	66	3	64	6	64	6	63	8	66	6	59	6	65	6
Imazaquin	83	5	72	12	80	5	85	4	84	5	85	3	88	5	84	5	77	4
Imazethapyr	86	3	74	5	80	3	82	6	80	4	83	6	83	7	82	5	79	4
Imazosulfuron	83	5	84	7	89	5	85	6	79	4	89	12	92	5	87	5	84	2
Iodosulfuron methyl	83	5	85	4	86	3	81	3	79	4	90	5	78	5	89	8	83	4
Ioxynil	87	5	90	5	85	4	85	6	89	3	87	7	94	2	88	5	89	2
MCPA	88	3	84	4	85	2	88	7	88	3	88	6	92	5	88	6	87	3
MCPB	84	7	93	6	83	6	84	8	85	7	84	8	88	6	91	8	79	3
Mecoprop	87	4	83	4	87	2	86	6	86	3	88	5	89	3	88	5	88	4
Mesosulfuron methyl	88	4	93	3	96	3	88	6	86	4	89	7	94	5	95	5	78	2
Mesotrione	85	4	90	3	86	3	84	6	85	3	86	3	89	4	81	5	82	4
Metazosulfuron	84	3	79	5	84	2	83	4	80	4	86	7	87	4	90	9	84	3
Metosulam	90	4	80	7	86	3	83	3	83	5	89	4	85	2	89	7	87	2
Metsulfuron methyl	86	3	85	2	85	4	85	5	86	2	90	4	91	6	85	6	82	1
Nicosulfuron	89	5	59	4	78	5	83	2	87	4	88	5	89	5	91	7	85	7
Penoxsulam	90	4	74	5	86	4	84	3	89	4	90	8	92	4	89	5	89	1
Primisulfuron	84	5	81	5	82	3	85	4	83	3	91	8	91	4	89	9	83	5

methyl																		
Propoxycarbazone	84	5	73	4	90	2	87	6	88	3	89	6	91	5	89	5	84	3
Propyrisulfuron	89	4	84	3	89	3	86	3	88	6	90	7	90	6	91	6	84	2
Prosulfuron	75	7	72	3	80	2	80	4	82	4	85	5	81	4	85	7	80	5
Pyrasulfotole	79	5	83	4	85	3	80	3	76	3	83	7	83	4	72	4	74	2
Pyrazosulfuron ethyl	80	5	81	3	91	4	84	4	87	4	86	6	88	4	94	7	84	2
Pyrimisulfan	92	5	90	3	88	2	86	2	96	5	92	5	92	4	92	3	89	4
Pyrithiobac sodium	86	5	72	7	84	3	85	8	84	8	89	7	87	6	91	7	86	4
Quinclorac	86	5	67	3	73	2	83	5	80	3	87	7	87	4	88	5	83	2
Quizalofop	84	4	75	3	87	3	81	6	80	3	80	6	82	4	87	6	81	6
Rimsulfuron	82	3	70	1	92	4	80	5	90	3	91	4	89	3	91	6	82	1
Saflufenacil	82	2	86	1	84	2	86	2	88	5	91	5	80	5	92	6	93	3
Sulfosulfuron	84	8	81	4	91	6	83	5	87	4	86	6	88	5	91	6	84	3
Thifensulfuron methyl	90	3	70	2	88	2	88	6	88	3	92	3	91	4	89	7	90	4
Triasulfuron	87	4	59	5	84	4	87	4	85	3	90	8	94	5	90	10	84	2
Tribenuron methyl	76	8	71	13	106	6	70	10	68	4	95	2	81	6	96	7	63	5
Triclopyr	94	7	84	6	84	4	83	9	86	4	90	6	85	6	91	5	86	6
Trifloxysulfuron	88	4	85	4	93	5	81	5	89	4	89	3	88	3	94	4	87	3
Triflusulfuron methyl	87	5	77	3	110	5	85	5	105	4	92	5	85	6	113	4	102	3
Warfarin	82	4	81	3	80	6	85	3	82	5	86	2	78	6	91	8	84	3

