Fast Multiresidue Method for the Determination of Pesticide Residues in Crops Using Mini Solid-Phase Extraction and Liquid Chromatography with Tandem Mass Spectrometry

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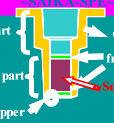
In order to respond to the increase in the number of pesticides subject to inspection according to the adoption of the Positive List system in Japan, the simultaneous analysis of more than 300 compounds is required for GC/MS and LC/MS(MS). However, it is difficult to greatly increase the number of samples inspected, because a pretreatment process of each sample is time consuming using current analysis techniques. Therefore, in order to accelerate the pretreatment process, a simple and fast method was developed for the determination of 124 pesticides/metabolites(127 compounds) in a wide variety crops, using solid-phase extraction (SPE) and liquid chromatography with tandem mass spectrometry (LC/MS/MS). After extraction with acetonitrile, the filtered extracts were made up to 50 mL and a 1 mL aliquot was followed with SPE in method-1, and another 1 mL aliquot was followed with SPE in method-2 for high polar pesticides. By using aliquots of extracts with small-scale mini-columns, purified samples could be obtained. And these methods were not needed liquid-liquid distribution and concentration by evaporator long time. So after fractioning, a single chemist could do pretreatment process in 10～20 minutes per sample. This proposed method with small matrix effects, is effective and suitable for the determination of multiple residual pesticides.

Structure for New Type Solid Phase Cartridge ~SAIKA-SPE~



★Three Kinds of Using Solid-phase in SAIKA-SPE

① C18(30mg)
Octadecile Silica $Si\text{-}C_{18}H_{37}$ by Varian corporation

② HBS(20mg)
Hydrophilic-Lipophilic Balanced Copolymer by Waters corporation

③ PSA(30mg)
Primary Secondery Amine $Si\text{-}CH_2CH_2CH_2NHCH_2NH_2$ by Varian corporation

Tools of Pretreatment by SAIKA Technological Method in LC-Method

Original Test Tube Rack

Original Flask Rack

Extraction process

Common extraction GC and LC

Sample 10g (Neutralization for acidic samples, and add 10mL water to dry samples)
Acetonitrile 25 mL
Homogenize 2min
Filtrate
Wash with Acetonitrile 10mL
Add water, make up to 50mL
Fill up(50mL) → GC method

LC method- I

Pretreatment time 20min/sample

Fraction 1mL (equivalent of 0.2g sample)
C18 (30mg): purification
wash with acetonitlile:water(80:20) 0.5 mL
Out flow liquid
dilute by adding 2 mL water (31% acetonitrile/sample)
HBS (20mg): retention
Out flow liquid
dilute by adding 22 mL 20% saline solution(4.4% acetonitrile/sample)
HBS (20mg): second retention
wash water 2 mL×2 (for removing NaCl)
aspiration dry 3 min
PSA (30 mg) connect: purification
elute acetone: n-hexane(1:1) 2 mL *1
only PSA(30mg)
0.4%HCOOH/Methanol(pH2.5) 1 mL*2
DRY UP *1,*2 separately
methanol 0.5 mL + 1%CH_3COOH/water(pH3) 0.5 mL
LC/MS/MS (MRM determination)

LC method- II

Pretreatment time 20min/sample

Fraction 1mL (equivalent of 0.2g sample)
C18 (30mg): purification
wash with Acetonitlile:water(80:20) 0.5 mL
Out flow liquid
add acetonitrile 4 mL, and azeotrope
DRY UP
add methanol : water (25:75) 0.9 mL
C18 (30mg): purification wash methanol : water (25:75) 0.9 mL
Out flow liquid
add 20%CH_3COOH(pH3) 50μL
Fill up to 2 mL
LC/MS/MS (MRM determination)

