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Determination of Organochlorine and Nitrogen-Containing Pesticide Residues in Fish with Different Fat Content

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ABSTRACT

A rapid and multiresidue method for determination the residues of organochlorine and nitrogen-containing pesticides in the different fat content fish by solid-phase extraction (SPE) technique was developed. In the present procedures, samples were extracted with acetonitrile. The extracts were cleaned up by a tandem SPE column of C_{18} and florisil, using acetonitrile as the only solvent. Residues were determined by gas chromatography equipped with electron capture detector. Recovery was obtained by three concentration levels of 12 organochlorine pesticides and their metabolites (chlorobenzilate, dieldrin, endosulfan sulfate, endosulfan, endrin, heptachlor, heptachlor epoxide, lindane, methoxchlor, *p,p'*-DDE, *p,p'*-DDT) and two nitrogen-containing pesticides (alachlor and trifluralin) from 9 fishes with 1.2 - 23.3% of fat content, respectively. The recoveries of the spiked pesticides residues from the fishes of different fat contain (fat < 10%, fat ≥ 10 but < 20%, and fat ≥ 20%) were in the range from 73.4% to 119.6%. There were no significant difference in the recovery between pesticides concentration and fat content. The recoveries of two samples were not included because of contaminated by *p,p'*-DDT and endosulfan separately, or disruption by sample matrix.

Key words: **pesticide, residues, fish.**

INTRODUCTION

Since organochlorine (OC) pesticides, such as DDT, are known to spread far and wide because of their persistency to decompose and their lipophilic property, the global monitoring of these pesticides has become one of the world's most important priorities[1-4]. In the Republic of China, OC pesticides have been decreasing and replaced by organophosphates, carbamates, and synthesized pyrethroids. However, due to the persistency of OC, the opening of the market to imports, and consumer awareness to environmental pollutants, the development of a multi-residue analysis technique to monitor OC residues in fish in order to ensure food safety as well as identify the source of contamination, is therefore necessary[5]. Gas Chromatography (GC), equipped with an electron capture detector (ECD), is routinely used to analyze OC pesticides in fish. The isolation of compounds of interest from fish may bring out some fish lipids that could be harmful to the analytical

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column as well as contaminate the detector. The cleanup procedure prior to analysis is therefore important. The methods for sample cleanup are well documented. According to the AOAC method (1995)[6], the OC in high-fat samples is extracted and partitioned with acetonitrile and petroleum ether followed by a florisil column to remove residual oil. This method, however, is time-consuming. Armishaw & Millar (1993) have compared three cleanup methods including gel permeation chromatography (GPC), sweep codistillation, and florisil column adsorption chromatography, and have found all the methods capable of yielding satisfactory recovery. However, when using sweep the codistillation method, the temperature should be well controlled to prevent the analytes from decomposition resulting in a low recovery. The cleanup by GPC could yield much more interference on the GC chromatogram. The florisil column absorption chromatography is considered to be the fastest and most reliable method for sample cleanup among the above three methods, although it requires more solvents than the sweep codistillation method and is less automatic than the GPC[7]. Solid phase extraction (SPE) has been suggested as a reliable extraction method to analyze pesticides and environmental pollutants in aquatic[8-10]. Schenck (1996) and Schenck *et al*. (1996) have employed a tandem SPE column for sample cleanup to analyze the OC residues in fish. Compared to the AOAC method (1995), the tandem SPE method gives a better cleanup efficiency as well as saves time and solvent. However, the suitability of applying this method to fish is fat content dependent[11, 12]. The lipid content could be different between species. Even in the same species, the fat content could vary due to seasonal or physiological change[13]. To develop a multiresidue analysis method to simultaneously detect multiple pesticides in fish with different fat content is, therefore, necessary.