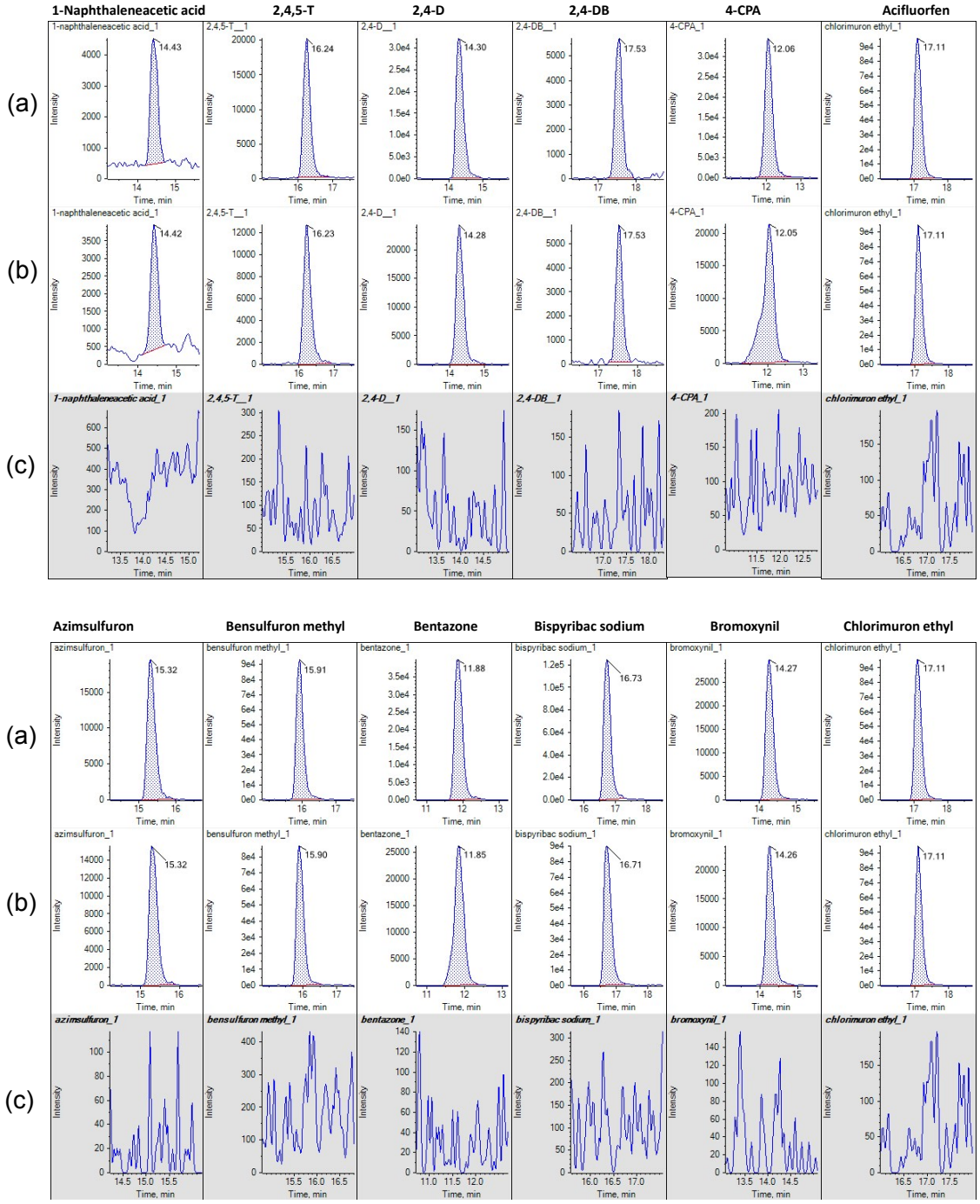

Fig. S4 LC-MS/MS chromatograms of (a) 5 ng/mL standard solutions, (b) an extract of the soybeans sample spiked with 0.01 mg/kg standard solution, and (c) an extract of the soybeans blank sample.