LC/MS/MS条件

LC conditions:
system : ACQUITY UPLC (Waters,USA)
column : Inertsil ODS-SP, 2.1×150mm,5μm (GLSciences,Japan)
mobile phase: A is 2mM ammonium acetate in water,
B is 2mM ammonium acetate in methanol.
gradient mode (%;concentration of B)
method-1: 10%(0-1min)→90%(20min)→90%(24min)→10%(24-31min)
method-2: 10%(0-1min)→60%(9min)→60%(10min)→10%(10-15min)
flow rate: 0.2mL/min, injection volume: 10μL, column temp.: 40℃,
detector: PDA 210-400nm
MS conditions:
system : Quattro Premier (Waters-Micromass, UK)
ionization mode: ESI(+),(-), capillary voltage: 3.5kV(+), 3.0kV(-),
analytical mode: MRM(Multiple Reaction Monitoring), source temp.: 120℃,
cone gas: N_2,50L/hr, desolvation temp.: 400℃, desolvation gas: N_2,1000L/hr

★ C18 (30mg): purification

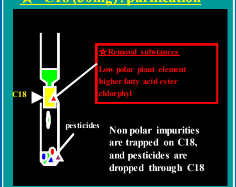
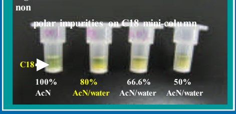


Fig. Effect of Acetonitrile ratio on the removal non polar impurities on C18 mini-column



★ PSA (30 mg): purification

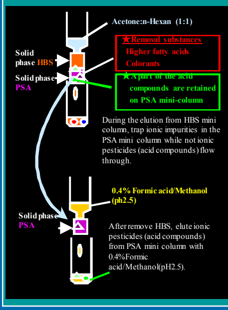


Table. Recovery of acid compounds from HBS+PSA and PSA after remove HBS

Pesticide	Acid-Amino bond (O=C-NH-)	pKa	HBS+PSA: Acetone : n-hexane (1:1) 2mL	HBS+PSA: Acetone 1mL	only PSA: 0.4% Formic acid/Methanol(pH2.5)	Total Recovery (%)
Pyrazosulfuron-ethyl	○	3.7	0	0.4	104.0	104.4
Cyclosulfamuron	○	5.04	0	0	96.8	96.8
Fluazifop-acid	-(-COOH)	3.2	0	0	96.8	96.8
Haloxyfop	-(-COOH)	2.9	0	0	96.0	96.0
Chlorimuron-ethyl	○	4.2	0	0	95.2	95.2
Bensulfuron-methyl	○	5.2	0	0	93.6	93.6

*Elution sequence: acetone-hexane(1:1) → acetone → 0.4% formic acid(pH2.5)
*○: with acid-amino bond and
*total acid compounds are 25 compounds/ 124 compounds

★ HBS (20mg): retention & second retention

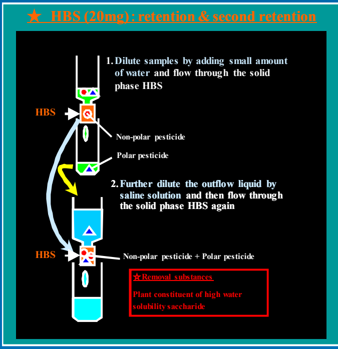
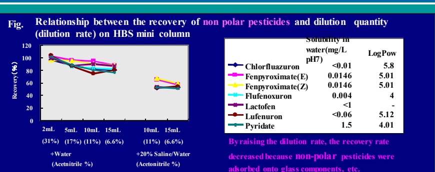
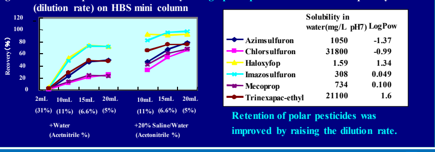


Fig. Relationship between the recovery of non polar pesticides and dilution quantity (dilution rate) on HBS mini column



By raising the dilution rate, the recovery rate decreased because non-polar pesticides were adsorbed onto glass components, etc.

Fig. Relationship between the recovery of high polar pesticides and dilution quantity (dilution rate) on HBS mini column



Retention of polar pesticides was improved by raising the dilution rate.

