Appendix D

Summary of Analytical Methods for Environmental Surveys

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1. Initial Environmental Survey

Development and study of analytical methods for the target substances in the FY2002 initial environmental survey was conducted in FY2001. For surface water and bottom sediment, screening tests for degradability were conducted prior to the development of analytical methods by the following procedure to identify the suitability of the method.

(1) Degradability screening test (rapid method)

As some of the chemical substances degrade under various environmental conditions, it was necessary to conduct screening tests for degradability under the assumed environmental condition and develop appropriate analytical methods. Screening tests were conducted establishing water and light conditions simultaneously since both conditions are considered very important in environmental degradation (in the light-related degradation test, only one pH condition was tested). For volatile substances, concentrations of the substances in the void space of the vials were properly measured so as not to misinterpret degradation of the substances.

<Preparation>

A volume of 100 ml of distilled water (pH: 5, 7 and 9) was added to 130-mL vials containing a stir bar (for magnetic stirrer) after which the vials were sealed. Next, a standard solution dissolved in hydrophilic solvent such as acetone (% order concentration recommended) was added to these vials using a microsyringe so that the concentration of the solution would be below 100 ppm, followed by 10 minutes of stirring by magnetic stirrer.

<Experiment>

a) Test solutions with the respective pH values were removed from the vials one hour after the preparation and analyzed immediately (Concentration A).
b) The solutions were analyzed after 5 days of storage in a dark place (Concentration B).
c) In order to examine the occurrence of degradation by light, analysis of the test solution with pH 7 (stored for 5 days) was conducted in a sunny room (Concentration C).

The above experiments were conducted at the temperature of 20 ±5°C.

<Result>

Degradability of the test substances was examined by calculating B/A×100, C/A×100 for the respective pH.

The combinations of experiments are shown below.

pH	*Initial concentration (μg/mL)*	*Residual rate after one hour (%)*	*Residual rate after 5 days*	
			Dark place (%)	Light emission (%)
5	✓	✓	✓	–
7	✓	✓	✓	✓
9	✓	✓	✓	–

Furthermore, in the course of development of analytical methods for surface water and bottom sediment, recovery experiments were conducted to determine the detection limit and recovery rate.

(2) Additional recovery experiment at low concentration

<Distilled water>

Calibration curves were obtained setting the sensitivity of the analytical instrument as high as practically allowable.

Samples were prepared by dissolving standard samples of target substances corresponding to three different concentrations including the lowest concentration in the range of a positive linear regression relationship. And, total analysis was conducted four times for each concentration and the measured values were obtained. Based upon the results, the power of test D was calculated by the following equation after obtaining the standard deviation of the measured values at each concentration.

$$D = t(n-1, 0.05) \bullet \frac{\sigma_R}{n} \bullet \frac{dC}{dR}$$

σ_R: standard deviation C: concentration R: measured value (response value)

The power of test D for the established analytical method was obtained by calculating the average value of the power of test D for three different concentrations. The detection limit was defined as three times ($3\times D$) and the quantitation limit was defined as ten times ($10\times D$) the power of the test.

<Bottom sediment>

Assuming a concentration in bottom sediment corresponding to the detection limit ($3\times D$) obtained in the above-mentioned method as the estimated detection limit, a standard sample of the target substance was added to the common bottom sediment so that the concentration would be 2-5 times the estimated detection limit, and the hermetically sealed sample was stored overnight at 4°C. Next, all procedures for the analysis of the bottom sediment sample were conducted and it was confirmed that the target chemical substance would be properly detected. When the substance was detected, 5 additional recovery experiments were conducted at the same concentration and the detection limit of the common bottom sediment was calculated by the following equation based on the total 7 measured values.

Detection limit (DL) $= t(n-1, 0.01) \bullet Sc$

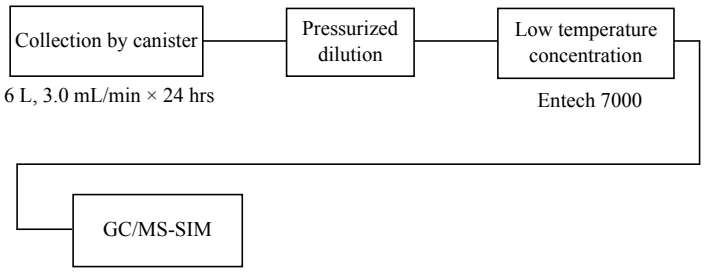
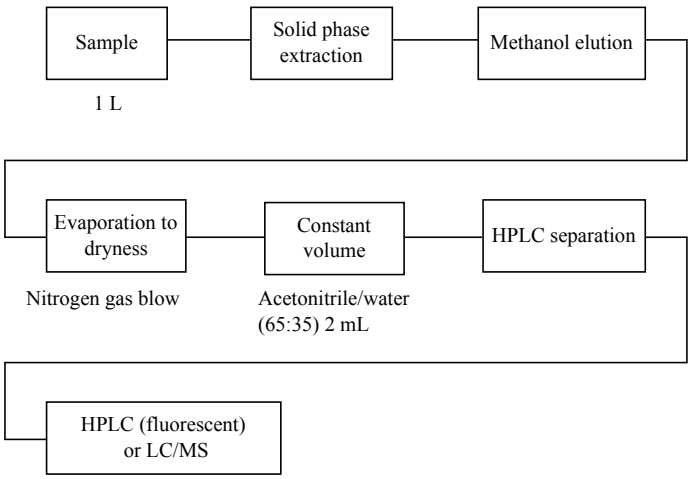
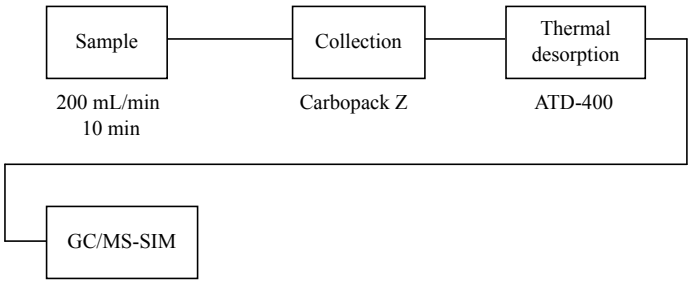
Sc: estimated value of the standard deviation

<River and sea water>

Ten times the detection limit amount of standard substances was added to the river water sample (from the Class B Water Area of Environmental Quality Standards) and the sea water sample (from the Class B of the Environmental Quality Standards, or, when not available, from the Class A Water Area or artificially prepared sea water) and they were analyzed immediately (more than twice). In addition, analysis was conducted on the river water and sea water without the addition of standard substances (more than twice for both samples). Recovery rate was calculated by subtracting the measured value (mean) of the sample water without the addition from the measured value (mean) with the addition.

In the practical survey, various studies such as extraction method, separation method and measurement conditions were conducted in parallel, in consideration of the existence of substances that interfere with the analysis.

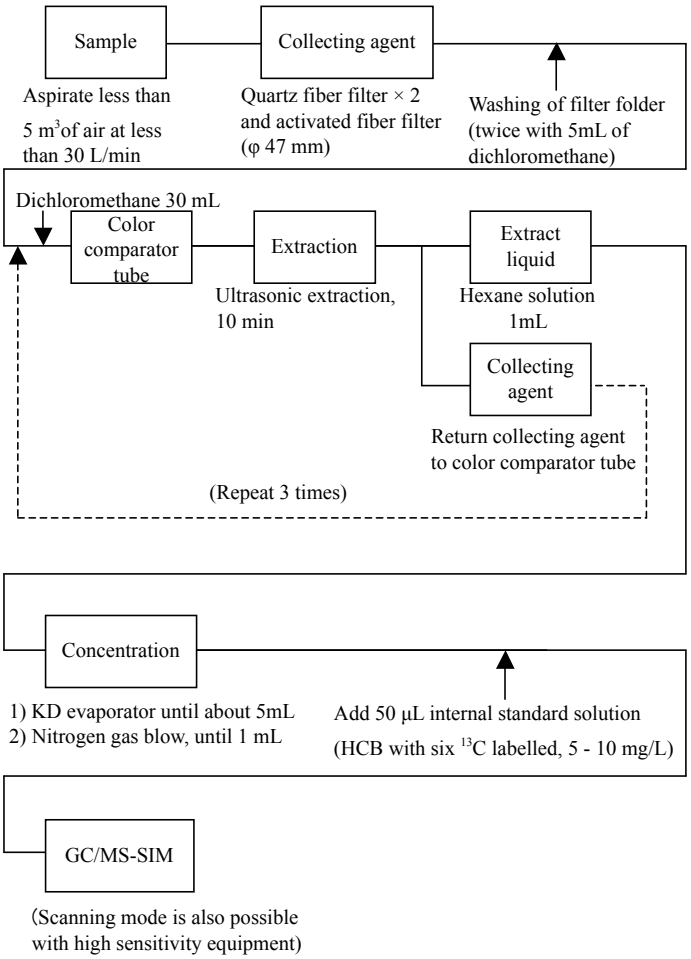
Analytical Method for the FY2003 Initial Environmental Survey

Substance	Analytical Method/Flow Chart	Remarks
(1) HCFCs (1.1) HCFC-141b (1.2) HCFC-22 (1.3) HCFC-123 (1.4) HCFC-142b (1.5) HCFC-225ca (1.6) HCFC-225cb (1.7) HFC-134a	**Air**  	GC/MS-SIM Column: HP-VOC Column length: 60 m Column I.D.: 0.32 mm Film thickness: 1.8 μm Detection limit: Air (ng/m^3) (1.1) 4 (1.2) 6 (1.3) 3 (1.4) 3 (1.5) 4 (1.6) 15 (1.7) 7
(2) Linear alkylbenzene sulfonic acid and its salt (LAS, carbon number of alkyl group: 10 - 14) (2.1) LAS_{10} (2.2) LAS_{11} (2.3) LAS_{12} (2.4) LAS_{13} (2.5) LAS_{14}	**Surface water**  	LC/MS Column: C_8 Column length: 0.25 m Column I.D.: 3.0 mm Detection limit: Surface water (μg/L) (2.1) 0.2 (2.2) 0.2 (2.3) 0.2 (2.4) 0.2 (2.5) 0.2
(3) Isoprene	**Air**  	GC/MS-SIM Column: DB-1 Column length: 60 m Column I.D.: 0.32 mm Film thickness: 3 μm Detection limit: Air (ng/m^3) (3) 12