MATERIALS AND METHODS

I. *Materials*

(I) *Organochlorine and Nitrogen-Containing Pesticide Standards*

1. Pesticide standards. Eleven pesticide standards were used in this study: alachlor (of purity 98.8%, Dr. Ethernstorfer); aldrin (of purity 98.7%, Dr. Ethernstorfer GmbH); chlorobenzilate (of purity 97%, Wako); dieldrin (of purity 98.4%, Dr. Ethernstorfer GmbH); endosulfan (of purity 99%, RDH); endrin (of purity 99.9%, Dr. Ethernstorfer GmbH); heptachlor (of purity 99.1%); lindane (gamm-HCH)(of purity 99.7%, Dr. Ethernstorfer GmbH); methoxychlor (of purity 99%, RDH); *p,p'*-DDT (of purity 99.7%, Dr. Ethernstorfer GmbH); trifluralin (of purity 99.5%, Lilly).

2. Metabolite standards including endosulfan sulfate (of purity 98%, RDH); heptachlor epoxide (of purity 99%, RDH) and *p,p'*-DDE (of purity 99.4%, Dr. Ethernstorfer GmbH) were used.

(II) *Equipment and Materials*

1. Equipment. Centrifuge (Backman, model J 2-MC with JA-14 toto); homogenizer (Kinematica, polytron®); SPE device (J&W Scientific, SPE vacuum manifold and accessories); rotary evaporator (Heidolph model VV2011); gas chromatography instrument (Varian 3400 equipped with ECD and a DB-608 capillary column 0.53 mm i.d. x 30 m).

2. Materials. SPE cartridges with 3 specifications: Bakerbond spe™ reservoirs (75 mL), octadecyl (C_{18}) cartridge (6 mL, 1000mg), and Florisil cartridge (6 mL, 1000mg).

3. Reagents. Acetone (Merck, extra pure), acetonitrile (Merck, isocratic grade for chromatography), sodium sulfate (Merck, anhydrous extra pure), ethyl ether (Merck, GR), isopropyl alcohol (Merck, GR), methanol (Merck, GR), n-hexane (Merck, for organic trace analysis), petroleum ether (RDH, extra pure), toluene (Merck, GR), and double distilled water.

(III) *Fish Samples*

Nine species were collected: Pacific saury (*Cololabis saira*), Common carp (*Cyprinus carpio*), Sea Perch (*Lateolabrax japonicus*), Daggertooth pike eel (*Muraenesox cinereus*), Golden