(Unit:%)

Pesticide	Spinach (n=5) 0.1μg/g Recovery	RSD	Spike Recovery	Spinach (n=5) 0.01μg/g Recovery	RSD	Spike Recovery	Brown Rice (n=3) 0.1μg/g Recovery	RSD	Spike Recovery	Orange (n=3) 0.1μg/g Recovery	RSD	Spike Recovery
3-OH carbofuran	93.4	4.0	105.0	90.0	7.9	100.0	122.3	1.9	127.0	107.3	1.4	116.0
4-CPA	80.2	15.2	90.0	78.0	5.7	80.0	127.3	2.4	145.0	134.3	5.6	137.0
Acephate	74.6	2.0	84.0	70.0	0.0	80.0	101.7	3.5	116.0	88.0	2.3	97.0
Acephate-d	60.0	3.3	59.0	50.0	0.0	50.0	87.0	3.4	92.0	72.3	0.8	66.0
Acetamiprid	71.4	2.7	91.5	79.0	11.3	100.0	71.2	1.1	93.0	64.7	5.0	90.5
Acibenzolar acid	139.4	3.2	159.0	254.0	13.8	250.0	162.0	1.1	185.0	148.3	7.4	153.0
Acibenzolar-S-methyl	89.4	1.8	98.5	99.0	4.2	105.0	95.2	2.4	100.5	86.5	2.6	95.5
Acifluorfen	7.2	15.2	89.5	0.0	—	135.0	0.0	—	112.0	0.0	—	81.5
Aldicarb	71.6	7.5	99.0	68.0	6.6	90.0	73.7	5.5	113.0	76.3	2.7	110.0
Aldicarb-sulfone	81.8	4.0	96.0	74.0	7.4	80.0	105.3	2.0	116.0	87.0	2.0	96.0
Aldicarb-sulfoxide	81.4	2.8	91.0	0.0	—	0.0	85.3	7.5	95.0	74.7	2.0	77.0
Azamethiphos	86.4	2.6	97.0	98.0	6.8	115.0	85.8	3.2	90.5	79.5	0.6	87.0
Azimsulfuron	88.6	4.7	92.0	80.0	6.3	110.0	84.5	6.4	109.0	63.3	6.0	83.5
Azoxystrobin	96.2	2.9	102.5	105.0	5.8	100.0	103.0	1.8	101.5	80.5	0.0	85.0
Bendiocarb	87.5	2.4	97.0	89.0	2.5	115.0	87.3	1.8	90.0	82.7	2.5	88.5
Benomyl	38.8	3.8	55.0	40.0	17.7	70.0	69.7	3.0	114.0	72.7	3.5	115.0
Bensulfuron-methyl	88.3	1.2	91.5	83.0	6.9	95.0	85.3	3.9	93.0	83.2	4.9	89.0
Bentazone	101.2	2.6	99.0	90.0	7.9	100.0	122.0	1.6	140.0	124.0	1.4	134.0
Benzobicyclon	106.2	2.8	116.5	105.0	5.8	110.0	116.2	4.7	115.5	109.5	4.1	121.0
Benzofenap	94.0	2.1	96.5	95.0	7.4	115.0	103.0	2.2	108.5	100.2	2.1	107.0
Bromoxynil	91.2	4.9	100.0	46.0	142.2	0.0	109.3	5.4	125.0	47.7	7.4	74.0
Butafenacil	97.3	5.6	99.0	100.0	11.2	110.0	105.7	4.3	113.0	83.5	6.7	81.0
Carbaryl	84.4	2.5	93.5	90.0	3.9	125.0	89.3	0.3	91.0	81.0	1.6	87.0
Carbaryl-d	85.7	4.1	80.5	90.0	5.6	105.0	85.7	3.5	80.0	153.5	40.1	88.0
Carbofuran	79.8	2.0	95.5	83.0	3.3	90.0	74.8	2.1	91.0	79.5	3.8	95.5
Carpropamid	88.6	4.0	97.0	95.0	10.5	105.0	88.3	3.3	88.0	58.2	2.5	61.0