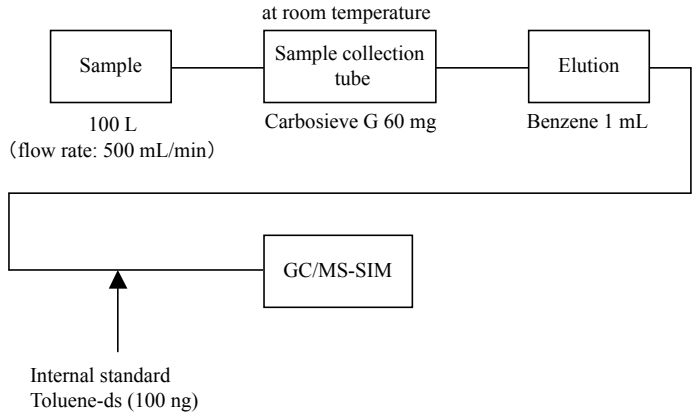
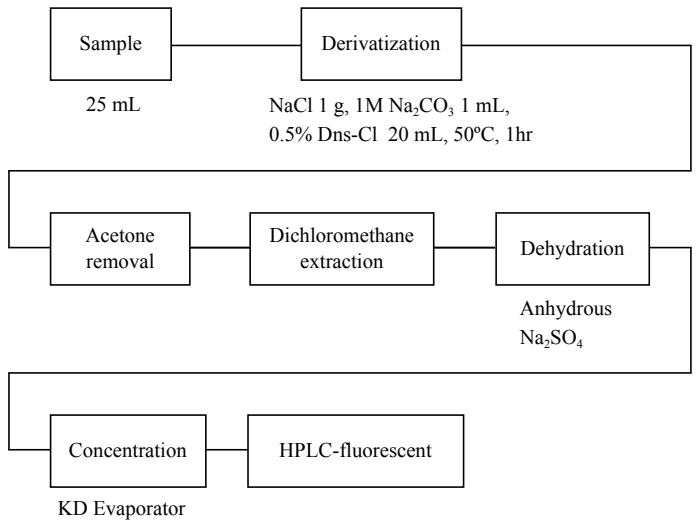
Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(4) Chlordecone	**Air** Sample → Collection (10 L/min × 24 hrs, Quartz fiber filter) → Extraction (Acetone 5 mL) → Concentration (Nitrogen gas blow) → Redissolution (Methanol 1.0 mL) → LC/MS/MS-MRM (ESI-negative)	LC/MS Column: C30-UG-5 Column length: 0.15 m Column I.D.: 2.0 mm Detection limit: Air (ng/m^3) (4) 0.0005
(5) Chlorpyrifos	**Wildlife** Sample (20 g) → Solid-liquid extraction (1) Acetone 50 mL × 2 times, (2) Centrifuging (3000 rpm) → Dilution (Pour into 1% Na_2SO_4 400 mL) → Solvent redissolution (Dichloromethane 100mL, 50mL) → Concentration (1) Rotary evaporator (Below 40ºC, until 5 mL), (2) Nitrogen gas blow (until 1 mL) → Acetonitrile - *n*-hexane partition (1) Dissolve in *n*-hexane 15 mL, (2) *n*-Hexane saturated acetonitrile 50 mL × 2 times) → Concentration (1) Rotary evaporator (Below 40ºC, until 5 mL), (2) Nitrogen gas blow (until 1 mL) → Column chromatography (5% Hydrated Florisil 3 g (I.D.: 1 cm), Wash column with 50 mL *n*-hexane, Elute with 30 mL benzene) → Concentration (1) Rotary evaporator (Below 40ºC, until 5 mL), (2) Nitrogen gas blow (until 1 mL) → GC/MS-SIM or GC-FPD	GC/MS-SIM Column: DB-5 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection limit: Wildlife (ng/g-wet) (5) 3

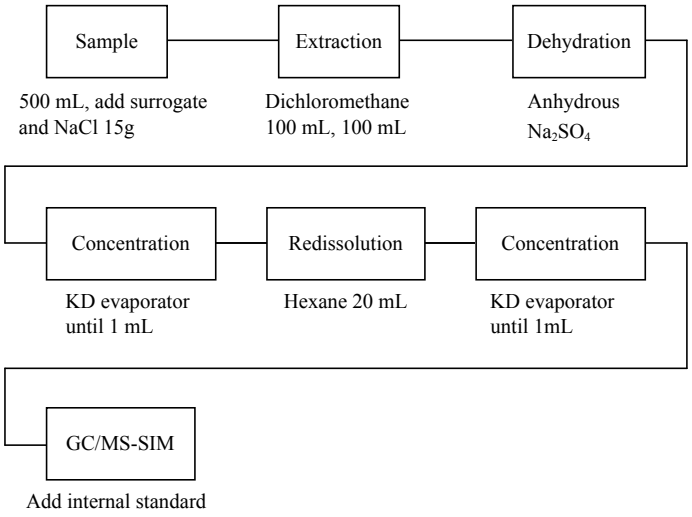
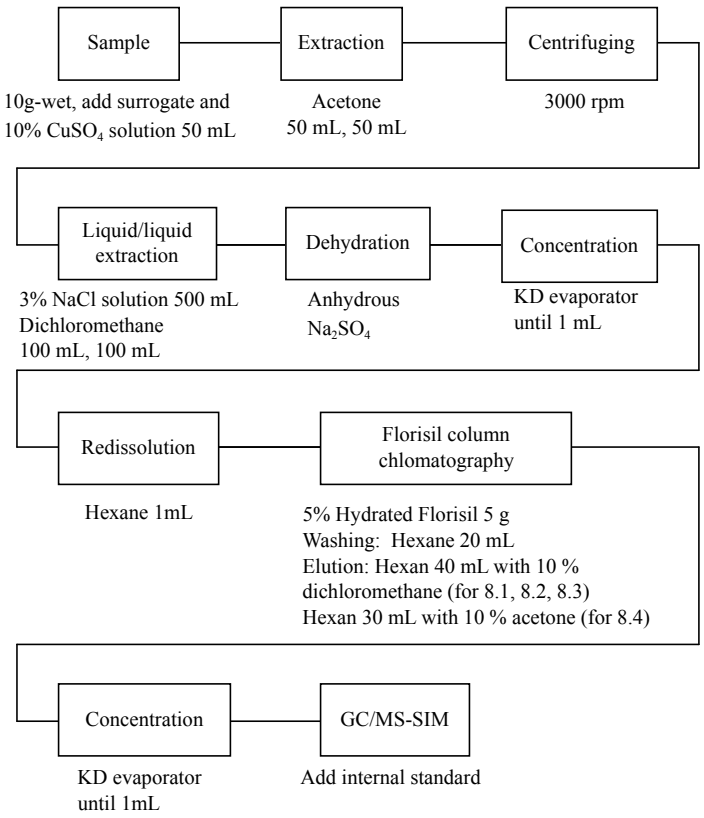
Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(5) Chlorpyrifos (continued)	**Air**  	GC/MS-SIM Column: DB1-MS Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm Detection limit: Air (ng/m3) (5) 2

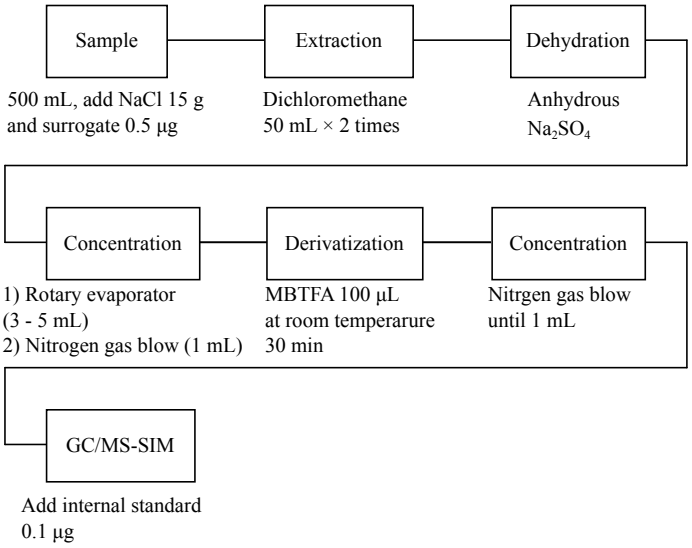
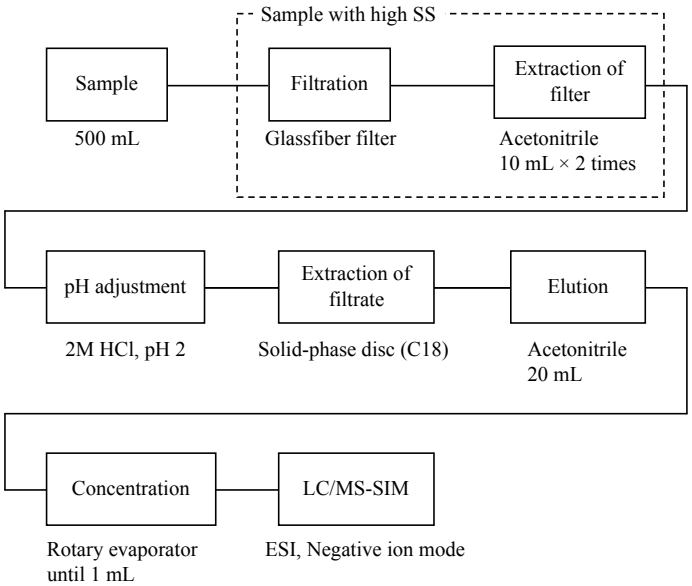
Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(6) Chloropicrin	**Air** 	GC/MS-SIM Column: DB-624 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 1.4 μm Detection limit: Air (ng/m^3) (6) 220
(7) Diethylenetriamine and an another substance (7.1) Diethylenetriamine (7.2) Triethylenetetramine	**Surface water** 	HPLC-fluorescent Column: Lichrosorb RP18 (5 μm) Column length: 0.25 m Column I.D.: 4 mm Detection limit: Surface water (μg/L) (1) 2 (2) 8

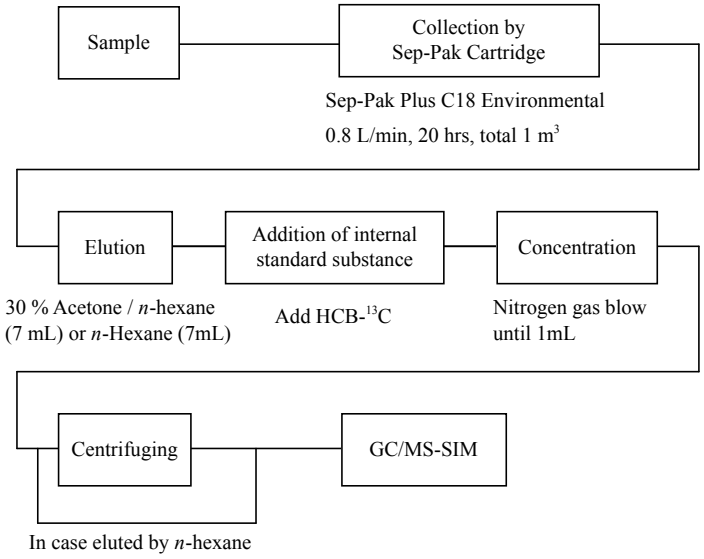
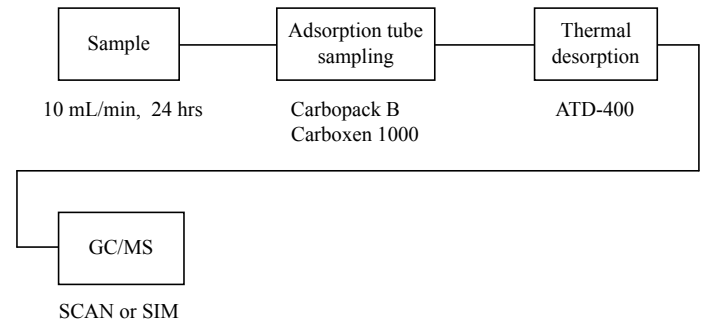
Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(8) 1,4-Dichloro-2-nitrobenzene and 3 other substances (8.1) 1,4-Dichloro-2-nitrobenzene (8.2) 1,3 -Dichloro-4-nitrobenzene (8.3) 1-Chloro-3-nitrobenzene (8.4) 1,4-Dinitrobenzene	**Surface water**  **Bottom sediment**  	GC/MS-SIM Column: DB-1701 Column length: 30 m Column I.D.: 0.32 mm Film thickness: 0.25 μm Detection limit: Surface water (μg/L) (8.1) 0.05 (8.2) 0.06 (8.3) 0.05 (8.4) 0.05 Bottom sediment (ng/g-dry) (8.1) 2.5 (8.2) 1.9 (8.3) 3.2 (8.4) 3.1

