

Research Article



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오비트랩 고분해능 질량분석기를 이용한 폐사 조류 중 다성분 잔류 농약 스크리닝 기법

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Residual Multi Pesticides Screening of Dead Birds by Orbitrap High Resolution Mass Spectrometry

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Received: 27 September 2017/ Revised: 30 October 2017/ Accepted: 8 November 2017

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Abstract

BACKGROUND: The objective of this study was to evaluate screening method of residual multi pesticides in dead birds by Orbitrap high resolution mass spectrometry (HRMS) to identify the cause of death for birds .

METHODS AND RESULTS: Extraction and clean-up method of residual pesticides in liver of dead birds was used QuEChERS (Quick Easy Cheap Effective Rugged and Safe) and method validations was conducted using liquid chromatography and gas chromatography with triple-quadrupole mass spectrometer (LC/MS/MS and GC/MS/MS) Also, we were evaluated screening method for the determination of residual pesticides in liver of dead birds by LC and GC Orbitrap Mass Spectrometry. Results of method validations, Correlation coefficients of the matrix matched calibration curves were >0.978, and the method detection limits (MDLs) and limits of quantitation (LOQ) were 2.8~72.1 ng/g (18.4 ng/g on average) and 9.0~230 ng/g (58.5 ng/g on average). The accuracy ranged from 69.1% to 130%

(103% on average), and the precision values were less than 14.8%(3.8% on average). The screening of residual pesticides in liver of dead birds by LC and GC Orbitrap HRMS was detected monocrotophos, carbofuran, carbosulfan, deltametrin, benfuracarb, carbofuran, phosphamidon, prochloraz in investigated samples.

CONCLUSION: This results showed that accurate mass were extraction of residual pesticides in dead birds by Orbitrap HRMS. It suggested that this screening method is applicable to the residual pesticide analysis for the cause of death as a main tool.

Key words: Dead Birds, Orbitrap high resolution mass spectrometry, Residual pesticides, QuEChERS

서 론

가

(Jang *et al.*, 2010).

가

(Park *et al.*, 1998; Kwon *et al.*, 2003)

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397 320 (liquid phase micro-extraction, LPME), (solid phase micro-extraction, SPME), (Stir bar sorptive extraction, SBSE), QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) (Ock, 2009). QuEChERS QuEChERS GC-ECD (electron capture detector), GC-NPD (nitrogen phosphorous detector), GC-FPD (flame photometric detector), LC-FLD (fluorescence detector) LC-UVD (UV photometric detecto) (Park *et al.*, 2014).

가 LC/MS/MS GC/MS/MS 가 가 가 GC/MS full scan (deconvolution) (non-target) (Meng *et al.*, 2010; Shaikh *et al.*, 2014). 가 Orbitrap-HRMS TOF-HRMS (Garcia-Reyes *et al.*, 2007; Alder *et al.*, 2011; Cervera *et al.*, 2012; Huerta *et al.*, 2013; Farre *et al.*, 2014). 4 3~10 ppm (Bletsou *et al.*, 2015) 가

가 (Trace Finder, MassHunter Workstation) (MZ Cloud, Chemspider, PubChem, Metlin)가 가 가 (Zedda and Zwiener, 2012; Jeon *et al.*, 2016). 가 가가 가 QuEChERS

재료 및 방법

폐사 조류 시료

2017 1 5 (*Corvus fugilegus*), (*Hypsipetes amaurotis*), (*Anas platyrhynchos*), (*Columba livia var. domestica*) (*Anser albifrons*) 7

표준물질 및 시약

PAN (Pesticide Action Network) 238 (Table 1) Accustandard (USA), Sigma-Aldrich (USA), Dr. Ehrenstorfer GmbH (Germany) (Table 1). QuEChERS AOAC (Na-acetate 1.5 g, MgSO₄ 6 g) , EMR-Lipid dSPE (Enhanced Matrix Removal-Lipid dispersive Solid-phase Extraction) EMR-Lipid Final Polish (MgSO₄ 1.6 g, NaCl 0.4 g) Agilent (USA) (methanol), (acetonitrile) 3 Honeywell B&J (USA) HPLC , QuEChERS (acetic acid) LC-MS/MS LC-Orbitrap (formic acid) (ammonium formate) Sigma-Aldrich (USA)