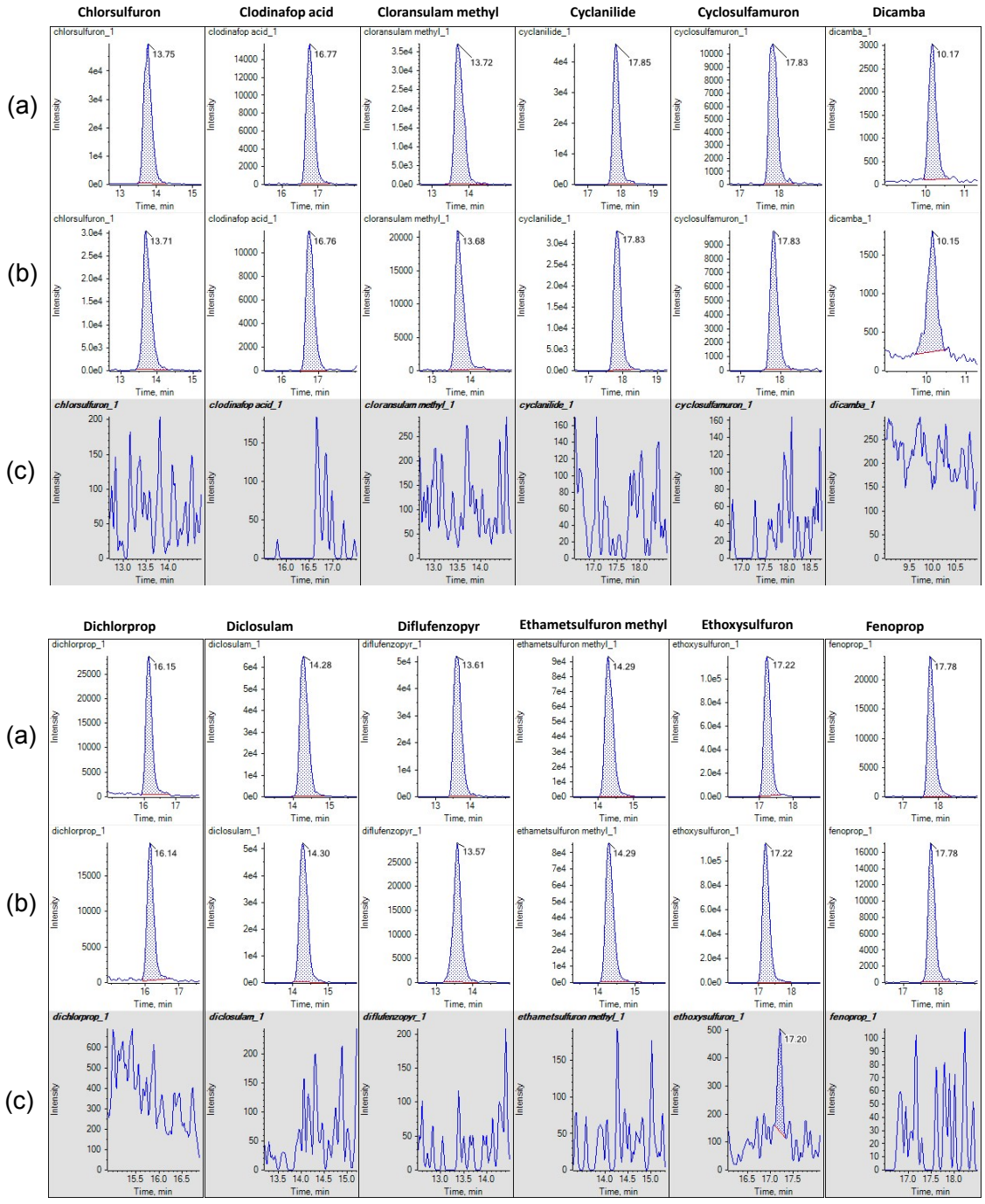

Fig. S4 (continued)

