

## **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.

<i>pH</i>	<i>Initial concentration (µg/mL)</i>	<i>Residual rate after one hour (%)</i>	<i>Residual rate after 5 days</i>	
			<i>Dark place (%)</i>	<i>Light emission (%)</i>
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.

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

$\sigma_R$ : 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>**

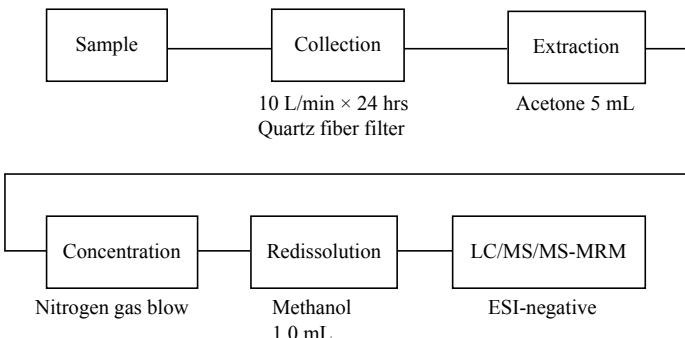
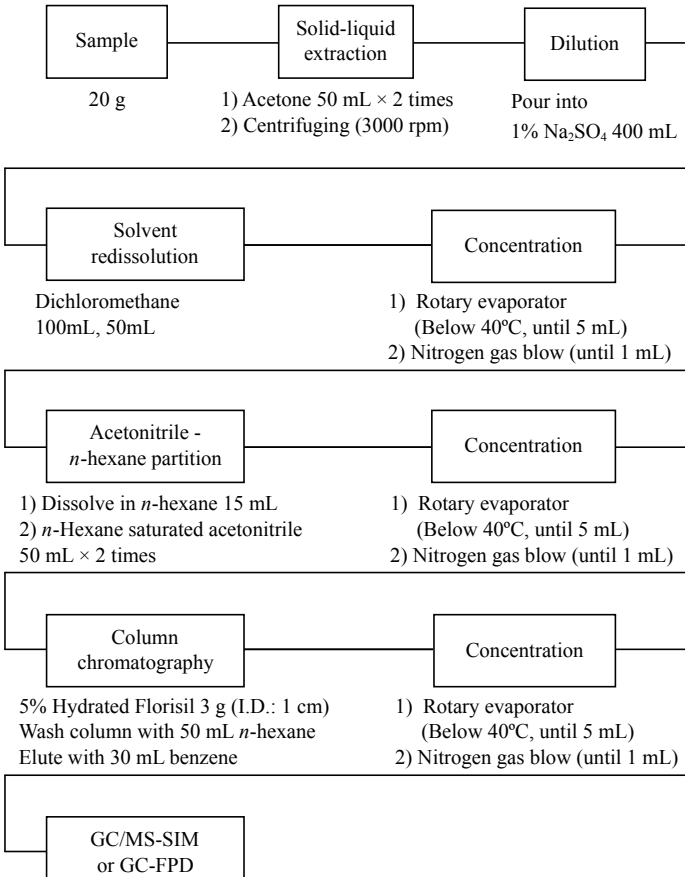
Ten times the detection limit amount of standard substances was added to the river water sample (from the Class B Water Area of Environmental Quality Standards) and the sea water sample (from the Class B of the Environmental Quality Standards, or, when not available, from the Class A Water Area or artificially 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 FY2003 Initial Environmental Survey