QuEChERS 전처리 방법

QuEChERS AOAC 2007.01

Table 1. Classification of residual pesticides for QA/QC

Classification	Residual Pesticides
Amide (18)	alachlor, bromobutide, butachlor, cyflufenamid, dichlofluanid, dimethenamid, diphenamid, fenoxanil, fentrazamide, flubendiamide, fluopicolide, fluopyram, mandipropamid, naprophamid, penthiopyrad, prochloraz, tolyfluanid, zoxamide
Anilide (9)	boscalid, flutolanil, mefenacet, mepronil, ofurace, propanil, pyrimisulfan, thifluzamide, tiadinil
Azine (9)	clofentezine, dimethametryn, metribuzine, prometryn, simazine, simetryn, tebufenozide, terbutryn, terbutylazine
Azole (27)	bitertanol, carbendazim, cyazofamid, cyproconazole, difenoconazole, diniconazole, etoxazole, fenbuconazole, fipronil, flusilazole, hexaconazole, imazalil, isopyrazam, metconazole, myclobutanil, paclobutrazol, penconazole, probenazole, propiconazole (2 isomers), tebuconazole, tebufenpyrad, tetraconazole, thiabendazole, triadimefon, tricyclazole, triflumizole, uniconazole
Benzoate (2)	pyriminobac-methyl (E), pyriminobac-methyl (Z)
Carbamate (22)	aldicarb, bendiocarb, benthialdicarb-isopropyl, carbaryl, carbofuran, chlorpropham, esprocarb, ethiofencarb, fenobucarb, fenothiocarb, furathiocarb, iprovalicarb, isoprocarb, methiocarb, methomyl, metolcarb, molinate, oxamyl, pirimicarb, propoxur, pyributicarb, thiobencarb,
Dicarboximide (2)	fenamidone, iprodione
Dinitroaniline (2)	ethalfluralin, pendimethalin
Diphenyl ether (2)	bifenox, oxyfluorfen
Keto-enol (1)	spiromesifen
Morpholine (1)	dimethomorph
Neonicotinoid (6)	acetamiprid, clothianidin, dinotefuran, imidacloprid, thiacloprid, thiamethoxam
Organochlorine (22)	aldrin, α -BHC, β -BHC, γ -BHC, δ -BHC, chlordane-cis, chlordane-trans, 2,4'-DDD, 4,4'-DDD, 2,4'-DDE, 4,4'-DDE, 2,4'-DDT, 4,4'-DDT, dieldrin, endosulfan sulfate, α -endosulfan, β -endosulfan, endrin, heptachlor, heptachlor epoxide, methoxychlor, methyl-pentachlorophenyl sulfide
Organophosphorus (44)	anilofos, azinphos-methyl, cadusafos, carbophenothion, chlorfenvinphos, chlorpyrifos, chlorpyrifos-methyl, diazinon, dimethoate, dimethylvinphos, edifenphos, EPN, ethion, ethoprophos, etrimfos, fenamiphos, fenitrothion, fenthion, fosthiazate, imicyafos, iprobenfos, isofenphos, malathion, mecarbam, methidathion, mevinphos, monocrotophos, parathion, parathion-methyl, phenthoate, phorate, phosalone, piperophos, pirimiphos-ethyl, pirimiphos-methyl, profenofos, prothiofos, pyraclofos, pyrazophos, pyridaphenthione, tebupirimfos, terbufos, tolclofos-methyl, tralomethrin
Phenoxy (4)	cyhalofop-butyl, diclofop methyl, fenoxaprop-p-ethyl, metamifop
phthalimide (1)	folpet
Pyrethroid (12)	acrinathrin, bifenthrin, cyfluthrin (4 isomers), cyhalothrin-L, cypermethrin, deltamethrin, fenpropathrin, fenvalerate (2 isomers), flucythrinate (2 isomers), permethrin (2 isomers), silafluofen, tefluthrin
Pyridine (2)	dithiopyr, thiazopyr
Pyrimidine (6)	cyprodinil, fenarimol, ferimzone, nuarimol, pyrimethanil, pyrimidifen
Pyrrole (2)	chlorfenapyr, fludioxonil
Quinoline (1)	pyriproxyfen
strobil (6)	azoxystrobin, fluacrypyrim, kresoxim-methyl, picoxystrobin, pyraclostrobin, trifloxystrobin,
Substituted Benzene (4)	chlorothalonil, dicloran, pentachloroaniline, quintozen
Urea (9)	chlorfluazuron, diuron, dymron, forchlorfenuron, metazosulfuron, methabenzthiazuron, metabromuron, pencycuron, triflumuron,
Xylyalanine (1)	metalaxyl
Unclassified (23)	bentazone, benzobicyclon, benzoximate, buprofezin, chinomethionat, chlorobenzilate, clomazone, dicofol, dimepiperate, diphenylamine, fenazaquin, fthalide, hexythiazox, indanofan, mepanipyrim, oxadiazon, oxaziclomefon, pyribenzoxim, pyridaben, pyroquilon, quinoclamine, sulfoxaflor, tetradifon