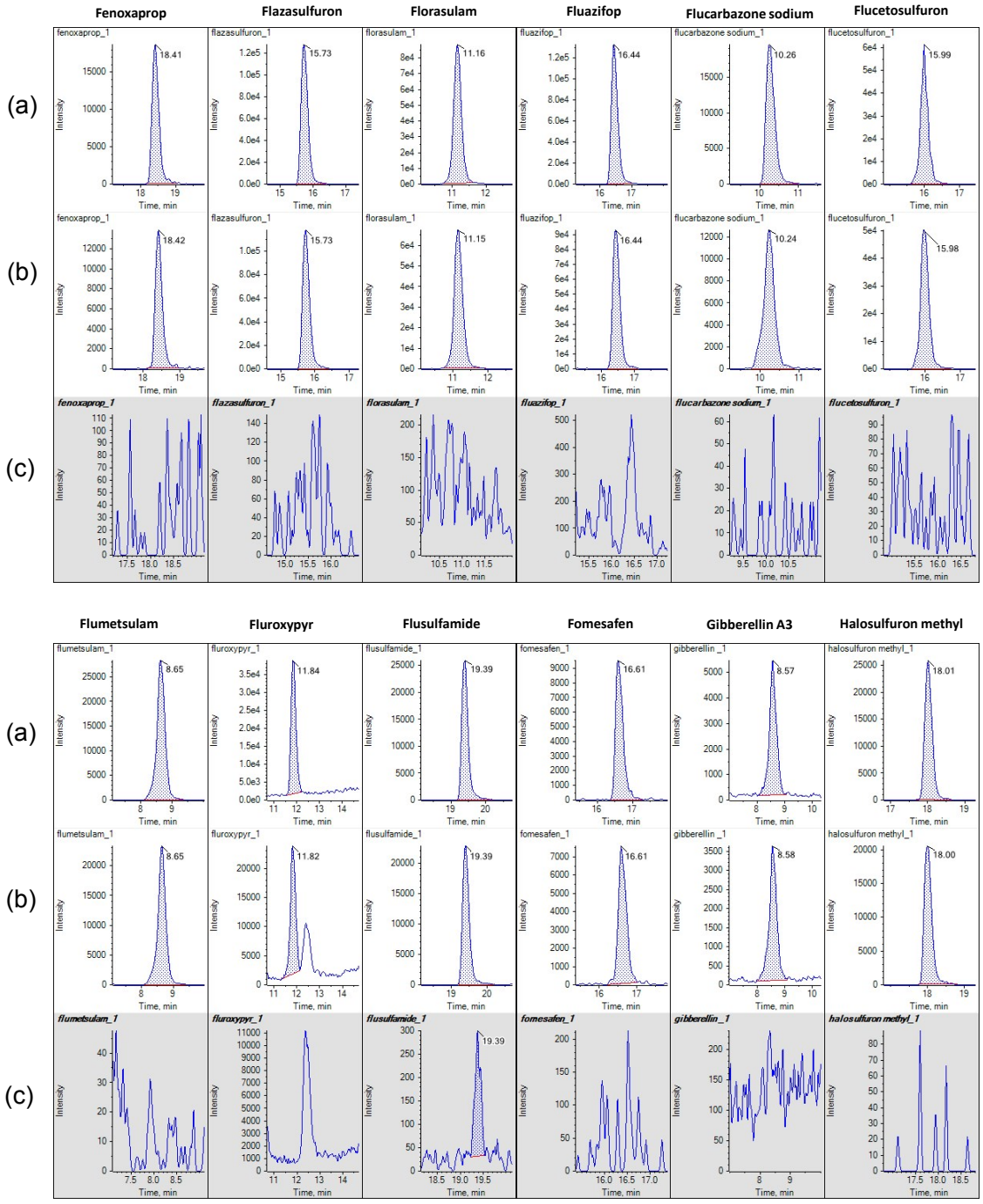

Fig. S4 (continued)