Substance	Analytical Method/Flow Chart	Remarks
<p>(1) HCFCs</p> <p>(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</p>	<p><b>Air</b></p> <pre>                     graph LR                     A[Collection by canister 6 L, 3.0 mL/min x 24 hrs] --&gt; B[Pressurized dilution]                     B --&gt; C[Low temperature concentration Entech 7000]                     C --&gt; D[GC/MS-SIM]                     </pre>	<p>GC/MS-SIM</p> <p>Column: HP-VOC                      Column length: 60 m                      Column I.D.: 0.32 mm                      Film thickness: 1.8 µm</p> <p>Detection limit:</p> <p>Air (ng/m<sup>3</sup>)</p> <p>(1.1) 4                      (1.2) 6                      (1.3) 3                      (1.4) 3                      (1.5) 4                      (1.6) 15                      (1.7) 7</p>
<p>(2) Linear alkylbenzene sulfonic acid and its salt (LAS, carbon number of alkyl group: 10 - 14)</p> <p>(2.1) LAS<sub>10</sub>                      (2.2) LAS<sub>11</sub>                      (2.3) LAS<sub>12</sub>                      (2.4) LAS<sub>13</sub>                      (2.5) LAS<sub>14</sub></p>	<p><b>Surface water</b></p> <pre>                     graph LR                     A[Sample 1 L] --&gt; B[Solid phase extraction]                     B --&gt; C[Methanol elution]                     C --&gt; D[Evaporation to dryness Nitrogen gas blow]                     D --&gt; E[Constant volume Acetonitrile/water 65:35) 2 mL]                     E --&gt; F[HPLC separation]                     F --&gt; G[HPLC fluorescent or LC/MS]                     </pre>	<p>LC/MS</p> <p>Column: C<sub>8</sub>                      Column length: 0.25 m                      Column I.D.: 3.0 mm</p> <p>Detection limit:</p> <p>Surface water (µg/L)</p> <p>(2.1) 0.2                      (2.2) 0.2                      (2.3) 0.2                      (2.4) 0.2                      (2.5) 0.2</p>
<p>(3) Isoprene</p>	<p><b>Air</b></p> <pre>                     graph LR                     A[Sample 200 mL/min 10 min] --&gt; B[Collection Carbopack Z]                     B --&gt; C[Thermal desorption ATD-400]                     C --&gt; D[GC/MS-SIM]                     </pre>	<p>GC/MS-SIM</p> <p>Column: DB-1                      Column length: 60 m                      Column I.D.: 0.32 mm                      Film thickness: 3 µm</p> <p>Detection limit:</p> <p>Air (ng/m<sup>3</sup>)</p> <p>(3) 12</p>

Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(4) Chlordecone	<p><b>Air</b></p>  <pre> graph LR     Sample[Sample] --&gt; Collection[Collection 10 L/min x 24 hrs Quartz fiber filter]     Collection --&gt; Extraction[Extraction Acetone 5 mL]     Extraction --&gt; Concentration[Concentration Nitrogen gas blow]     Concentration --&gt; Redissolution[Redissolution Methanol 1.0 mL]     Redissolution --&gt; LCMSMS[LC/MS/MS-MRM ESI-negative]     </pre>	<p>LC/MS            Column: C30-UG-5            Column length: 0.15 m            Column I.D.: 2.0 mm</p> <p>Detection limit:            Air (ng/m<sup>3</sup>)            (4) 0.0005</p>
(5) Chlorpyrifos	<p><b>Wildlife</b></p>  <pre> graph TD     Sample[Sample 20 g] --&gt; Extraction[Solid-liquid extraction 1) Acetone 50 mL x 2 times 2) Centrifuging (3000 rpm)]     Extraction --&gt; Dilution[Dilution Pour into 1% Na2SO4 400 mL]     Dilution --&gt; Redissolution[Solvent redissolution Dichloromethane 100mL, 50mL]     Redissolution --&gt; Conc1[Concentration 1) Rotary evaporator (Below 40°C, until 5 mL) 2) Nitrogen gas blow (until 1 mL)]     Conc1 --&gt; Partition[Acetonitrile - n-hexane partition 1) Dissolve in n-hexane 15 mL 2) n-Hexane saturated acetonitrile 50 mL x 2 times]     Partition --&gt; Conc2[Concentration 1) Rotary evaporator (Below 40°C, until 5 mL) 2) Nitrogen gas blow (until 1 mL)]     Conc2 --&gt; Chrom[Column chromatography 5% Hydrated Florisil 3 g (I.D.: 1 cm) Wash column with 50 mL n-hexane Elute with 30 mL benzene]     Chrom --&gt; Conc3[Concentration 1) Rotary evaporator (Below 40°C, until 5 mL) 2) Nitrogen gas blow (until 1 mL)]     Conc3 --&gt; GC[GC/MS-SIM or GC-FPD]     </pre>	<p>GC/MS-SIM            Column: DB-5            Column length: 30 m            Column I.D.: 0.25 mm            Film thickness: 0.25 µm</p> <p>Detection limit:            Wildlife (ng/g-wet)            (5) 3</p>

Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
<p>(5) Chlorpyrifos (continued)</p>	<p><b>Air</b></p> <p>Aspirate less than 5 m<sup>3</sup> of air at less than 30 L/min</p> <p>Quartz fiber filter × 2 and activated fiber filter (φ 47 mm)</p> <p>Washing of filter folder (twice with 5mL of dichloromethane)</p> <p>Dichloromethane 30 mL</p> <p>Color comparator tube</p> <p>Extraction</p> <p>Ultrasonic extraction, 10 min</p> <p>Extract liquid</p> <p>Hexane solution 1mL</p> <p>Collecting agent</p> <p>Return collecting agent to color comparator tube</p> <p>(Repeat 3 times)</p> <p>Concentration</p> <p>1) KD evaporator until about 5mL 2) Nitrogen gas blow, until 1 mL</p> <p>Add 50 µL internal standard solution (HCB with six <sup>13</sup>C labelled, 5 - 10 mg/L)</p> <p>GC/MS-SIM</p> <p>(Scanning mode is also possible with high sensitivity equipment)</p>	<p>GC/MS-SIM</p> <p>Column: DB1-MS</p> <p>Column length: 30 m</p> <p>Column I.D.: 0.25 mm</p> <p>Film thickness: 0.25 µm</p> <p>Detection limit:</p> <p>Air (ng/m<sup>3</sup>)</p> <p>(5) 2</p>

Analytical Method for the FY2003 Initial Environmental Survey (continued)

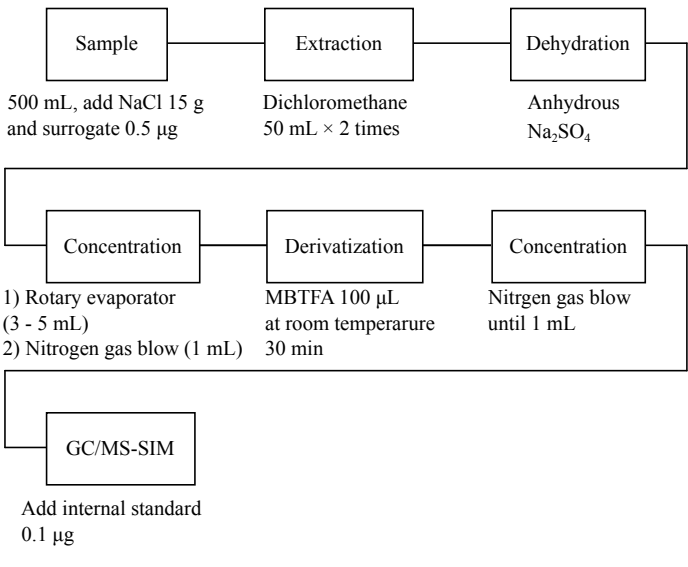
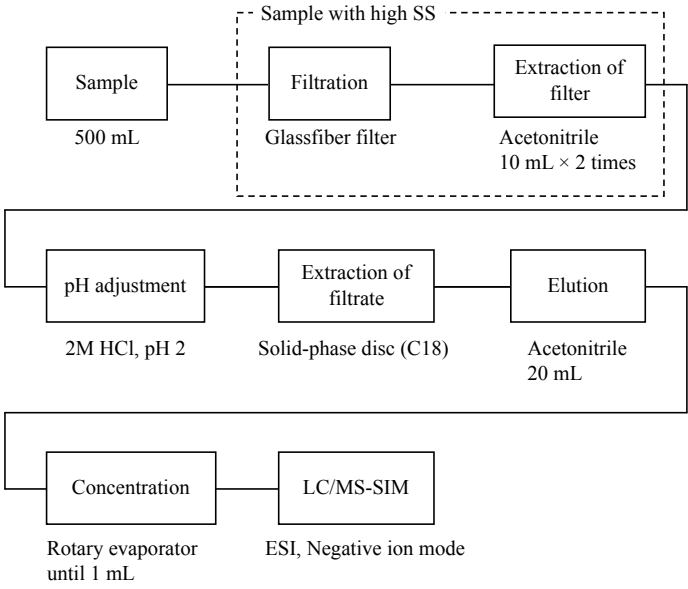
Substance	Analytical Method/Flow Chart	Remarks
<p>(6) Chloropicrin</p>	<p><b>Air</b></p> <p>at room temperature</p> <pre> graph LR     Sample[Sample 100 L (flow rate: 500 mL/min)] --&gt; Tube[Sample collection tube Carbosieve G 60 mg]     Tube --&gt; Elution[Elution Benzene 1 mL]     Elution --&gt; GC[GC/MS-SIM]     IS[Internal standard Toluene-ds (100 ng)] --&gt; GC     </pre>	<p>GC/MS-SIM</p> <p>Column: DB-624 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 1.4 <math>\mu\text{m}</math></p> <p>Detection limit:</p> <p>Air (<math>\text{ng}/\text{m}^3</math>)</p> <p>(6) 220</p>
<p>(7) Diethylenetriamine and another substance</p> <p>(7.1) Diethylenetriamine (7.2) Triethylenetetramine</p>	<p><b>Surface water</b></p> <pre> graph LR     Sample[Sample 25 mL] --&gt; Derivatization[Derivatization NaCl 1 g, 1M Na2CO3 1 mL, 0.5% Dns-Cl 20 mL, 50°C, 1hr]     Derivatization --&gt; Acetone[Acetone removal]     Acetone --&gt; DCM[Dichloromethane extraction]     DCM --&gt; Dehydration[Dehydration Anhydrous Na2SO4]     Dehydration --&gt; Concentration[Concentration KD Evaporator]     Concentration --&gt; HPLC[HPLC-fluorescent]     </pre>	<p>HPLC-fluorescent</p> <p>Column: Lichrosorb RP18 (5 <math>\mu\text{m}</math>) Column length: 0.25 m Column I.D.: 4 mm</p> <p>Detection limit:</p> <p>Surface water (<math>\mu\text{g}/\text{L}</math>)</p> <p>(1) 2 (2) 8</p>



Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
<p>(8) 1,4-Dichloro-2-nitrobenzene and 3 other substances</p> <p>(8.1) 1,4-Dichloro-2-nitrobenzene</p> <p>(8.2) 1,3 -Dichloro-4-nitrobenzene</p> <p>(8.3) 1-Chloro-3-nitrobenzene</p> <p>(8.4) 1,4-Dinitrobenzene</p>	<p><b>Surface water</b></p> <pre> graph LR     subgraph SW_Step1 [ ]         S1[Sample] --&gt; E1[Extraction] --&gt; D1[Dehydration]     end     S1["500 mL, add surrogate and NaCl 15g"]     E1["Dichloromethane 100 mL, 100 mL"]     D1["Anhydrous Na2SO4"]          subgraph SW_Step2 [ ]         C1[Concentration] --&gt; R1[Redissolution] --&gt; C2[Concentration]     end     C1["KD evaporator until 1 mL"]     R1["Hexane 20 mL"]     C2["KD evaporator until 1 mL"]          subgraph SW_Final [ ]         G1[GC/MS-SIM]     end     G1["Add internal standard"]          S1 --- E1 --- D1 --- C1 --- R1 --- C2 --- G1                     </pre> <p><b>Bottom sediment</b></p> <pre> graph LR     subgraph BS_Step1 [ ]         S2[Sample] --&gt; E2[Extraction] --&gt; C3[Centrifuging]     end     S2["10g-wet, add surrogate and 10% CuSO4 solution 50 mL"]     E2["Acetone 50 mL, 50 mL"]     C3["3000 rpm"]          subgraph BS_Step2 [ ]         L1[Liquid/liquid extraction] --&gt; D2[Dehydration] --&gt; C4[Concentration]     end     L1["3% NaCl solution 500 mL Dichloromethane 100 mL, 100 mL"]     D2["Anhydrous Na2SO4"]     C4["KD evaporator until 1 mL"]          subgraph BS_Step3 [ ]         R2[Redissolution] --&gt; F1[Florisil column chromatography]     end     R2["Hexane 1mL"]     F1["5% Hydrated Florisil 5 g Washing: Hexane 20 mL Elution: Hexan 40 mL with 10 % dichloromethane (for 8.1, 8.2, 8.3) Hexan 30 mL with 10 % acetone (for 8.4)"]          subgraph BS_Final [ ]         C5[Concentration] --&gt; G2[GC/MS-SIM]     end     C5["KD evaporator until 1 mL"]     G2["Add internal standard"]          S2 --- E2 --- C3 --- L1 --- D2 --- C4 --- R2 --- F1 --- C5 --- G2                     </pre>	<p>GC/MS-SIM</p> <p>Column: DB-1701</p> <p>Column length: 30 m</p> <p>Column I.D.: 0.32 mm</p> <p>Film thickness: 0.25 µm</p> <p>Detection limit:</p> <p>Surface water (µg/L)</p> <p>(8.1) 0.05</p> <p>(8.2) 0.06</p> <p>(8.3) 0.05</p> <p>(8.4) 0.05</p> <p>Bottom sediment (ng/g-dry)</p> <p>(8.1) 2.5</p> <p>(8.2) 1.9</p> <p>(8.3) 3.2</p> <p>(8.4) 3.1</p>

Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(9) 3,3'-Dichlorobenzidine	<p><b>Surface water</b></p>  <pre> graph TD     S[Sample 500 mL, add NaCl 15 g and surrogate 0.5 µg] --&gt; E[Extraction Dichloromethane 50 mL x 2 times]     E --&gt; D[Dehydration Anhydrous Na2SO4]     D --&gt; C1[Concentration 1) Rotary evaporator (3 - 5 mL) 2) Nitrogen gas blow (1 mL)]     C1 --&gt; Der[Derivatization MBTFA 100 µL at room temperature 30 min]     Der --&gt; C2[Concentration Nitrogen gas blow until 1 mL]     C2 --&gt; GC[GC/MS-SIM Add internal standard 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)            (9) 0.010</p>
(10) Pyridine-triphenylborane	<p><b>Surface water</b></p>  <pre> graph TD     S[Sample 500 mL] --&gt; F[Filtration Glassfiber filter]     F --&gt; EF[Extraction of filter Acetonitrile 10 mL x 2 times]     EF --&gt; PA[pH adjustment 2M HCl, pH 2]     PA --&gt; ECF[Extraction of filtrate Solid-phase disc C18]     ECF --&gt; EL[Elution Acetonitrile 20 mL]     EL --&gt; C[Concentration Rotary evaporator until 1 mL]     C --&gt; LC[LC/MS-SIM ESI, Negative ion mode]     </pre>	<p>LC/MS-SIM            Column: Inertsil ODS-80A            Column length: 0.25 m            Column I.D.: 1.5 mm</p> <p>Surface water (µg/L)            (10) 0.030</p>

Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
(11) 2,4,6-Tri- <i>tert</i> -butylphenol	<p><b>Air</b></p> <pre> graph TD     Sample[Sample] --&gt; Collection[Collection by Sep-Pak Cartridge Sep-Pak Plus C18 Environmental 0.8 L/min, 20 hrs, total 1 m³]     Collection --&gt; Elution[Elution 30 % Acetone / n-hexane (7 mL) or n-Hexane (7mL)]     Elution --&gt; AddStd[Addition of internal standard substance Add HCB-13C]     AddStd --&gt; Concentration[Concentration Nitrogen gas blow until 1mL]     Concentration --&gt; Centrifuging[Centrifuging In case eluted by n-hexane]     Centrifuging --&gt; GCMS[GC/MS-SIM]     </pre>	<p>GC/MS-SIM            Column: HP-5MS            Column length: 30 m            Column I.D.: 0.25 mm            Film thickness: 0.25 µm</p> <p>Detection limit:            Air (ng/m<sup>3</sup>)            (11) 0.9</p>
(12) Bromomethane	<p><b>Air</b></p> <pre> graph TD     Sample[Sample] --&gt; Adsorption[Adsorption tube sampling 10 mL/min, 24 hrs Carboxen 1000]     Adsorption --&gt; Desorption[Thermal desorption ATD-400]     Desorption --&gt; GCMS[GC/MS SCAN or SIM]     </pre>	<p>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</p> <p>Detection limit:            Air (ng/m<sup>3</sup>)            (12) 0.027</p>

Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
<p>(13) 1,2,5,6,9,10-Hexabromocyclododecane</p>	<p><b>Surface water</b></p> <pre> graph TD     subgraph SW_Step1 [ ]         S1[Sample 1000 mL NaCl 30 g] --&gt; S2[Solid phase extraction Pass through]         S2 --&gt; S3[Elution Acetone 2 mL Dichloromethane 4 mL, 2 mL]     end     S3 --&gt; S4[Dehydration Anhydrous Na2SO4]     S4 --&gt; S5[Concentration Rotary evaporator until 1 mL]     S5 --&gt; S6[Hexane redissolution Hexane 20 mL]     S6 --&gt; S7[Concentration Rotary evaporator until 1 mL]     S7 --&gt; S8[Florisil column chromatography Florisil 910 mg Washing: hexane 20 mL Elution: 5% ethylether - hexane 10 mL]     S8 --&gt; S9[Concentration Nitrogen gas blow until 1 mL]     S9 --&gt; S10[GC/MS-SIM Add internal standard]     </pre> <p><b>Bottom sediment</b></p> <pre> graph TD     subgraph BS_Step1 [ ]         B1[Sample 20 g-wet] --&gt; B2[Extraction Acetone 20 mL x 3 times Centrifuging (3000 rpm)]         B2 --&gt; B3[Hexane extraction 5% NaCl solution 500 mL Hexane 50 mL x 2 times]     end     B3 --&gt; B4[H2SO4 treatment 10 mL + 5 mL]     B4 --&gt; B5[Hexane extraction 5% NaCl solution 30 mL Hexane 20 mL x 2 times]     B5 --&gt; B6[Dehydration Anhydrous Na2SO4]     B6 --&gt; B7[Concentration Rotary evaporator until 1 mL]     B7 --&gt; B8[Florisil column chromatography Florisil 910 mg Washing: hexane 20 mL Elution: 5% ethylether - hexane 10 mL]     B8 --&gt; B9[Concentration Nitrogen gas blow until 1 mL]     B9 --&gt; B10[GC/MS-SIM Add internal standard]     </pre>	<p>GC/MS-SIM Column: DB-5ms Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.1 µm</p> <p>Detection limit: Surface water (µg/L) (13) 0.087</p> <p>Bottom sediment (ng/g-dry) (13) 23</p>