Chloransulam-methyl	87.5	4.0	98.5	77.0	7.4	105.0	77.5	3.4	92.0	70.5	12.1	106.5
Chlorfluazuron	97.0	3.9	101.5	130.0	3.8	95.0	132.7	7.5	199.0	115.0	8.8	122.5
Chloridazon	75.4	2.0	98.0	76.0	9.8	155.0	75.2	5.5	93.5	74.5	6.0	94.5
Chlorimuron-ethyl	89.5	2.9	94.5	88.0	17.3	105.0	90.5	8.9	99.0	89.5	2.4	104.5
Chlorsulfuron	78.0	5.5	92.5	76.0	11.8	110.0	61.3	8.6	113.0	44.3	9.9	80.0
Chromafenozide	94.6	1.6	103.0	96.0	6.8	110.0	107.3	2.2	108.0	34.7	3.0	36.0
Clethodim	33.7	6.3	101.0	30.0	39.1	110.0	36.7	20.5	101.5	26.0	5.1	107.5
Clethodim-sulfone	79.5	4.8	92.0	74.0	10.0	165.0	83.0	5.5	86.5	75.3	2.3	102.5
Cloprop	89.8	3.3	96.0	96.0	9.3	90.0	108.0	3.3	128.0	81.0	8.6	90.0
Clothianidin	121.2	3.4	130.0	92.0	9.1	90.0	85.3	5.8	93.0	63.0	4.2	74.0
Cumyluron	94.0	4.6	102.0	96.0	4.4	110.0	93.5	1.9	86.0	92.0	1.9	94.5
Cyazofamid	99.2	2.1	96.5	99.0	7.5	115.0	88.5	4.5	83.5	54.2	2.3	57.5
Cyclosulfamuron	94.1	4.0	97.0	105.0	4.8	125.0	98.7	2.5	107.5	98.8	4.3	103.5
Cycloxydim	24.7	9.2	99.0	25.0	40.0	105.0	25.7	29.2	96.0	17.5	7.6	104.5
Cymoxanil	117.4	6.6	132.0	120.0	15.6	160.0	102.7	2.2	116.0	83.0	9.4	92.0
Cyprodinil	85.5	3.5	94.5	88.0	11.8	90.0	84.2	2.2	87.5	83.3	2.8	94.5
Diclosulam	82.3	3.6	95.5	72.0	7.9	105.0	70.8	4.3	93.0	60.5	10.8	93.0
Diflubenzuron	91.2	3.4	98.0	87.0	13.2	100.0	92.3	7.2	93.5	89.3	2.3	93.5
Dimethirimol	76.5	7.0	97.5	72.0	16.0	95.0	80.0	6.6	90.5	51.5	6.8	92.5
Dimethoate	80.4	1.1	90.0	70.0	10.1	100.0	88.7	4.7	99.0	83.7	2.5	96.0
Dimethomorph(E)	94.9	6.6	103.5	111.0	8.7	105.0	104.7	1.2	105.5	86.3	2.2	93.5
Dimethomorph(Z)	94.4	3.6	110.0	97.0	7.8	125.0	101.5	1.5	106.5	65.5	2.3	63.5
Dimron	101.8	4.6	115.0	99.0	6.6	115.0	105.2	1.5	110.5	109.5	1.2	117.0
Diuron	85.8	2.3	90.5	90.0	10.4	110.0	87.7	1.4	86.0	76.5	3.4	84.0
Ethiofencarb	29.5	9.0	101.0	24.0	57.8	90.0	8.8	33.2	93.5	25.7	8.1	98.5
Ethiofencarb-sulfone	84.2	4.2	90.0	90.0	7.9	90.0	112.0	3.2	121.0	86.7	3.5	110.0
Ethiofencarb-sulfoxide	100.0	6.7	82.0	72.0	6.2	70.0	99.7	3.1	104.0	73.7	5.1	85.0
Famoxadone	108.5	3.9	108.5	125.0	8.0	150.0	122.8	2.1	118.0	89.8	6.3	94.0
Fenbucarb	83.1	3.8	97.5	88.0	3.1	170.0	93.3	1.3	92.0	79.5	0.6	89.5