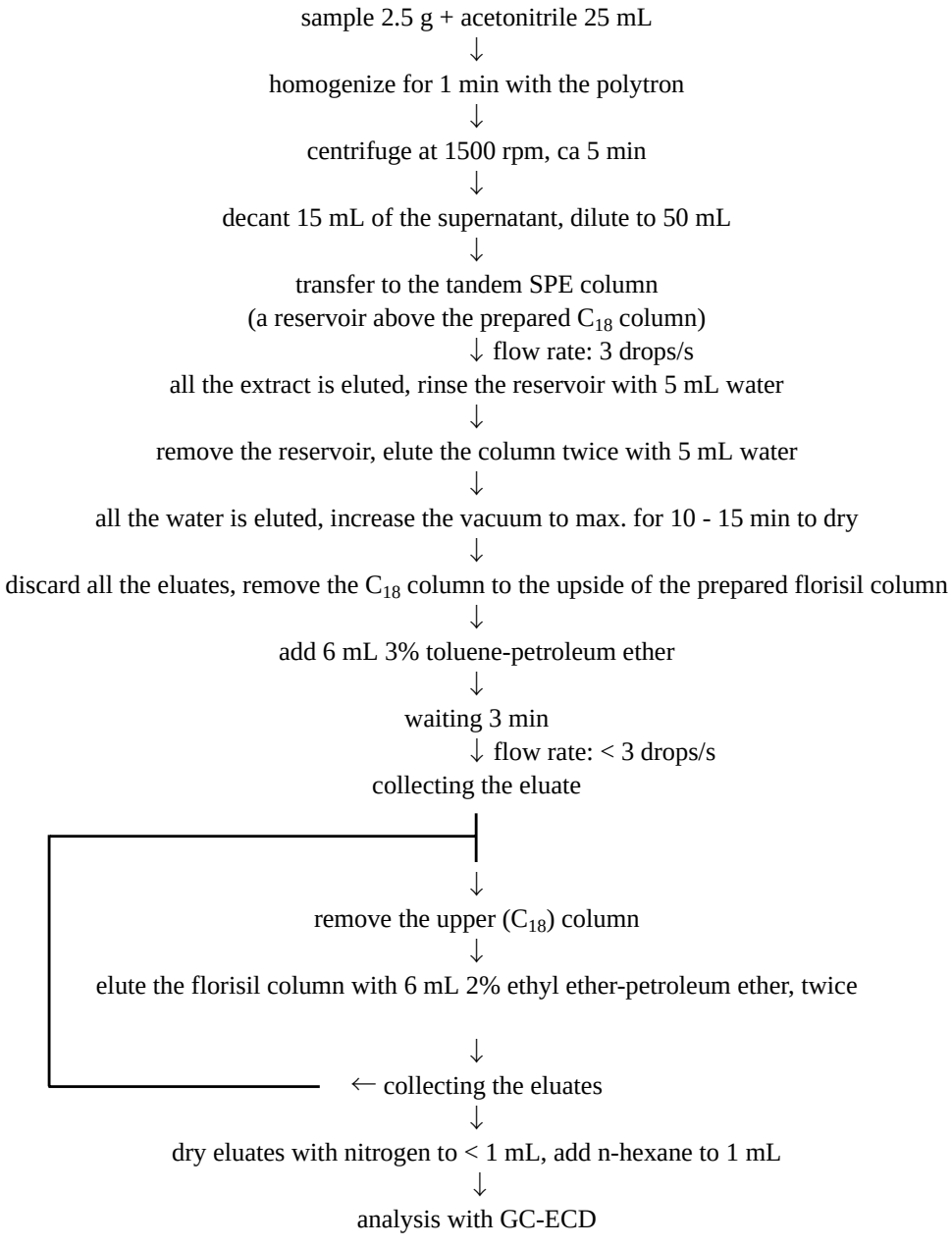

Figure 1. Analytical procedure for determining pesticides residues in fish tissue according to Schenck, *et al.*, (1996) method.

threadfin bream (*Nemipterus virgatus*), Atlantic salmon (*Salmo salar*), pollack (*Theragra sp.*), Tilapia (*Tilapia nilotica*), and Large head hairtail (*Trichiurus lepturus*). Most of the fish were purchased from supermarkets in the Taichung area, except for the common carp, which was purchased from a fish culture center located at Wu-Shan-Tao when it was 3-months old and then cultured for more than one year.

II. *Methods*

(I) *Instrument Conditions*

The GC oven temperature was initiated at 140˚C for 2 min, raised to 200˚C at 4˚C/min, kept at 200˚C for 5 min, raised to 230˚C at 5˚C/min, kept at 230˚C for 5 min, raised to 260˚C at 2˚C/min, and then kept at 260˚C for 6 min. The temperatures of the injector and detector were 250 ˚C

and 320˚C, respectively. The injection volume was 1 μL. The flow rates of carrier gas (N_2) and makeup gas (N_2) were kept at 1.9 and 30 mL/min, respectively.

(II) *The Procedure for the Extraction and Cleanup of Organochlorine and Nitrogen-Containing Pesticides from Fish*

The head, tail, and scales of the fish were removed, and the fish body was then chopped and transferred into a bottle.

1. Method 1. Method for the analysis of 9 chlorinated pesticides in beef fat developed by Long (1991)[15] was followed.

2. Method 2. Method for the simultaneous analysis of 21 organophosphate pesticides in an aquatic environment reported by Sun *et al*. (1997) [16] was followed.