(Association of Analytical Communities Official Method	1%	가	15 mL	50 mL
2007.01, Lehotay <i>et al.</i> , 2007)	,	1	,	Na-acetate 1.5 g
	EMR	(Han <i>et al.</i> ,	MgSO ₄ 6 g	가 250 rpm
2016)	.	5 g	10	5,000 rpm 10

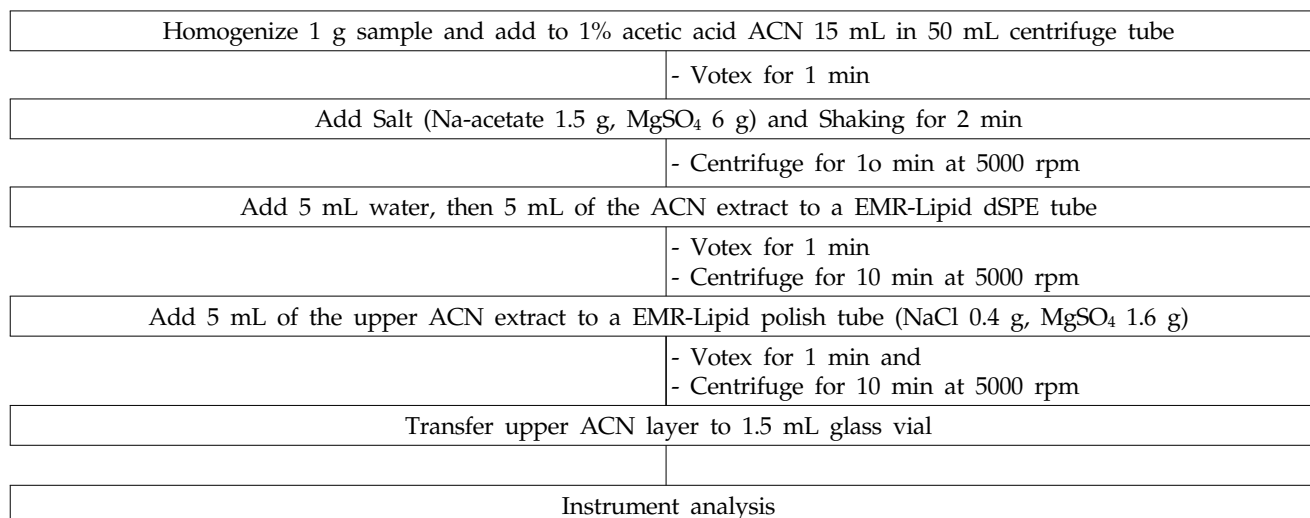


Fig. 1. Flow chart for residual pesticides in liver of dead birds.

