

Appendix D

Summary of Analytical Methods for Environmental Surveys

Appendix D Summary of Analytical Methods for Environmental Surveys

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 $20 \pm 5^{\circ}\text{C}$.

<Result>

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

The combinations of experiments are shown below.

pH	Initial concentration ($\mu\text{g/mL}$)	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.

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

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.

$$\text{Detection limit (DL)} = t(n-1, 0.01) \cdot S_c$$

S_c : 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	<p>Surface water</p> <pre> graph LR S1[Sample] --> V[Vials] --> H1[Headspace GC/MS] S1 --- S1T[Add surrogate substance] V --- V1[NaCl, Add internal standard substance] H1 --- H1T[Hermetical sealing, shaking, warming] </pre> <p>Bottom sediment</p> <pre> graph LR S2[Sample] --> SLE[Solid-liquid extraction] --> P[Purge] --> H2[Headspace GC/MS] S2 --- S2T[Add surrogate] SLE --- SLE1[Methanol] P --- P1[NaCl, water, Add surrogate] </pre>	<p>GC/MS</p> <p>Column: Column length: 25 - 120 m Column I.D.: 0.2 - 0.75 mm Film thickness: 0.1 - 3.0 µm</p> <p>Detection limit: Surface water (µg/L) (1) 0.1</p> <p>Bottom sediment (ng/g-dry) (1) 10</p>
(2) Epichlorohydrin	<p>Air</p> <pre> graph LR S3[Sample] --> ATS[Adsorption tube sampling] --> TD[Thermal desorption] --> GMSIM[GC/MS-SIM] S3 --- S3T[80 mL/min, 5 hrs] </pre>	<p>GC/MS (SIM)</p> <p>Column: HP-VOC Column length: 60 m Column I.D.: 0.32 mm Film thickness: 1.0 µm</p> <p>Detection limit: Air (ng/m³) (2) 0.14</p>

Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(3) 1-Octanol	<p>Surface water</p> <pre> graph LR A[Sample 1,000 mL] --> B[Extraction] B --> C[Dehydration/Concentration/ Evaporation to dryness] C --> D[Derivatization] D --> E[Extraction] E --> F[Dehydration/Concentration] F --> G[GC/MS-SIM] </pre> <p>Add surrogate</p> <p>NaCl 50 g Dicyclomethane 50 mL×2</p> <p>Anhydrous Na₂SO₄ KD Evaporator Nitrogen stream</p> <p>DMF 0.2 mL BSTFA 0.2 mL Room temp.: 30 min</p> <p>5% NaOH 2 mL <i>n</i>-Hexane 2 mL</p> <p>Suck up the hexane layer with a Pasteur pipette and concentrate to 0.5 mL in nitrogen stream</p> <p>Bottom sediment</p> <pre> graph LR H[Sample 10 g] --> I[Extraction] I --> J[Redissolution] J --> K[Dehydration/Concentration/ Evaporation to dryness] K --> L[Derivatization] L --> M[Alkali decomposition] M --> N[Extraction] N --> O[Cleanup] O --> P[Concentration] P --> Q[GC/MS-SIM] </pre> <p>NaCl 15 g Purified water 500 mL Surrogate solution Hexane 5 mL</p> <p>Methanol 30 mL×2 Ultrasonic wave 10 min. Centrifugation 3000 rpm 10 min.</p> <p>5% NaCl 100 mL Dicyclomethane 50 mL×2</p> <p>DMF 0.2 mL BSTFA 0.2 mL Room temp.: 30 min</p> <p>5% NaOH 2 mL 70°C 1 hr</p> <p><i>n</i>-Hexane 2 mL Suck up the hexane layer with a Pasteur pipette</p> <p>Sep-Pak Florisil <i>n</i>-Hexane 2 mL (discard) 4% ether/hexane 5 mL (collection)</p> <p>Until 0.5 mL in nitrogen stream</p> <p>Wildlife</p> <pre> graph LR R[Sample 10 g] --> S[Extraction] S --> T[Redissolution] T --> U[Dehydration/ dry and harden] U --> V[Derivatization] V --> W[Alkali decomposition] W --> X[Extraction] X --> Y[Dehydration / dry and harden] Y --> Z[Hexane dissolution] Z --> AA[Cleanup] AA --> AB[Concentration] AB --> AC[GC/MS-SIM] </pre> <p>Add surrogate</p> <p>Methanol 30 mL×2 Homogenizer Centrifuging</p> <p>5% NaCl 100 mL Dichloromethane 50 mL×2</p> <p>Anhydrous Na₂SO₄</p> <p>DMF 0.2 mL BSTFA 1 mL Room temperature 30 min</p> <p>5% NaOH 8 mL 70°C 1hr</p> <p>Hexane 20 mL</p> <p>Na₂SO₄</p> <p>1 mL</p> <p>Florisil 7 g (internal diameter 1 cm) 4% Ether/hexane 40 mL (collection)</p> <p>0.5 mL</p>	<p>GC/MS (SIM)</p> <p>Column: Ultra-2 (HP)</p> <p>Column length: 25 m</p> <p>Column I.D.: 0.32 mm</p> <p>Film thickness: 0.52 μm</p> <p>Detection limit:</p> <p>Surface water (μg/L)</p> <p>(3) 0.002</p> <p>Bottom sediment (ng/g-dry)</p> <p>(3) 0.24</p> <p>Wildlife (ng/g-wet)</p> <p>(3) 0.77</p>

Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(4) Chlorodifluoromethane	<p>Air</p> <pre> graph LR A[Canister 6 L] --> B[Collection 3.0 mL/min x 24 hrs] B --> C[Pressurized dilution] C --> D[Low-temperature concentration Entech 7000] D --> E[GC/MS-SIM] </pre>	<p>GC/MS (SIM) Column: HP-VOC Column length: 60 m Column I.D.: 0.32 mm Film thickness: 1.8 µm</p> <p>Detection limit: Air (ng/m³) (4) 6</p>
(5) <i>p</i> -Chloronitrobenzene	<p>Wildlife</p> <pre> graph LR A[Sample 10 g] --> B[Extraction by continuous steam distillation NaCl 15 g Purified water 500 mL Surrogate solution Hexane 5 mL Essential oil measuring apparatus] B --> C[Dehydration Anhydrous Na2SO4] C --> D[Concentration] D --> E[Column cleanup Florisil cartridge column] E --> F[Concentration 1 mL Internal standard solution] F --> G[GC/MS-SIM] </pre>	<p>GC/MS (SIM) Column: DB-17 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.5 µm</p> <p>Detection limit: Wildlife (ng/g-wet) (5) 7.8</p>
(6) Dinitrotoluene	<p>Air</p> <pre> graph LR A[Sample] --> B[Adsorption tube sampling 200 mL/min 24 hrs Tenax TA 60 - 80 mesh 200 mg] B --> C[Thermal desorption ATD-400] C --> D[GC/MS-SIM] </pre>	<p>GC/MS (SIM) Column: HP Ultra-2 Column length: 25 m Column I.D.: 0.20 mm Film thickness: 0.33 µm</p> <p>Detection limit: Air (ng/m³) (6) 2,4-Dinitrotoluene: 0.95 (6) 2,6-Dinitrotoluene: 0.89</p>
(7) Methylbromide	<p>Surface water</p> <pre> graph LR A[Sample 10 - 100 mL Add surrogate] --> B[Vials NaCl, Add internal standard] B --> C[Headspace GC/MS Hermetical sealing, shaking, warming] C --> D[GC/MS-SIM] </pre>	<p>GC/MS (SIM) Column: Column length: 25 - 120 m Column I.D.: 0.2 - 0.75 mm Film thickness: 0.1 - 3.0 µm</p> <p>Detection limit: Surface water (µg/L) (7) 0.1</p>

Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(8) Terephthalic acid	<p>Surface water</p> <pre> graph LR A[Sample 500 mL Adjust pH to 1.0] --> B[PS-2 Pass through] B --> C[Leaching Ethyl acetate 5 mL] C --> D[Acylation 2% PFBB, K2CO3 80°C, 60 min.] D --> E[Extraction Hexane 1 mL p-Terphenyl-d14 0.1 µg] E --> F[GC/MS-SIM] </pre> <p>Bottom sediment</p> <pre> graph LR G[Sample 5 g] --> H[Extraction twice 0.05N HCl 30% water-methanol solution 20 mL Shaking 5 min. Ultrasonic wave 10 min.] H --> I[Centrifuging 3000 rpm 10 min.] I --> J[Concentration To about 10 mL] J --> K[Water layer Milli-Q water 150 mL pH 1.0] K --> L[PS-2 Pass through] L --> M[Leaching Ethyl acetate 5 mL] M --> N[Acylation 2% PFBB, K2CO3 80°C, 60 min.] N --> O[Dissolution Hexane 1 mL] O --> P[Silica cartridge 0.5% Ethyl acetate/hexane 10 mL rinse 5% Ethyl acetate/hexane 10 mL leaching] P --> Q[GC/MS-SIM p-Terphenyl-d14 0.1 µg] </pre>	<p>GC/MS (SIM) Column: HP-5 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm</p> <p>Detection limit: Surface water (µg/L) (8) 0.048</p> <p>Bottom sediment (ng/g-dry) (8) 8.6</p>

Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(9) 2,4,6-Tri- <i>tert</i> -butylphenol	<p>Surface water</p> <pre> graph LR A[Sample 500 mL] --> B[Solid phase extraction ODS cartridge] B --> C[Dissolution Hexane 5 mL] C --> D[Dehydration Na2SO4] D --> E[Concentration] E --> F[Add internal standard HCB-13C8] F --> G[GC/MS-SIM] </pre> <p>Bottom sediment</p> <pre> graph LR A[Sample 20 g] --> B[Extraction Acetone 50 mL] B --> C[Ultrasonic shaking] C --> D[Centrifuging 2000 rpm] D --> E[Hexane redissolution Hexane 100 mL x 2] E --> F[Dehydration Na2SO4] F --> G[Concentration] G --> H[Continue on * (surface water)] </pre> <p>Wildlife</p> <ul style="list-style-type: none"> • ASE extraction → Acetonitrile extraction <pre> graph LR A[Sample 5 g] --> B[ASE extraction] B --> C[Acetonitrile extracts] C --> D[Continue on * *] </pre> <ul style="list-style-type: none"> • Solvent extraction → Acetonitrile extraction × 2 <pre> graph LR A[Sample 5 g] --> B[Ultrasonic wave Acetonitrile 50 mL] B --> C[Homogenization] C --> D[Centrifuging 2000 rpm] D --> E[Acetonitrile extracts] E --> F[Acetonitrile layer 2% NaCl solution 500 mL Hexane 100 mL] F --> G[Hexane transfer] G --> H[Hexane layer Water rinse 3 times] H --> I[Dehydration/Concentration Na2SO4] I --> J[Cleanup] J --> K[Concentration] K --> L[Continue on * (surface water)] </pre>	<p>GC/MS (SIM) Column: Agilent Ultra-2 Column length: 25 m Column I.D.: 0.2 mm Film thickness: 0.33 μm</p> <p>Detection limit: Surface water (μg/L) (9) 0.020</p> <p>Bottom sediment (ng/g-dry) (9) 6.5</p> <p>Wildlife (ng/g-wet) ASE extraction (9) 20</p> <p>Solvent extraction (9) 21</p>

Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(10) Nitrobenzene	<p>Surface water</p> <pre> graph LR A[Sample 500 mL NaCl 15 g Surrogate solution Hexane 5 mL] --> B[Extraction by continuous steam distillation Essential oil measuring apparatus] B --> C[Dehydration Na2SO4] C --> D[Concentration] D --> E[GC/MS-SIM] </pre> <p>After the addition of internal standard, 1 mL</p> <p>Bottom sediment</p> <pre> graph LR A[Sample 20 g NaCl 15 g Purified water 500 mL Surrogate solution Hexane 5 mL] --> B[Extraction by continuous steam distillation Essential oil measuring apparatus] B --> C[Dehydration Na2SO4] C --> D[Purification Reduced copper] D --> E[Concentration] E --> F[Column cleanup Silica or florisil cartridge column] F --> G[Concentration] G --> H[GC/MS-SIM] </pre> <p>After the addition of internal standard 1 mL</p> <p>Air</p> <pre> graph LR A[Sample 1 L/min. 30 min.] --> B[Collection Tenax TA] B --> C[Thermal desorption] C --> D[GC/MS-SIM] </pre>	<p>GC/MS (SIM) Column: DB-17 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.5 µm</p> <p>Detection limit: Surface water (µg/L) (10) 0.037</p> <p>Bottom sediment (ng/g-dry) (10) 1.4</p> <p>GC/MS (SIM) Column: DB-17 Column length: 15 m Column I.D.: 0.53 mm Film thickness: 1.0 µm</p> <p>Detection limit: Air (ng/m³) (10) 0.7</p>

Analytical Method for the FY2002 Initial Environmental Survey (continued)

