

Supplementary Materials

Multi-Residue Screening of Pesticides in Aquaculture Waters through Ultra-High-Performance Liquid Chromatograph-Q/Orbitrap Mass Spectrometry

Shou-Ying Wang ^{1,2}, Essy Kouadio Fodjo ³, Cong Kong ^{1,4,*} and Hui-Juan Yu ^{1,*}

¹ Key Laboratory of East China Sea Fishery Resources Exploitation, Ministry of Agriculture and Rural Affairs, East China Sea Fisheries Research Institute, Chinese Academy of Fishery Sciences, Shanghai 200090, China; magnolia7319@163.com

² College of Food Science & Technology, Shanghai Ocean University, Shanghai 201306, China

³ Laboratory of Physical Chemistry, Université Felix Houphouët-Boigny, 22 BP 582 Abidjan 22, Cote d'Ivoire; kouadio.essy@univ-fhb.edu.ci

⁴ Shanghai Key Laboratory of Forensic Medicine (Academy of Forensic Science), Shanghai 200063, China

* Correspondence: kongc@ecsf.ac.cn (C.K.); yuhuj@ecsf.ac.cn (H.-J.Y.)

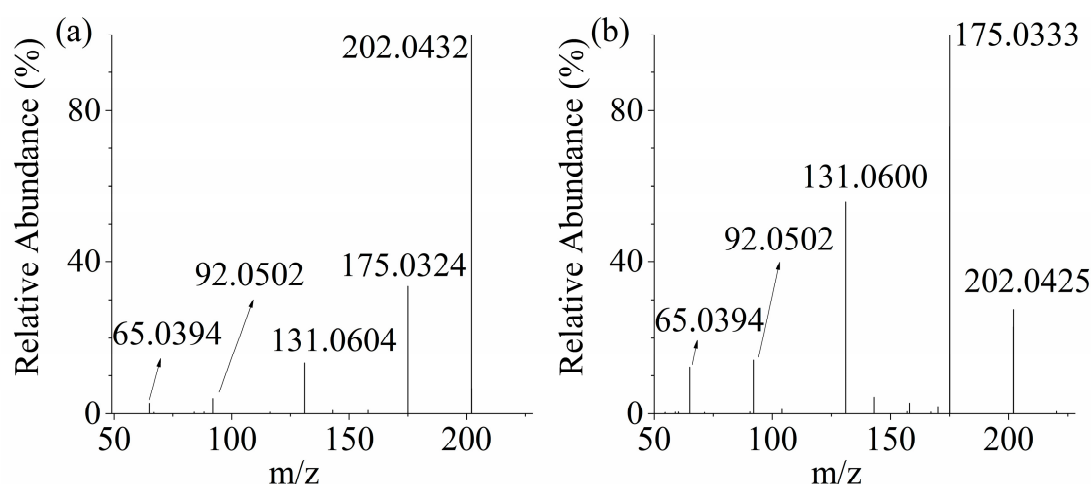


Figure S1. Comparison of MS/MS spectrum of thiabendazole generated by different NCE, (a) at NCE of 20, 50 and 80, (b) at NCE of 80.