Table 2. Analytical condition of LC/MS/MS for residual pesticides

LC parameters				
Instrument	Shimadzu 8030 with Nexera UHPLC			
Column	Phenomenex Kinetix, C18, 100 × 2.1 mm, 2.6 μm			
Run Time	20 min			
Injection volume	5 μL			
Oven temp.	40°C			
Mobile phase	A: 0.1% formic acid, 5 mM ammonium formate in water B: 0.1% formic acid, 5 mM ammonium formate in methanol			
Gradient condition	Time (min)	A (%)	B (%)	Flow (mL/min)
	0	95	5	0.2
	2	95	5	0.2
	7	5	95	0.2
	11.5	5	95	0.2
	12.5	95	5	0.2
15	95	5	0.2	
MS/MS parameters				
Ionization mode	Electrospray ionization (ESI)			
Nebulizing gas flow	3 L/min			
DL temp.	250°C			
Heat block temp.	400°C			
Drying gas flow	15 L/min			

1 g EMR-Lipid dSPE
5 mL 가 vortex 2 g EMR dSPE-
lipid kit (NaCl:MgSO₄=2:8) 1 vortex ,
5 mL EMR-lipid polish
kit 가 vortex

(Fig. 1).

전처리 방법의 유효성 검증

238
(method detection limit, MDL),
(limit of quantification, LOQ),
LC/MS/MS GC/MS/MS (Table 2, 3)
가 . 가 0.1~100 ng/mL
(matrix matched standard method)

Table 3. Analytical condition of GC/MS/MS for residual pesticides

GC parameters	
Instrument	TSQ8300 EVO with Trace 1310
Carrier gas flow	He, 1.0 mL/min
Column	DB-5MS (30 m×250 μm×0.25 μm)
Injection temp.	280°C
Injection mode	Splitless
Injection volume	1 μL
Oven temp.	50°C (2 min)→ 15°C/min → 140°C (15 min) → 5°C/min → 300°C (10 min)
MS/MS parameters	
Ionization mode	Electron ionization (EI)
Multiplier voltage	1,000 V
Source temperature	280°C
Scan time	1 sec
Interface temp.	280°C
Q2 collision gas	Argon

500 ng/mL	150 μL	가	×0.25 μm, J&W Scientific, USA)	GC
			(injection mode)	(splitless)
			가 He,	1.0 mL/min
7	3.14	10	1 μL	
	2000 ng/mL	150 μ	(transfer line)	280°C
L	가 5		60°C	10
	5		300°C	5°C
100		5		4
	100		GC-Orbitrap	(ionization
			mode) EI (Electron ionization)	
			280°C	(mass resolution) 60,000
고분해능 질량분석기 기기조건			45~650 m/z	full scan
		LC-Orbitrap		
GC-Orbitrap		LC-Orbitrap		
LC	Dionex Ultimate 3000-Q	Q		
Exactive Plus	Thermo Scientific (USA)			
	Phenomenex			
Kinetex C18	(100×2.1 mm, 2.6 μm)		accurate mass	LC-Orbitrap
	40°C	0.1%	TraceFinder 3.3	Thermo Scientific (USA)
5 mM	가		NiST	MZ Cloud
200 μL/min				MS
	2	0%		ddMS2
100%	30	35		GC-Orbitrap NIST
	40		gc-orbitrap contaminants library	
			TraceFinder 4.1	
ionization)		LC-Orbitrap		
	(ionization mode)	ESI (electospray		
		300°C		
	(mass resolution) 70,000	50~1000		
m/z	full scan	ddMS2		
(data dependent MS/MS)				
GC-Orbitrap	GC	TRACE 1300		Q
Exactive GC	Thermo Scientific (USA)			
		DB-5MS (30 m×250 μm		
				QuEChERS

결과 해석

LC-Orbitrap

Thermo Scientific (USA)

MZ Cloud
MS

ddMS2

GC-Orbitrap NIST

gc-orbitrap contaminants library

TraceFinder 4.1

결과 및 고찰**전처리 방법 유효성 검증**

QuEChERS

Table 4. Method detection limit (MDL), limit of quantitation (LOQ), accuracy and precision for residual pesticides by AOAC QuChERS method for liver of dead birds

