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×D) and the quantitation limit was defined as ten times (10×D) the power of the test.

<Bottom sediment>

Assuming a concentration in bottom sediment corresponding to the detection limit (3×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)= $t\,(n-1, 0.01) \bullet Sc$

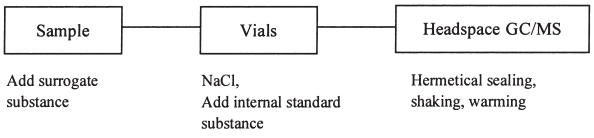
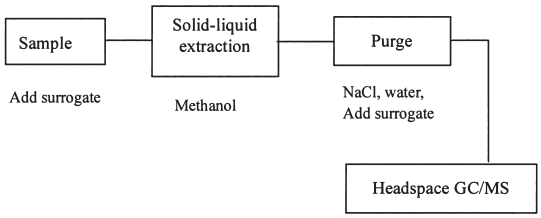
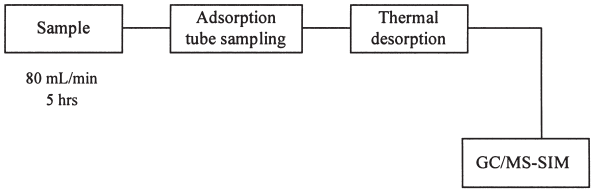
Sc : estimated value of the standard deviation

<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 FY2002 Initial Environmental Survey

Substance	Analytical Method / Flow Chart	Remarks
(1) Isoprene	**Surface water**  **Bottom sediment**  	GC/MS Column: Column length: 25 - 120 m Column I.D.: 0.2 - 0.75 mm Film thickness: 0.1 - 3.0 μm Detection limit: Surface water (μg/L) (1) 0.1 Bottom sediment (ng/g-dry) (1) 10
(2) Epichlorohydrin	**Air**  	GC/MS (SIM) Column: HP-VOC Column length: 60 m Column I.D.: 0.32 mm Film thickness: 1.0 μm Detection limit: Air (ng/m^3) (2) 0.14

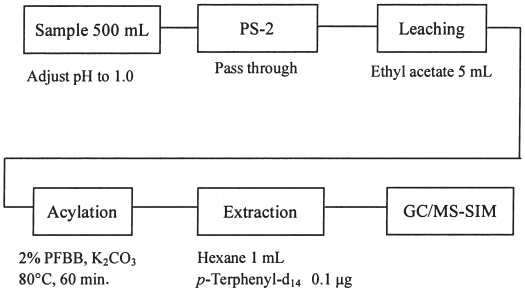
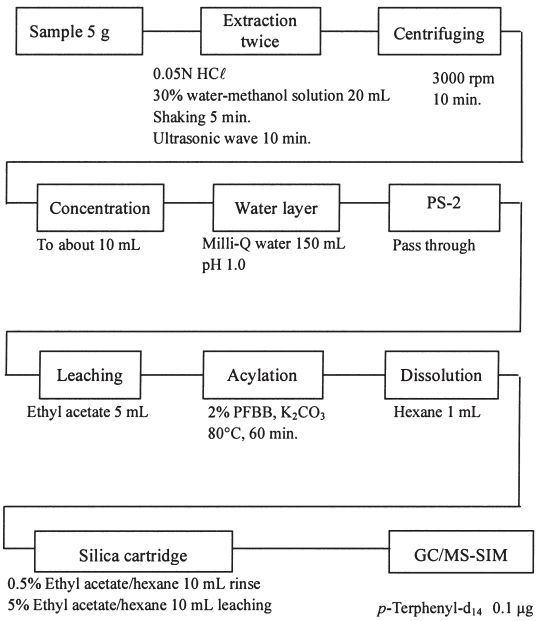
Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(3) 1-Octanol	**Surface water** Sample 1,000 mL — Add surrogate Extraction — NaCℓ 50 g, Dicyclomethane 50 mL×2 Dehydration/Concentration/Evaporation to dryness — Anhydrous Na_2SO_4, KD Evaporator, Nitrogen stream Derivatization — DMF 0.2 mL, BSTFA 0.2 mL, Room temp.: 30 min Extraction — 5% NaOH 2 mL, *n*-Hexane 2 mL Dehydration/Concentration — Suck up the hexane layer with a Pasteur pipette and concentrate to 0.5 mL in nitrogen stream GC/MS-SIM **Bottom sediment** Sample 10 g — NaCℓ 15 g, Purified water 500 mL, Surrogate solution, Hexane 5 mL Extraction — Methanol 30 mL×2, Ultrasonic wave 10 min., Centrifugation, 3000 rpm 10 min. Redissolution — 5% NaCℓ 100 mL, Dicyclomethane 50 mL×2 Dehydration/Concentration/Evaporation to dryness Derivatization — DMF 0.2 mL, BSTFA 0.2 mL, Room temp.: 30 min Alkali decomposition — 5% NaOH 2 mL, 70°C 1 hr Extraction — *n*-Hexane 2 mL, Suck up the hexane layer with a Pasteur pipette Cleanup — Sep-Pak Florisil, *n*-Hexane 2 mL (discard), 4% ether/hexane 5 mL (collection) Concentration — Until 0.5 mL in nitrogen stream GC/MS-SIM **Wildlife** Sample 10 g — Add surrogate Extraction — Methanol 30 mL×2, Homogenizer, Centrifuging Redissolution — 5% NaCl 100 mL, Dichloromethane 50 mL×2 Dehydration/dry and harden — Anhydrous Na_2SO_4 Derivatization — DMF 0.2 mL, BSTFA 1 mL, Room temperature 30 min Alkali decomposition — 5% NaOH 8 mL, 70°C 1hr Extraction — Hexane 20 mL Dehydration / dry and harden — Na_2SO_4 Hexane dissolution — 1 mL Cleanup — Florisil 7 g (internal diameter 1 cm), 4% Ether/hexane 40 mL (collection) Concentration — 0.5 mL GC/MS-SIM	GC/MS (SIM) Column: Ultra-2 (HP) Column length: 25 m Column I.D.: 0.32 mm Film thickness: 0.52 μm Detection limit: Surface water (μg/L) (3) 0.002 Bottom sediment (ng/g-dry) (3) 0.24 Wildlife (ng/g-wet) (3) 0.77