Pesticide	Spinach (n=5) 0.1μg/g Recovery	RSD	Spike Recovery	Spinach (n=5) 0.01μg/g Recovery	RSD	Spike Recovery	Brown Rice (n=3) 0.1μg/g Recovery	RSD	Spike Recovery	Orange (n=3) 0.1μg/g Recovery	RSD	Spike Recovery
Fenhexamid	92.5	3.0	95.0	95.0	11.8	105.0	85.3	2.1	83.5	85.3	4.0	89.0
Fenpyroximate(E)	102.9	4.2	112.5	123.0	2.2	105.0	110.0	4.3	142.5	117.0	2.6	134.0
Fenpyroximate(Z)	98.0	2.8	104.5	113.0	4.0	155.0	98.7	2.0	118.0	105.8	1.8	112.5
Ferimzone(E+Z)	85.8	2.7	99.5	87.0	5.1	135.0	85.5	2.0	93.5	73.7	1.0	82.0
Flazasulfuron	85.5	1.8	95.0	83.0	3.3	110.0	85.7	7.0	112.5	83.0	4.2	109.5
Fluazifop-acid	87.8	1.8	94.5	96.0	8.6	115.0	84.8	7.5	100.0	69.3	5.8	92.5
Fluazifop-buthyl	100.7	1.9	103.0	106.0	3.9	105.0	107.2	1.8	118.0	107.8	1.9	110.0
Flufenoxuron	108.4	3.4	101.5	153.0	8.5	130.0	136.3	4.5	180.5	124.2	5.6	133.5
Flumetsulam	72.2	2.6	90.0	65.0	10.9	105.0	48.5	9.0	106.5	98.2	14.1	137.5
Flusulfamide	96.0	5.1	109.5	119.0	7.5	165.0	129.2	5.5	150.0	101.8	8.4	128.0
Fomesafen	31.3	10.1	67.5	11.0	20.3	70.0	5.5	24.1	75.5	3.3	17.3	60.5
Halosulfuron-methyl	80.1	3.4	86.0	70.0	12.4	75.0	83.0	4.2	104.5	57.8	11.8	93.5
Haloxyfop	89.5	3.4	97.0	98.0	10.6	115.0	96.7	4.2	109.0	73.0	11.0	104.0
Hexaflumuron	101.8	3.2	102.0	130.0	4.7	125.0	122.5	4.6	132.5	113.0	3.8	115.0
Imazalil	78.5	2.6	99.5	89.0	4.7	150.0	60.7	6.0	85.5	356.7	4.1	350.5
Imazosulfuron	81.6	5.4	97.0	72.0	27.6	115.0	69.3	6.5	110.0	72.0	10.9	114.0
Imidacloprid	85.6	2.6	93.0	80.0	8.8	90.0	95.3	6.8	115.0	77.3	3.3	88.0
Inabenfide	91.0	3.1	109.5	380.0	43.2	100.0	97.5	1.8	96.0	86.8	4.1	101.0
Indanofan	88.2	3.7	95.5	98.0	12.8	125.0	76.0	3.4	76.0	44.3	3.6	46.5
Indoxacarb MP	99.9	4.6	104.5	123.0	13.0	115.0	113.5	1.8	122.5	110.0	4.0	115.5
Isoxaflutole	80.0	2.0	83.0	105.0	4.8	105.0	97.2	1.8	90.0	68.8	4.1	93.0
Lactofen	111.1	1.8	105.5	135.0	3.7	90.0	127.0	6.5	147.5	117.5	6.3	123.5
Linuron	89.0	2.9	89.5	92.0	7.3	160.0	89.8	0.9	89.5	43.2	5.5	47.5
Lufenuron	108.3	4.1	98.5	138.0	13.5	110.0	130.2	5.0	154.5	119.2	5.5	130.0
MBC	61.0	3.5	91.5	66.0	8.3	160.0	71.0	2.4	90.5	46.5	4.7	96.5
MCPA	112.4	3.0	118.0	116.0	4.7	120.0	110.0	2.4	129.0	115.3	3.3	129.0