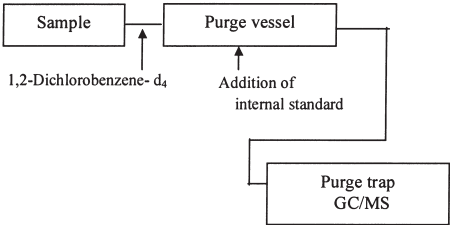
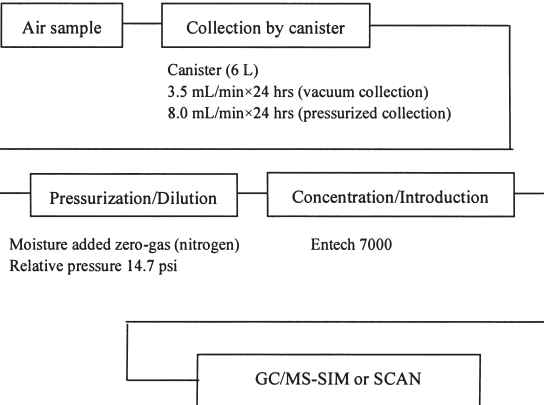
Substance	Analytical Method / Flow Chart	Remarks
<p>(11) Polychlorinated terphenyls (PCT)</p> <p>(11-1) 4-Monochloro-o-terphenyl</p> <p>(11-2) 4-Monochloro-p-terphenyl</p> <p>(11-3) 2,5-Dichloro-o-terphenyl</p> <p>(11-4) 2,5-Dichloro-m-terphenyl</p> <p>(11-5) 2,4-Dichloro-p-terphenyl + 2,5-Dichloro-p-terphenyl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

Analytical Method for the FY2002 Initial Environmental Survey (continued)

Substance	Analytical Method / Flow Chart	Remarks
(12) Metacrylic acid	<p>Air</p> <pre> graph LR Sample[Sample] -- "1 L / min. 24 hrs" --> SPE[Solid phase extraction] SPE --> Diss[Dissolution] Diss -- "Acetone 2 mL" --> Der[Derivatization] Der --> Ext[Extraction] Ext -- "Water 10 mL Hexane 1 mL" --> GC[GC/MS-SCAN] </pre> <p>PFBBR 18-crown-6 Potassium carbonate</p>	<p>GC/MS (SCAN) Column: HP-1MS Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm</p> <p>Detection limit: Air (ng/m³) (12) 0.77</p>
(13) Methyl- <i>tert</i> -butylether	<p>Surface water</p> <pre> graph LR Sample[Sample 5 mL] --> Vials[Vials] Vials -- "Add surrogate Add internal standard" --> GC[Purge trap GC/MS] </pre> <p>Bottom sediment</p> <pre> graph LR Sample[Sample 10 g] -- "Add surrogate" --> SLE[Solid-liquid extraction] SLE -- "Methanol 10 mL x twice 25 mL constant volume" --> Vials[Vials] Vials -- "Extract solution (1/50 of the vessel surface area) Add water and internal standard" --> GC[Purge trap GC/MS] </pre>	<p>GC/MS (SIM) Column: DB-VRX Column length: 60 m Column I.D.: 0.25 mm Film thickness: 1.4 µm</p> <p>Detection limit: Surface water (µg/L) (13) 0.006</p> <p>Bottom sediment (ng/g-dry) (13) 0.70</p>

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)	<p>Surface water</p>  <p>Bottom sediment</p> <p>Sample 50 g (20 - 25 g for silt)</p> <p>Purified water 300 mL, 10% Copper sulfate solution 50 mL Add surrogate 50 ng (1,4-Dichlorobenzene -¹³C₆ 5 ng/μL solution) Gently stir the mixture of sample and surrogate to accelerate dispersion Hexane 7 mL</p> <p>Setup Keep the temperature of the cooling water at about 2°C</p> <p>Steam distillation Distillation: 1 hr (after confirming boiling)</p> <p>Hexane layer → Dehydration (Na₂SO₄) → Concentration (1 mL) → GC/MS-SIM</p> <p>Add internal standard 50 ng (4-Bromofluorobenzene)</p> <p>Air</p> 	<p>GC/MS-SIM</p> <p>Column: HP-5973 BPX-5 Column length: 60 m Column I.D.: 0.25 mm Film thickness: 0.25 μm</p> <p>Detection limit Bottom sediment (ng/g-dry) (1) 0.02</p> <p>GC/MS-SIM or SCAN</p> <p>Column: Hp-1 Column length: 60 m Column I.D.: 0.32 mm Film thickness: 1.0 μm</p> <p>Detection limit Air (ng/m³) (1) 29</p>