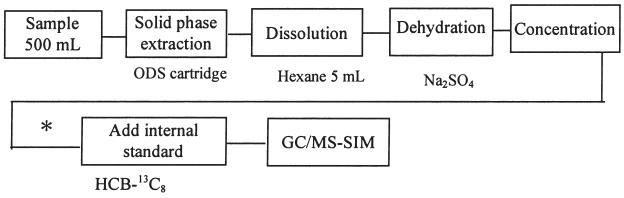
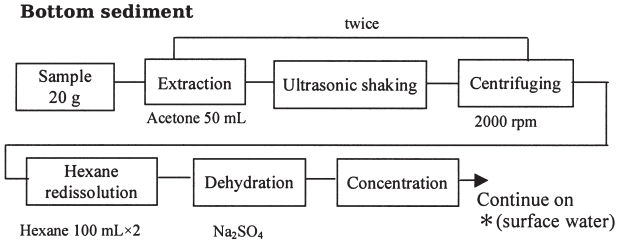
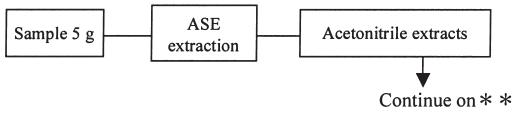
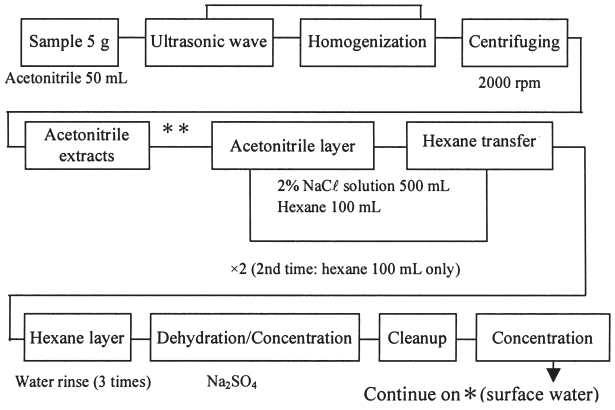
Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(4) Chlorodifluoromethane	**Air** Canister (6 L) → Collection (3.0 mL/min× 24 hrs) → Pressurized dilution → Low-temperature concentration (Entech 7000) → GC/MS-SIM	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) (4) 6
(5) *p*-Chloronitrobenzene	**Wildlife** Sample 10 g (NaCℓ 15 g, Purified water 500 mL, Surrogate solution, Hexane 5 mL) → Extraction by continuous steam distillation (Essential oil measuring apparatus) → Dehydration (Anhydrous Na_2SO_4) → Concentration → Column cleanup (Florisil cartridge column) → Concentration 1 mL (Internal standard solution) → GC/MS-SIM	GC/MS (SIM) Column: DB-17 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.5 μm Detection limit: Wildlife (ng/g-wet) (5) 7.8
(6) Dinitrotoluene	**Air** Sample (200 mL/min 24 hrs) → Adsorption tube sampling (Tenax TA (60 - 80 mesh) 200 mg) → Thermal desorption (ATD-400) → GC/MS-SIM	GC/MS (SIM) Column: HP Ultra-2 Column length: 25 m Column I.D.: 0.20 mm Film thickness: 0.33 μm Detection limit: Air (ng/m^3) (6) 2,4-Dinitrotoluene: 0.95 (6) 2,6-Dinitrotoluene: 0.89
(7) Methylbromide	**Surface water** Sample 10 - 100 mL (Add surrogate) → Vials (NaCℓ, Add internal standard) → Headspace GC/MS (Hermetical sealing, shaking, warming)	GC/MS (SIM) Column: Column length: 25 - 120 m Column I.D.: 0.2 - 0.75 mm Film thickness: 0.1 - 3.0 μm Detection limit: Surface water (μg/L) (7) 0.1

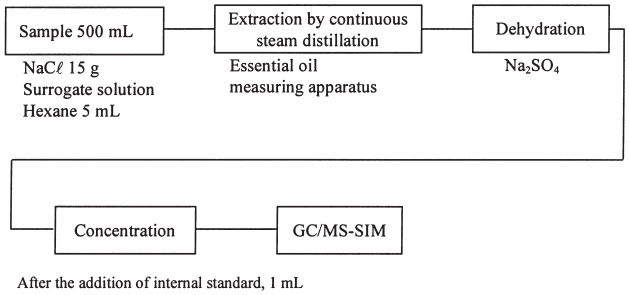
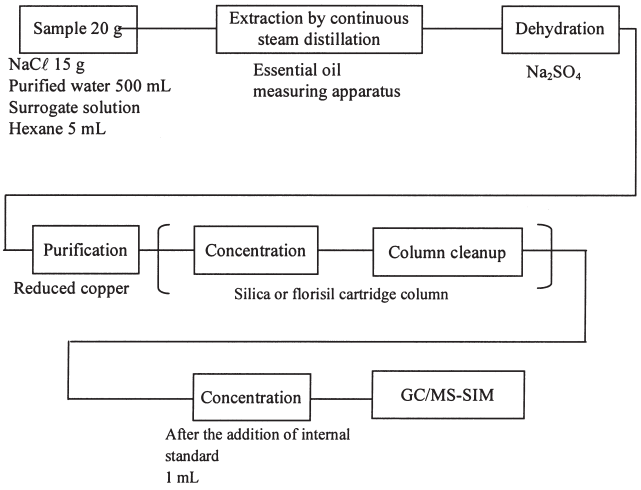
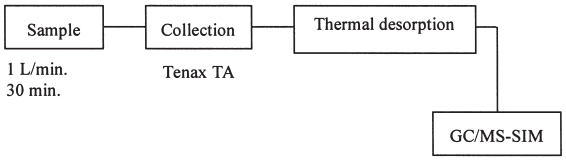
Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(8) Terephthalic acid	**Surface water**  **Bottom sediment**  	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) (8) 0.048 Bottom sediment (ng/g-dry) (8) 8.6

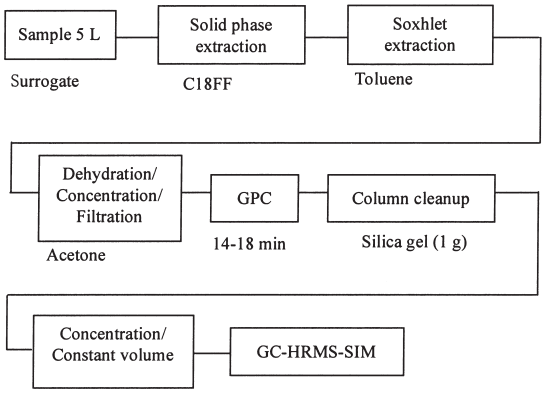
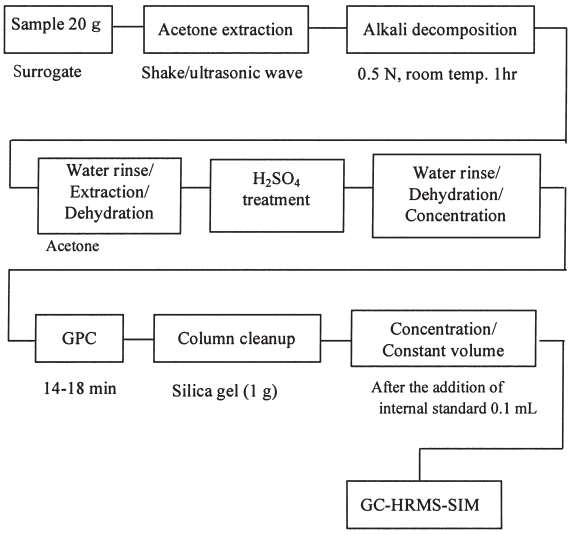
Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(9) 2,4,6-Tri-*tert*-butylphenol	**Surface water**  **Bottom sediment**  **Wildlife** ・ASE extraction→Acetonitrile extraction  ・Solvent extraction→Acetonitrile extraction ×2  	GC/MS (SIM) Column: Agilent Ultra-2 Column length: 25 m Column I.D.: 0.2 mm Film thickness: 0.33 μm Detection limit: Surface water (μg/L) (9) 0.020 Bottom sediment (ng/g-dry) (9) 6.5 Wildlife (ng/g-wet) ASE extraction (9) 20 Solvent extraction (9) 21

