

Supporting Information

for

**Determination of Polychlorinated Biphenyls and
Organochlorine Pesticides in Chinese mitten Crab
(*Eriocheir sinensis*) using modified QuEChERS
followed by GC–MS**

Ting Liu, Jianguang Zhou, Li He, Jinhua Gan*

Yangtze River Fisheries Research Institute, Chinese Academy of Fishery Sciences, Wuhan, China

* Corresponding author: Jinhua Gan. Tel: +86-10-81780165; E-mail: gjh@yfi.ac.cn

The Supporting Information includes following items:

Page S3	Table S1. The information of collected Chinese mitten crabs.
Page S4	Table S2. Target compounds, retention time and and selected SIM ions for the analysis of pollutants residues by mass spectrometry.
Page S5	Table S3. Average recovery and relative standard deviation (RSD) of 8 PCBs and 10 OCPs spiked in white meat of mitten crabs at three different concentrations by GC–MS analysis.
Page S6	Table S4. Average recovery and relative standard deviation (RSD) of 8 PCBs and 10 OCPs spiked in brown meat of mitten crabs at three different concentrations by GC–MS analysis.
Page S7	Table S5. Comparison of the analytical features of the method proposed with other previously reported studies.

Table S1. The information of collected Chinese mitten crabs.

Number	Average weight	Location
01	2.8 kg	29°59'41"N, 116°40'3"E
02	2.5 kg	29°59'19"N, 116°39'47"E
03	2.4 kg	29°16'35"N, 116°12'38"E
04	2.6 kg	29°43'35"N, 116°18'26"E
05	2.7 kg	29°35'13"N, 116°20'40"E
06	2.9 kg	29°42'59"N, 115°48'2"E
07	2.9 kg	29°46'49"N, 115°39'28"E
08	2.4 kg	29°2'22"N, 115°49'13"E
09	2.8 kg	29°3'13"N, 115°51'31"E
10	3.0 kg	29°15'1"N, 115°49'27"E
11	2.5 kg	29°59'26"N, 116°43'7"E
12	2.7 kg	29°59'7"N, 116°44'1"E

Table S2. Target compounds, retention time and selected SIM ions for the analysis of pollutants residues by mass spectrometry.

Compounds	Retention Time (min)	SIM ions
α -HCH	8.78	218.9/180.9
β -HCH	9.46	218.9/182.9
γ -HCH	9.57	218.9/180.9
δ -HCH	10.28	218.9/180.9
<i>p,p'</i> -DDT	18.40	234.9
<i>o,p'</i> -DDT	17.23	236.9
<i>p,p'</i> -DDD	17.15	234.9
<i>o,p'</i> -DDD	16.01	236.9
<i>p,p'</i> -DDE	15.73	245.9
<i>o,p'</i> -DDE	14.70	317.8
PCB 28	11.09	255.9/185.9
PCB 52	12.08	291.8/219.9
PCB 101	14.80	325.8
PCB 118	16.86	325.7/183.9
PCB 153	17.61	359.7/289.8
PCB 138	18.55	359.7/289.8
PCB 180	20.60	393.7/323.7
PCB 198	21.33	429.6/357.7

Table S3. Average recovery and relative standard deviation (RSD) of 8 PCBs and 10 OCPs spiked in white meat of mitten crabs at three different concentrations by GC–MS analysis (n = 6).