Classification	MDL (ng/g)	LOQ (ng/g)	Accuracy (%)	Precision (%)
Amide (18)	19.4 (8.57~33.6)	62.1 (27.3~107)	106 (69.1~128)	3.9 (0.2~10.2)
Anilide (9)	14.8 (6.00~23.2)	47.1 (19.1~73.9)	106 (7700~122)	3.6 (1.1~7.9)
Azine (9)	11.1 (2.83~18.5)	35.2 (9.00~58.9)	114 (106~122)	3.7 (1.9~5.3)
Azole (27)	19.3 (7.16~55.3)	61.5 (22.8~176)	98.4 (77.4~122)	3.4 (0.6~14.2)
Benzoate (2)	19.4 (17.9~20.9)	61.8 (57.0~66.6)	92.9 (90.0~95.9)	6.4 (4.3~8.4)
Carbamate (22)	12.5 (4.55~40.3)	39.7 (14.5~129)	112 (91.1~125)	3.1 (0.3~6.8)
Dicarboximide (2)	14.8 (8.20~21.4)	47.1 (26.1~68.1)	118 (108~127)	4.9 (1.0~8.8)
Dinitroaniline (2)	12.9 (2.86~23.0)	41.2 (9.10~73.2)	97.6 (97.4~97.8)	2.7 (0.8~4.5)
Diphenyl ether (2)	52.0 (32.0~72.1)	165.8 (102~230)	90.4 (85.2~95.5)	10.6 (9.2~12.0)
Keto-enol (1)	12.1	38.5	114	2.2
Morpholine (1)	6.19	19.7	96.3	3.5
Neonicotinoid (6)	21.5 (15.4~27.2)	68.4 (49.0~86.6)	106 (102~118)	4.9 (1.8~14.8)
Organochlorine (22)	24.5 (7.38~71.0)	77.9 (23.5~226)	96.1 (81.0~112)	3.7 (0.4~14.8)
Organophosphorus (44)	18.4 (7.13~40.4)	58.5 (22.7~129)	105 (82.1~125)	3.7 (0.1~9.2)
Phenoxy (4)	8.99 (5.28~16.8)	28.6 (16.8~53.5)	101 (86.5~114)	2.8 (0.8~5.3)
phthalimide (1)	33.0	105.1	110	5.0
Pyrethroid (12)	21.2 (5.02~37.8)	67.6 (16.0~120)	88.7 (74.4~105)	4.4 (1.3~9.4)
Pyridine (2)	23.3 (21.9~24.7)	74.3 (69.7~77.4)	104 (103~106)	2.0 (1.0~3.0)
Pyrimidine (6)	15.7 (10.1~24.3)	50.0 (32.3~77.4)	97.8 (83.5~114)	4.2 (1.2~6.8)
Pyrrole (2)	38.5 (26.0~51.0)	122.6 (82.9~162)	93.6 (91.2~96.0)	3.3 (1.6~5.1)
Quinoline (1)	7.38	23.5	116	2.0
Strobin (6)	17.3 (12.4~25.9)	55.0 (39.6~82.5)	113 (107~120)	3.1 (0.9~5.0)
Substituted Benzene(4)	25.9 (17.6~30.3)	82.6 (56.1~96.5)	88.5 (78.6~105)	4.8 (1.2~7.0)
Urea (9)	12.8 (6.59~34.5)	40.8 (21.0~110)	113.3 (101~130)	4.7 (1.6~8.7)
Xylyalanine (1)	9.23	29.4	108	3.1
Unclassified (23)	17.3 (2.92~49.6)	54.9 (9.3~158)	104 (75.5~126.3)	3.3 (0.5~8.9)
Total	18.4 (2.8~72.1)	58.5 (9.0~230)	103 (69.1~130)	3.8 (0.1~14.8)

(238) LC/MS/MS (139) (2017) (: 70~130%, : 30%
 GC/MS/MS (99) MRM) zoxamide (69.1%)
 1 2 . , EU 가 (SANCO/
 (Fig. 1). 825/00 rev.8.1, 2010) (: 70~120%,
 , 0.1~100 g/mL : 20%) Cadusafos 17
 USDA method validation
 (r^2) 0.978~0.999 \pm guideline (2000) (50%~150%,
 15% . Table 4 20%)
 QuEChERS AOAC
 , EMR
 , 2.8~72.1 ng/g “
 9.0~230 ng/g (18.4 ng/g 58.5 ng/g) (I)” (2015) Han (2016)
 가 26
 . 69.1~130% 0.1~14.8%
 103% 3.8% . Log Kow가 1
 가 “ 5 ” PPCPs ,