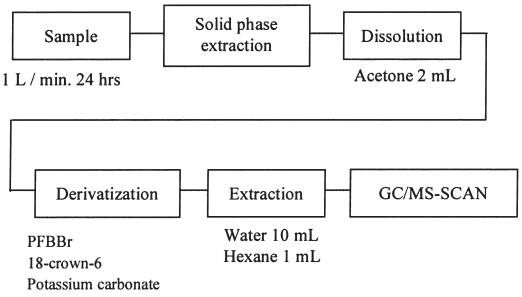
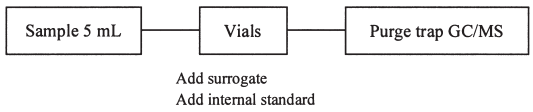
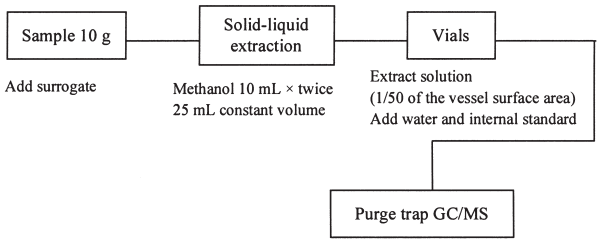
Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(10) Nitrobenzene	**Surface water**  **Bottom sediment**  **Air**  	GC/MS (SIM) Column: DB-17 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.5 μm Detection limit: Surface water (μg/L) (10) 0.037 Bottom sediment (ng/g-dry) (10) 1.4 GC/MS (SIM) Column: DB-17 Column length: 15 m Column I.D.: 0.53 mm Film thickness: 1.0 μm Detection limit: Air (ng/m^3) (10) 0.7

Analytical Method for the FY2002 Initial Environmental Survey (continued)

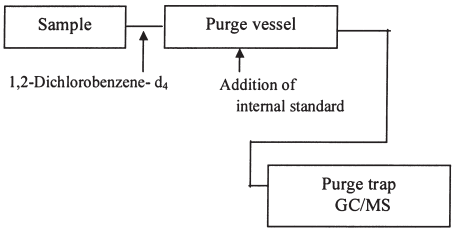
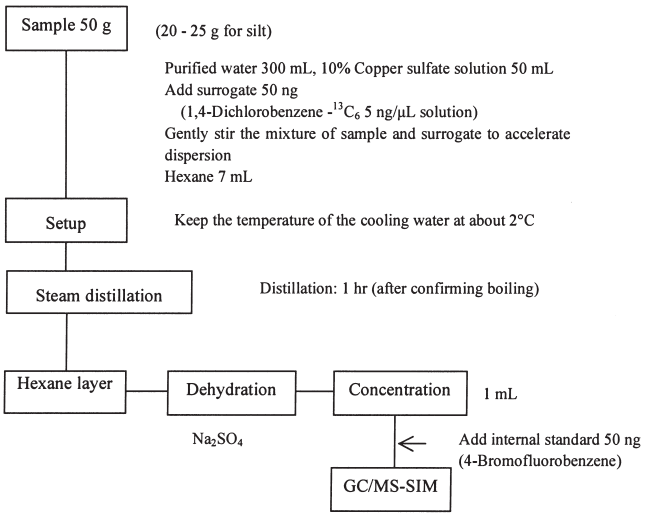
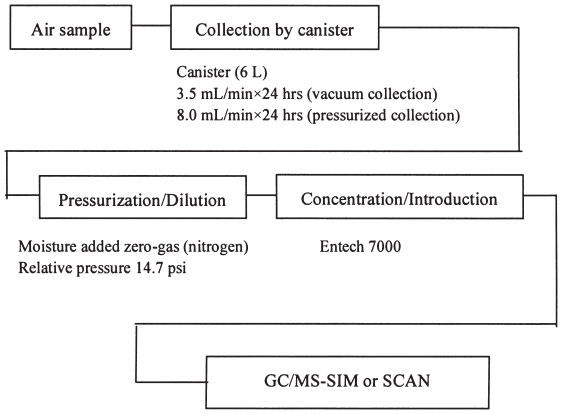
Substance	Analytical Method / Flow Chart	Remarks
(11) Polychlorinated terphenyls (PCT) (11-1) 4-Monochloro-o-terphenyl (11-2) 4-Monochloro-p-terphenyl (11-3) 2,5-Dichloro-o-terphenyl (11-4) 2,5-Dichloro-m-terphenyl (11-5) 2,4-Dichloro-*p*-terphenyl + 2,5-Dichloro-*p*-terphenyl (11-6) 2,4,6-Trichloro-*p*-terphenyl (11-7) 2,3,5,6-Tetrachloro -*p*-terphenyl (11-8) 2,4,4",6-Tetrachloro -*p*-terphenyl (11-9) 2,3,4,5,6-Pentachloro -*p*-terphenyl	**Surface water**  After the addition of internal standard 0.1 mL **Bottom sediment / Wildlife**  	GC-HRMS (SIM) Column: DB-5HT Column length: 15 m Column I.D.: 0.25 mm Film thickness: 0.1 μm Detection limit: Surface water (ng/L) (11-1) 0.023 (11-2) 0.013 (11-3) 0.021 (11-4) 0.016 (11-5) 0.023 (11-6) 0.022 (11-7) 0.024 (11-8) 0.026 (11-9) 0.024 Bottom sediment (ng/g-dry) (11-1) 0.029 (11-2) 0.019 (11-3) 0.019 (11-4) 0.019 (11-5) 0.021 (11-6) 0.0091 (11-7) 0.017 (11-8) 0.019 (11-9) 0.020 Wildlife (ng/g-wet) (11-1) 0.0078 (11-2) 0.026 (11-3) 0.016 (11-4) 0.016 (11-5) 0.016 (11-6) 0.0078 (11-7) 0.020 (11-8) 0.020 (11-9) 0.021

Analytical Method for the FY2002 Initial Environmental Survey (continued)