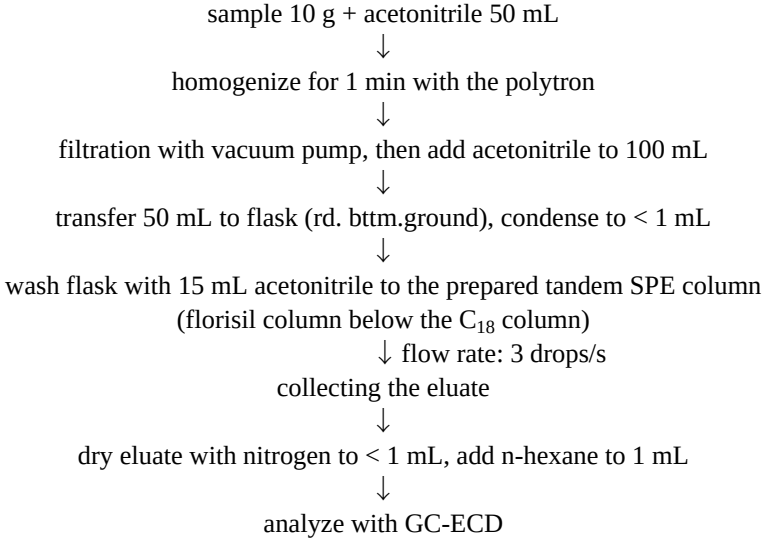

Figure 2. Analytical procedure for determining pesticides residues in fish tissue modified from Schenck, *et al*., (1996) method.

Table 1. Comparison of different pre-treatment method on the recoveries of organochlorine pesticides in fish tissue

Pesticides	Recovery (%)[a]		
	Spiked level(μg/g)	Method 2[b]	Method 3[c]
Alachor	0.5	68.1 (12.1)	0.0
Aldrin	0.1	41.7 (11.5)	63.6 (36.3)
Chlorobenzilate	0.8	81.2 (7.4)	0.0
Dieldrin	0.03	81.5 (5.3)	93.6 (3.4)
α-Endosulfan	0.11	56.0 (14.0)	75.6 (13.7)
β-Endosulfan	0	68.3 (6.9)	0.0
Endosulfan sulfate	0.11	70.2 (7.8)	84.0 (9.8)
Endrin	0.1	81.3 (4.5)	62.7 (13.3)
Heptachlor	0.1	56.9 (5.3)	54.3 (50.9)
Heptachlor epoxide	0.1	62.6 (5.8)	77.9 (19.1)
p,p'-DDE	0.1	43.6 (7.8)	65.2 (20.7)
p,p'-DDT	0.1	68.2 (11.2)	63.2 (12.1)

[a] n=3; number in parenthesis is the coefficient of variation (CV, %).
[b] Sun, *et al*. (1998) method. [c] Schenck, *et al*. (1996) method.

3. Method 3. The method based on a report of Schenck *et al.* (1996)[11] is schematically listed in Figure 1. SPE cartridges were conditioned with the solvents as follows. The reservoir was rinsed with acetone. Then the C_{18} cartridge was stepwise washed with 6 mL of petroleum ether, 6 mL of acetone, 12 of mL methanol, and 12 mL of distilled water (retain about 1-2 mL of water in car-