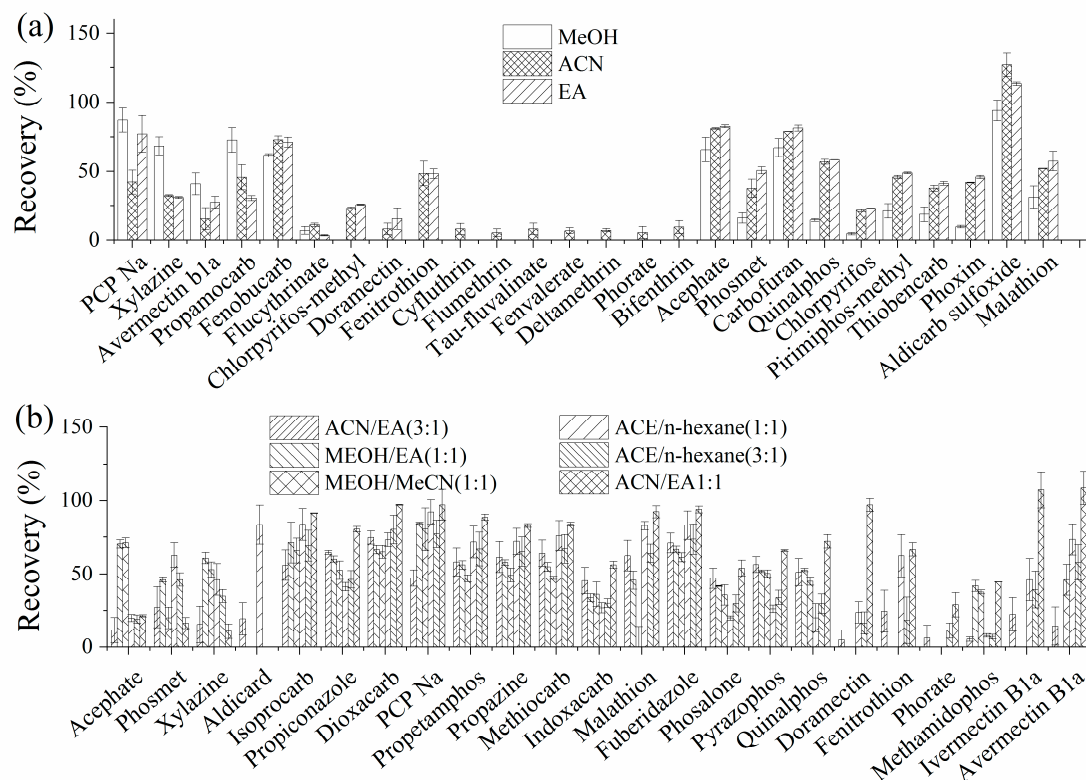


Figure S2. Compounds of significant recovery differences with single (a) or mixed (b) solvents as eluents after loading on HLB columns. Spiked level of $2.5 \mu\text{g}\cdot\text{L}^{-1}$.

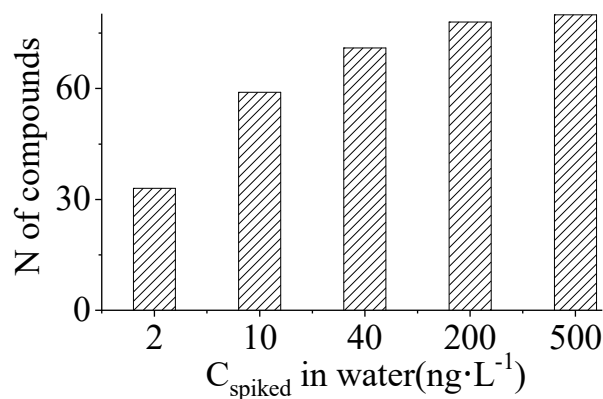


Figure S3. Number of compounds identified at different spiked concentration in blank aquaculture water samples.

Table S1. Chromatographic mass spectrometry parameters and instrument detection limits (IDL) of 80 pesticides.