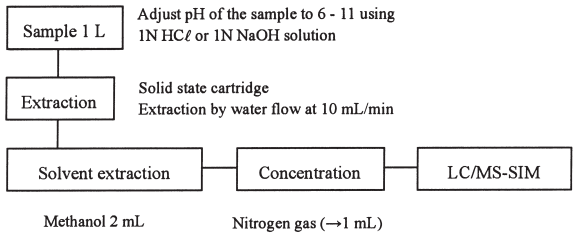
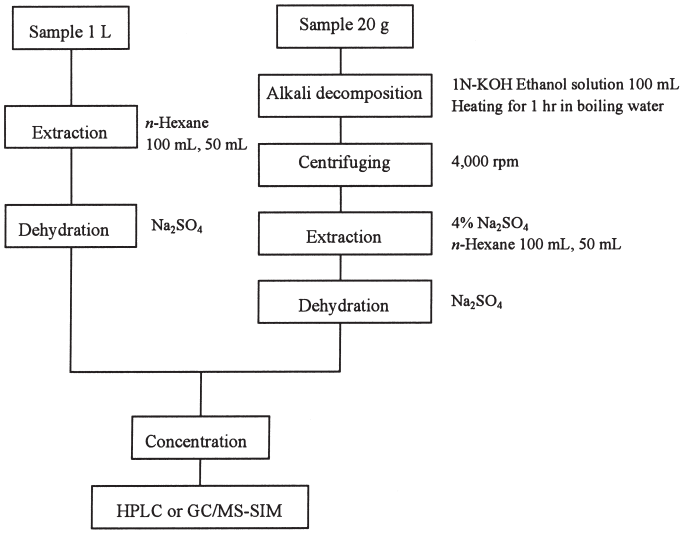
Substance	Analytical Method / Flow Chart	Remarks
(12) Metacrylic acid	**Air**  	GC/MS (SCAN) Column: HP-1MS Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection limit: Air (ng/m^3) (12) 0.77
(13) Methyl-*tert*-butylether	**Surface water**  	GC/MS (SIM) Column: DB-VRX Column length: 60 m Column I.D.: 0.25 mm Film thickness: 1.4 μm Detection limit: Surface water (μg/L) (13) 0.006
	Bottom sediment  	Bottom sediment (ng/g-dry) (13) 0.70

2. Environmental Survey for Exposure Study

Analytical Method for the FY2002 Environmental Survey for Exposure Study

Substance	Analytical Method / Flow Chart	Remarks
(1) 1,2-Dichlorobenzene (*o*-Dichlorobenzene)	**Surface water**  **Bottom sediment**  **Air**  	GC/MS-SIM Column: HP-5973 BPX-5 Column length: 60 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection limit Bottom sediment (ng/g-dry) (1) 0.02 GC/MS-SIM or SCAN Column: Hp-1 Column length: 60 m Column I.D.: 0.32 mm Film thickness: 1.0 μm Detection limit Air (ng/m^3) (1) 29

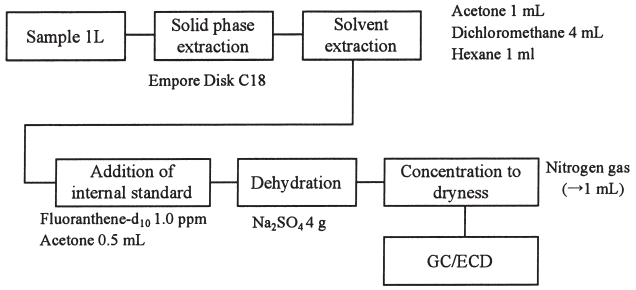
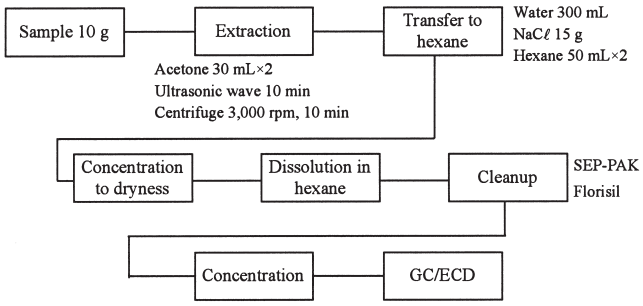
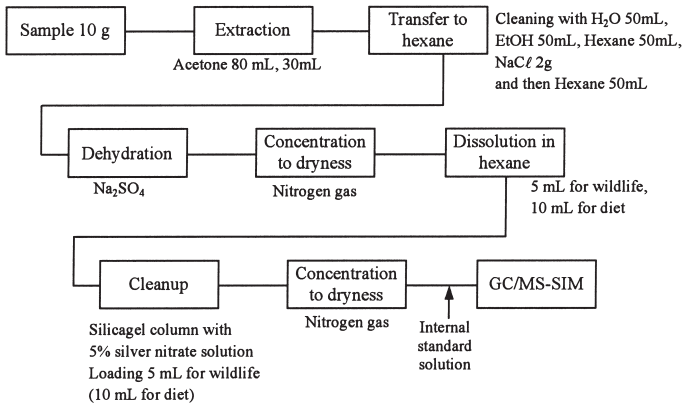
Analytical Method for the FY2002 Environmental Survey for Exposure Study (continued)

Substance	Analytical Method / Flow Chart	Remarks
(2) Perfluorooctane sulfonic acid (PFOS) (3) Perfluorooctanic acid (PFOA)	**Surface water**  	LC/MS-SIM Agilent 1100 Column: Zorbox XDBC-18 3.5 μm 2.1 mm x 150 mm Detection limit Surface water (ng/L) (2) 0.05
(4) Benzo[*a*]pyrene	**Surface water** **Bottom sediment / Wildlife**  	GC/MS-SIM Column: Ultra-2 Column length: 50 m Column I.D.: 0.31 mm Film thickness: 0.52 μm HPLC Column: Perkin Elmer PAH 2.6 mm x 250 mm Guard column: Nucleosil C18 4.6 mm×33 mm Detection limit Surface water (ng/L) (4) HPLC: 50 GC/MS: 60 Bottom sediment (ng/g-dry) / wildlife (ng/g-wet) (4) HPLC: 1 GC/MS: 3

Analytical Method for the FY2002 Environmental Survey for Exposure Study (continued)