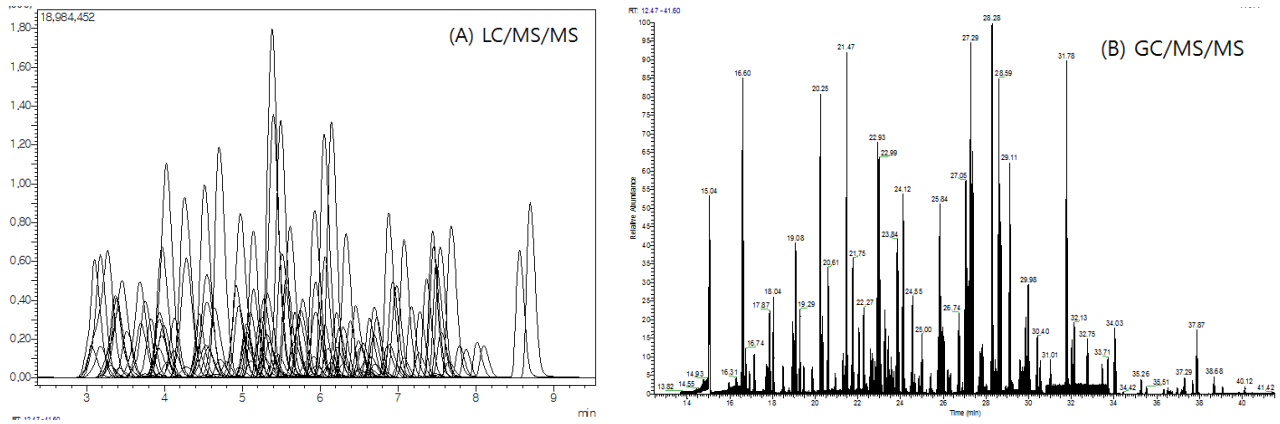


Fig. 2. Chromatograms for residual pesticides by (A) LC/ MS/MS and (B) GC/MS/MS

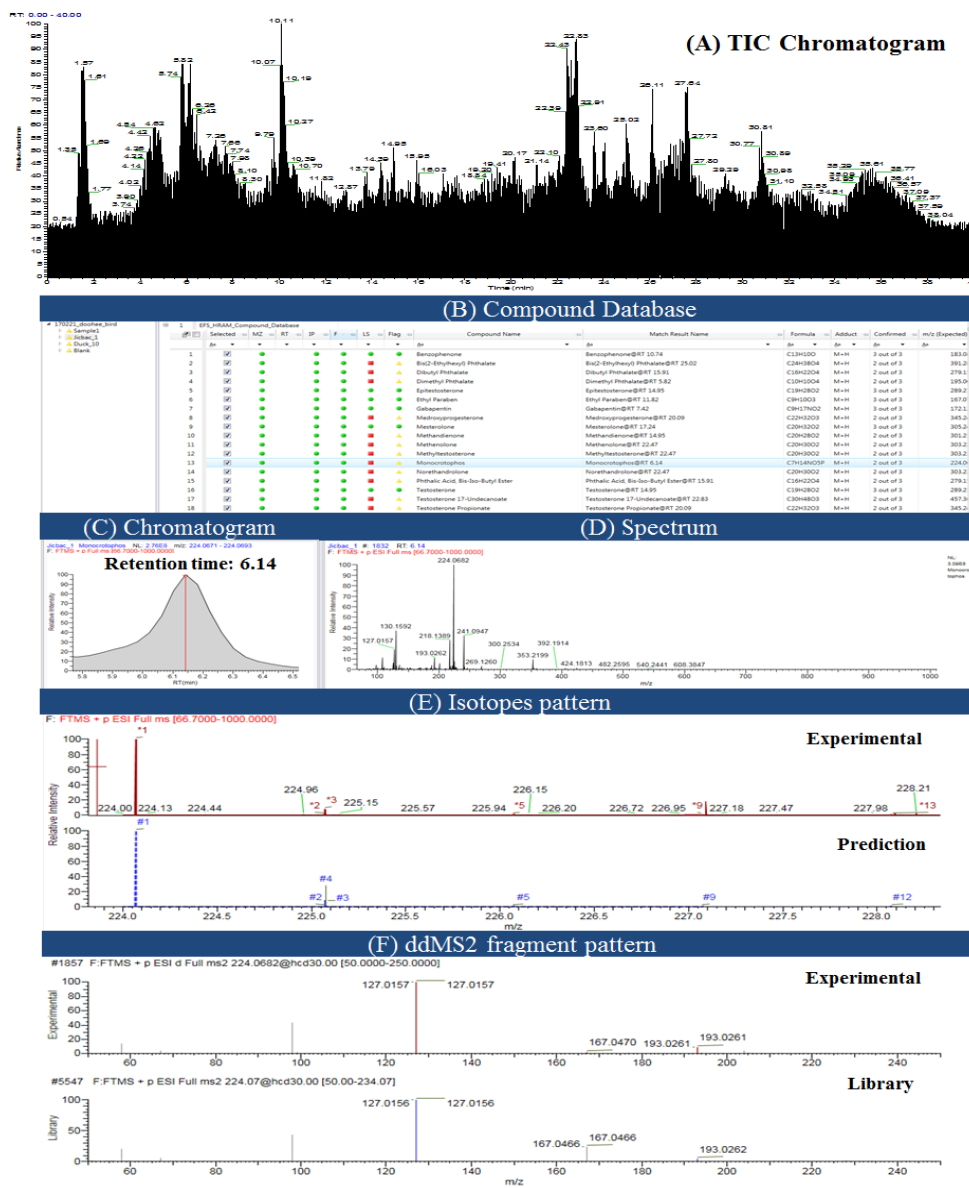


Fig. 3. (A) Total ion chromatogram of residual pesticides in liver of dead birds and Tracefinder3.3 browser by LC-Orbitrap. (B) Candidate list with a library matching compound, (C) its chromatogram, (D) mass spectrum, (D) isotopes pattern of the compared with experimental and prediction, (E) datadependent MS/MS of the compared with experimental and library.