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

Substance	Analytical Method / Flow Chart	Remarks
<p>(2) Perfluorooctane sulfonic acid (PFOS)</p> <p>(3) Perfluorooctanic acid (PFOA)</p>	<p>Surface water</p> <pre> graph TD A[Sample 1 L] --> B[Adjust pH of the sample to 6 - 11 using 1N HCl or 1N NaOH solution] B --> C[Extraction] C --> D[Solvent extraction] D --> E[Concentration] E --> F[LC/MS-SIM] </pre> <p>Solid state cartridge Extraction by water flow at 10 mL/min</p> <p>Methanol 2 mL Nitrogen gas (→1 mL)</p>	<p>LC/MS-SIM</p> <p>Agilent 1100</p> <p>Column: Zorbox XDDBC-18 3.5 μm 2.1 mm x 150 mm</p> <p>Detection limit Surface water (ng/L) (2) 0.05</p>
<p>(4) Benzo[a]pyrene</p>	<p>Surface water Bottom sediment / Wildlife</p> <pre> graph TD subgraph Surface_water [Surface water] SW_S[Sample 1 L] --> SW_E[Extraction] SW_E --> SW_D[Dehydration] end subgraph Bottom_sediment_wildlife [Bottom sediment / Wildlife] BS_S[Sample 20 g] --> BS_AD[Alkali decomposition] BS_AD --> BS_C[Centrifuging] BS_C --> BS_E[Extraction] BS_E --> BS_D[Dehydration] end SW_D --> C[Concentration] BS_D --> C C --> HPLC[HPLC or GC/MS-SIM] </pre> <p><i>n</i>-Hexane 100 mL, 50 mL</p> <p>1N-KOH Ethanol solution 100 mL Heating for 1 hr in boiling water</p> <p>4,000 rpm</p> <p>4% Na₂SO₄ <i>n</i>-Hexane 100 mL, 50 mL</p> <p>Na₂SO₄</p> <p>Na₂SO₄</p>	<p>GC/MS-SIM</p> <p>Column: Ultra-2 Column length: 50 m Column I.D.: 0.31 mm Film thickness: 0.52 μm</p> <p>HPLC</p> <p>Column: Perkin Elmer PAH 2.6 mm x 250 mm Guard column: Nucleosil C18 4.6 mm x 33 mm</p> <p>Detection limit Surface water (ng/L) (4) HPLC: 50 GC/MS: 60</p> <p>Bottom sediment (ng/g-dry) / wildlife (ng/g-wet) (4) HPLC: 1 GC/MS: 3</p>

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

Substance	Analytical Method / Flow Chart	Remarks
(5) Polychlorinated naphthalene	<p>Wildlife / Diet ([] for diet)</p> <p>Air</p>	<p>HRGC/MS-SIM</p> <p>Resolution: 10,000</p> <p>Column: J&W DB-5MS</p> <p>Column length: 60 m</p> <p>Column I.D.: 0.32 mm</p> <p>Film thickness: 0.25 µm</p> <p>Detection limit</p> <p>Wildlife (pg/g-wet)</p> <p>(5) Monochloride: 1</p> <p>Dichloride: 0.5</p> <p>Trichloride: 0.5</p> <p>Tetrachloride: 0.6</p> <p>Pentachloride: 0.9</p> <p>Hexachloride: 0.7</p> <p>Heptachloride: 0.9</p> <p>Octachloride: 0.8</p> <p>Diet (pg/g-fresh weight)</p> <p>(5) Monochloride: 5</p> <p>Dichloride: 1</p> <p>Trichloride: 1</p> <p>Tetrachloride: 1</p> <p>Pentachloride: 1</p> <p>Hexachloride: 1</p> <p>Heptachloride: 1</p> <p>Octachloride: 1</p> <p>GC/MS-SIM</p> <p>JEOL JMS-700</p> <p>Column: HP Ultra-2</p> <p>Column length: 25 m</p> <p>Column I.D.: 0.20 mm</p> <p>Film thickness: 0.33 µm</p> <p>Detection limit</p> <p>Air (pg/m³)</p> <p>(5) Monochloride: 0.01</p> <p>Dichloride: 0.04</p> <p>Trichloride: 0.04</p> <p>Tetrachloride: 0.03</p> <p>Pentachloride: 0.1</p> <p>Hexachloride: 0.1</p> <p>Heptachloride: 0.1</p> <p>Octachloride: 0.2</p>