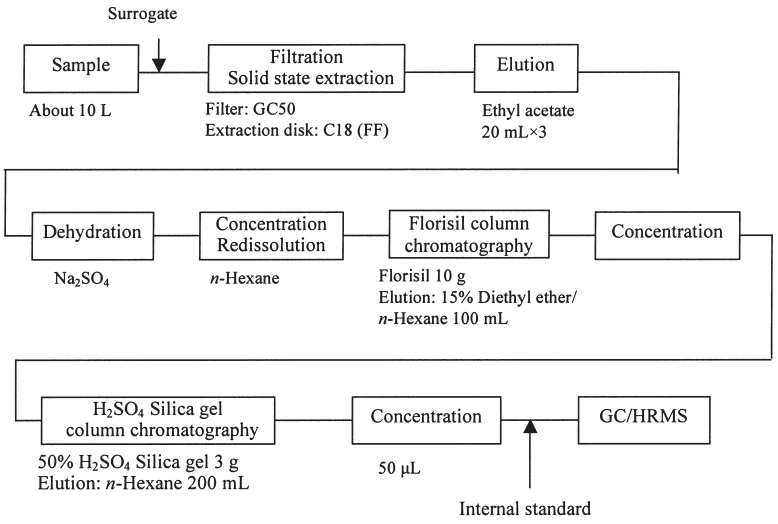
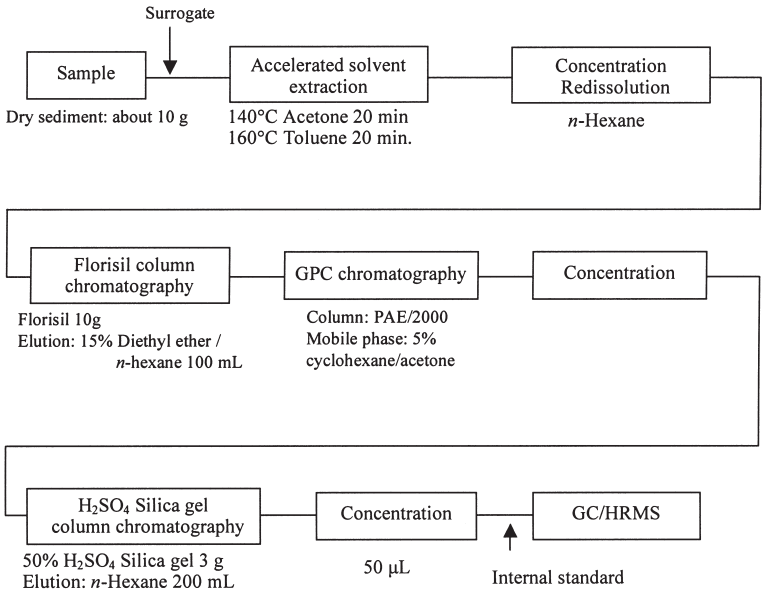
Substance	Analytical Method / Flow Chart	Remarks
(5) Polychlorinated naphthalene	**Wildlife / Diet** ([ ] for diet) Sample 20 g [10 g] → Extraction → Alkali decomposition Homogenization with Hexane, Na_2SO_4 Centrifuging 2,000 rpm, 5 min Extraction twice by hexane 1 mol/L KOH/Ethanol 25 mL 1 hr at room temperature H_2O treatment → Sulfuric acid treatment Dehydration / Concentration Na_2SO_4 Vacuum concentration 10 mL constant volume by acetone GPC CLNpak PAE-2000 Load 5 mL Aceton / cyclohexane (95:5) Column cleanup → Concentration → HRGC/MS-SIM Florisil cartridge column Nitrogen gas, 1mL Add internal standard solution **Air** Air sample High-volume air sampler 1,000 m^3 Particulate: Quartz microfiber filter (QMF) → Soxhlet extraction → Acetone, 24 hrs → Transfer to hexane → Sulfuric acid treatment → Column cleanup → GC/MS-SIM Vapor phase: Polyurethane foam (PUF) → Soxhlet extraction → Hexane, 24 hrs → Sulfuric acid treatment → Column cleanup → GC/MS-SIM	HRGC/MS-SIM Resolution: 10,000 Column: J&W DB-5MS Column length: 60 m Column I.D.: 0.32 mm Film thickness: 0.25 μm Detection limit Wildlife (pg/g-wet) (5) Monochloride: 1 Dichloride: 0.5 Trichloride: 0.5 Tetrachloride: 0.6 Pentachloride: 0.9 Hexachloride: 0.7 Heptachloride: 0.9 Octachloride: 0.8 Diet (pg/g-fresh weight) (5) Monochloride: 5 Dichloride: 1 Trichloride: 1 Tetrachloride: 1 Pentachloride: 1 Hexachloride: 1 Heptachloride: 1 Octachloride: 1 GC/MS-SIM JEOL JMS-700 Column: HP Ultra-2 Column length: 25 m Column I.D.: 0.20 mm Film thickness: 0.33 μm Detection limit Air (pg/m^3) (5) Monochloride: 0.01 Dichloride: 0.04 Trichloride: 0.04 Tetrachloride: 0.03 Pentachloride: 0.1 Hexachloride: 0.1 Heptachloride: 0.1 Octachloride: 0.2

FY2002 Environmental Survey for Exposure Study (continued)

Substance	Analytical Method / Flow Chart	Remarks
(6) Polybrominated diphenylether (6-1) Octabromodiphenyl ether (6-2) Decabromodiphenyl ether	**Surface water**  **Bottom sediment**  **Wildlife / Diet**  	GC/ECD Column: Hp-5890II DB1 Column length: 5 m Column I.D.: 0.32 mm Film thickness: 0.1 μm Detection limit Surface water (ng/L) (6-2) 120 Bottom sediment (ng/g-dry) (6-2) 9.7 Wildlife (ng/g-wet) (6-1) 0.20 Diet (ng/g-fresh weight) (6-2) 0.5

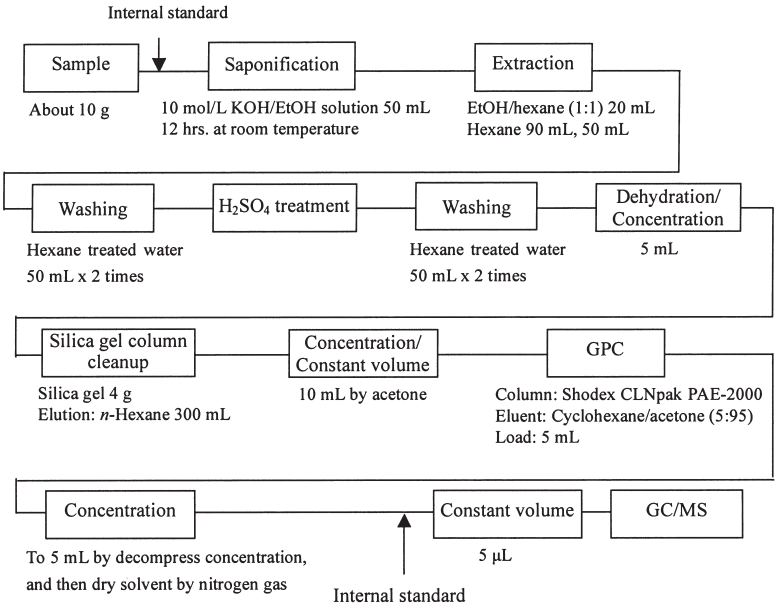
3. Monitoring Investigation

Analytical Method for the FY2002 Monitoring Investigation

Substance	Analytical Method / Flow Chart	Remarks
(1) PCBs	**Surface water**  **Bottom sediment**  	GC/MS 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 0.06 / 0.12 Di-chloride 0.2 / 0.6 Tri-chloride 0.3 / 0.9 Tetra-chloride 0.3 / 0.9 Penta-chloride 0.2 / 0.6 Hexa-chloride 0.3 / 0.9 Hepta-chloride 0.2 / 0.6 Octa-chloride 0.3 / 0.9 Nona-chloride 0.3 / 0.9 Deca-chloride 0.3 / 0.9 Bottom sediment (pg/g-dry) (1) Detection limit / Quantitation limit Mono-chloride 0.06 / 0.12 Di-chloride 0.2 / 0.6 Tri-chloride 0.3 / 0.9 Tetra-chloride 0.3 / 0.9 Penta-chloride 0.2 / 0.6 Hexa-chloride 0.3 / 0.9 Hepta-chloride 0.2 / 0.6 Octa-chloride 0.3 / 0.9 Nona-chloride 0.3 / 0.9 Deca-chloride 0.3 / 0.9