Compound	Formula	CAS Number	Classification	Retention Time (min)	Extracted Mass (m/z)	Fragment Ion (m/z)	Adduct	IDL (ng/mL)	NCE
Avermectin B1a	C48H72O14	71751-41-2	avermectins/I	11.81	895.48143	123.11683/153.05462	M+Na	5	50
Doramectin	C50H74O14	117704-25-3	avermectins/I	12.00	921.49708	777.41843/449.25097	M+Na	2	50
Ivermectin B1a	C48H74O14	70288-86-7	avermectins/I	12.20	897.49708	753.41843/329.20872	M+Na	5	50
Propamocarb	C9H20N2O2	24579-73-5	carbamates/F	4.05	189.15975	102.05496/74.02365	M+H	1	20, 50, 80
Aminocarb	C11H16N2O2	2032-59-9	carbamates/I	4.23	209.12845	152.10699/137.08352	M+H	1	20, 50, 80
Aldicarb sulfoxide	C7H14N2O3S	1646-87-3	carbamates/I	5.00	207.07979	89.04195/69.0573	M+H	20	40
Aldicarb sulfone	C7H14N2O4S	1646-88-4	carbamates/I	4.95	223.0747	86.06004/76.0393	M+H	1	40
Methomyl	C5H10N2O2S	16752-77-5	carbamates/I	5.71	163.05357	88.02155/106.03211	M+H	50	20, 50, 80
Thiofanox sulfoxide	C9H18N2O3S	39184-27-5	carbamates/I	7.10	235.11109	57.06988/104.01646	M+H	2	40
Thiofanox sulphone	C9H18N2O4S	39184-59-3	carbamates/I	7.01	251.106	251.106/57.06988	M+H	5	30
Dioxacarb	C11H13NO4	6988-21-2	carbamates/I	7.27	224.09173	123.04406/167.07027	M+H	1	20, 50, 80
Pirimicarb	C11H18N4O2	23103-98-2	carbamates/I	8.06	239.15025	182.12879/72.04439	M+H	1	20, 50, 80
Propoxur	C11H15NO3	114-26-1	carbamates/I	8.52	210.11247	111.04406/95.04914	M+H	1	40
Carbofuran	C12H15NO3	1563-66-2	carbamates/I	8.60	222.11247	123.04406/165.09101	M+H	1	20, 50, 80
Bendiocarb	C11H13NO4	22781-23-3	carbamates/I	8.57	224.09173	167.07027/109.02841	M+H	1	20
Carbaryl	C12H11NO2	63-25-2	carbamates/I	9.06	202.08626	132.04439/124.08827	M+H	1	40
XMC	C10H13NO2	2655-14-3	carbamates/I	9.03	180.10191	123.08044/113.99744	M+H	2	40
Isoprocarb	C11H15NO2	2631-40-5	carbamates/I	9.21	194.11756	137.09609/95.04914	M+H	2	20, 50, 80
Thiofanox	C9H18N2O2S	39196-18-4	carbamates/I,A	9.04	241.09812	58.06513/184.07906	M+Na	5	20, 50, 80
2,3,5-Trimethacarb	C11H15NO2	2655-15-4	carbamates/I,A,N	9.21	194.11756	135.04406/107.04914	M+H	2	20, 50, 80
3,4,5-Trimethylphenol	C11H15NO2	527-54-8	carbamates/I,A,N	9.50	194.11756	135.04406/107.04914	M+H	2	20, 50, 80
Fenobucarb	C12H17NO2	3766-81-2	carbamates/I	9.68	208.13321	208.13321/95.04914	M+H	1	40
Methiocarb	C11H15NO2S	2032-65-7	carbamates/M	9.90	226.08963	169.06816/121.06479	M+H	1	20
Promecarb	C12H17NO2	2631-37-0	carbamates/I	9.95	208.13321	208.13321/109.06479	M+H	1	40
Carbendazim	C9H9N3O2	10605-21-7	imidazoles/F	5.89	192.07675	160.05054/132.05562	M+H	1	20, 50, 80
Thiabendazole	C10H7N3S	148-79-8	imidazoles/F	7.10	202.04334	175.03245/131.06037	M+H	1	80
Fuberidazole	C11H8N2O	3878-19-1	imidazoles/F	7.17	185.07094	157.07602/156.0682	M+H	1	60
Imazalil	C14H14Cl2N2O	35554-44-0	imidazoles/F	9.17	297.0556	158.97628/69.04472	M+H	1	50
Dodemorph	C18H35NO	1593-77-7	imidazoles/F	9.41	282.27914	116.10699/98.09643	M+H	1	40
Chlordimeform	C10H13ClN2	6164-98-3	organochlorines/I	6.35	197.084	117.0573/152.02615	M+H	1	60