FY2002 Environmental Survey for Exposure Study (continued)

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

3. Monitoring Investigation

Analytical Method for the FY2002 Monitoring Investigation

Substance	Analytical Method / Flow Chart	Remarks																																																																		
(1) PCBs	<p>Surface water</p> <p>Surrogate</p> <p>Bottom sediment</p> <p>Surrogate</p>	<p>GC/MS</p> <p>Column: HT8-PCB Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm</p> <p>Detection/Quantitation limit</p> <p>Surface water (pg/L)</p> <table border="1"> <thead> <tr> <th>(1)</th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr><td>Mono-chloride</td><td>0.06</td><td>0.12</td></tr> <tr><td>Di-chloride</td><td>0.2</td><td>0.6</td></tr> <tr><td>Tri-chloride</td><td>0.3</td><td>0.9</td></tr> <tr><td>Tetra-chloride</td><td>0.3</td><td>0.9</td></tr> <tr><td>Penta-chloride</td><td>0.2</td><td>0.6</td></tr> <tr><td>Hexa-chloride</td><td>0.3</td><td>0.9</td></tr> <tr><td>Hepta-chloride</td><td>0.2</td><td>0.6</td></tr> <tr><td>Octa-chloride</td><td>0.3</td><td>0.9</td></tr> <tr><td>Nona-chloride</td><td>0.3</td><td>0.9</td></tr> <tr><td>Deca-chloride</td><td>0.3</td><td>0.9</td></tr> </tbody> </table> <p>Bottom sediment (pg/g-dry)</p> <table border="1"> <thead> <tr> <th>(1)</th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr><td>Mono-chloride</td><td>0.06</td><td>0.12</td></tr> <tr><td>Di-chloride</td><td>0.2</td><td>0.6</td></tr> <tr><td>Tri-chloride</td><td>0.3</td><td>0.9</td></tr> <tr><td>Tetra-chloride</td><td>0.3</td><td>0.9</td></tr> <tr><td>Penta-chloride</td><td>0.2</td><td>0.6</td></tr> <tr><td>Hexa-chloride</td><td>0.3</td><td>0.9</td></tr> <tr><td>Hepta-chloride</td><td>0.2</td><td>0.6</td></tr> <tr><td>Octa-chloride</td><td>0.3</td><td>0.9</td></tr> <tr><td>Nona-chloride</td><td>0.3</td><td>0.9</td></tr> <tr><td>Deca-chloride</td><td>0.3</td><td>0.9</td></tr> </tbody> </table>	(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	(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
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(1) PCBs (continued)	<p>Wildlife</p> <p>Internal standard</p> <p>Air</p> <p>Sampled by high-volume air sampler (HV) with quartz-fiber-filter (QFF), polyurethane foam (PUF) and active carbon felt (ACF) sorbent media.</p>	<p>GC/MS</p> <p>Column: HT8-PCB Column length: 50 m Column I.D.: 0.25 mm Film thickness: 0.25 µm</p> <p>Detection/Quantitation limit Wildlife (pg/g-wet)</p> <table border="1"> <thead> <tr> <th>(1)</th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr><td>Mono-chloride</td><td>0.7</td><td>2.1</td></tr> <tr><td>Di-chloride</td><td>0.9</td><td>2.7</td></tr> <tr><td>Tri-chloride</td><td>0.8</td><td>2.4</td></tr> <tr><td>Tetra-chloride</td><td>1.0</td><td>3.0</td></tr> <tr><td>Penta-chloride</td><td>1.0</td><td>3.0</td></tr> <tr><td>Hexa-chloride</td><td>1.0</td><td>3.0</td></tr> <tr><td>Hepta-chloride</td><td>1.0</td><td>3.0</td></tr> <tr><td>Octa-chloride</td><td>1.0</td><td>3.0</td></tr> <tr><td>Nona-chloride</td><td>0.6</td><td>1.8</td></tr> <tr><td>Deca-chloride</td><td>0.4</td><td>1.2</td></tr> </tbody> </table> <p>GC/HRMS</p> <p>Resolution: 