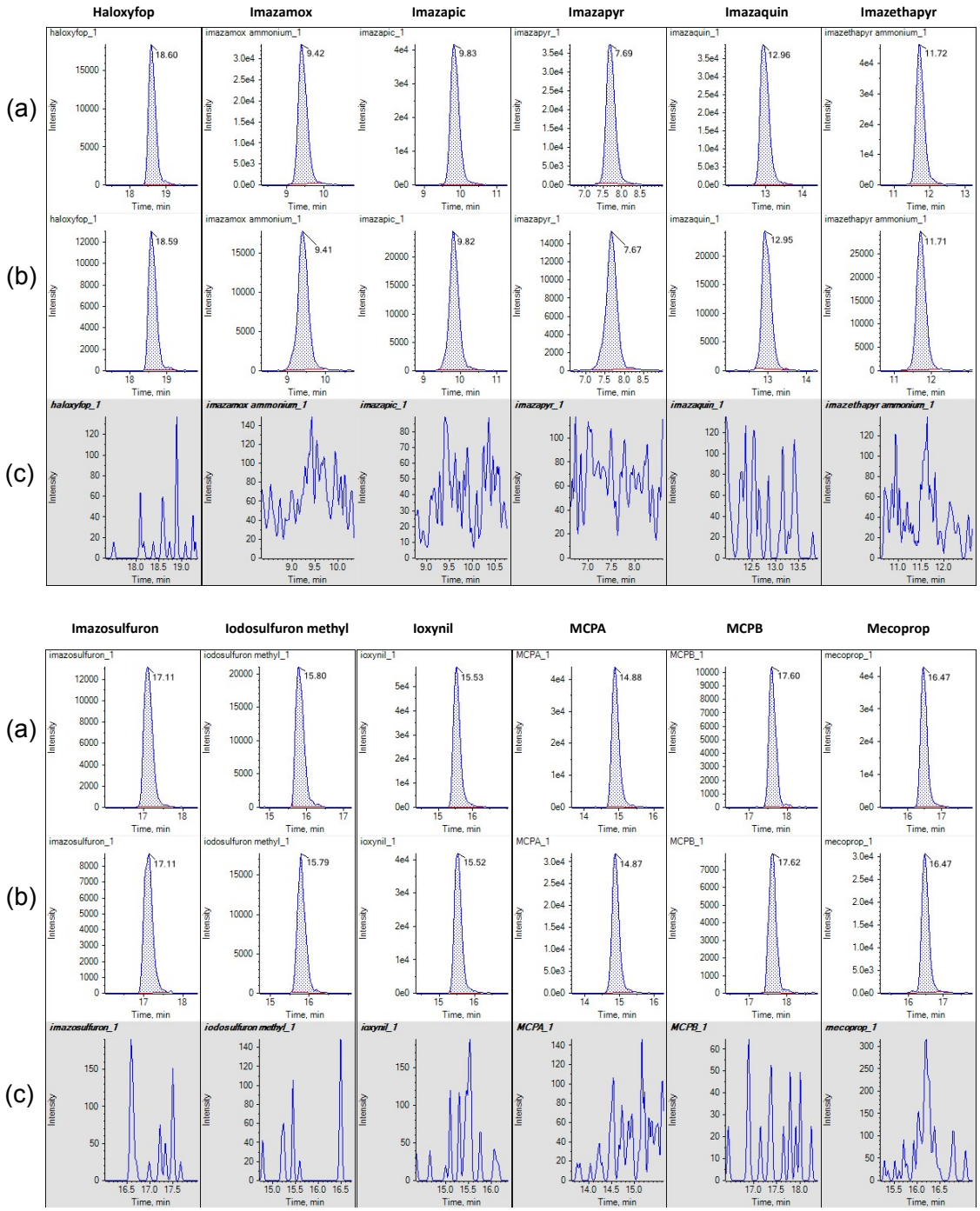

Fig. S4 (continued)