Thiobencarb	C12H16ClNOS	28249-77-6	organochlorines/H	10.90	258.07139	258.07139/125.01525	M+H	1	20, 50, 80
PCP Na ¹	C6Cl5HO	131-52-2	organochlorines/H	11.48	262.83973	262.83973/262.83973	M-H	1	20, 50, 80
Methamidophos	C2H8NO2PS	10265-92-6	organophosphorus/I	2.50	142.00861	142.00861/112.01598	M+H	5	50
Acephate	C4H10NO3PS	30560-19-1	organophosphorus/I	3.23	184.01918	113.0025/142.99263	M+H	20	20, 50, 80
Omethoate	C5H12NO4PS	1113-02-6	organophosphorus/I	4.20	214.02974	214.02974/182.98754	M+H	1	20, 50, 80
Monocrotophos	C7H14NO5P	6923-22-4	organophosphorus/I	6.58	224.06824	127.01547/58.02874	M+H	1	35
Dichlorvos	C4H7Cl2O4P	62-73-7	organophosphorus/I	8.67	220.95318	127.01565/78.99452	M+H	5	20, 50, 80
trichlorfon	C4H8Cl3O4P	52-68-6	organophosphorus/I	6.65	256.92985	127.01547/220.95318	M+H	1	20, 50, 80
Dimethoate	C5H12NO3PS2	60-51-5	organophosphorus/I	7.15	230.0069	142.99623/170.96978	M+H	1	35
Phoratoxon sulfoxide	C7H17O4PS2	2588-05-8	organophosphorus/I	7.48	261.03786	114.96133/128.97698	M+H	1	20, 50, 80
Mevinphos	C7H13O6P	7786-34-7	organophosphorus/I	7.89	225.05225	127.01547/67.01784	M+H	1	30
Phorate sulfoxide	C7H17O3PS3	2588-03-6	organophosphorus/I,A	9.20	277.01502	114.96133/142.93848	M+H	1	30
Famphur	C10H16NO5PS2	52-85-7	organophosphorus/I	9.25	326.02803	93.00999/142.99263	M+H	1	20, 50, 80
Phorate sulfone	C7H17O4PS3	2588-04-7	organophosphorus/I,A	9.20	293.00993	246.96807/114.96133	M+H	2	20, 50, 80
Isocarbophos	C11H16NO4PS	24353-61-5	organophosphorus/I	9.35	312.04299	269.99844/236.00554	M+Na	1	20, 50, 80
Methidathion	C6H11N2O4PS3	950-37-8	organophosphorus/I	9.55	302.96913	145.00662/71.02399	M+H	2	20
Phosmet	C11H12NO4PS2	732-11-6	organophosphorus/I	9.73	318.00181	160.0393/133.02841	M+H	1	40
Fenitrothion	C9H12NO5PS	122-14-5	organophosphorus/I	9.96	278.02466	142.99263/149.02332	M+H	50	30
Malathion	C10H19O6PS2	121-75-5	organophosphorus/I	9.94	353.02529	195.06031/227.03238	M+Na	1	20, 50, 80
Propetamphos	C10H20NO4PS	31218-83-4	organophosphorus/I	10.05	282.09234	138.0137/156.02395	M+H	5	40
Triazophos	C12H16N3O3PS	24017-47-8	organophosphorus/I	10.20	314.07228	162.06619/114.96133	M+H	1	20, 50, 80
Quinalphos	C12H15N2O3PS	13593-03-8	organophosphorus/I	10.58	299.06138	147.05529/163.03245	M+H	1	20, 50, 80
Coumaphos	C14H16ClO5PS	56-72-4	organophosphorus/I	10.72	363.02174	226.99263/306.95913	M+H	1	20, 50, 80
Pirimiphos-methyl	C11H20N3O3PS	29232-93-7	organophosphorus/I	10.72	306.10358	108.05562/67.02907	M+H	1	50
Phosalone	C12H15ClNO4PS2	2310-17-0	organophosphorus/I	10.80	367.99414	114.9615/138.01033	M+H	1	40
Pyrazophos	C14H20N3O5PS	13457-18-6	organophosphorus/F	10.83	374.0934	194.05602/222.08732	M+H	1	20, 50, 80
Phoxim	C12H15N2O3PS	14816-18-3	organophosphorus/I	10.80	299.06138	129.04472/95.04945	M+H	1	20, 50, 80
Chlorpyrifos-methyl	C7H7Cl3NO3PS	5598-13-0	organophosphorus/I	11.07	321.90226	142.99263/289.87605	M+H	10	20, 50, 80
Chlorpyrifos	C9H11Cl3NO3PS	2921-88-2	organophosphorus/I	11.49	349.93356	114.9615/197.9273	M+H	1	30
Tributylphosphorotrithioate	C12H27OPS3	78-48-8	organophosphorus/GR	11.77	315.10344	57.06988/168.99052	M+H	1	20, 50, 80
Prothiofos	C11H15Cl2O2PS2	34643-46-4	organophosphorus/I	11.98	344.97009	258.92025/132.96046	M+H	100	20, 50, 80
Xylazine	C12H16N2S	7361-61-7	others	6.93	221.1107	90.0372/164.05285	M+H	1	50
Ethoxyquin	C14H19NO	91-53-2	others/F	9.15	218.15394	148.07569/190.12264	M+H	1	20, 50, 80
Propiconazole	C15H17Cl2N3O2	60207-90-1	others/F	10.79	342.07706	158.97628/69.06988	M+H	1	20, 50, 80