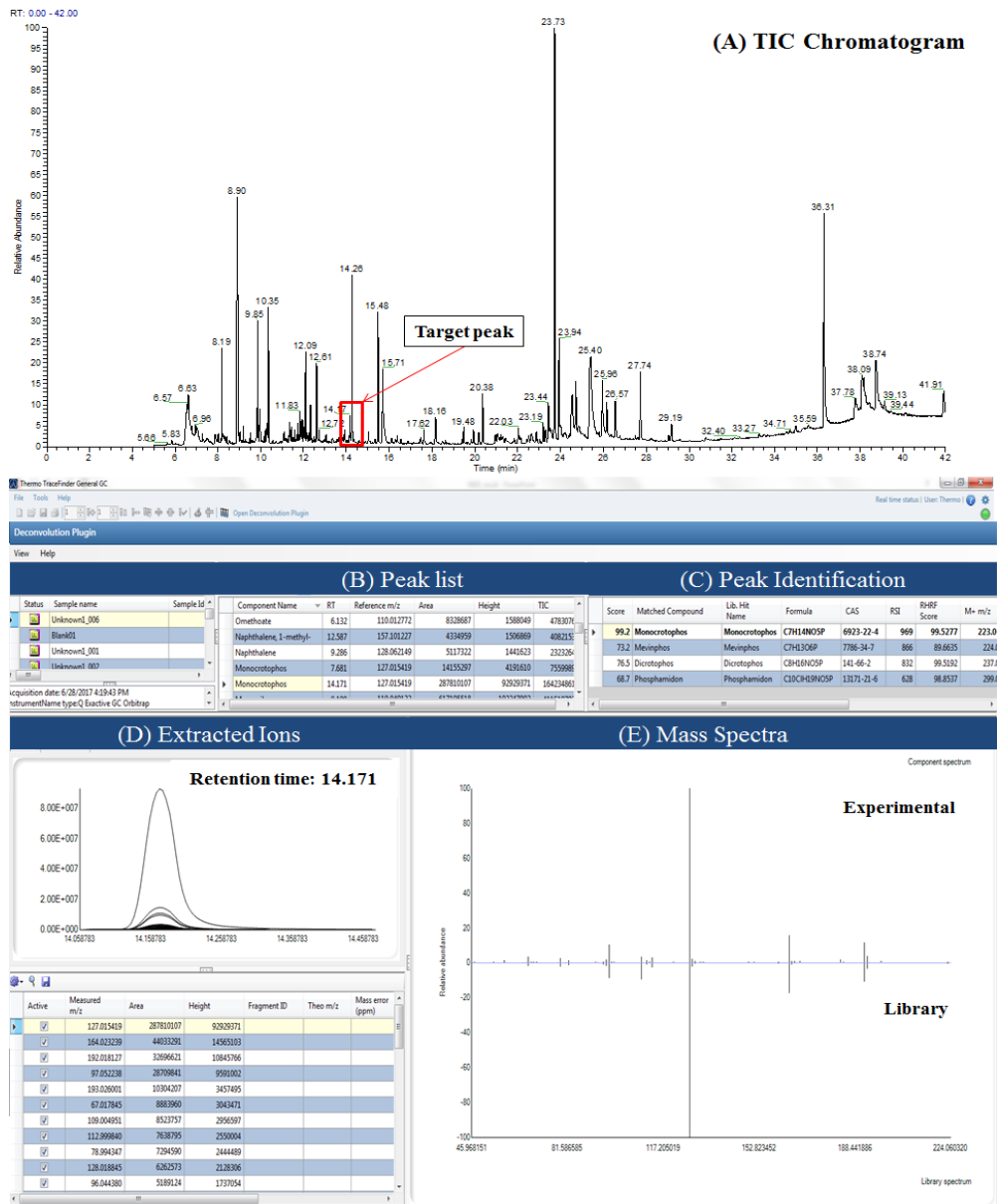


Fig. 4. (A) Total ion chromatogram of residual pesticides in liver of dead birds and Tracefinder4.1 browser by GC-Orbitrap. (B) Candidate list with a library matching compound, (C) peak identification, (D) its chromatogram and extracted ions, (E) mass spectrum of the compared with experimental and library.

Kow가 1 acephate (-0.85) dicrotophos (-0.50) Log

가 가 . LC/MS GC/MS

고분해능 질량분석기 스크리닝 결과

LC GC

(target)/ (suspect)/ (non-target)

(Krauss *et al.*, 2010). m/z

가 m/z 가

가 full 가 (Hernández *et al.*, 2015).

scan m/z 가

Table 5. Detection of residual pesticides in liver of dead birds

	Sample	Residual Pesticides
1	<i>Corvus fugilegus</i>	not detecting
2	<i>Hypsipetes amaurotis1</i>	monocrotophos
3	<i>Anas platyrhynchos</i>	carbofuran, carbosulfan, deltamethrin
4	<i>Columba livia var. domestica</i>	benfuracarb, carbofuran
5	<i>Hypsipetes amaurotis2</i>	carbofuran
6	<i>Hypsipetes amaurotis3</i>	phosphamidon
7	<i>Anser albifrons</i>	monocrotophos, prochloraz, fenitrothion