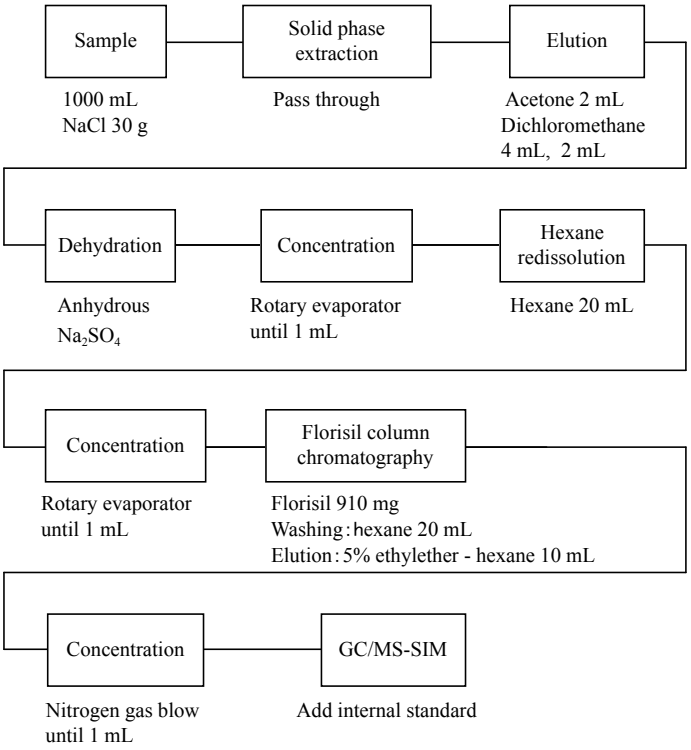
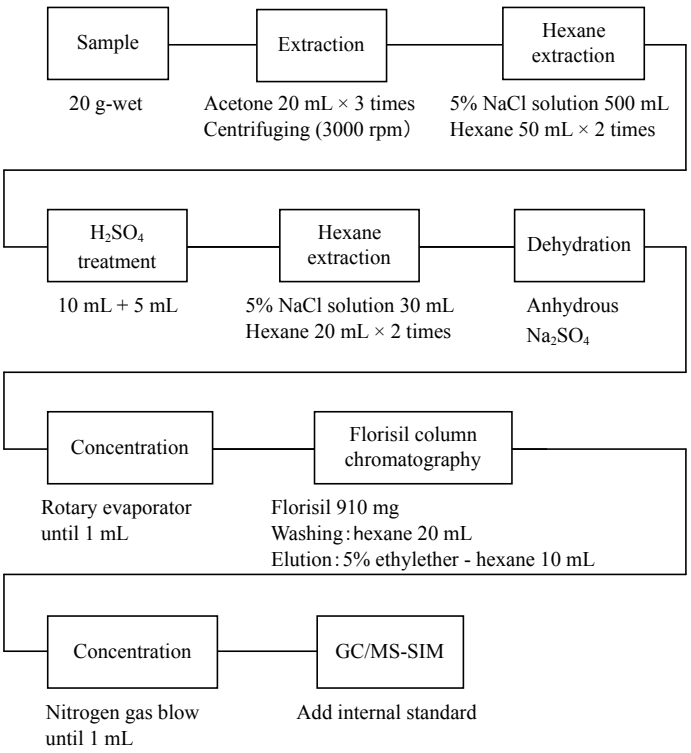
Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(9) 3,3'-Dichlorobenzidine	**Surface water** 	GC/MS-SIM Column: HP-5 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm Detection limit: Surface water (µg/L) (9) 0.010
(10) Pyridine-triphenylborane	**Surface water** 	LC/MS-SIM Column: Inertsil ODS-80A Column length: 0.25 m Column I.D.: 1.5 mm Surface water (µg/L) (10) 0.030

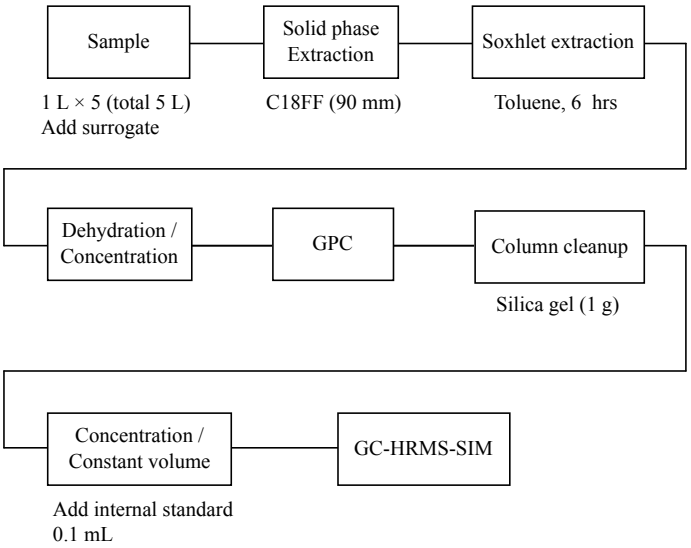
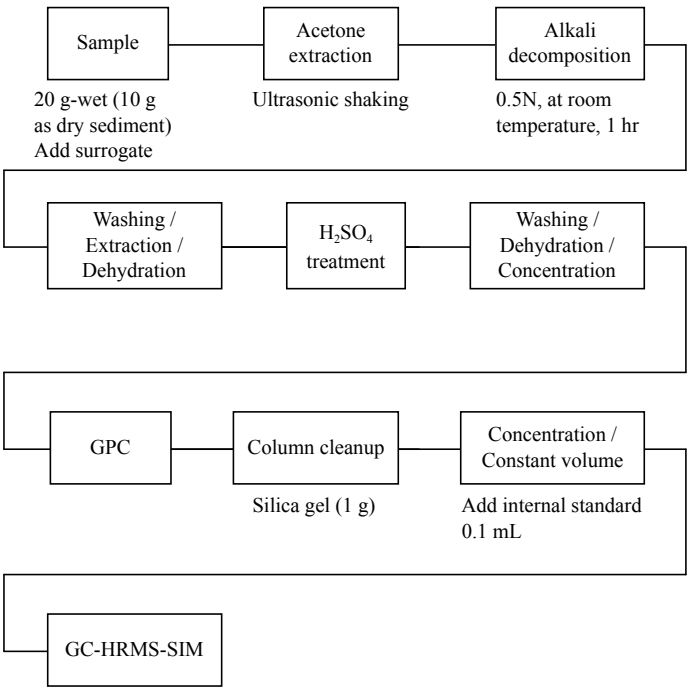
Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(11) 2,4,6-Tri-*tert*-butylphenol	**Air**  	GC/MS-SIM Column: HP-5MS Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection limit: Air (ng/m^3) (11) 0.9
(12) Bromomethane	**Air**  	GC/MS-SIM or GC/MS-SCAN Column: SPB-HAP Column length: 60 m Column I.D.: 0.32 mm Film thickness: 4.0 μm Detection limit: Air (ng/m^3) (12) 0.027

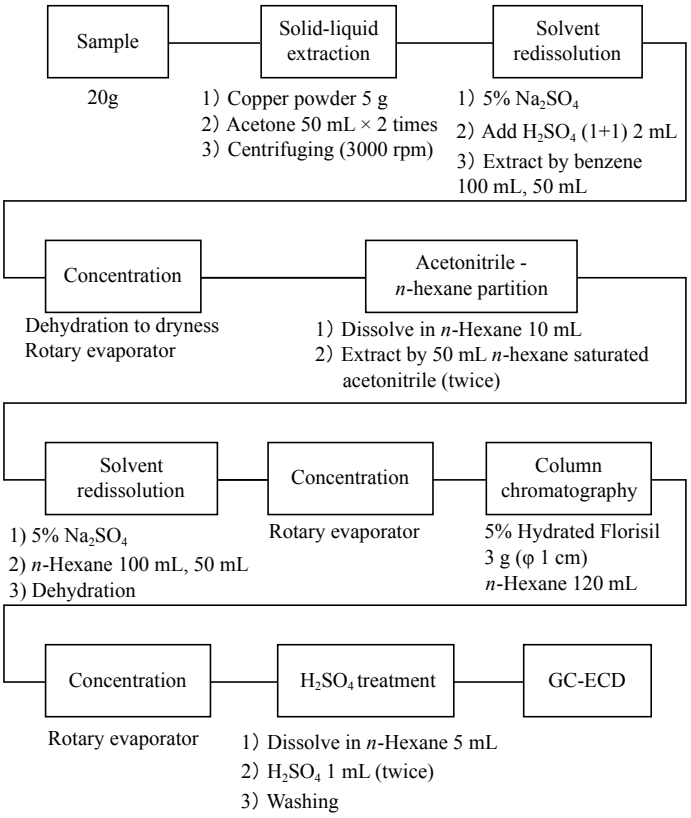
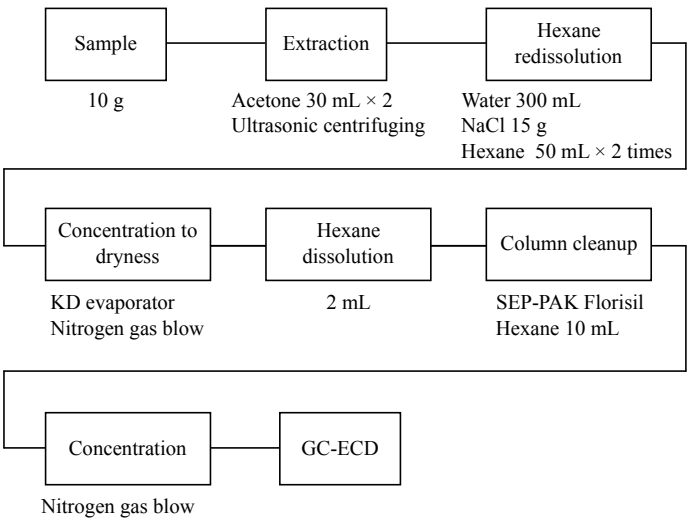
Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(13) 1,2,5,6,9,10-Hexabromocyclododecane	**Surface water**  **Bottom sediment**  	GC/MS-SIM Column: DB-5ms Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.1 μm Detection limit: Surface water (μg/L) (13) 0.087 Bottom sediment (ng/g-dry) (13) 23

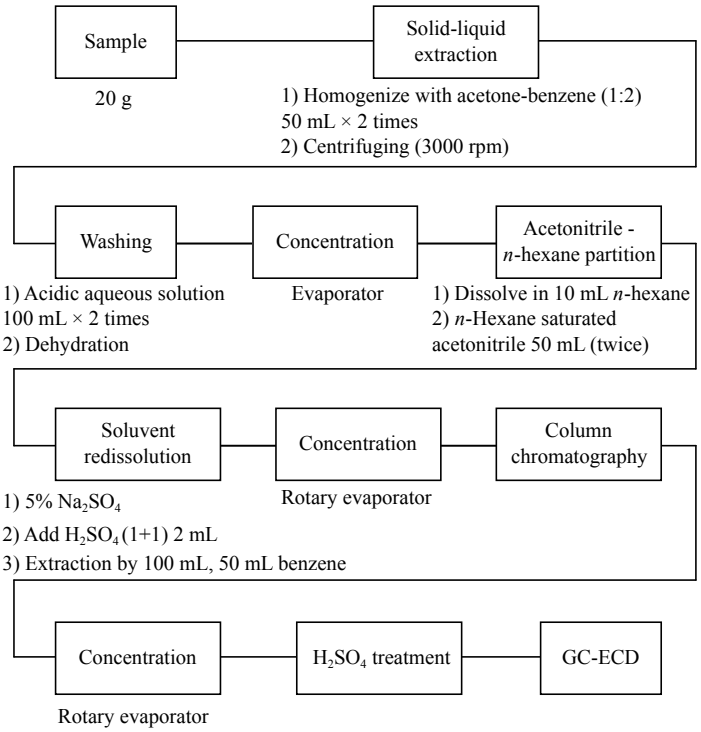
Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(14) Hexabromobiphenyl (14.1) 2,2',4,4',6,6'-Hexabromobiphenyl (14.2) 2,2',4,4',5,5'-Hexabromobiphenyl (14.3) 3,3',4,4',5,5'-Hexabromobiphenyl	**Surface water**  **Bottom sediment**  	GC-HRMS-SIM Column: DB-1HT Column length: 15m Column I.D.: 0.25mm Film thickness: 0.1μm Column: HP-5MS Column length: 30m Column I.D.: 0.32mm Film thickness: 0.1μm Detection limit: Surface water (μg/L) (14.1) 0.012 (14.2) 0.019 (14.3) 0.012 Bottom sediment (ng/g-dry) (14.1) 0.0087 (14.2) 0.014 (14.3) 0.023

Analytical Method for the FY2003 Initial Environmental Survey (continued)