Indoxacarb	C22H17ClF3N3O7	144171-61-9	others/I	10.91	528.07799	203.01866/168.02107	M+H	1	20, 50, 80
Fipronil-desulfinyl	C12H4Cl2F6N4	205650-65-3	phenylpyrazoles/I	10.25	386.96444	350.98667/281.99146	M-H	1	40
Fipronil	C12H4Cl2F6N4OS	120068-37-3	phenylpyrazoles/I	10.36	434.93143	329.95845/183.01646	M-H	1	40
Fipronil-sulfide	C12H4Cl2F6N4S	120067-83-6	phenylpyrazoles/I	10.47	418.93651	57.9746/170.00863	M-H	1	40
Fipronil-sulfone	C12H4Cl2F6N4O2S	120068-36-2	phenylpyrazoles/I	10.58	450.92634	414.94857/243.98839	M-H	1	40
flucythrinate	C26H23F2NO4	70124-77-5	pyrethroids/I,A	11.42	469.19334	114.09134/199.0929	M+NH4	5	20, 50, 80
Cyfluthrin	C22H18Cl2FNO3	68359-37-5	pyrethroids/I,A	11.60	451.0986	191.0025/114.09134	M+NH4	500	20, 50, 80
Deltamethrin	C22H19Br2NO3	52918-63-5	pyrethroids/I,A	11.74	521.00699	278.90762/89.05971	M+NH4	5	20, 50, 80
Fenvalerate	C25H22ClNO3	51630-58-1	pyrethroids/I,A	11.74	437.16265	437.16265/114.09134	M+NH4	20	20, 50, 80
Tau-fluvalinate	C26H22ClF3N2O3	102851-06-9	pyrethroids/I,A	11.85	503.13438	181.06479/114.09134	M+H	20	40
Flumethrin	C28H22Cl2FNO3	69770-45-2	pyrethroids/I,A	12.03	532.0853	114.09134/73.02982	M+Na	5	20, 50, 80
Bifenthrin	C23H22ClF3O2	82657-04-3	pyrethroids/I,A	12.18	440.15987	181.10118/166.0777	M+NH4	10	20, 50, 80
Simazine	C7H12ClN5	122-34-9	triazines/H	8.72	202.0854	132.0323/124.08692	M+H	1	50
Simetryne	C8H15N5S	1014-70-6	triazines/H	8.80	214.11209	96.05562/124.08692	M+H	1	60
Propazine	C9H16ClN5	139-40-2	triazines/H	9.78	230.1167	188.06975/146.0228	M+H	1	50
Prometryn	C10H19N5S	7287-19-6	triazines/H	9.83	242.14339	200.09644/158.04949	M+H	1	50

‡: Sodium pentachlorophenol; A: Acaricide; F: Fungicide; GR: Growth regulator; H: herbicide; I: insecticide; N: Nematicide

Table S2. The recovery, relative standard deviation (RSD) at three different levels SDL, and matrix effect of eighty compounds.