10,000 Column: DB-5MS Column length: 60 m Column I.D.: 0.32 mm Film thickness: 0.25 µm</p> <p>Detection/Quantitation limit Air (pg/m³)</p> <table border="1"> <thead> <tr> <th>(1)</th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr><td>Mono-chloride</td><td>30</td><td>90</td></tr> <tr><td>Di-chloride</td><td>1.0</td><td>3.0</td></tr> <tr><td>Tri-chloride</td><td>0.5</td><td>1.5</td></tr> <tr><td>Tetra-chloride</td><td>0.9</td><td>2.7</td></tr> <tr><td>Penta-chloride</td><td>0.4</td><td>1.2</td></tr> <tr><td>Hexa-chloride</td><td>0.2</td><td>0.6</td></tr> <tr><td>Hepta-chloride</td><td>0.007</td><td>0.021</td></tr> <tr><td>Octa-chloride</td><td>0.01</td><td>0.03</td></tr> <tr><td>Nona-chloride</td><td>0.01</td><td>0.03</td></tr> <tr><td>Deca-chloride</td><td>0.005</td><td>0.015</td></tr> </tbody> </table>	(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	(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
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(2) HCB (3) Drins (3-1) Aldrin (3-2) Dieldrin (3-3) Endrin (4) DDTs (4-1) <i>p,p'</i> -DDT (4-2) <i>p,p'</i> -DDE (4-3) <i>p,p'</i> -DDD (4-4) <i>o,p'</i> -DDT (4-5) <i>o,p'</i> -DDE (4-6) <i>o,p'</i> -DDD (5) Chlordanes (5-1) <i>trans</i> -Chlordane (5-2) <i>cis</i> -Chlordane (5-3) <i>trans</i> -Nonachlor (5-4) <i>cis</i> -Nonachlor (5-5) Oxychlordane (6) Heptachlor (7) HCH (7-1) α -HCH (7-2) β -HCH	<p>Wildlife</p> <p>Air</p> <p>Sampled by high-volume air sampler (HV) with quartz-fiber-filter (QFF), polyurethane foam (PUF) and active carbon felt (ACF) sorbent media.</p>	<p>GC/MS Column: DB-5MS Column length: 60 m Column I.D.: 0.25 mm Film thickness: 0.25 μm</p> <p>Detection/Quantitation limit Wildlife (pg/g-wet)</p> <table border="1"> <thead> <tr> <th></th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr> <td>(2)</td> <td>0.2</td> <td>0.6</td> </tr> <tr> <td>(3-1)</td> <td>1.4</td> <td>4.2</td> </tr> <tr> <td>(3-2)</td> <td>4</td> <td>12</td> </tr> <tr> <td>(3-3)</td> <td>6</td> <td>18</td> </tr> <tr> <td>(4-1)</td> <td>0.2</td> <td>0.6</td> </tr> <tr> <td>(4-2)</td> <td>0.2</td> <td>0.6</td> </tr> <tr> <td>(4-3)</td> <td>0.08</td> <td>0.24</td> </tr> <tr> <td>(4-4)</td> <td>0.4</td> <td>1.2</td> </tr> <tr> <td>(4-5)</td> <td>0.3</td> <td>0.9</td> </tr> <tr> <td>(4-6)</td> <td>0.2</td> <td>0.6</td> </tr> <tr> <td>(5-1)</td> <td>0.5</td> <td>1.5</td> </tr> <tr> <td>(5-2)</td> <td>0.3</td> <td>0.9</td> </tr> <tr> <td>(5-3)</td> <td>0.4</td> <td>1.2</td> </tr> <tr> <td>(5-4)</td> <td>0.6</td> <td>1.8</td> </tr> <tr> <td>(5-5)</td> <td>0.4</td> <td>1.2</td> </tr> <tr> <td>(6)</td> <td>0.5</td> <td>1.5</td> </tr> <tr> <td>(7-1)</td> <td>0.3</td> <td>0.9</td> </tr> <tr> <td>(7-2)</td> <td>0.3</td> <td>0.9</td> </tr> </tbody> </table> <p>GC/HRMS Column: DB-17HT Column length: 30 m Column I.D.: 0.32 mm Film thickness: 0.15 μm</p> <p>Detection/Quantitation limit Air (pg/m³)</p> <table