Analytical Method for the FY2003 Initial Environmental Survey (continued)

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

Analytical Method for the FY2003 Initial Environmental Survey (continued)

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

Analytical Method for the FY2003 Initial Environmental Survey (continued)

Substance	Analytical Method/Flow Chart	Remarks
<p>(15) Polybrominated diphenyl ethers (continued)</p> <p>(15.1) Hexabromodiphenyl ether</p> <p>(15.2) Decabromodiphenyl ether</p>	<p><b>Wildlife</b></p> <pre> graph TD     Sample[Sample 20 g] --&gt; SLE[Solid-liquid extraction]     SLE --&gt; W[Washing]     W --&gt; C1[Concentration Evaporator]     C1 --&gt; AP[Acetonitrile - n-hexane partition]     AP --&gt; SR[Solvent redissolution]     SR --&gt; C2[Concentration Rotary evaporator]     C2 --&gt; CC[Column chromatography]     CC --&gt; C3[Concentration Rotary evaporator]     C3 --&gt; H2SO4[H2SO4 treatment]     H2SO4 --&gt; GC_ECD[GC-ECD]     </pre> <p>1) Homogenize with acetone-benzene (1:2) 50 mL × 2 times 2) Centrifuging (3000 rpm)</p> <p>1) Acidic aqueous solution 100 mL × 2 times 2) Dehydration</p> <p>1) Dissolve in 10 mL <i>n</i>-hexane 2) <i>n</i>-Hexane saturated acetonitrile 50 mL (twice)</p> <p>1) 5% Na<sub>2</sub>SO<sub>4</sub> 2) Add H<sub>2</sub>SO<sub>4</sub> (1+1) 2 mL 3) Extraction by 100 mL, 50 mL benzene</p>	<p>GC-ECD</p> <p>Column: Glass column Column length: 0.5 m Column I.D.: 3 mm</p> <p>Detection limit:</p> <p>Wildlife (ng/g-wet)</p> <p>(15.1) 0.5 (15.2) 1</p>

2. Environmental Survey for Exposure Study

Analytical Method for the FY2003 Environmental Survey for Exposure Study

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



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

Substance	Analytical Method/Flow Chart	Remarks
(2) <i>o</i> -Chloroaniline	<p><b>Surface water</b></p> <pre> graph TD     S[Sample 500 mL] --&gt; A[Aniline-d5 100 ng NaCl 30 g] --&gt; SPE[Solid phase extraction Sep-Pak PS-2 Water flow 20 mL/min]     SPE --&gt; AD[Air dehydration]     AD --&gt; E[Elution Methyl acetate 3 mL]     E --&gt; C1[Concentration until about 2 mL]     C1 --&gt; D[Dehydration Hexane a few mL Anhydrous Na2SO4]     D --&gt; C2[Concentration Nitrogen gas blow until 1 mL]     C2 --&gt; GC[GC/MS]     GC --&gt; A10[Acenaphthene-d10 100 ng] --&gt; C2     </pre>	<p>GC/MS-SIM          Column: SGE BP-20          Column length: 30 m          Column I.D.: 0.25 mm          Film thickness: 0.25 μm</p> <p>Detection limit:          Surface water (μg/L)          (2) 25</p>
(3) 1-Chloro-2,4-dinitrobenzene	<p><b>Surface water</b></p> <pre> graph TD     S[Sample 1 L] --&gt; A[2,4-Dinitrotoluene-ring-d3 100 ng] --&gt; E[Extraction Benzene 100 mL x 2 times]     E --&gt; D[Dehydration Anhydrous Na2SO4]     D --&gt; CR[Concentration / Redissolution n-Hexane 100 mL]     CR --&gt; C1[Concentration Nitrogen gas blow]     C1 --&gt; FC[Florisil column chromatography (only for colored sample) Florisil cartridge treatment Elution: 5% acetone / n-hexane 10 mL]     FC --&gt; CC[Concentration / Constant volume Nitrogen gas blow until 1 mL]     CC --&gt; GC[GC/MS]     A10[Phenanthrene-d10 100 ng] --&gt; GC     </pre>	<p>GC/MS          Column: SGE BPX-5          Column length: 30 m          Column I.D.: 0.25 mm          Film thickness: 0.25 μm</p> <p>Detection limit:          Surface water (ng/L)          (3) 10</p>