Compounds	SDLs (ng·L ⁻¹)	ME (%)	200 ng·L ⁻¹		40 ng·L ⁻¹		10 ng·L ⁻¹	
			Recovery	RSD	Recovery	RSD	Recovery	RSD
Triazophos	2.00	17	80%	12%	89%	7%	89%	12%
Propiconazole	2.00	13	97%	12%	101%	9%	109%	15%
Monocrotophos	2.00	−2	99%	12%	101%	7%	94%	9%
Dimethoate	2.00	7	98%	14%	108%	6%	105%	10%
Phosalone	2.00	14	97%	15%	115%	8%	108%	14%
Pyrazophos	2.00	12	102%	10%	100%	14%	101%	9%
Quinalphos	2.00	18	85%	14%	82%	12%	89%	10%
Thiabendazole	2.00	37	72%	10%	92%	6%	87%	10%
Bendiocarb	2.00	7	90%	12%	103%	9%	86%	14%
Propazine	2.00	12	75%	12%	91%	10%	93%	8%
Prometryn	2.00	15	82%	13%	101%	10%	89%	9%
Imazalil	2.00	8	86%	9%	84%	9%	75%	11%
Trichlorfon	2.00	11	84%	13%	84%	7%	72%	14%
Methidathion	2.00	6	99%	15%	88%	13%	79%	8%
Chlordimeform	2.00	25	87%	12%	79%	7%	60%	12%
Fipronil-desulfinyl	2.00	9	105%	12%	99%	9%	103%	9%
Fipronil	2.00	−50	100%	15%	104%	15%	111%	8%
Fipronil-sulfone	2.00	9	98%	15%	104%	12%	107%	13%
Fipronil-sulfide	2.00	0	102%	12%	114%	10%	110%	13%
Phoratoxon sulfoxide	2.00	18	102%	12%	117%	8%	101%	7%
Famphur	2.00	8	83%	11%	91%	14%	96%	15%
Pirimiphos-methyl	2.00	2	91%	20%	88%	9%	93%	13%
Phorate sulfoxide	2.00	13	119%	9%	119%	8%	118%	13%
Phorate sulfone	2.00	15	90%	14%	103%	12%	94%	9%
Methiocarb	2.00	26	67%	19%	77%	9%	60%	10%
Carbaryl	2.00	22	86%	8%	95%	8%	85%	13%
Tributylphosphorotrithioate	2.00	13	82%	15%	90%	8%	81%	10%
Indoxacarb	2.00	16	85%	11%	89%	9%	87%	9%
Coumaphos	2.00	0	97%	17%	97%	9%	102%	8%
Simazine	2.00	24	85%	9%	95%	6%	79%	14%
Simetryne	2.00	14	90%	9%	95%	8%	72%	6%
Phoxim	2.00	18	85%	14%	82%	12%	88%	10%
Fuberidazole	2.00	20	109%	7%	71%	7%	109%	9%
2,3,5-Trimethacarb	10.0	14	86%	7%	103%	10%	98%	7%
3,4,5-Trimethylphenol	10.0	4	92%	13%	96%	10%	74%	10%
Thiofanox sulfoxide	10.0	2	125%	6%	78%	14%	44%	13%
Thiofanox sulphone	10.0	16	98%	11%	105%	9%	82%	8%
Acephate	10.0	−9	16%	8%	10%	12%	10%	16%
Dioxacarb	10.0	4	91%	15%	100%	7%	97%	10%
Fenobucarb	10.0	−3	77%	9%	75%	9%	103%	14%
Carbofuran	10.0	−1	97%	14%	105%	8%	91%	10%
Dodemorph	10.0	38	116%	12%	110%	15%	99%	10%
Carbendazim	10.0	4	102%	4%	113%	3%	119%	8%
Propetamphos	10.0	24	81%	13%	80%	13%	60%	7%
Isoprocarb	10.0	14	86%	7%	96%	10%	98%	7%
Pirimicarb	10.0	28	96%	15%	93%	8%	86%	8%
Propoxur	10.0	−3	87%	9%	88%	9%	72%	9%