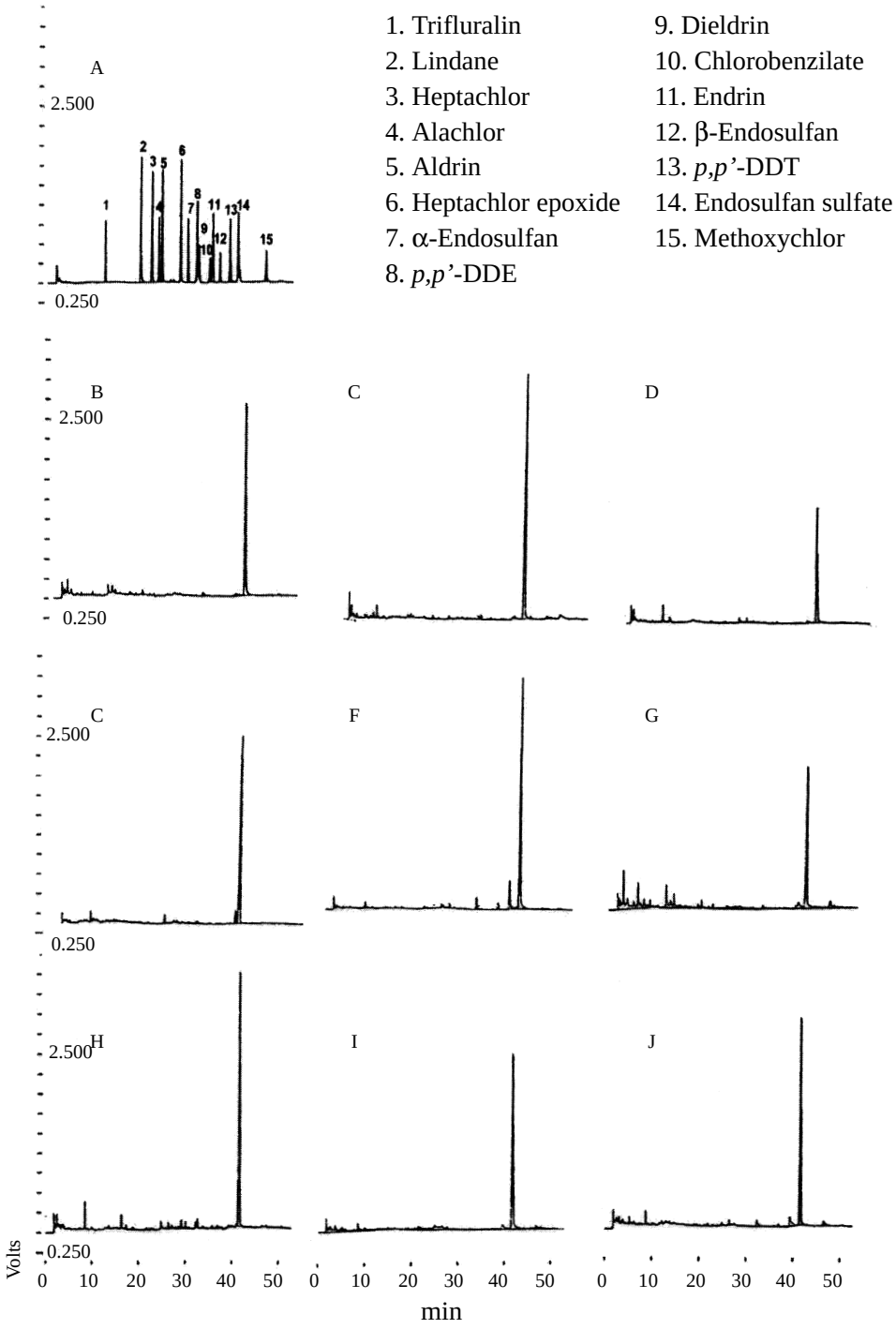

Figure 3. Chromatograms of 14 organochlorine and nitrogen-containing pesticide for pesticides standard and its metabolite (A); and chromatograms of sample of *Cololabis saira* (B), *Cyprinus carpio* (C), *Lateolabrax japonicus* (D), *Muraenesox cinereus* (E), *Nemipterus virgatus* (F), *Salmo salar* (G), *Theragra sp.* (H), *Tilapia nilotica* (I), *Trichiurus lepturus* (J).

Table 2. Recovery of spiking different organochlorine pesticides to common carp (*Cyprinus carpio*) tissue by cleanup with solid-phase extraction method

Pesticides	Spiked level	Recovery[a] (%)			Detection
	(μg/g)	Spiked level x 1	Spiked level x 2	Spiked level x 4	limit (ppb)
Alachlor	0.125	88.9 (9.4)	97.5 (2.0)	105.4 (10.6)	225
Aldrin	0.025	116.0 (1.4)	94.8 (2.7)	97.8 (12.4)	28
Chlorobenzilate	0.2	97.9 (3.1)	93.1 (1.3)	92.0 (3.7)	3510
Dieldrin	0.0075	95.4 (1.6)	89.7 (1.7)	98.0 (2.1)	3
α-Endosulfan	0.025	94.3 (3.0)	103.8 (3.7)	94.4 (4.1)	44
β-Endosulfan	0	98.9 (1.6)	91.4 (2.2)	93.8 (5.8)	24
Endosulfan sulfate	0.025	73.7 (2.4)	90.2 (1.3)	84.5 (7.7)	–
Endrin	0.025	86.6 (1.1)	90.6 (1.6)	92.3 (7.0)	35
Heptachlor	0.025	91.5 (1.8)	93.8 (2.2)	102.6 (11.5)	25
Heptachlor epoxide	0.025	98.8 (1.6)	99.0 (2.3)	98.8 (6.4)	16
Lindane	0.025	87.2 (1.7)	97.0 (2.1)	87.8 (1.6)	20
Methoxychlor	0.025	74.2 (5.0)	73.4 (1.9)	105.2 (2.8)	99
p,p'-DDE	0.025	90.5 (1.7)	91.3 (2.1)	99.7 (3.1)	34
p,p'-DDT	0.025	119.5 (1.5)	93.8 (1.3)	119.6 (8.6)	30
Trifluralin	0.025	112.6 (2.5)	117.4 (1.0)	84.5 (0.7)	120