Orbitrap	Fig. 3-A	LC-Orbitrap	(Fig. 4-D)	(Fig. 4-E)
full scan	ddMS2	가	monocrotophos	
m/z	4		1 monocrotophos, carbofuran, carbosulfan, deltamethrin, benfuracarb,	2 carbofuran, 3 monocrotophos, prochloraz, fenitrothion
accurate mass			Jang (2010) 1998	2009
TraceFinder3.3	Fig. 3-B~F	Fig. 3-B	MZ Cloud	monocrotophos, phosphamidon, carbofuran, fenitrothion
, NIST		ddMS2		
epitestosterone, testosterone			LC-Orbitrap	GC-Orbitrap
		6.14	가	가
monocrotophos		monocrotophos		가
MS	ddMS2	NIST	가	
		(Fig. 3-E), MZ Cloud		
		(Fig. 3-F)		
		ddMS2		
127.0157 m/z	127.0156 m/z	(exact mass - accurate mass)가		
	-0.0001	0.79 ppm		
	Fig. 4	GC-Orbitrap		
		LC-Orbitrap		
m/z				
가				
700		TraceFinder 4.1		
contaminants library		gc-orbitrap		
Fig. 4-B				
monocrotophos			(Fig. 4-C)	
		score		

Notes

The author declare no conflict of interest.

Acknowledgement

This work was supported by the National Institute of Environmental Research R&D (1900-1946-303-210-13) of Korea Government (NIER).

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