Chlorpyrifos	10.0	9	79%	13%	70%	18%	63%	15%
Flucythrinate	10.0	−2	61%	11%	64%	18%	24%	17%
Omethoate	10.0	−1	62%	14%	61%	8%	87%	9%
Aldicarb sulfoxide	10.0	−2	78%	5%	78%	7%	72%	9%
Aldicarb sulfone	10.0	−3	86%	15%	24%	14%	76%	12%
Methomyl	10.0	−10	110%	7%	76%	9%	107%	12%
Aminocarb	10.0	17	82%	15%	61%	11%	95%	2%
Promecarb	10.0	9	71%	15%	82%	5%	82%	13%
Methamidophos	10.0	−6	5%	15%	5%	9%	7%	5%
Thiobencarb	10.0	22	84%	9%	79%	8%	72%	8%
Propamocarb	10.0	6	61%	12%	54%	15%	48%	14%
Mevinphos	10.0	19	104%	14%	101%	15%	71%	14%
Phosmet	40.0	49	79%	10%	68%	19%		
Thiofanox	40.0	−18	20%	11%	32%	14%		
PCP Na	40.0	−11	115%	7%	92%	23%		
Ivermectin B1a	40.0	−6	74%	14%	76%	3%		
Xylazine	40.0	15	21%	19%	6%	15%		
Doramectin	40.0	−9	70%	18%	26%	11%		
Dichlorvos	40.0	13	86%	11%	76%	18%		
Isocarbophos	40.0	37	65%	9%	62%	10%		
Deltamethrin	40.0	5	64%	14%	63%	11%		
XMC	40.0	9	83%	9%	85%	9%		
Avermectin B1a	40.0	−88	57%	14%	29%	20%		
Malathion	40.0	44	100%	11%	71%	12%		
Ethoxyquin	200	34	1%	9%				
Fenitrothion	200	3	70%	15%				
Flumethrin	200	32	118%	15%				
Tau-fluvalinate	200	28	118%	12%				
Fenvalerate	200	2	75%	19%				
Chlorpyrifos-methyl	200	4	82%	14%				
Bifenthrin	200	40	78%	9%				
Prothiofos ¹	1000	11	38%	11%				
Cyfluthrin ¹	1000	18	35%	18%				

¹: the compound of spiked level is 1000 ng·L^{−1}.

Table S3. Screened results for 30 real samples at different aquaculture environment of Shanghai.

Samples	Detected Compounds	Content (ng/L)
1	propiconazole, dimethoate, carbendazim , prometryn , fipronil-desulfinyl, methomyl	<2.00, <2.00, 81.9 , 5.98 , <2.00, 10.0
2	carbendazim , prometryn	32.1 , <2.0
3	triazophos, propiconazole, dimethoate , carbendazim , propazine, prometryn, fipronil-desulfinyl, fipronil, fipronil-sulfone, fipronil-sulfide, methomyl , simazine , simetryne, propamocarb	<2.00, <2.00, 3.58 , 220 , <2.00, 20.5, <2.00, <2.00, <2.00, <2.00, 10.0 , 7.11 , <2.00, 10.0
4	thiabendazole , carbendazim , prometryn , fipronil-desulfinyl, fipronil-sulfone, fipronil-sulfide	2.00 , 58.7 , 9.50 , <2.00, <2.00, <2.00
5	carbendazim , propazine, prometryn , simazine, simetryne dioxacarb , thiabendazole, carbendazim , prometryn ,	13.2 , <2.00, 2.56 , <2.00, 21.8 8.39 , <2.00, 16.0 , 10.0 , 6.26 ,
6	chlordimeform , fipronil-desulfinyl, fipronil, fipronil-sulfone, fipronil-sulfide, famphur	<2.00, <2.00, <2.00, <2.00, <2.00
7	triazophos, propiconazole, prometryn , fipronil-desulfinyl, fipronil-sulfone, fipronil-sulfide	<2.00, <2.00, 2.00 , <2.00, <2.00, <2.00