[a] n=3; number in parenthesis is the coefficient of variation (CV, %).

Table 3. Fat content of fish muscle

Fish species	Fat (%)[a]
Pacific saury (*Cololabis saira*)	23.3 ± (1.6)
Common carp (*Cyprinus carpio*)	11.7 ± (1.0)
Sea Perch (*Lateolabrax japonicus*)	2.8 ± (0.8)
Daggertooth pike eel (*Muraenesox cinereus*)	1.2 ± (0.2)
Golden threadfin bream (*Nemipterus virgatus*)	2.1 ± (0.3)
Atlantic salmon (*Salmo salar*)	21.9 ± (0.9)
Pollack (*Theragra sp.*)	19.4 ± (1.3)
Tilapia (*Tilapia nilotica*)	5.4 ± (1.3)
Largehead hairtail (*Trichiurus lepturus*)	3.4 ± (0.6)

[a] n=3; number in parenthesis is the standard deviation.

tridge). A florisil cartridge was packed with sodium sulfate (2-cm), and then eluted with 5 mL of petroleum ether. The tandem SPE was lined up with a reservoir, florisil cartridge, and C_{18} cartridge.

4. Method 4. Modified from method 3. This method only requires 2 cartridges: C_{18} and florisil. The procedure is listed in Figure 2. A florisil cartridge was packed tightly with 2 g sodium sulfate and then inserted with glass cotton. These two cartridges were connected in an order of C_{18} and florisil. The C_{18} cartridge was rinsed with 6 mL of acetonitrile, which was then retained in the florisil cartridge waiting for use.

(III) *Analysis of the Total Fat Content in Fish*

The method developed by Undeland *et al*. (1998)[14] was followed. Fish meat (10 g) was chopped and then mixed with 16 mL of isopropyl alcohol. The mixture was ground for 30 sec while kept in an ice bath. After adding 32 mL of n-hexane, the mixture was ground for another 30 sec and

then centrifuged at 19,600 g (11500 rpm) for 15 min at 4˚C. The n-hexane layer was collected and dried over nitrogen gas in a 28˚C water bath. The fat content of the fish sample was thus calculated on the basis of the mass of dried residue.

RESULTS AND DISCUSSION

The GC chromatograms of 14 organochlorine and nitrogen-containing pesticide standards and their metabolites are shown in Figure 3 (A). The standard of endosulfan is a mixture of α-endosulfan (peak 7) and β-endosulfan (peak 12); while the endosulfan sulfate (peak 14) is a metabolite of endosulfan. Heptachlor epoxide (peak 6) and *p',p'*-DDE are the metabolites of heptachlor and *p',p'*-DDT.

The common carp was used to test for the effects of different extraction and cleanup methods on the recovery of pesticides. Our preliminary study showed that Method 1 was not capable of effectively removing the fish lipid. This could minimize the applicability for the pesticide analysis in animal tissues with different fat content. Method 2 could give a better lipid-removing efficiency and has been reported to be a method of cleanup for simultaneous analysis of 21 organophosphate pesticides[16]. However, a satisfactory recovery ranging from 70% to 120% was barely achieved, except for chlorobenzilate, dieldrin, and endrin (Table 1). Both spiked alachor and chlorobenzilate were not detected by using Method 3. In addition, the high coefficient of variation (>20%) on the recoveries of heptachlor, aldrin, and *p',p'*-DDE indicates that Method 3 is low in repeatability. Schenck *et al*.[12] also reported that this method gave a low pesticide recovery (< 70%) when the fish samples with lipid content up to 22.6% were used. By using Method 3, a centrifugation procedure is applied to separate the supernatant which contains the compounds of interest from the sample matrix. This step could