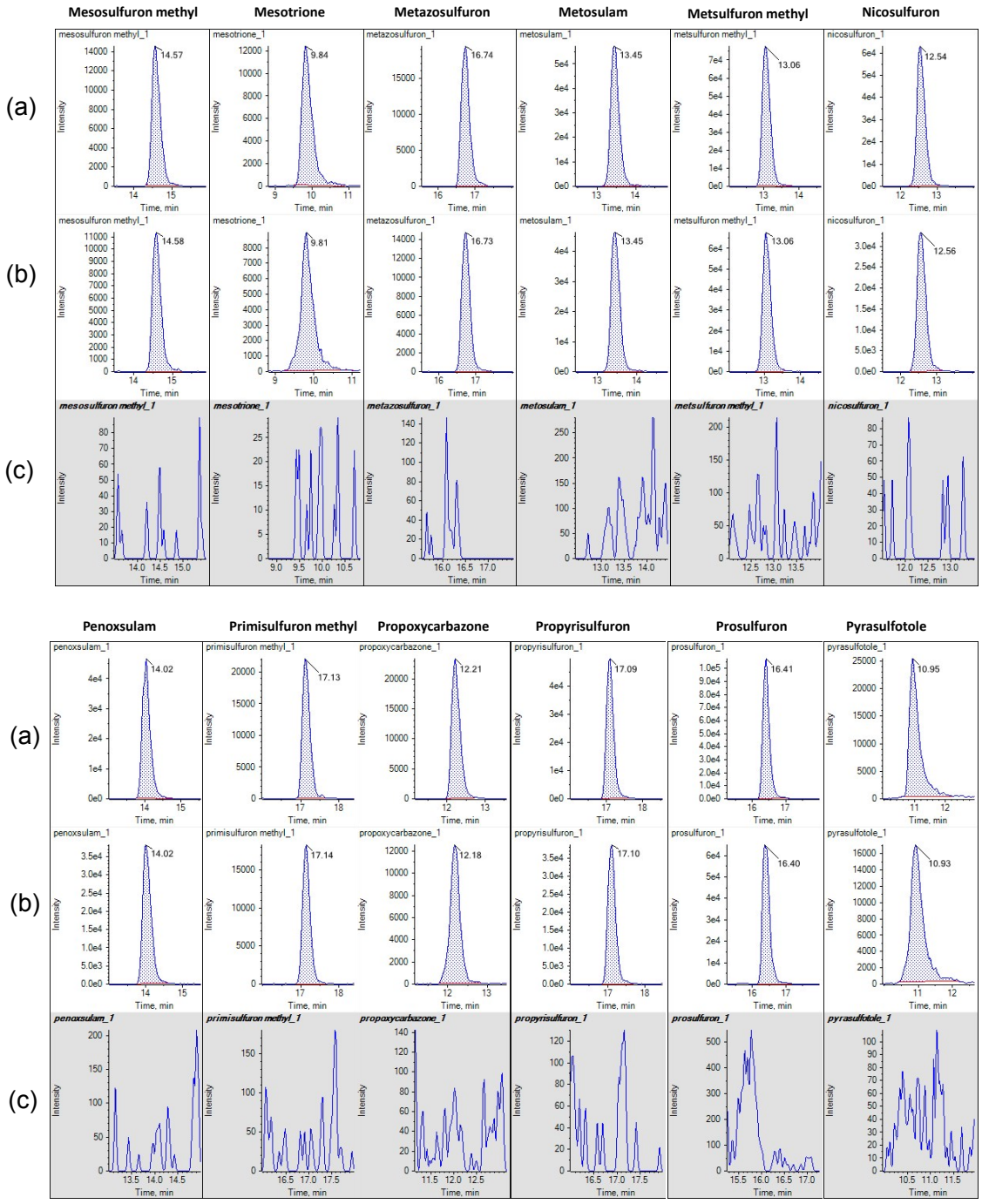

Fig. S4 (continued)