8	carbendazim , prometryn, fipronil-desulfinyl	76.6, <2.00, <2.00
9	propiconazole, carbendazim , prometryn , fipronil-desulfinyl, simetryne , avermectin b1a	<2.00, 50.4, 54.1, <2.00, 34.1, 40.0
10	propiconazole, carbendazim , prometryn , methomyl, simetryne	<2.00, 59.6, 33.9, <2.00, 20.8
11	carbendazim , prometryn , fipronil-desulfinyl	25.1, 5.45, <2.00
12	propiconazole, carbendazim , prometryn , trichlorfon , fipronil-desulfinyl, fipronil-sulfone	<2.00, 43.7, 5.32, 29.9, <2.00, <2.00
13	propiconazole, dimethoate, carbendazim , prometryn , fipronil-desulfinyl	<2.00, <2.00, 43.2, 9.23, <2.00
14	triazophos, propiconazole , dimethoate, prometryn , fipronil-desulfinyl, fipronil, fipronil-sulfone, fipronil-sulfide	<2.00, 5.48, <2.00, 7.34, <2.00, <2.00, <2.00, <2.00
15	triazophos, propiconazole, carbendazim , prometryn , fipronil-desulfinyl, fipronil, fipronil-sulfone, fipronil-sulfide, methomyl	<2.00, <2.00, 224, 9.32, <2.00, <2.00, <2.00, <2.00, 10.0
16	carbendazim , prometryn	10.0, <2.00
17	propiconazole, carbendazim , prometryn, imazalil , fipronil-desulfinyl, fipronil-sulfone	<2.00, 4233, <2.00, 27.4, <2.00, <2.00
18	no detected pesticides	-
19	propiconazole, carbendazim , prometryn , fipronil-desulfinyl	<2.00, 85.0, 7.64, <2.00
20	carbendazim , prometryn	12.3, 18.3
21	dimethoate, carbendazim , prometryn, simetryne	<2.00, 32.6, <2.00, <2.00
22	triazophos, propiconazole , carbendazim , prometryn , fipronil-desulfinyl, fipronil, fipronil-sulfone, fipronil-sulfide	<2.00, 4.72, 10.0, 2.62, <2.00, <2.00, <2.00, <2.00
23	propiconazole, carbendazim , prometryn , trichlorfon , fipronil-desulfinyl, simetryne	<2.00, 37.7, 26.6, 219, <2.00, <2.00
24	carbendazim , prometryn , dichlorvos , trichlorfon	33.2, 9.35, 896, 1020
25	triazophos, propiconazole , dimethoate, carbendazim , prometryn , fipronil-desulfinyl, fipronil-sulfone, fipronil-sulfide	<2.00, 5.53, <2.00, 53.5, 25.3, <2.00, <2.00, <2.00
26	carbendazim , prometryn	16.6, 24.7
27	propiconazole, dimethoate, carbendazim , prometryn , fipronil-sulfone, simazine	2.78, <2.00, 69.9, 8.89, <2.00, <2.00
28	propiconazole, carbendazim , prometryn , simazine, simetryne	<2.00, 26.2, 16.5, <2.00, <2.00
29	triazophos, propiconazole, carbendazim , prometryn , fipronil-desulfinyl, fipronil, fipronil-sulfone, fipronil-sulfide	<2.00, <2.00, 69.0, 3.34, <2.00, <2.00, <2.00, <2.00
30	carbendazim , prometryn	14.8, <2.00

Note: <2.00 represent the content of compounds below SDLs of corresponding compounds, which were qualitatively screened out without quantification.



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