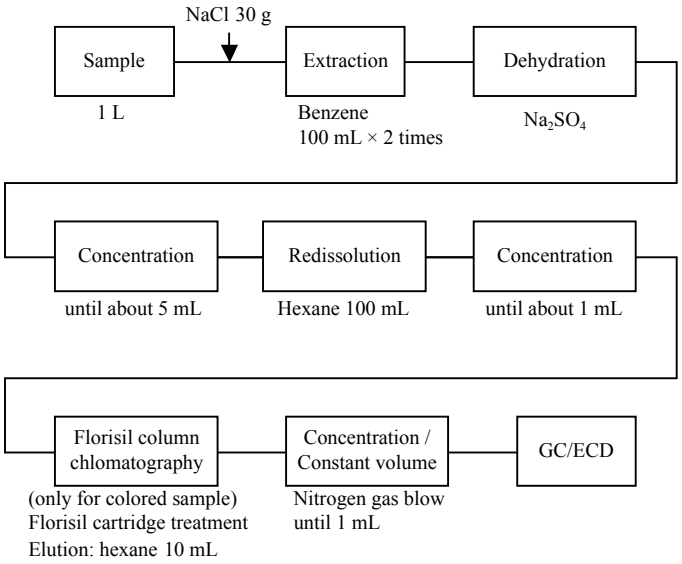
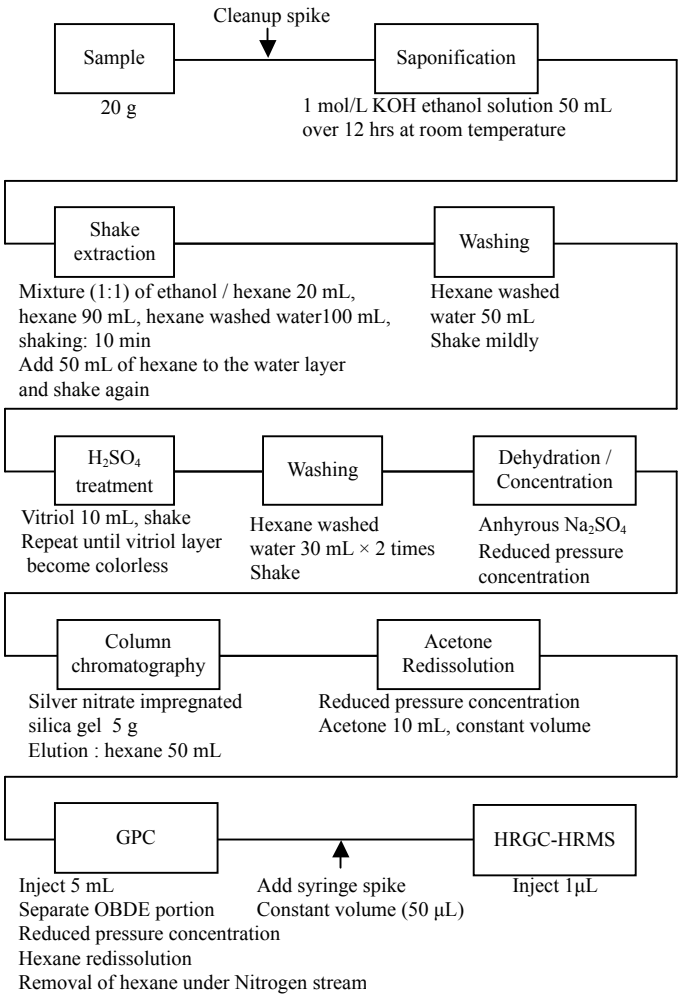
Substance	Analytical Method/Flow Chart	Remarks
(15) Polybrominated diphenyl ethers (15.1) Hexabromodiphenyl ether	**Bottom sediment** 	GC-ECD Column: Glass column Column length: 0.5 m Column I.D.: 3 mm Detection limit: Bottom sediment (ng/g-dry) (15.1) 0.5
(15.2) Decabromodiphenyl ether	**Bottom sediment** 	GC-ECD Column: DB-1 Column length: 5 m Column I.D.: 0.32 mm Film thickness: 0.1 μm Bottom sediment (ng/g-dry) (15.2) 8.7

Analytical Method for the FY2003 Initial Environmental Survey (continued)

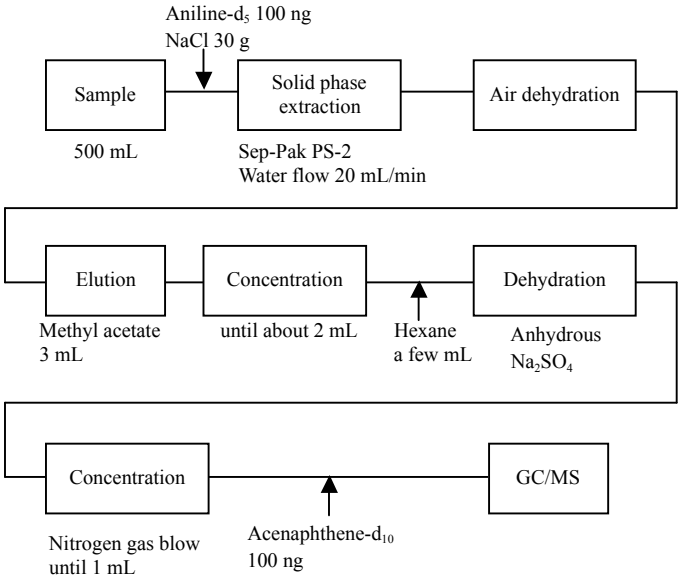
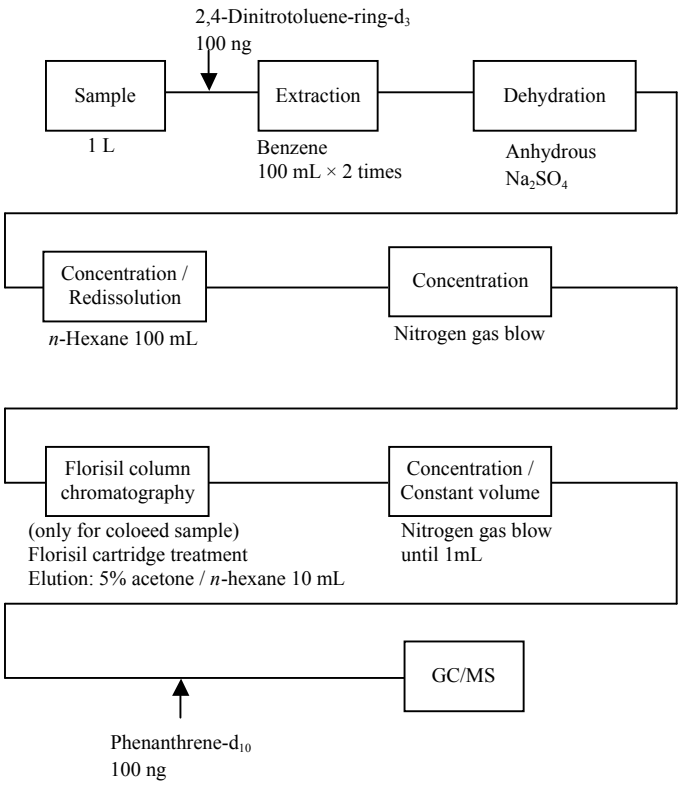
Substance	Analytical Method/Flow Chart	Remarks
(15) Polybrominated diphenyl ethers (continued) (15.1) Hexabromodiphenyl ether (15.2) Decabromodiphenyl ether	**Wildlife**  	GC-ECD Column: Glass column Column length: 0.5 m Column I.D.: 3 mm Detection limit: Wildlife (ng/g-wet) (15.1) 0.5 (15.2) 1

2. Environmental Survey for Exposure Study

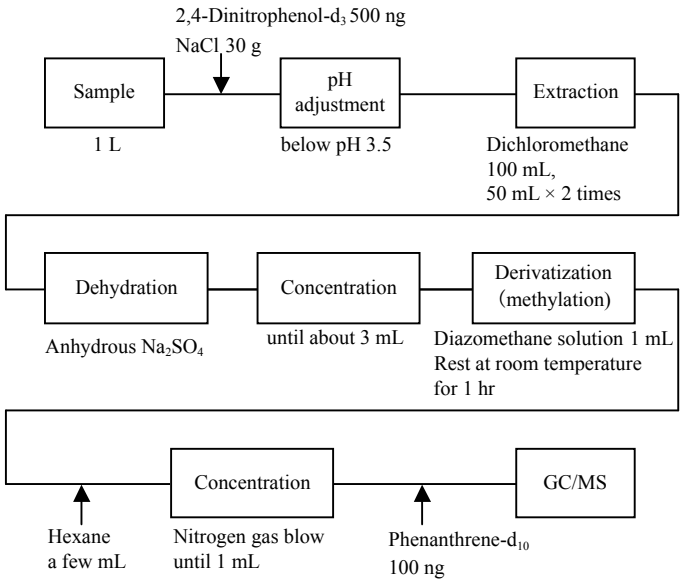
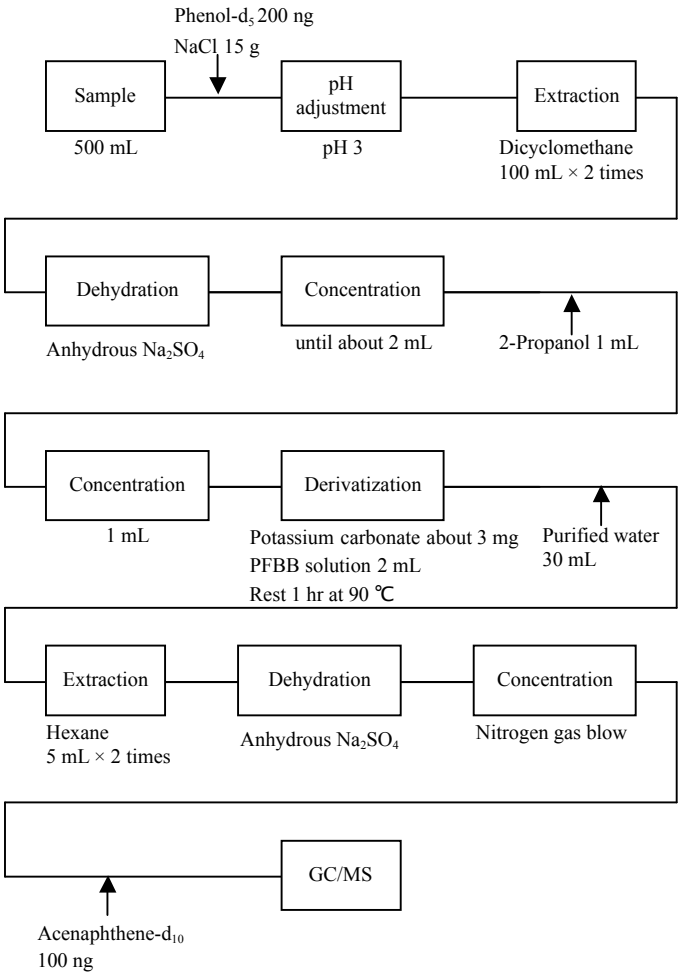
Analytical Method for the FY2003 Environmental Survey for Exposure Study

Substance	Analytical Method/Flow Chart	Remarks
(1) Octabromodiphenylether	**Surface water**  **Wildlife**  	GC/ECD Column: SGE BP-1 Column length: 12 m Column I.D.: 0.22 mm Film thickness: 0.1 µm Detection limit: Surface water (ng/L) (1) 3 HRGC-HRMS Detection limit: Wildlife (ng/g-wet) (1) 0.0007