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

Substance	Analytical Method/Flow Chart	Remarks
(4) 2,4-Dinitrophenol	<p><b>Surface water</b></p> <pre> graph TD     S[Sample 1 L] --&gt; A[2,4-Dinitrophenol-d3 500 ng NaCl 30 g] --&gt; B[pH adjustment below pH 3.5]     B --&gt; C[Extraction Dichloromethane 100 mL, 50 mL x 2 times]     C --&gt; D[Dehydration Anhydrous Na2SO4]     D --&gt; E[Concentration until about 3 mL]     E --&gt; F[Derivatization (methylation) Diazomethane solution 1 mL Rest at room temperature for 1 hr]     F --&gt; G[Concentration Nitrogen gas blow until 1 mL]     G --&gt; H[GC/MS Phenanthrene-d10 100 ng]     I[Hexane a few mL] --&gt; J[Concentration]     J --&gt; H     </pre>	<p>GC/MS          Column: SGE BPX-5          Column length: 30 m          Column I.D.: 0.25 mm          Film thickness: 0.25 μm</p> <p>Detection limit:          Surface water (ng/L)          (4) 19</p>
(5) Phenol	<p><b>Surface water</b></p> <pre> graph TD     S[Sample 500 mL] --&gt; A[Phenol-d3 200 ng NaCl 15 g] --&gt; B[pH adjustment pH 3]     B --&gt; C[Extraction Dichloromethane 100 mL x 2 times]     C --&gt; D[Dehydration Anhydrous Na2SO4]     D --&gt; E[Concentration until about 2 mL]     E --&gt; F[Derivatization Potassium carbonate about 3 mg PFBB solution 2 mL Rest 1 hr at 90 °C]     F --&gt; G[Concentration 1 mL]     G --&gt; H[Extraction Hexane 5 mL x 2 times]     H --&gt; I[Dehydration Anhydrous Na2SO4]     I --&gt; J[Concentration Nitrogen gas blow]     J --&gt; K[GC/MS Acenaphthene-d10 100 ng]     L[2-Propanol 1 mL] --&gt; E     </pre>	<p>GC/MS          Column: SGE BP-10          Column length: 30 m          Column I.D.: 0.25 mm          Film thickness: 0.25 μm</p> <p>Detection limit:          Surface water (ng/L)          (5) 28</p>