Compounds	10 µg kg ⁻¹ spike level		20 µg kg ⁻¹ spike level		50 µg kg ⁻¹ spike level	
	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
α-HCH	108.9	14.3	107.6	12.4	114.7	12.2
β-HCH	112.2	1.9	119.9	4.3	104.3	4.6
γ-HCH	103.7	10.3	104.1	14.4	108.1	10.4
δ-HCH	105.8	4.6	100.8	2.7	107.5	2.8
<i>p,p'</i> -DDT	110.1	1.6	106.7	1.9	109.2	4.4
<i>o,p'</i> -DDT	115.3	3.7	103.7	1.3	110.8	6.7
<i>p,p'</i> -DDD	111.4	8.8	110.4	11.9	114.9	11.2
<i>o,p'</i> -DDD	93.5	12.4	98.4	11.6	96.2	7.4
<i>p,p'</i> -DDE	80.2	12.8	89.2	10.4	95.3	9.3
<i>o,p'</i> -DDE	107.4	3.2	91.7	9.2	101.4	10.6
PCB 28	100.9	2.1	102.6	0.2	99.2	0.1
PCB 52	102.8	4.9	108.7	5.4	100.6	4.3
PCB 101	111.6	3.3	119.7	0.6	113.4	0.9
PCB 118	107.7	6.4	109.5	2.6	101.5	4.8
PCB 153	105.9	5.7	105.4	7.7	102.6	11.5
PCB 138	107.6	8.2	106.2	6.3	105.3	9.5
PCB 180	99.8	7.9	101.6	2.0	100.2	6.9
PCB 198	116.3	12.1	118.6	11.3	114.5	6.8

Table S4. Average recovery and relative standard deviation (RSD) of 8 PCBs and 10 OCPs spiked in brown meat of mitten crabs at three different concentrations by GC–MS analysis (n = 6).

Compounds	10 µg kg ⁻¹ spike level		20 µg kg ⁻¹ spike level		50 µg kg ⁻¹ spike level	
	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
α-HCH	110.2	11.2	102.7	8.9	105.1	1.6
β-HCH	99.6	4.2	108.2	7.4	110.5	3.4
γ-HCH	102.3	6.6	97.3	3.5	113.2	2.9
δ-HCH	96.7	7.7	105.9	9.1	100.3	5.7
<i>p,p'</i> -DDT	100.3	8.2	111.6	2.9	104.8	3.6
<i>o,p'</i> -DDT	104.2	4.0	102.3	2.6	100.9	5.5
<i>p,p'</i> -DDD	110.4	7.2	114.8	3.9	101.1	6.6
<i>o,p'</i> -DDD	99.2	11.7	103.8	8.7	92.9	5.8
<i>p,p'</i> -DDE	89.3	4.5	94.2	6.8	87.3	7.4
<i>o,p'</i> -DDE	102.9	5.6	109.0	3.7	99.3	5.2
PCB 28	108.1	10.7	102.1	9.5	109.2	3.8
PCB 52	117.3	8.4	112.5	8.9	110.5	6.6
PCB 101	101.8	5.3	103.2	7.7	89.3	9.4
PCB 118	114.6	5.5	111.5	8.6	119.2	3.7
PCB 153	93.6	1.7	99.7	3.2	106.8	1.9
PCB 138	98.9	1.9	106.2	1.2	101.6	3.6
PCB 180	99.3	7.4	108.3	5.8	92.1	6.9
PCB 198	102.8	2.2	115.2	6.8	104.3	3.6

Table S5. Comparison of the analytical features of the method proposed with other previously reported studies.