border="1"> <thead> <tr> <th></th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr> <td>(2)</td> <td>0.3</td> <td>0.9</td> </tr> <tr> <td>(3-1)</td> <td>0.02</td> <td>0.06</td> </tr> <tr> <td>(3-2)</td> <td>0.2</td> <td>0.6</td> </tr> <tr> <td>(3-3)</td> <td>0.03</td> <td>0.09</td> </tr> <tr> <td>(4-1)</td> <td>0.08</td> <td>0.24</td> </tr> <tr> <td>(4-2)</td> <td>0.03</td> <td>0.09</td> </tr> <tr> <td>(4-3)</td> <td>0.006</td> <td>0.018</td> </tr> <tr> <td>(4-4)</td> <td>0.05</td> <td>0.15</td> </tr> <tr> <td>(4-5)</td> <td>0.01</td> <td>0.03</td> </tr> <tr> <td>(4-6)</td> <td>0.007</td> <td>0.021</td> </tr> <tr> <td>(5-1)</td> <td>0.2</td> <td>0.6</td> </tr> <tr> <td>(5-2)</td> <td>0.2</td> <td>0.6</td> </tr> <tr> <td>(5-3)</td> <td>0.1</td> <td>0.3</td> </tr> <tr> <td>(5-4)</td> <td>0.01</td> <td>0.03</td> </tr> <tr> <td>(5-5)</td> <td>0.008</td> <td>0.024</td> </tr> <tr> <td>(6)</td> <td>0.04</td> <td>0.12</td> </tr> <tr> <td>(7-1)</td> <td>0.2</td> <td>0.6</td> </tr> <tr> <td>(7-2)</td> <td>0.03</td> <td>0.09</td> </tr> </tbody> </table>		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		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
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(8) Organotin compounds (8-1) TBT (8-2) TPT	<p>Bottom sediment</p> <pre> graph LR A[Sample 2 g Add surrogate Purified water 20 mL] --> B[Derivatization pH5 Buffer solution 2 mL 2% NaBEt4 5 mL] B --> C[Extraction/ Centrifuging EtOH/hexane (1:1) 20 mL Hexane 90 mL, 50 mL] C --> D[Extraction 10% NaCl 500 mL Hexane 50 mL x twice] D --> E[Dehydration Na2SO4] E --> F[Sulfur removal Reduced copper] F --> G[Concentration Reduced pressure KD] G --> H[Column cleanup Sep-Pak + Florisil] H --> I[Concentration Nitrogen stream 0.3 mL] I --> J[GC/MS-SIM] </pre> <p>Wildlife</p> <pre> graph LR K[Sample 5 g Add surrogate substance] --> L[Extraction 1M HBr-Methanol/ Ethyl acetate (1:1) 70 mL] L --> M[Suction filtration 1M HBr-Methanol/ Ethyl acetate (1:1) 30 mL] M --> N[Redissolution Ethyl acetate/Hexane (3:2) Saturated NaBr solution 100 mL] N --> O[Dehydration Na2SO4] O --> P[Concentration Rotary evaporator Nitrogen stream] P --> Q[Derivatization pH5 Buffer solution 5 mL 10% NaB-Et4 solution 1 mL Purified water 15 mL] Q --> R[Alkali composition 1M KOH/Ethanol 40 mL] R --> S[Extraction Hexane 40 mL, 40 mL Purified water 20 mL] S --> T[Concentration Reduced pressure KD Nitrogen stream] T --> U[Column cleanup Sep-Pak + Florisil Hexane (containing 5% ether) 6 mL] U --> V[GC/MS-SIM] </pre>	<p>GC/MS (quadrupole type) Column: Capillary column Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm</p> <p>Detection/Quantitation limit Bottom sediment (ng/g-dry)</p> <table border="1"> <thead> <tr> <th></th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr> <td>(8-1)</td> <td>0.27</td> <td>0.81</td> </tr> <tr> <td>(8-2)</td> <td>0.11</td> <td>0.33</td> </tr> </tbody> </table> <p>Detection/Quantitation limit Wildlife (ng/g-wet)</p> <table border="1"> <thead> <tr> <th></th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr> <td>(8-1)</td> <td>1.3</td> <td>3.9</td> </tr> <tr> <td>(8-2)</td> <td>0.16</td> <td>0.48</td> </tr> </tbody> </table>		Detection limit	Quantitation limit	(8-1)	0.27	0.81	(8-2)	0.11	0.33		Detection limit	Quantitation limit	(8-1)	1.3	3.9	(8-2)	0.16	0.48
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