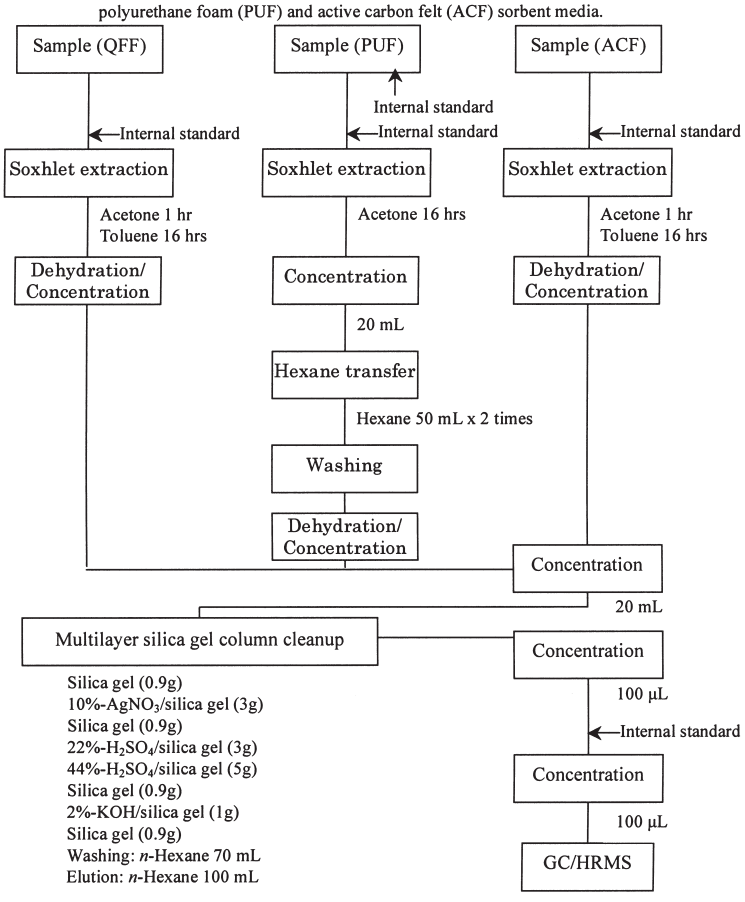
Analytical Method for the FY2002 Monitoring Investigation

Substance	Analytical Method / Flow Chart	Remarks
(1) PCBs (continued)	**Wildlife**	GC/MS Column: HT8-PCB Column length: 50 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection/Quantitation limit Wildlife (pg/g-wet)



(1)	Detection limit	Quantitation limit
Mono-chloride	0.7	2.1
Di-chloride	0.9	2.7
Tri-chloride	0.8	2.4
Tetra-chloride	1.0	3.0
Penta-chloride	1.0	3.0
Hexa-chloride	1.0	3.0
Hepta-chloride	1.0	3.0
Octa-chloride	1.0	3.0
Nona-chloride	0.6	1.8
Deca-chloride	0.4	1.2

Air Sampled by high-volume air sampler (HV) with quartz-fiber-filter (QFF), polyurethane foam (PUF) and active carbon felt (ACF) sorbent media.

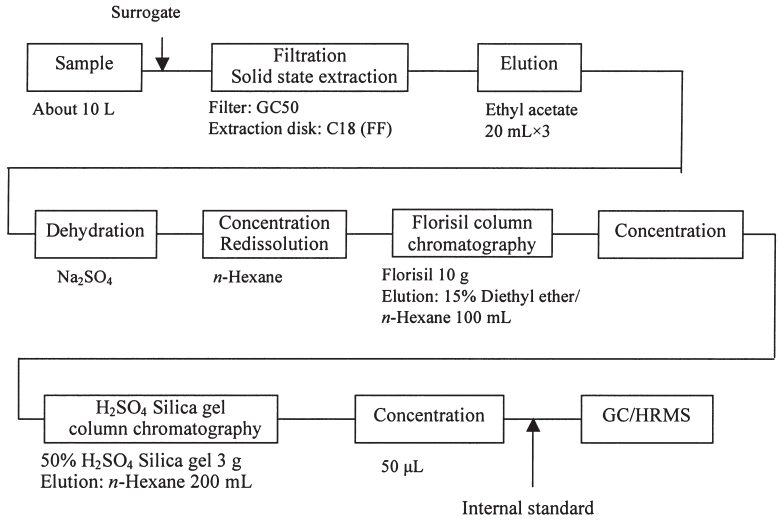
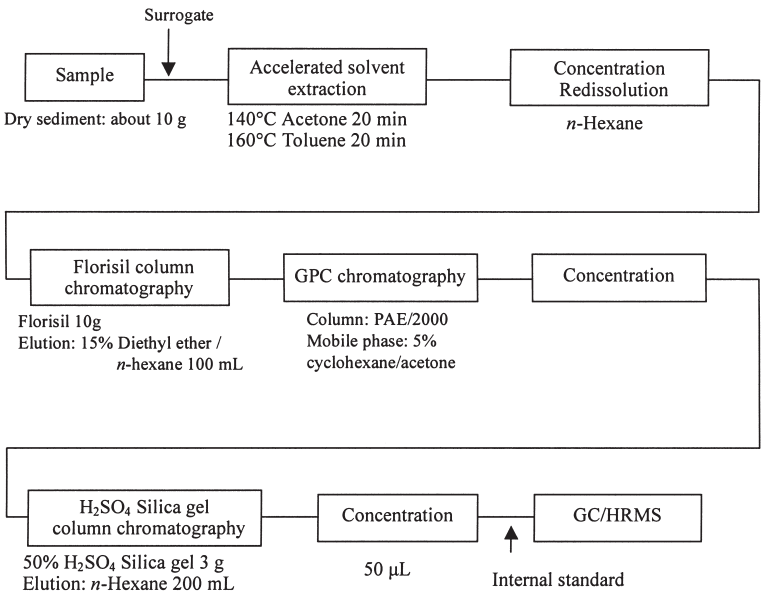


GC/HRMS
Resolution: 10,000
Column: DB-5MS
Column length: 60 m
Column I.D.: 0.32 mm
Film thickness: 0.25 μm