Analytes	Matrix	Extraction time	Extraction solvent	Solvent comsumption	LOD ($\mu\text{g kg}^{-1}$)	LOQ ($\mu\text{g kg}^{-1}$)	Ref.
PCBs	Chinese mitten crabs	Not mentioned	Hexane-acetone (1:1)	20 mL	0.003-0.03	0.01-0.1	Ron L.A.P. Hoogenboom ¹
pharmaceutical and other residues	Crabs	15 min	MeCN	10 mL	3.3-15	10-50	Cong Kong ²
DDTs and PCBs	Water	5 h	n-Hexane	40 mL	1	3.3	A. Aguilar ³
			Hexane-	80 mL	0.01	0.03	Jing Chen ⁴
OCPs	Shrimps	>24 h	dichloromethane mixture (1:1v/v)				
OCPs, PCBs and PBDEs	Crabs	8 h	n-hexane-dichloromethane (1:1)	100 mL	0.03-0.33	0.1-1.1	Marta Commendatorea ⁵
OCPs, PCBs and PAHs	Sediment	20 min	hexane/acetone, dichloromethane/acetone	20 mL	0.003-2.8	0.01-9.6	Fida Ben Salem ⁶
PCBs and PAHs	Blue crabs	36 h	acetone and n-hexane (1:1, V/V)	300 mL	0.02	0.07	Mansoreh Ghaeni ⁷
PCBs	catfish	30 min.	Water and acetonitrile	35 mL	0.3	1	Narong Chamkasem ⁸
PCBs and PCDD/Fs	Chinese mitten crabs	Not mentioned	dichloromethane and hexane (1:1, v:v)	100 mL	0.08-0.8	0.3-2.5	Ying Han ⁹
OCPs and PCBs	Chinese mitten crabs	15 min	MeCN	15 mL	0.03-1.08	0.1-3.6	The proposed method

References:

1. Hoogenboom, R. L.; Kotterman, M. J.; Hoek-van Nieuwenhuizen, M.; van der Lee, M. K.; Mennes, W. C.; Jeurissen, S. M.; van Leeuwen, S. P., Dioxins, PCBs and heavy metals in Chinese mitten crabs from Dutch rivers and lakes. *Chemosphere* **2015**, *123*, 1-8.
2. Kong, C.; Wang, Y.; Huang, Y.; Yu, H., Multiclass screening of >200 pharmaceutical and other residues in aquatic foods by ultrahigh-performance liquid chromatography-quadrupole-Orbitrap mass spectrometry. *Anal Bioanal Chem* **2018**, *410* (22), 5545-5553.
3. Aguilar, A.; Borrell, A., DDT and PCB reduction in the western Mediterranean from 1987 to 2002, as shown by levels in striped dolphins (*Stenella coeruleoalba*). *Mar Environ Res* **2005**, *59* (4), 391-404.
4. Chen, J.; Chen, L.; Liu, D.; Zhang, G., Organochlorine pesticide contamination in marine organisms of Yantai coast, northern Yellow Sea of China. *Environ Monit Assess* **2014**, *186* (3), 1561-8.
5. Commendatore, M.; Yorio, P.; Scenna, L.; Ondarza, P. M.; Suarez, N.; Mariano, C.; Miglioranza, K.S.B., Persistent organic pollutants in sediments, intertidal crabs, and the threatened Olrog's gull in a northern Patagonia salt marsh, Argentina. *Mar Pollut Bull* **2018**, *136*, 533-546.
6. Ben Salem, F.; Ben Said, O.; Duran, R.; Monperrus, M., Validation of an Adapted QuEChERS Method for the Simultaneous Analysis of Polycyclic Aromatic Hydrocarbons, Polychlorinated Biphenyls and Organochlorine Pesticides in Sediment by Gas Chromatography–Mass Spectrometry. *Bulletin of Environmental Contamination and Toxicology* **2016**, *96* (5), 678-684.
7. Ghaeni, M.; Pour, N.A.; Hosseini, M., Bioaccumulation of polychlorinated biphenyl (PCB), polycyclic aromatic hydrocarbon (PAH), mercury, methyl mercury, and arsenic in blue crab *Portunus segnis* from Persian Gulf. *Environmental Monitoring & Assessment* **2015**, *187* (5), 253.
8. Chamkasem, N.; Lee, S.; Harmon, T., Analysis of 19 PCB congeners in catfish tissue using a modified QuEChERS method with GC-MS/MS. *Food Chem* **2016**, *192*, 900-6.
9. Han, Y.; Liu, W.; Zhu, W.; Rao, K.; Xiao, K.; Gao, L.; Su, G.; Liu, G., Sources of polychlorinated dibenzo-p-dioxins and dibenzofurans, and biphenyls in Chinese mitten crabs. *Chemosphere* **2018**, *196*, 522-530.