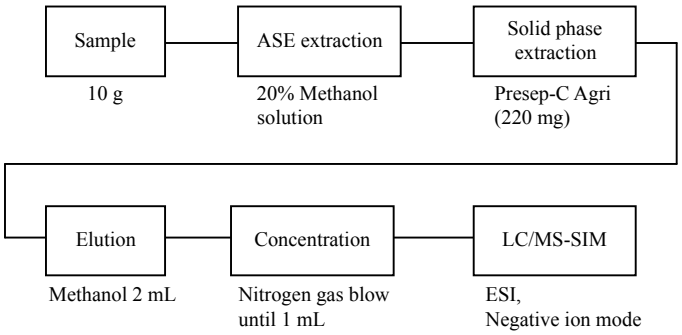
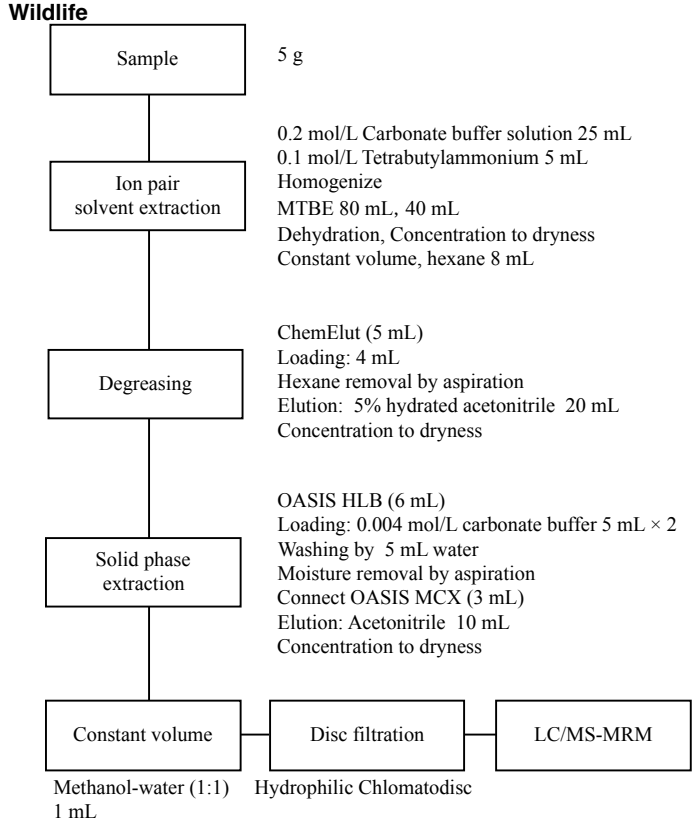
Analytical Method for the FY2003 Environmental Survey for Exposure Study (continued)

Substance	Analytical Method/Flow Chart	Remarks
(2) *o*-Chloroaniline	**Surface water** 	GC/MS-SIM Column: SGE BP-20 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection limit: Surface water (μg/L) (2) 25
(3) 1-Chloro-2,4-dinitrobenzene	**Surface water** 	GC/MS Column: SGE BPX-5 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection limit: Surface water (ng/L) (3) 10

Analytical Method for the FY2003 Environmental Survey for Exposure Study (continued)

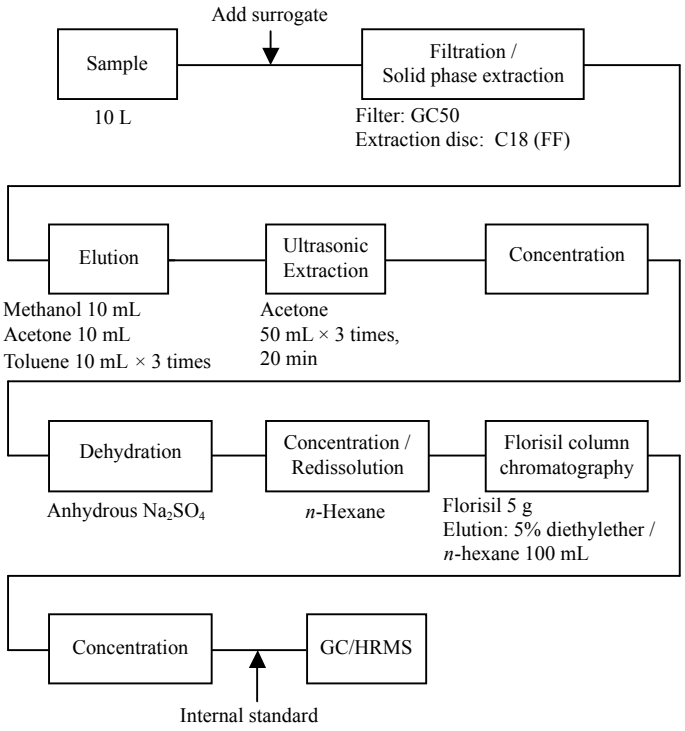
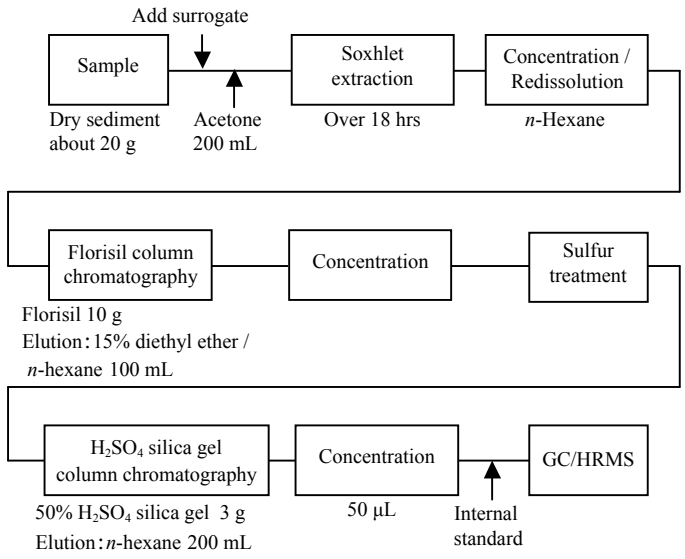
Substance	Analytical Method/Flow Chart	Remarks
(4) 2,4-Dinitrophenol	**Surface water** 	GC/MS Column: SGE BPX-5 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection limit: Surface water (ng/L) (4) 19
(5) Phenol	**Surface water** 	GC/MS Column: SGE BP-10 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection limit: Surface water (ng/L) (5) 28

Analytical Method for the FY2003 Environmental Survey for Exposure Study (continued)

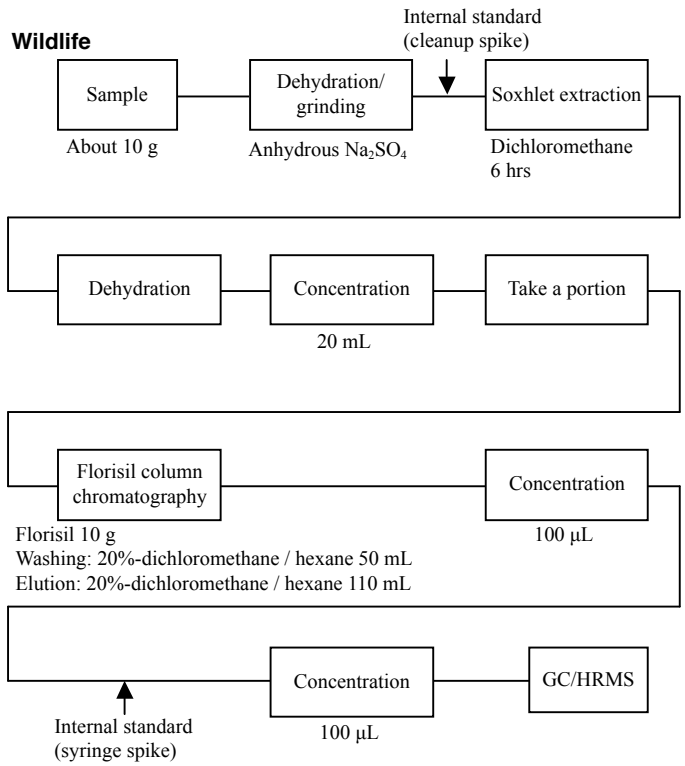
Substance	Analytical Method/Flow Chart	Remarks
(6) Perfluorooctane sulfonic acid (PFOS) (7) Perfluorooctanoic acid (PFOA)	**Bottom sediment**  **Wildlife**  	LC/MS-SIM Column: Zorbax XDB C-18 Column length: 150 mm Column I.D.: 2.1 mm Particle diameter: 3.5 μm Detection limit: Bottom sediment (ng/g-dry) (6) 0.022 (7) 0.016 LC/MS/MS-MRM Column: CAPCEL PAK C18 MG-II Column length: 150 mm Column I.D.: 2 mm Particle diameter: 5 μm Detection limit: Wildlife (ng/g-wet) (6) 0.033 (7) 0.059

3. Monitoring Investigation

Analytical Method for the FY2003 Monitoring Investigation

Substance	Analytical Method/Flow Chart	Remarks
(1) PCBs	**Surface water**  **Bottom sediment**  	GC/HRMS Resolution: 10,000 Column: HT8-PCB Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm Detection/Quantitation limit: Surface water (pg/L) (1) Detection limit / Quantitation limit Mono-chloride 0.4 / 2 Di-chloride 0.2 / 0.5 Tri-chloride 0.2 / 0.4 Tetra-chloride 0.09 / 0.3 Penta-chloride 0.07 / 0.3 Hexa-chloride 0.09 / 0.3 Hepta-chloride 0.07 / 0.3 Octa-chloride 0.07 / 0.3 Nona-chloride 0.4 / 2 Deca-chloride 0.9 / 3 PCB 77, 81 0.3, 0.2 / 0.8, 0.6 PCB 105, 114 0.7, 0.1 / 3, 0.4 PCB 118, 123 2, 0.1 / 6, 0.4 PCB 126, 156 0.1. 0.2 / 0.4, 0.5 PCB 157, 167 0.2, 0.09 / 0.5, 0.3 PCB 169, 170 0.2, 0.3 / 0.4, 0.9 PCB 180, 189 0.5, 0.2 / 2, 0.5 Bottom sediment (pg/g-dry) (1) Detection limit / Quantitation limit Mono-chloride 0.4 / 2 Di-chloride 0.2 / 0.7 Tri-chloride 0.2 / 0.6 Tetra-chloride 0.2 / 0.4 Penta-chloride 0.2 / 0.5 Hexa-chloride 0.2 / 0.4 Hepta-chloride 0.3 / 0.7 Octa-chloride 0.3 / 0.8 Nona-chloride 0.6 / 2 Deca-chloride 0.6 / 2 PCB 77, 81 0.3, 0.3 / 0.8, 0.8 PCB 105, 114 2, 0.3 / 4, 0.8 PCB 118, 123 2, 0.3 / 6, 0.8 PCB 126, 156 0.2, 2 / 0.6, 5 PCB 157, 167 0.4, 0.2 / 2, 0.7 PCB 169, 170 0.4, 2 / 2, 5 PCB 180, 189 0.2, 0.4 / 0.4, 2

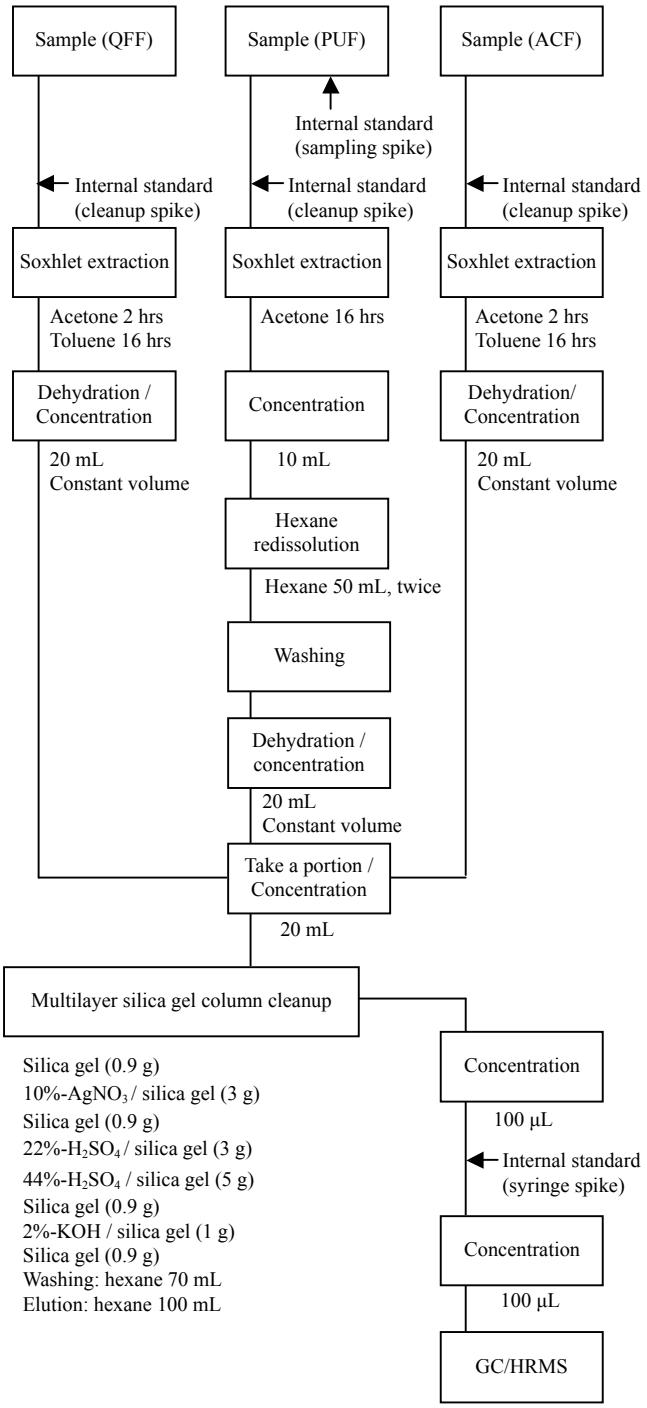
Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks
(1) PCBs (continued)		GC/HRMS Resolution: 10,000 Column: DB-5MS Column length: 60 m Column I.D.: 0.32 mm Film thickness: 0.25 μm Detection/Quantitation limit: Wildlife (pg/g-wet)