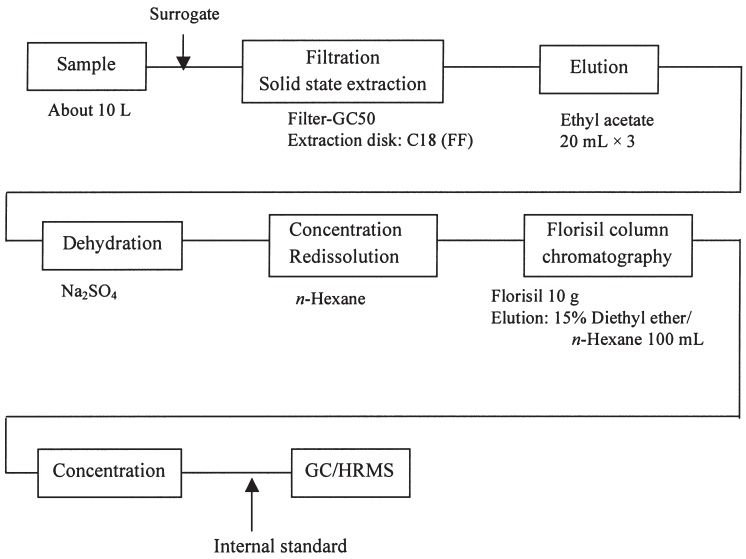
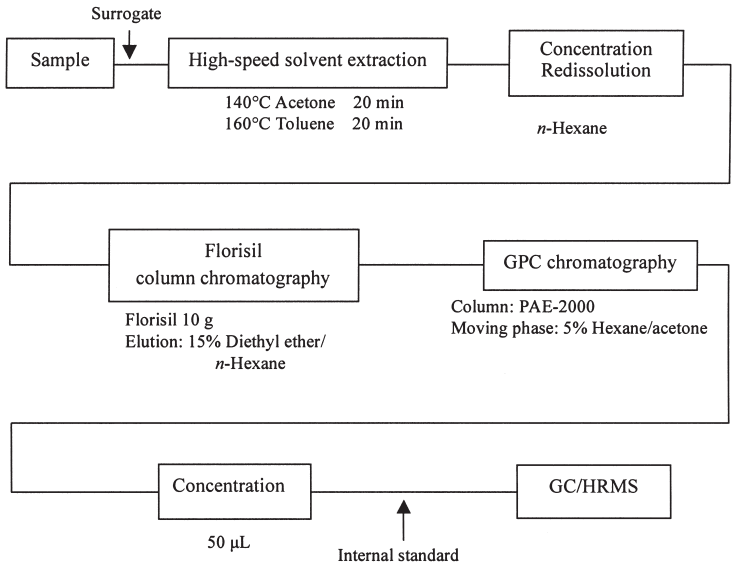
Detection/Quantitation limit
Air (pg/m^3)

(1)	Detection limit	Quantitation limit
Mono-chloride	30	90
Di-chloride	1.0	3.0
Tri-chloride	0.5	1.5
Tetra-chloride	0.9	2.7
Penta-chloride	0.4	1.2
Hexa-chloride	0.2	0.6
Hepta-chloride	0.007	0.021
Octa-chloride	0.01	0.03
Nona-chloride	0.01	0.03
Deca-chloride	0.005	0.015

Analytical Method for the FY2002 Monitoring Investigation

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) Heptachlor (7) HCH (7-1) α-HCH (7-2) β-HCH	**Surface water**  **Bottom sediment**  	GC/MS Column: HT8-PCB Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm Column: RH Column length: 30 m Column I.D.: 0.25 mm Film thickness: 250nm Detection/Quantitation limit Surface water (pg/L) (2) 0.2 / 0.6 (4-1) 0.2 / 0.6 (4-2) 0.2 / 0.6 (4-3) 0.08 / 0.24 (4-4) 0.4 / 1.2 (4-5) 0.3 / 0.9 (4-6) 0.2 / 0.6 (5-1) 0.5 / 1.5 (5-2) 0.3 / 0.9 (5-3) 0.4 / 1.2 (5-4) 0.6 / 1.8 (5-5) 0.4 / 1.2 (6) 0.5 / 1.5 (7-1) 0.3 / 0.9 (7-2) 0.3 / 0.9 Bottom sediment (pg/g-dry) (2) 0.3 / 0.9 (4-1) 2 / 6 (4-2) 0.9 / 2.7 (4-3) 0.8 / 2.4 (4-4) 2 / 6 (4-5) 1 / 3 (4-6) 2 / 6 (5-1) 0.6 / 1.8 (5-2) 0.3 / 0.9 (5-3) 0.5 / 1.5 (5-4) 0.7 / 2.1 (5-5) 0.5 / 1.5 (6) 0.6 / 1.8 (7-1) 0.4 / 1.2 (7-2) 0.3 / 0.9 (Detection limit / Quantitation limit)

Analytical Method for the FY2002 Monitoring Investigation

Substance	Analytical Method / Flow Chart	Remarks
(3) Drins (3-1) Aldrin (3-2) Dieldrin (3-3) Endrin	**Surface water**  **Bottom sediment**  	GC/MS Column: HT8-PCB Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm Column: RH17 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 250 nm Detection/Quantitation limit Surface water (pg/L) (3-1) Detection limit 0.2, Quantitation limit 0.6 (3-2) Detection limit 0.6, Quantitation limit 1.8 (3-3) Detection limit 2, Quantitation limit 6 Bottom sediment (pg/g-dry) (3-1) Detection limit 2, Quantitation limit 6 (3-2) Detection limit 1, Quantitation limit 3 (3-3) Detection limit 2, Quantitation limit 6

Analytical Method for the FY2002 Monitoring Investigation

Substance	Analytical Method / Flow Chart	Remarks

Substance

(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

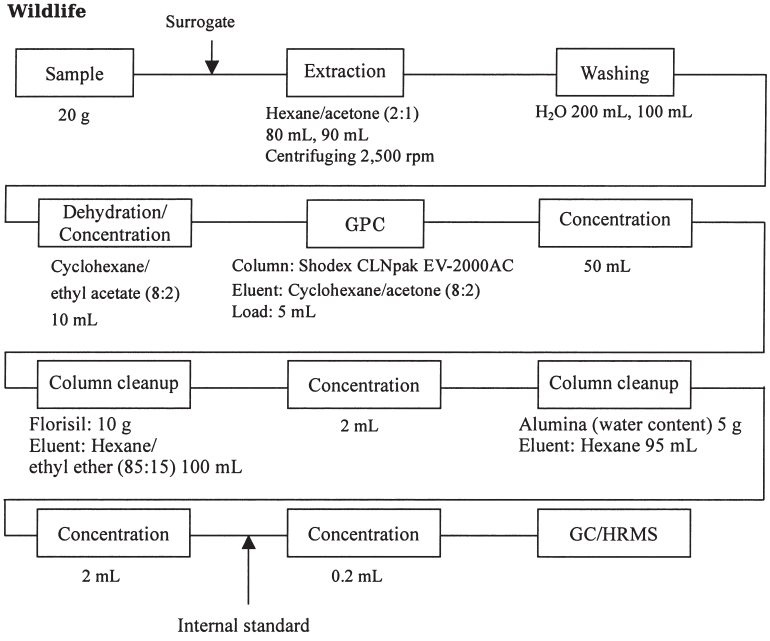
(5) Chlordanes
- (5-1) *trans*-Chlordane
- (5-2) *cis*-Chlordane
- (5-3) *trans*-Nonachlor
- (5-4) *cis*-Nonachlor
- (5-5) Oxychlordane