Mecoprop	79.0	5.0	94.5	84.0	5.0	120.0	64.2	6.6	95.0	75.0	9.7	109.5
Mepanipyrim	85.9	3.0	95.0	92.0	4.9	110.0	80.8	2.5	85.5	59.3	1.3	65.0
Mepanipyrim_metabolite	75.7	2.2	81.5	93.0	4.8	105.0	82.7	2.3	87.0	82.7	4.0	84.5
Methabenzthiazuron	97.4	3.3	108.0	97.0	5.9	100.0	101.2	3.6	101.5	89.2	0.6	99.5
Methamidophos	66.2	3.4	69.0	52.0	8.6	60.0	61.7	3.7	72.0	59.7	2.6	67.0
Methamitron	47.0	3.4	56.0	56.0	9.8	50.0	65.7	5.8	79.0	33.0	3.0	42.0
Methiocarb	87.9	2.6	98.0	92.0	4.9	110.0	90.3	0.6	95.0	34.8	0.8	37.5
Methiocarb-sulfone	85.8	0.7	94.5	89.0	8.3	100.0	81.5	3.8	93.0	88.0	1.1	93.5
Methiocarb-sulfoxide	3.4	1.6	79.0	68.0	6.6	90.0	97.7	2.6	111.0	84.7	3.4	92.0
Methomyl	82.2	5.0	93.0	86.0	6.4	90.0	104.3	5.9	114.0	84.0	3.6	90.0
Methomyl-d	84.4	3.1	85.0	74.0	7.4	80.0	104.3	7.3	96.0	80.7	4.7	70.0
Methoxyfenozide	94.2	3.1	109.0	100.0	5.0	100.0	98.7	2.3	96.5	96.7	2.4	103.5
Methyl-Dimron	98.0	6.4	108.0	102.0	4.4	115.0	103.5	2.2	101.5	46.7	1.6	46.5
Metsulfuron-methyl	86.7	2.4	100.5	78.0	9.7	115.0	68.5	11.0	120.0	89.3	15.0	127.0
Monocrotophos	88.0	5.9	84.0	74.0	7.4	70.0	101.0	6.0	117.0	76.7	6.2	72.0
Naproanilide	100.1	1.1	105.5	103.0	4.3	100.0	104.7	2.8	102.0	93.2	0.3	100.0
Nitenpyram	70.8	9.7	94.0	82.0	13.4	80.0	118.0	0.8	140.0	66.7	4.8	84.0
Omethoate	90.4	2.8	97.0	78.0	5.7	90.0	94.0	2.8	104.0	77.7	4.5	86.0
Oryzalin	91.8	6.6	100.0	93.0	9.8	115.0	93.0	1.4	91.0	84.7	5.8	95.0
Oxamyl	88.2	2.0	97.0	94.0	9.5	90.0	104.7	6.5	119.0	99.3	2.3	109.0
Oxaziclomefone	89.8	2.7	96.5	97.0	2.8	115.0	92.0	1.1	98.0	93.8	2.5	98.5
Oxycarboxin	80.9	4.0	93.0	84.0	6.5	105.0	83.2	3.0	90.0	77.5	4.2	93.0
Oxydemeton-methyl	[illegible]5.0	1.9	84.0	82.0	5.5	90.0	92.3	4.4	108.0	76.7	3.3	84.0
Pencycuron	92.6	2.0	99.0	100.0	6.1	110.0	94.0	0.5	96.5	93.3	3.6	94.5
Phenmedipham	91.6	3.0	98.5	93.0	8.2	105.0	95.0	2.8	96.5	90.8	2.2	97.0
pymetrozine	85.2	4.7	93.0	100.0	7.1	100.0	128.0	5.5	133.0	97.0	7.1	104.0
Pyrazosulfuron-ethyl	90.8	2.4	95.0	89.0	4.7	110.0	91.0	4.5	101.0	85.0	4.7	87.5
Pyrazoxyfen	96.8	3.1	106.5	104.0	5.3	120.0	104.8	2.4	107.0	70.7	4.1	75.5
Pyributicarb	99.1	1.6	105.5	109.0	6.0	130.0	103.7	2.0	118.0	97.7	3.8	110.0