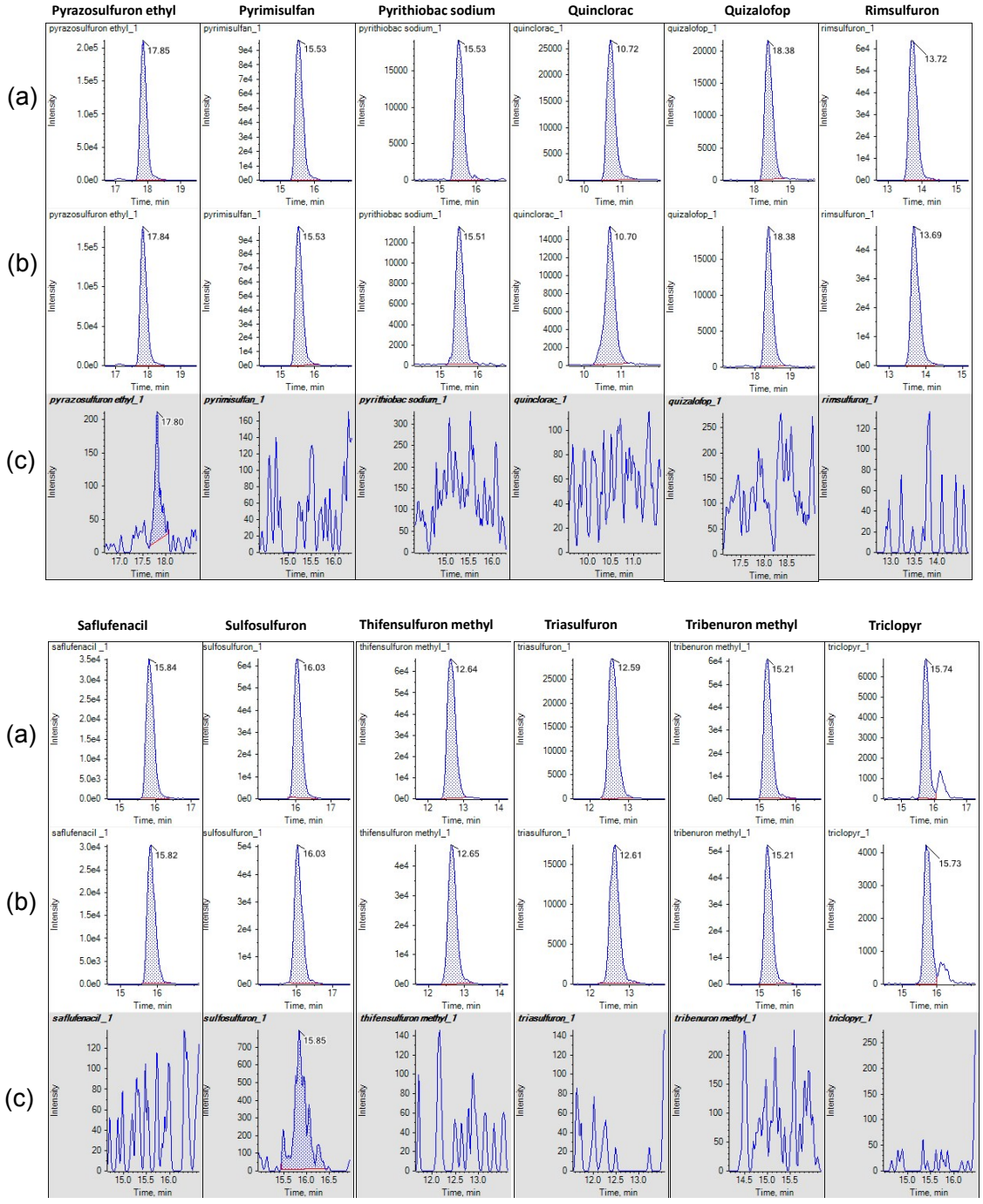

Fig. S4 (continued)

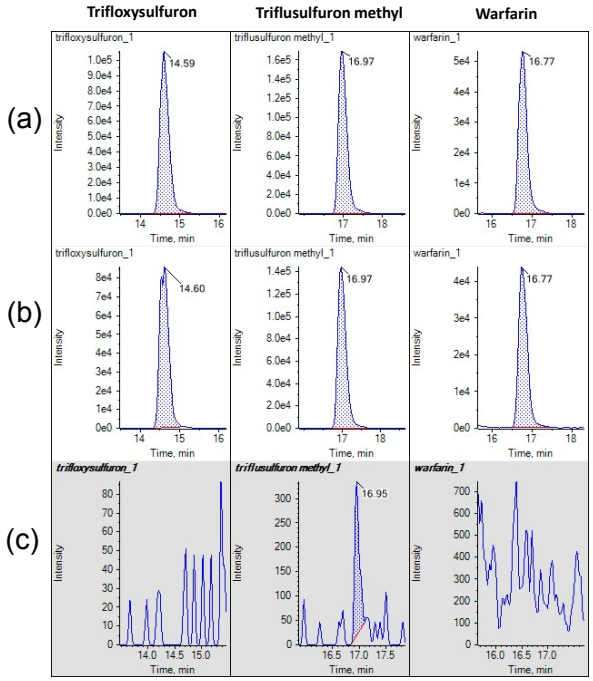

Fig. S4 (continued)