Table 4. Recovery of spiked[a] organochlorine pesticide in fish tissue containing different fat content

Pesticides	Recovery (%)[b]								
	< 10% fat			≧ 10% < 20% fat			≧ 20% fat		
	mean	min	max	mean	min	max	mean	min	max
Alachlor	97.2	86.1	104.3	97.2	88.9	105.4	93.5	90.3	97.5
Aldrin	83.5	78.0	90.4	88.2	73.7	116.0	72.3	70.7	74.6
Chlorobenzilate	95.4	79.1	108.7	90.9	89.5	97.9	77.4	73.5	80.3
Dieldrin	91.4	80.2	99.0	92.2	89.7	98.0	84.6	75.4	92.7
α-Endosulfan	93.9	84.1	98.8	100.3	94.3	113.9	87.2	71.9	106.0
β-Endosulfan	94.9	41.5	119.0	86.0	80.8	98.9	81.3	71.2	93.6
Endosulfan sulfate	116.1	70.6	266.5[c]	79.9	73.7	94.6	85.9	74.7	105.8
Endrin	95.6	82.5	105.2	93.2	86.6	104.0	80.3	77.3	84.0
Heptachlor	100.2	81.6	116.4	104.7	91.5	114.0	83.8	71.3	99.0
Heptachlor epoxide	93.4	85.8	99.0	95.1	90.6	99.0	86.0	71.8	99.8
Lindane	88.2	81.7	92.9	86.5	84.8	97.0	88.1	80.2	96.3
Methoxychlor	93.0	75.4	106.2	82.8	73.4	105.2	78.5	72.2	92.6
p,p'-DDE	98.2	81.8	127.6[d]	95.6	90.5	106.9	75.8	73.4	79.6
p,p'-DDT	92.7	70.4	139.4[d]	98.3	93.8	119.5	73.4	71.5	75.5
Trifluralin	88.2	78.9	100.3	94.0	82.9	117.4	94.0	88.7	106.3

[a] Spike level : see table 2.

[b] The minimum and maximum value of different fish which are mean of three different spike level and each spike level repeat three times.

[c] Recovery of lowest spiked level in daggertooth pike eel.

[d] Recovery of lowest spiked level in largehead hairtail.

easily lead to an error in operation. According to Schenck[11], toluene and 3% toluene-petroleum ether used for the cleanup process in Method 3 were only suitable for the extraction of organochlorines from low-fat fish. Extracting the pesticides from high-fat fish could bring out some fat from test samples that could result in interference in instrumental analysis. According to Sapp[17], spiking 1 or 2% diethyl ether in hexane or petroleum ether as an elution solvent was capable of eluting the organochlorines from the cleanup cartridge. Schenck *et al.*[12] suggested that depending on the fat content of fish, the concentration of diethyl ether in petroleum ether could be adjusted up to 10% allowing the fat to be retained in the florisil cartridge. A method modified from Schenck *et al.* was developed in this study. Two SPE cartridges for sample cleanup were connected, while using the acetonitrile as an only solvent for washing and elution (refer to Method 4). Fourteen pesticide standards with different levels were tested for recovery. The results of this test are shown in Table 2. The recoveries of 14 pesticides at three spiked levels (low, median, and high) were in the range of 73.7%-119.5% (CV% ranged from 1.1% to 9.4%), 73.4%-117.4% (CV% ranged from 1.0% to 3.7%), and 84.5%-119.6% (CV% ranged from 0.7% to 12.4%), respectively. This modified method is superior to the method proposed by Schenck *et al.* (1996) in terms of the analyses of the organochlorine and nitrogen-containing pesticides from common carp. In addition, the developed method is simple in sample preparation, low in coefficient of variation, and uses a minimum amount of solvent.