(6) Heptachlor

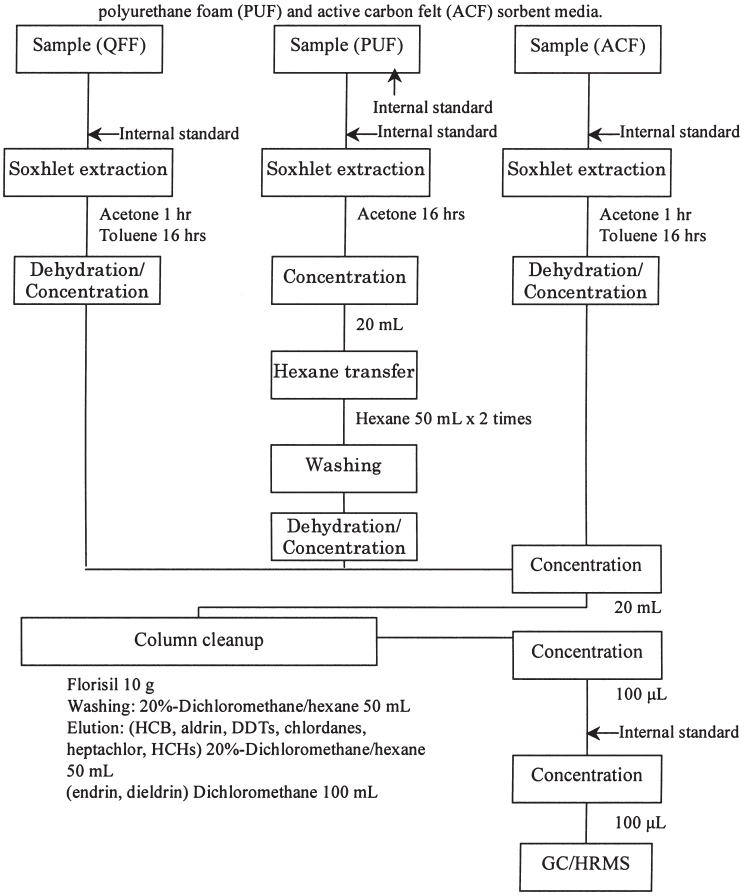
(7) HCH
- (7-1) *α*-HCH
- (7-2) *β*-HCH

Analytical Method / Flow Chart

Wildlife



Air Sampled by high-volume air sampler (HV) with quartz-fiber-filter (QFF), polyurethane foam (PUF) and active carbon felt (ACF) sorbent media.



Remarks

GC/MS
Column: DB-5MS
Column length: 60 m
Column I.D.: 0.25 mm
Film thickness: 0.25 μm

Detection/Quantitation limit
Wildlife (pg/g-wet)

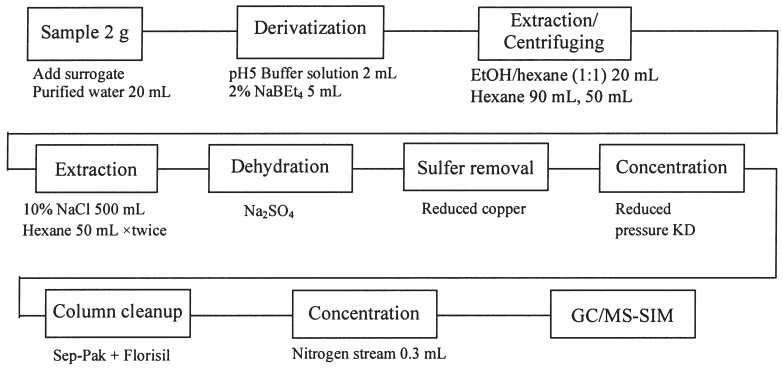
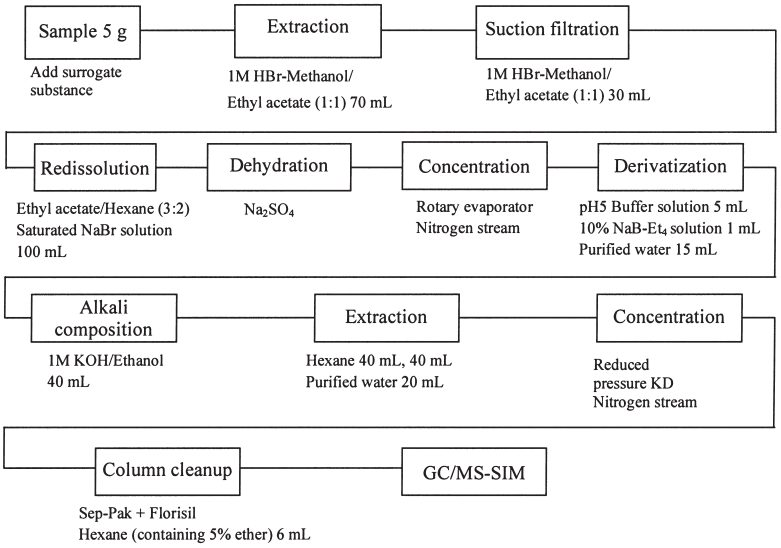
	Detection limit	Quantitation limit
(2)	0.2	0.6
(3-1)	1.4	4.2
(3-2)	4	12
(3-3)	6	18
(4-1)	0.2	0.6
(4-2)	0.2	0.6
(4-3)	0.08	0.24
(4-4)	0.4	1.2
(4-5)	0.3	0.9
(4-6)	0.2	0.6
(5-1)	0.5	1.5
(5-2)	0.3	0.9
(5-3)	0.4	1.2
(5-4)	0.6	1.8
(5-5)	0.4	1.2
(6)	0.5	1.5
(7-1)	0.3	0.9
(7-2)	0.3	0.9

GC/HRMS
Column: DB-17HT
Column length: 30 m
Column I.D.: 0.32 mm
Film thickness: 0.15μm

Detection/Quantitation limit
Air (pg/m^3)

	Detection limit	Quantitation limit
(2)	0.3	0.9
(3-1)	0.02	0.06
(3-2)	0.2	0.6
(3-3)	0.03	0.09
(4-1)	0.08	0.24
(4-2)	0.03	0.09
(4-3)	0.006	0.018
(4-4)	0.05	0.15
(4-5)	0.01	0.03
(4-6)	0.007	0.021
(5-1)	0.2	0.6
(5-2)	0.2	0.6
(5-3)	0.1	0.3
(5-4)	0.01	0.03
(5-5)	0.008	0.024
(6)	0.04	0.12
(7-1)	0.2	0.6
(7-2)	0.03	0.09

Analytical Method for the FY2002 Monitoring Investigation

Substance	Analytical Method / Flow Chart	Remarks
(8) Organotin compounds (8-1) TBT (8-2) TPT	**Bottom sediment**  **Wildlife**  	GC/MS (quadrupole type) Column: Capillary column Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 μm Detection/Quantitation limit Bottom sediment (ng/g-dry) (8-1) Detection limit 0.27, Quantitation limit 0.81 (8-2) Detection limit 0.11, Quantitation limit 0.33 Detection/Quantitation limit Wildlife (ng/g-wet) (8-1) Detection limit 1.3, Quantitation limit 3.9 (8-2) Detection limit 0.16, Quantitation limit 0.48