Table S10 LODs (μg/kg) of the target compounds

□	Brown rice	Soybeans	Peanuts	Spinach	Cabbage	Eggplant	Potatoes	Apples	Oranges
1-Naphthaleneacetic acid	2	2	3	2	2	2	2	2	2
2,4,5-T	0.4	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2
2,4-D	0.1	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1
2,4-DB	0.5	0.5	0.7	0.5	0.5	0.5	0.5	0.5	0.5
4-CPA	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Acifluorfen	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Azimsulfuron	0.3	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2
Bensulfuron methyl	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Bentazone	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Bispyribac sodium	0.1	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Bromoxynil	0.1	0.2	0.3	0.1	0.1	0.1	0.1	0.1	0.1
Chlorimuron ethyl	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Chlorsulfuron	0.2	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Clodinafop acid	0.3	0.3	0.4	0.3	0.3	0.3	0.3	0.3	0.3
Cloransulam methyl	0.4	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Cyclanilide	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Cyclosulfamuron	0.5	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
Dicamba	2	3	2	2	2	2	2	2	2
Dichlorprop	1	0.3	0.5	0.3	0.3	0.3	0.3	0.3	0.3
Diclosulam	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1

Diflufenzopyr	0.2	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Ethametsulfuron methyl	0.4	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Ethoxysulfuron	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Fenoprop	0.4	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2
Fenoxaprop	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Flazasulfuron	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Florasulam	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Fluazifop	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Flucarbazone sodium	0.2	0.3	1	0.2	0.2	0.2	0.2	0.2	0.2
Flucetosulfuron	0.3	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Flumetsulam	0.1	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Fluroxypyr	0.9	0.7	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Flusulfamide	0.1	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Fomesafen	0.4	0.2	0.4	0.2	0.2	0.2	0.2	0.2	0.2
Gibberellin A_3	0.6	0.6	2	0.6	0.6	0.6	0.6	0.6	0.6
Halosulfuron methyl	0.5	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Haloxyfop	0.4	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Imazamox	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Imazapic	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Imazapyr	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Imazaquin	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Imazethapyr	0.4	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Imazosulfuron	0.3	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3

Iodosulfuron methyl	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Ioxynil	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
MCPA	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
MCPB	0.6	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Mecoprop	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Mesosulfuron methyl	0.3	0.3	0.4	0.3	0.3	0.3	0.3	0.3	0.3
Mesotrione	0.4	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Metazosulfuron	0.3	0.5	0.4	0.3	0.3	0.3	0.3	0.3	0.3
Metosulam	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Metsulfuron methyl	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Nicosulfuron	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Penoxsulam	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Primisulfuron methyl	0.2	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Propoxycarbazone	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Propyrisulfuron	0.1	0.1	0.4	0.1	0.1	0.1	0.1	0.1	0.1
Prosulfuron	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Pyrasulfotole	0.2	0.3	0.3	0.2	0.2	0.2	0.2	0.2	0.2
Pyrazosulfuron ethyl	0.1	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Pyrimisulfan	0.1	0.2	0.4	0.1	0.1	0.1	0.1	0.1	0.1
Pyrithiobac sodium	0.3	0.3	0.6	0.3	0.3	0.3	0.3	0.3	0.3
Quinclorac	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Quizalofop	0.1	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Rimsulfuron	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2

Saflufenacil	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Sulfosulfuron	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Thifensulfuron methyl	0.2	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Triasulfuron	0.3	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Tribenuron methyl	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Triclopyr	2	2	2	2	2	2	2	2	2
Trifloxysulfuron	0.1	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1
Triflusulfuron methyl	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Warfarin	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3