To determine the applicability of the modified method on fish with different fat content, three levels of organochlorine pesticides were spiked and tested for the recovery. Table 3 lists the fat content in the test fish. Three categories were classified based on fat content. First, low-fat (fat content < 10%) fish included *Muraenesox cinereus, Trichiurus lepturus, and Tilapia nilotica*; second, median-fat fish (fat content ranges between 10% and 20%) included *Cyprinus carpio* and *Theragra chalcogramma*; and third, high-fat fish (fat content > 20%) included *Salmo salar* and *Cololabis saira*. The average recoveries of the pesticides from fish were in the range from 72.3% to 116.1% (Table 4). The maximum recovery from low-fat fish was found to be endosulfan sulfate (266.5%), the spiked level of which was 0.25 μg/g, from daggertooth pike eel (*Muraenesox cinereus*). The GC chromatogram (Figure 1 E) shows one peak appearing at the same retention time as the endosulfan sulfate peak. The exceptionally high recovery of endosulfan sulfate from daggertooth pike eel could result from the matrix effect or residual endosulfan sulfate in fish. The maximum recoveries of *p,p'*-DDE and *p,p'*-DDT from largehead hairtail (*Trichiurus lepturus*), which was classified into a low-fat fish, reached up to 127.6% and 139.4%, respectively. Two peaks corresponding to the retention times of *p,p'*-DDE and *p,p'*-DDT appear in the GC chromatogram of the *Trichiurus lepturus* extract as shown in Figure 3(J). This result suggests this fish sample could be contaminated with *p,p'*-DDT resulting in a high recovery of *p,p'*-DDT and its metabolite *p,p'*-DDE. The matrix effect could not be a cause in this case because of the lower possibility in occurrence of the two corresponding peaks at the same time.

A key point of residual analysis is the extraction technique, which allows the compounds of interest to be extracted from the sample matrix and minimizes the lipid interference in instrumental analysis. Therefore, the manipulation of the partition of fish oil and pesticide in solvent is an important point of concern. In this study, we have demonstrated a method which is not only able to simultaneously analyze 9 organochlorines and 2 nitrogen-containing pesticides in a variety of fish with wide ranges of fat content, but also simplifies the process of analysis. However, the matrix effect could occur when using the developed method. As can be seen from Figure 1(B) to 1(J), an unknown peak appears between the standard peaks of endosulfan sulfate and methoxychlor that could interfere with pesticide analysis. In this study, acetonitrile (boiling point = 82˚C) was applied as an elution solvent to replace acetone (boiling point =

55.5-57.5˚C), routinely used for pesticide extraction. This could reduce the efficiency in the sample concentration, and is a drawback.

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不同油脂魚體中有機氯及含氮農藥殘留偵測

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摘　要

建立一個快速的、利用多種殘留（multiresidue）分析及固相萃取（solid-phsae extraction）技術，偵測不同油脂含量魚體中有機氯及含氮農藥的方法。在此方法中，樣品以乙腈抽出，萃取物利用以C_{18}和矽酸鎂（florisil）管柱串聯的固相萃取管淨化，乙腈為淨化過程中唯一使用的溶劑。殘留藥劑利用氣相層析儀（gas chromatography, GC）附電子捕獲式檢出器（electron capture detector, ECD）偵測。回收率試驗以12種有機氯農藥及其代謝產物（aldrin, chlorobenzilate, dieldrin, endosulfan sulfate, endosulfan, endrin, heptachlor, heptachlor epoxide, lindane, methoxchlor, *p,p'*-DDE, *p,p'*-DDT）和2種含氮農藥（alachlor, trifluralin）共14種藥劑的不同濃度組合（一倍、兩倍及四倍添加量），在9種油脂含量範圍為1.2　3.3%的魚體中分別進行。魚體依油脂含量分為三組（油脂含量＜10%、≧10%但＜20%及≧20%）的試驗結果中，除了可能有一組樣品魚是已受到*p,p'*粄DT污染、有一組樣品魚可能受到endosulfan污染或圖譜受到基質干擾外，其餘樣品魚體內供試藥劑回收率在73.4%至119.6%間，且回收率在不同處理濃度及不同油脂含量魚體間並無顯著差異。

關鍵詞：農藥，殘留量，魚體。