Pesticide	Spinach (n=5) 0.1μg/g Recovery	RSD	Spike Recovery	Spinach (n=5) 0.01μg/g Recovery	RSD	Spike Recovery	Brown Rice (n=3) 0.1μg/g Recovery	RSD	Spike Recovery	Orange (n=3) 0.1μg/g Recovery	RSD	Spike Recovery
Pyridate	69.2	6.7	105.5	128.0	9.0	140.0	119.3	4.5	221.0	24.8	20.4	181.5
Pyridate_Metabolite	71.7	4.7	94.0	72.0	11.6	90.0	72.3	7.0	71.0	85.2	2.2	90.0
Pyriftalid	98.3	3.8	108.5	107.0	7.8	120.0	104.2	2.5	101.5	96.7	2.1	104.5
Quinclorac	84.4	4.8	88.0	54.0	21.1	40.0	114.0	3.5	127.0	76.0	1.3	80.0
Quizalofop-ethyl	103.4	1.9	107.5	125.0	4.9	150.0	126.8	6.0	135.0	115.7	0.9	120.0
Sethoxydim	32.7	7.8	102.5	33.0	40.9	145.0	32.2	15.6	95.0	25.5	2.0	95.0
TBZ	77.7	3.7	93.0	84.0	2.7	105.0	82.8	2.7	89.5	185.0	2.6	212.5
Tebufenozide	91.4	5.6	97.0	90.0	5.6	100.0	94.0	3.0	89.5	95.7	0.8	101.5
Teflubenzuron	103.3	2.6	102.5	114.0	7.2	130.0	116.8	3.5	129.0	110.7	2.3	118.5
Thiacloprid	81.8	1.9	92.0	93.0	6.1	95.0	92.3	2.8	94.5	93.0	2.5	89.5
thiamethoxam	93.4	3.5	87.0	90.0	13.6	100.0	111.0	4.1	129.0	92.7	6.6	102.0
Thifensulfuron-methyl	85.1	4.4	99.5	76.0	8.6	135.0	62.7	9.3	121.0	84.8	15.7	139.0
Thiodicarb	102.4	2.8	110.0	107.0	4.2	115.0	110.0	2.8	105.5	108.7	4.3	116.0
Tralkoxydim	88.9	3.5	100.0	79.0	30.2	110.0	84.2	1.2	95.5	71.0	2.4	107.5
Tribenuron-methyl	79.7	6.5	94.0	85.0	7.2	95.0	110.8	8.4	106.5	87.8	3.1	100.5
Trichlamide	88.3	4.9	95.5	91.0	4.6	90.0	91.0	0.5	92.0	91.5	3.3	100.0
Trichlorfon	83.2	6.2	87.0	90.0	13.6	100.0	88.3	7.5	102.0	81.0	5.4	88.0
Trifloxystrobin	88.7	4.5	100.0	100.0	6.1	115.0	101.8	0.8	109.5	93.3	0.8	98.0
Triflusulfuron-methyl	87.0	1.3	93.0	93.0	7.2	115.0	92.7	6.7	102.5	75.0	3.3	89.0
Trinexapac-ethyl	85.7	1.2	89.0	81.0	11.0	115.0	64.2	3.5	89.5	80.2	3.1	88.5

*Sample were spiked at 0.1μg/g of each pesticides(127 compounds).

* (magenta) pesticides were eluted PSA mini column (27 compounds).

* (blue) pesticides were analyzed using LC method-Ⅱ (33 compounds).

Summary

▮A lot of pesticides from the polar to non-polar were able to be re-concentrated by retaining in two stages of dilution via a solid phase HBS mini column.

▮Because of not necessary liquid-liquid distribution by a separatory funnel, it was related to fast and simple pretreatment process.

▮The pretreatment