(1)	Detection limit	Quantitation limit
Mono-chloride	0.69	2.1
Di-chloride	2.5	7.5
Tri-chloride	2	6
Tetra-chloride	2.3	6.9
Penta-chloride	1.9	5.7
Hexa-chloride	1.1	3.3
Hepta-chloride	1.6	4.8
Octa-chloride	1.8	5.4
Nona-chloride	1.3	3.9
Deca-chloride	1.5	4.5
PCB 77	0.69	2.1
PCB 81	1.5	4.5
PCB 105	2.2	6.6
PCB 114	1.1	3.3
PCB 118	3.7	11
PCB 123	0.97	2.9
PCB 126	0.96	2.9
PCB 156	0.84	2.5
PCB 157	1.2	3.6
PCB 167	0.71	2.1
PCB 169	1.4	4.2
PCB 170	1.8	5.4
PCB 180	1.5	4.5
PCB 189	1.5	4.5

Analytical Method for the FY2003 Monitoring Investigation (continued)

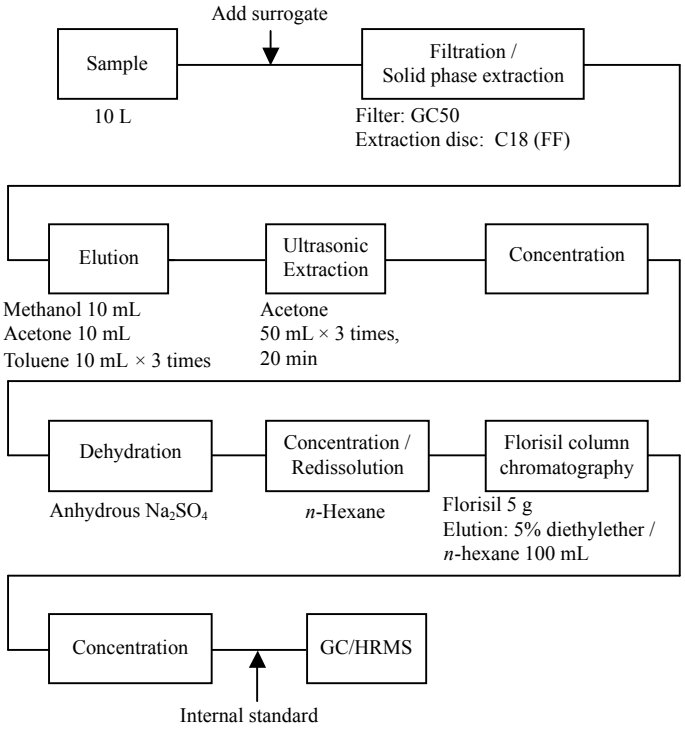
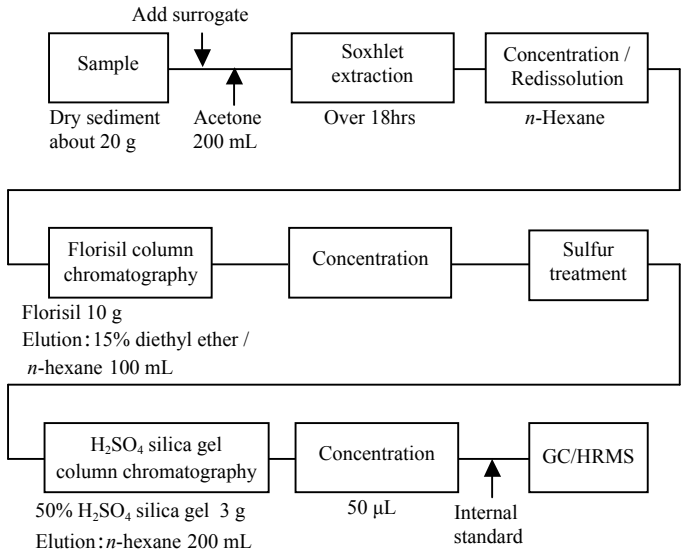
Substance	Analytical Method/Flow Chart	Remarks
(1) PCBs (continued)	**Air** Sampled by high-volume air sampler (HV) with quartz-fiber-filter (QFF), polyurethane form (PUF) and active carbon felt (ACF) sorbent media.	GC/HRMS Resolution: 10,000 Column: DB-5MS Column length: 60 m Column I.D.: 0.32 mm Film thickness: 0.25 μm Detection/Quantitation limit:



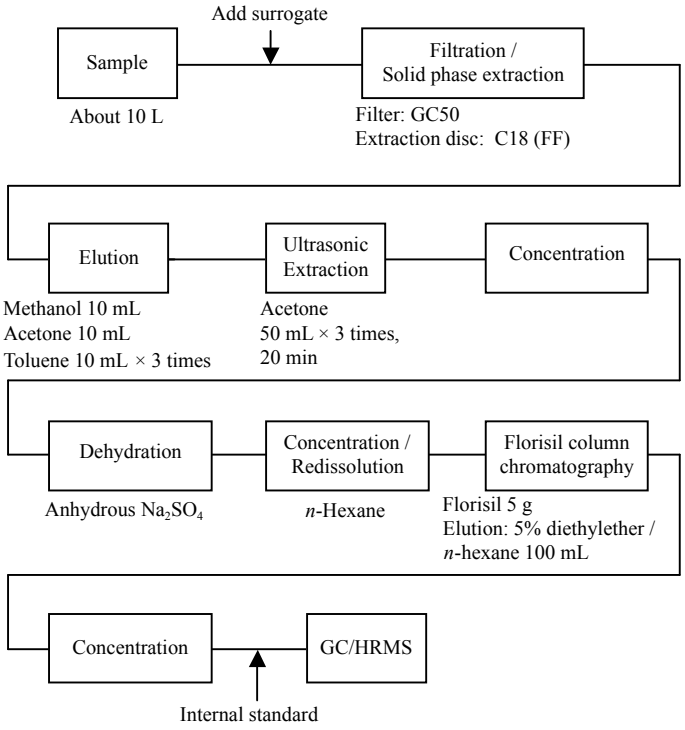
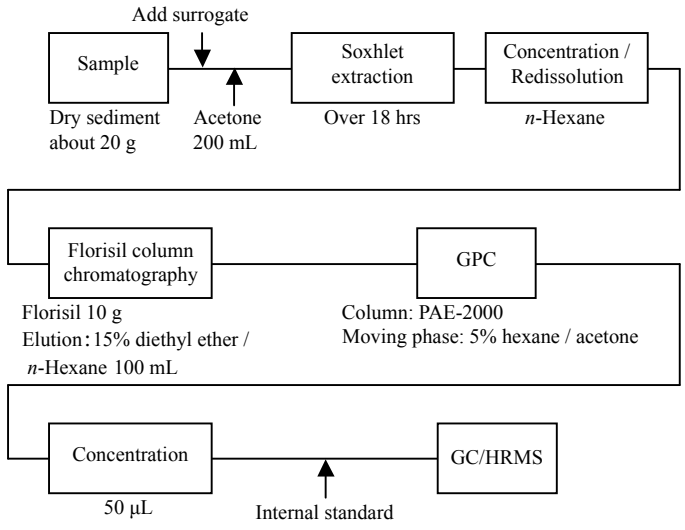
Air (pg/m^3)

(1)	Detection limit	Quantitation limit
Mono-chloride	0.041	0.12
Di-chloride	0.33	1.0
Tri-chloride	1.1	3.2
Tetra-chloride	0.58	1.7
Penta-chloride	0.11	0.32
Hexa-chloride	0.029	0.086
Hepta-chloride	0.01	0.03
Octa-chloride	0.019	0.057
Nona-chloride	0.013	0.039
Deca-chloride	0.0057	0.017
PCB 77	0.0043	0.013
PCB 81	0.0051	0.015
PCB 105	0.0072	0.022
PCB 114	0.0082	0.025
PCB 118	0.0050	0.015
PCB 123	0.0052	0.016
PCB 126	0.0089	0.027
PCB 156	0.0083	0.025
PCB 157	0.0077	0.023
PCB 167	0.007	0.021
PCB 169	0.0098	0.029
PCB 170	0.0098	0.029
PCB 180	0.016	0.048
PCB 189	0.0083	0.025

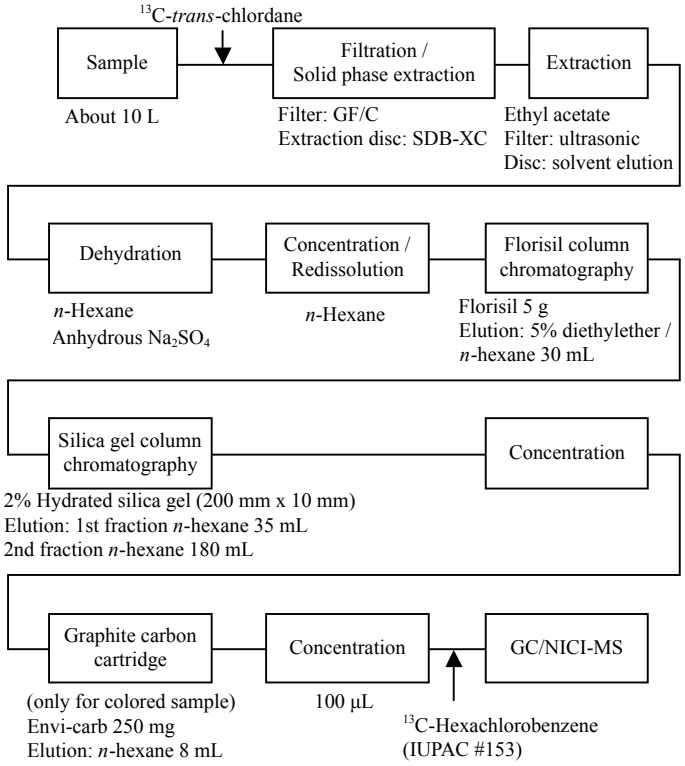
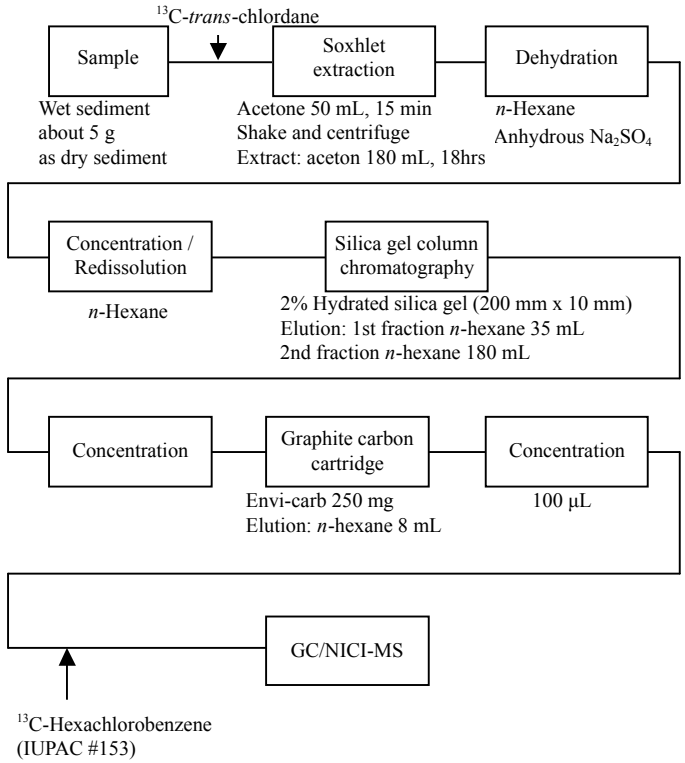
Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks
(2) HCB (4) DDTs (4.1) *p,p′*-DDT (4.2) *p,p′*-DDE (4.3) *p,p′*-DDD (4.4) *o,p′*-DDT (4.5) *o,p′*-DDE (4.6) *o,p′*-DDD (5) Chlordanes (5.1) *trans*-Chlordane (5.2) *cis*-Chlordane (5.3) *trans*-Nonachlor (5.4) *cis*-Nonachlor (5.5) Oxychlordane (6) Heptachlors (6.1) Heptachlor (8) Mirex (9) HCHs (9.1) α-HCH (9.2) β-HCH (9.3) γ-HCH (9.4) δ-HCH	**Surface water**  **Bottom sediment**  	GC/HRMS Resolution: 10,000 Column: HT8-PCB Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm Detection/Quantitation limit: Surface water (pg/L) (2) 2 5 (4.1) 0.9 3 (4.2) 2 4 (4.3) 0.5 2 (4.4) 0.7 3 (4.5) 0.3 0.8 (4.6) 0.3 0.8 (5.1) 2 5 (5.2) 0.9 3 (5.3) 0.5 3 (5.4) 0.1 0.3 (5.5) 0.5 2 (6.1) 0.5 2 (8) 0.09 0.3 (9.1) 0.9 3 (9.2) 0.7 3 (9.3) 2 7 (9.4) 0.5 2 Bottom sediment (pg/g-dry) (2) 2 4 (4.1) 0.4 2 (4.2) 0.3 0.9 (4.3) 0.3 0.9 (4.4) 0.3 0.8 (4.5) 0.2 0.6 (4.6) 0.5 2 (5.1) 2 4 (5.2) 2 4 (5.3) 0.6 2 (5.4) 0.9 3 (5.5) 0.4 1 (6.1) 1 3 (8) 0.4 2 (9.1) 0.5 2 (9.2) 0.7 2 (9.3) 0.4 2 (9.4) 0.7 2

Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks
(3) Drins (3.1) Aldrin (3.2) Dieldrin (3.3) Endrin (6) Heptachlors (6.2) *trans*-Heptachlor epoxide (6.3) *cis*-Heptachlor epoxide	**Surface water**  **Bottom sediment**  	GC/HRMS Resolution: 10,000 Column: RH17 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection/Quantitation limit: Surface water (pg/L) (3.1) Detection limit 0.2, Quantitation limit 0.6 (3.2) Detection limit 0.3, Quantitation limit 0.7 (3.3) Detection limit 0.3, Quantitation limit 0.7 (6.2) Detection limit 0.4, Quantitation limit 2 (6.3) Detection limit 0.2, Quantitation limit 0.7 Bottom sediment (pg/g-dry) (3.1) Detection limit 0.6, Quantitation limit 2 (3.2) Detection limit 2, Quantitation limit 4 (3.3) Detection limit 2, Quantitation limit 5 (6.2) Detection limit 3, Quantitation limit 9 (6.3) Detection limit 1, Quantitation limit 3

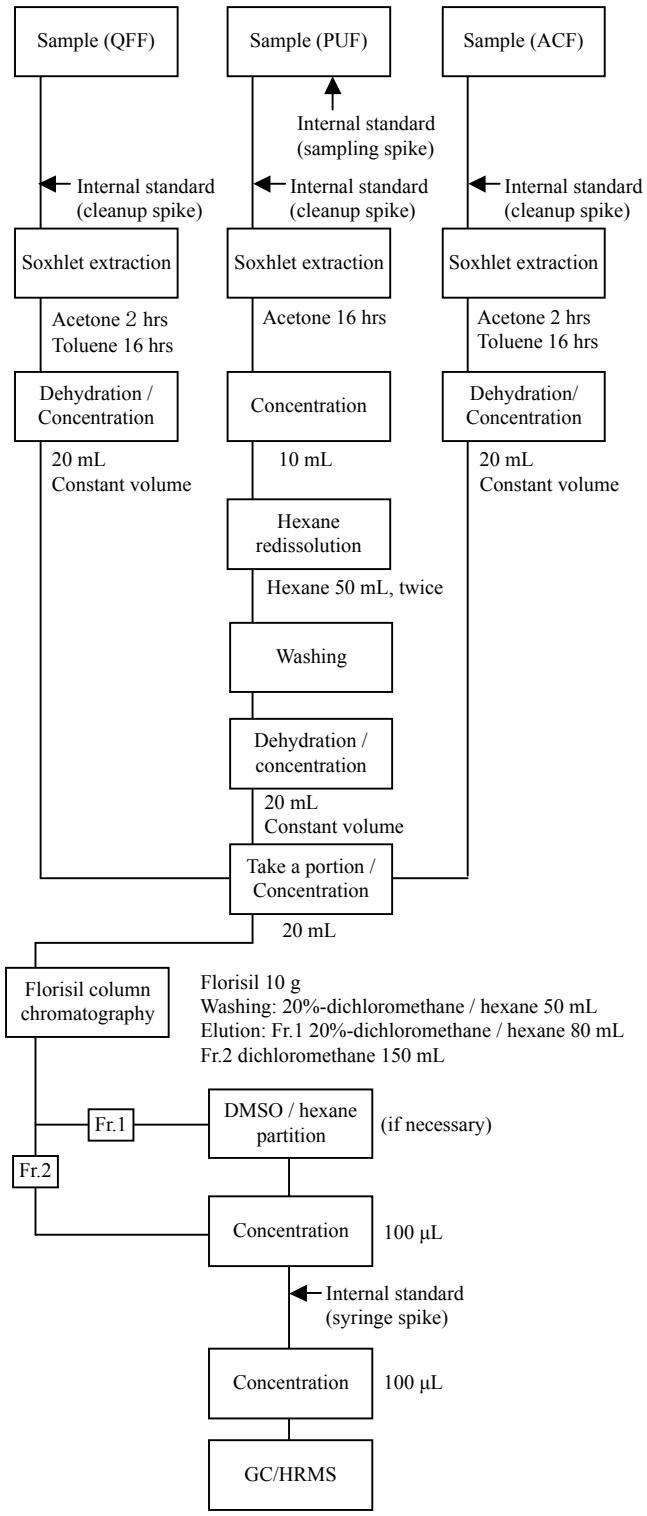
Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks
(7) Toxaphene (7.1) Parlar-26 (7.2) Parlar-50 (7.3) Parlar-62	**Surface water**  **Bottom sediment**  	GC/NICI-MS Column: RH12 Column length: 60 m Column I.D.: 0.25 mm Film thickness: 0.25 µm Detection/Quantitation limit: Surface water (pg/L) (7.1) Detection limit 20, Quantitation limit 40 (7.2) Detection limit 30, Quantitation limit 70 (7.3) Detection limit 90, Quantitation limit 300 Bottom sediment (pg/g-dry) (7.1) Detection limit 30, Quantitation limit 90 (7.2) Detection limit 50, Quantitation limit 200 (7.3) Detection limit 2000, Quantitation limit 4000

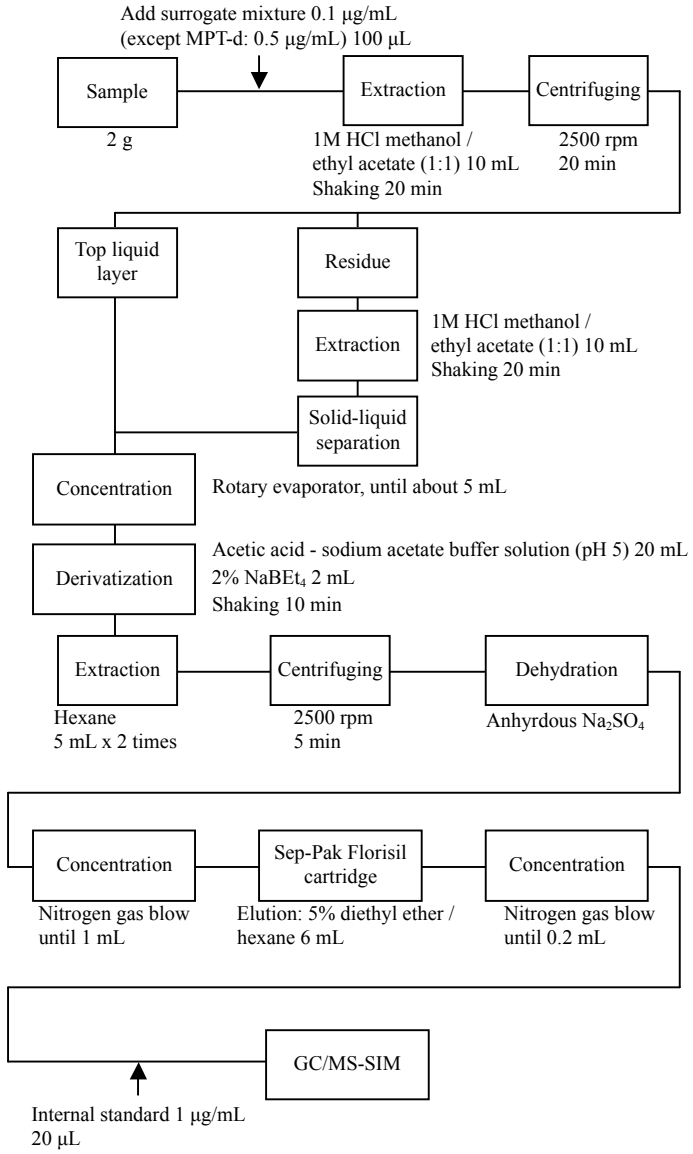
Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks
(2) HCB (3) Drins (3.1) Aldrin (3.2) Dieldrin (3.3) Endrin (4) DDTs (4.1) *p,p′*-DDT (4.2) *p,p′*-DDE (4.3) *p,p′*-DDD (4.4) *o,p′*-DDT (4.5) *o,p′*-DDE (4.6) *o,p′*-DDD (5) Chlordanes (5.1) *trans*-Chlordane (5.2) *cis*-Chlordane (5.3) *trans*-Nonachlor (5.4) *cis*-Nonachlor (5.5) Oxychlordane (6) Heptachlors (6.1) Heptachlor (6.2) *trans*-Heptachlor epoxide (6.3) *cis*-Heptachlor epoxide (7) Toxaphene (7.1) Parlar-26 (7.2) Parlar-50 (7.3) Parlar-62 (8) Mirex (9) HCHs (9.1) α-HCH (9.2) β-HCH (9.3) γ-HCH (9.4) δ-HCH	**Wildlife** Sample (About 10 g) → Dehydration/grinding (Anhydrous Na_2SO_4) ← Internal standard (cleanup spike) → Soxhlet extraction (Dichloromethane 6 hrs) → Dehydration → Concentration (20 mL) → Take a portion → Florisil column chromatography Florisil 10 g Washing: 20%-dichloromethane / hexane 50 mL Elution: Fr.1 20%-dichloromethane / hexane 110 mL Fr.2 dichloromethane 170 mL → Fr.1, Fr.2 → Further cleanup for samples containing a large amount of fat → Fr.2 † Fr.1 / Fr.2 → Concentration (100 µL) ← Internal standard (syringe spike) → Concentration (100 µL) → GC/HRMS Fr.1: HCB, DDTs, chlordanes, aldrin, heptachlor, HCHs, *trans*-heptachlor epoxide, mirex, toxaphene Note: GC/NICI-MS method is applied for toxaphene. Fr.2: endrin, dieldrin, *cis*-heptachlor epoxide Further cleanup of the Fr.2 is to be conducted by the following procedure for samples containing a large amount of fat Fr.2 † → DMSO / hexane partition (25 mL, 4 times) → DMSO layer (H_2O 100 mL) / Hexane layer → Hexane reverse-phase partition (75 mL, 3 times) → Hexane layer → Water washing (Twice) → Dehydration → †	Other than toxaphene GC/HRMS Column: DB-17HT Column length: 30 m Column I.D.: 0.32 mm Film thickness: 0.15 µm Toxaphene GC/NICI-MS Column: HT8 Column length: 60 m Column I.D.: 0.25 mm Film thickness: 0.15 µm Detection/Quantitation limit: Wildlife (pg/g-wet) (2) 7.5 / 23 (3.1) 0.84 / 2.5 (3.2) 1.6 / 4.8 (3.3) 1.6 / 4.8 (4.1) 3.5 / 11 (4.2) 1.9 / 5.7 (4.3) 3.3 / 9.9 (4.4) 0.97 / 2.9 (4.5) 1.2 / 3.6 (4.6) 2.0 / 6.0 (5.1) 2.4 / 7.2 (5.2) 1.3 / 3.9 (5.3) 1.2 / 3.6 (5.4) 1.6 / 4.8 (5.5) 2.8 / 8.4 (6.1) 2.2 / 6.6 (6.2) 4.4 / 13 (6.3) 2.3 / 6.9 (7.1) 15 / 45 (7.2) 11 / 33 (7.3) 40 / 120 (8) 0.81 / 2.4 (9.1) 0.61 / 1.8 (9.2) 3.3 / 9.9 (9.3) 1.1 / 3.3 (9.4) 1.3 / 3.9 (Detection limit / Quantitation limit)

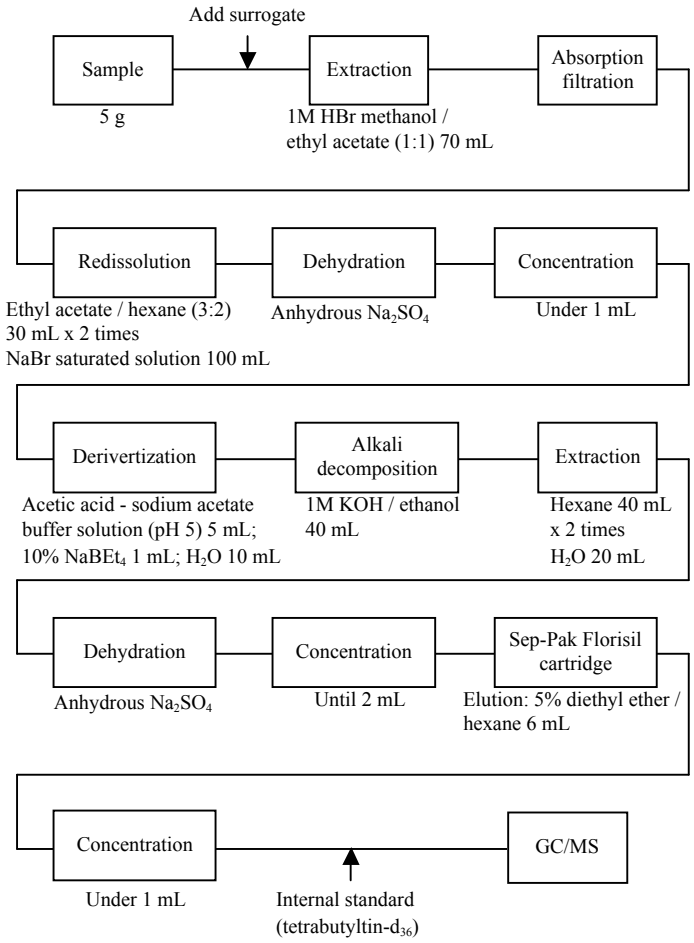
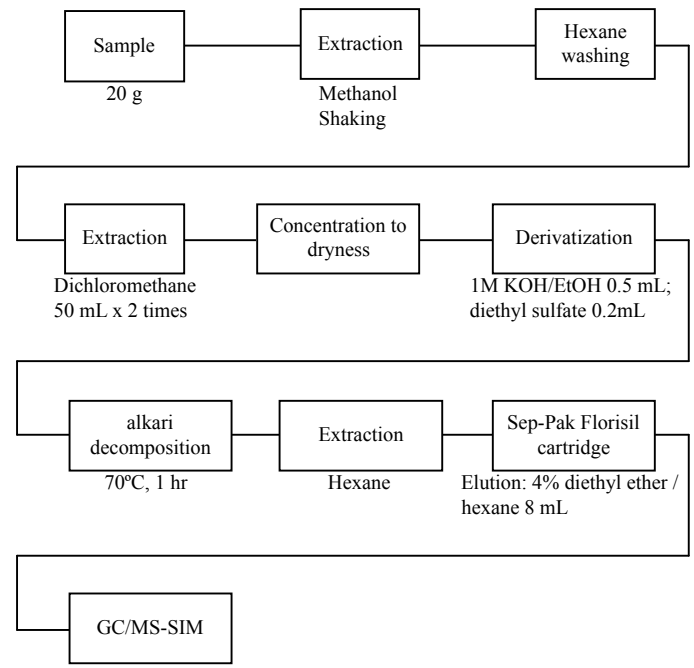
Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks
(2) HCB (3) Drins (3.1) Aldrin (3.2) Dieldrin (3.3) Endrin (4) DDTs (4.1) *p,p′*-DDT (4.2) *p,p′*-DDE (4.3) *p,p′*-DDD (4.4) *o,p′*-DDT (4.5) *o,p′*-DDE (4.6) *o,p′*-DDD (5) Chlordanes (5.1) *trans*-Chlordane (5.2) *cis*-Chlordane (5.3) *trans*-Nonachlor (5.4) *cis*-Nonachlor (5.5) Oxychlordane (6) Heptachlors (6.1) Heptachlor (6.2) *trans*-Heptachlor epoxide (6.3) *cis*-Heptachlor epoxide (7) Toxaphene (7.1) Parlar-26 (7.2) Parlar-50 (7.3) Parlar-62 (8) Mirex (9) HCHs (9.1) α-HCH (9.2) β-HCH (9.3) γ-HCH (9.4) δ-HCH	**Air** Sampled by high-volume air sampler (HV) with quartz-fiber-filter (QFF), polyurethane form (PUF) and active carbon felt (ACF) sorbent media.  Fr.1: HCB, DDTs, chlordanes, aldrin, heptachlor, HCHs, *trans*-heptachlor epoxide, mirex, toxaphene Note: GC/NICI-MS method is applied for toxaphene. Fr.2: endrin, dieldrin, *cis*-heptachlor epoxide	Other than toxaphene GC/HRMS Column: DB-17HT Column length: 30 m Column I.D.: 0.32 mm Film thickness: 0.15 µm Toxaphene GC/NICI-MS Column: HT8 Column length: 60 m Column I.D.: 0.25 mm Film thickness: 0.15 µm Detection/Quantitation limit: Air (pg/m^3) (2) 0.78 2.3 (3.1) 0.0077 0.023 (3.2) 0.70 2.1 (3.3) 0.014 0.042 (4.1) 0.046 0.14 (4.2) 0.13 0.40 (4.3) 0.018 0.054 (4.4) 0.040 0.12 (4.5) 0.0068 0.020 (4.6) 0.014 0.042 (5.1) 0.29 0.86 (5.2) 0.17 0.51 (5.3) 0.12 0.35 (5.4) 0.0088 0.026 (5.5) 0.015 0.045 (6.1) 0.085 0.25 (6.2) 0.033 0.099 (6.3) 0.0048 0.015 (7.1) 0.066 0.20 (7.2) 0.27 0.81 (7.3) 0.52 1.6 (8) 0.0028 0.0084 (9.1) 0.24 0.71 (9.2) 0.063 0.19 (9.3) 0.19 0.57 (9.4) 0.01 0.03 Detection limit / Quantitation limit

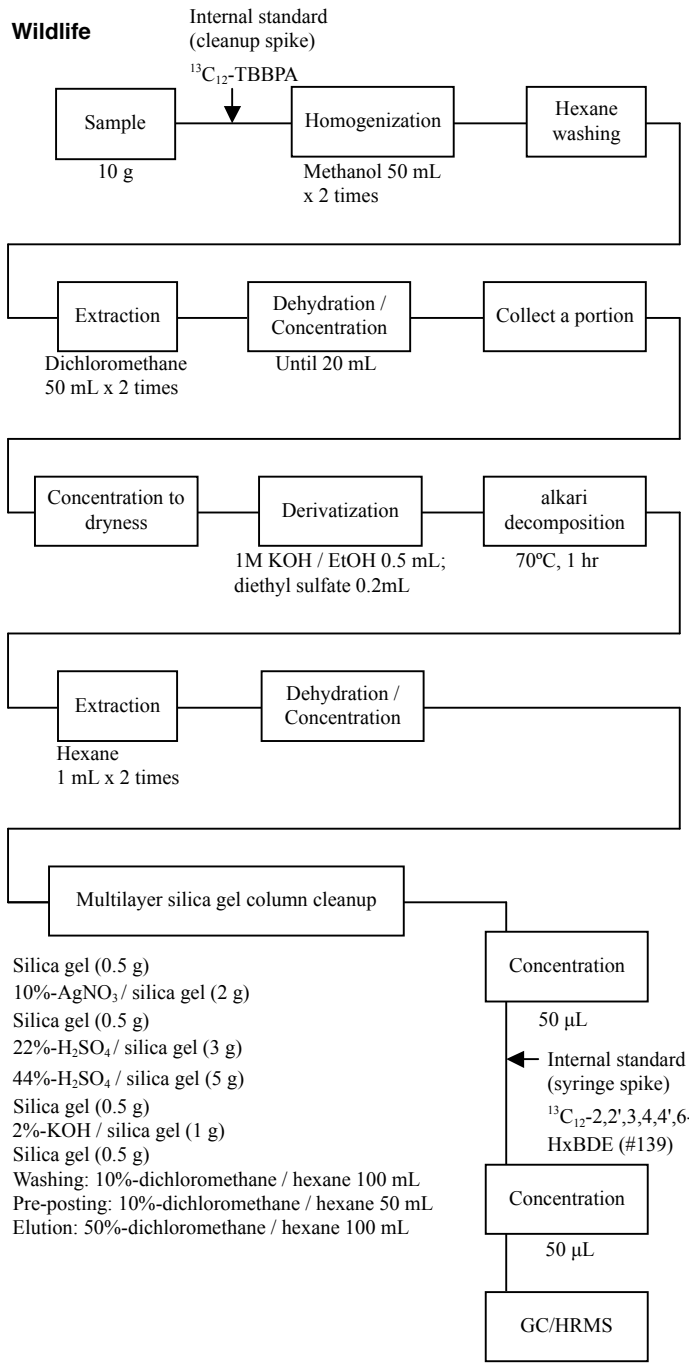
Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks
(10) Organotin compounds (10.1) TBT (10.2) DBT (10.3) TPT (10.4) DPT (10.5) MPT	**Bottom sediment**  	GC/MS (QP-MS) Column: DB-5MS Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm Detection/Quantitation limit: Bottom sediment (ng/g-dry) (10.1) Detection limit 0.4, Quantitation limit 1.2 (10.2) Detection limit 0.4, Quantitation limit 1.2 (10.3) Detection limit 0.09, Quantitation limit 0.28 (10.4) Detection limit 0.06, Quantitation limit 0.16 (10.5) Detection limit 0.8, Quantitation limit 2.4

Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks
(10) Organotin compounds (continued) (10.1) TBT (10.2) DBT (10.3) TPT (10.4) DPT (10.5) MPT	**Wildlife**  	GC/MS Column: DB-5MS Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm Detection/Quantitation limit: Wildlife (ng/g-wet) (10.1) Detection limit 1, Quantitation limit 3 (10.2) Detection limit 1, Quantitation limit 3 (10.3) Detection limit 0.5, Quantitation limit 1.5 (10.4) Detection limit 0.5, Quantitation limit 1.5 (10.5) Detection limit 5, Quantitation limit 15
(11) Tetrabromo bisphenol A	**Bottom sediment**  	GC/MS-SIM Column: SGE BPX-5 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm Detection/Quantitation limit: Bottom sediment (ng/g-dry) (11) Detection limit 5.5, Quantitation limit 18

Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks
(11) Tetrabromo bisphenol A (continued)		GC/HRMS Column: DB-5MS Column length: 60 m Column I.D.: 0.32 mm Film thickness: 0.25 μm or Column: DB-17HT Column length: 60 m Column I.D.: 0.32 mm Film thickness: 0.15 μm Detection/Quantitation limit: Wildlife (ng/g-wet) (11) Detection limit 0.030, Quantitation limit 0.090