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)	<p><b>Bottom sediment</b></p> <pre>                     graph LR                         S[Sample 10 g] --&gt; ASE[ASE extraction 20% Methanol solution]                         ASE --&gt; SPE[Solid phase extraction Presep-C Agri (220 mg)]                         SPE --&gt; E[Elution Methanol 2 mL]                         E --&gt; C[Concentration Nitrogen gas blow until 1 mL]                         C --&gt; LC[LC/MS-SIM ESI, Negative ion mode]                     </pre> <p><b>Wildlife</b></p> <pre>                     graph TD                         S[Sample 5 g] --&gt; IPE[Ion pair solvent extraction 0.2 mol/L Carbonate buffer solution 25 mL 0.1 mol/L Tetrabutylammonium 5 mL Homogenize MTBE 80 mL, 40 mL Dehydration, Concentration to dryness Constant volume, hexane 8 mL]                         IPE --&gt; D[Degreasing ChemElut (5 mL) Loading: 4 mL Hexane removal by aspiration Elution: 5% hydrated acetonitrile 20 mL Concentration to dryness]                         D --&gt; SPE[Solid phase extraction OASIS HLB (6 mL) Loading: 0.004 mol/L carbonate buffer 5 mL x 2 Washing by 5 mL water Moisture removal by aspiration Connect OASIS MCX (3 mL) Elution: Acetonitrile 10 mL Concentration to dryness]                         SPE --&gt; CV[Constant volume Methanol-water (1:1) 1 mL]                         CV --&gt; DF[Disc filtration Hydrophilic Chomatodisc]                         DF --&gt; LC[LC/MS-MRM]                     </pre>	<p>LC/MS-SIM Column: Zorbax XDB C-18 Column length: 150 mm Column I.D.: 2.1 mm Particle diameter: 3.5 µm</p> <p>Detection limit: Bottom sediment (ng/g-dry) (6) 0.022 (7) 0.016</p>
	<p><b>Wildlife</b></p> <pre>                     graph TD                         S[Sample 5 g] --&gt; IPE[Ion pair solvent extraction 0.2 mol/L Carbonate buffer solution 25 mL 0.1 mol/L Tetrabutylammonium 5 mL Homogenize MTBE 80 mL, 40 mL Dehydration, Concentration to dryness Constant volume, hexane 8 mL]                         IPE --&gt; D[Degreasing ChemElut (5 mL) Loading: 4 mL Hexane removal by aspiration Elution: 5% hydrated acetonitrile 20 mL Concentration to dryness]                         D --&gt; SPE[Solid phase extraction OASIS HLB (6 mL) Loading: 0.004 mol/L carbonate buffer 5 mL x 2 Washing by 5 mL water Moisture removal by aspiration Connect OASIS MCX (3 mL) Elution: Acetonitrile 10 mL Concentration to dryness]                         SPE --&gt; CV[Constant volume Methanol-water (1:1) 1 mL]                         CV --&gt; DF[Disc filtration Hydrophilic Chomatodisc]                         DF --&gt; LC[LC/MS-MRM]                     </pre>	<p>LC/MS/MS-MRM Column: CAPCEL PAK C18 MG-II Column length: 150 mm Column I.D.: 2 mm Particle diameter: 5 µm</p> <p>Detection limit: Wildlife (ng/g-wet) (6) 0.033 (7) 0.059</p>

### 3. Monitoring Investigation

#### Analytical Method for the FY2003 Monitoring Investigation

Substance	Analytical Method/Flow Chart	Remarks																																																																																																												
(1) PCBs	<p><b>Surface water</b></p> <p><b>Bottom sediment</b></p>	<p>GC/HRMS Resolution: 10,000 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>0.4</td><td>2</td></tr> <tr><td>Di-chloride</td><td>0.2</td><td>0.5</td></tr> <tr><td>Tri-chloride</td><td>0.2</td><td>0.4</td></tr> <tr><td>Tetra-chloride</td><td>0.09</td><td>0.3</td></tr> <tr><td>Penta-chloride</td><td>0.07</td><td>0.3</td></tr> <tr><td>Hexa-chloride</td><td>0.09</td><td>0.3</td></tr> <tr><td>Hepta-chloride</td><td>0.07</td><td>0.3</td></tr> <tr><td>Octa-chloride</td><td>0.07</td><td>0.3</td></tr> <tr><td>Nona-chloride</td><td>0.4</td><td>2</td></tr> <tr><td>Deca-chloride</td><td>0.9</td><td>3</td></tr> <tr><td>PCB 77, 81</td><td>0.3, 0.2</td><td>0.8, 0.6</td></tr> <tr><td>PCB 105, 114</td><td>0.7, 0.1</td><td>3, 0.4</td></tr> <tr><td>PCB 118, 123</td><td>2, 0.1</td><td>6, 0.4</td></tr> <tr><td>PCB 126, 156</td><td>0.1, 0.2</td><td>0.4, 0.5</td></tr> <tr><td>PCB 157, 167</td><td>0.2, 0.09</td><td>0.5, 0.3</td></tr> <tr><td>PCB 169, 170</td><td>0.2, 0.3</td><td>0.4, 0.9</td></tr> <tr><td>PCB 180, 189</td><td>0.5, 0.2</td><td>2, 0.5</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>0.4</td><td>2</td></tr> <tr><td>Di-chloride</td><td>0.2</td><td>0.7</td></tr> <tr><td>Tri-chloride</td><td>0.2</td><td>0.6</td></tr> <tr><td>Tetra-chloride</td><td>0.2</td><td>0.4</td></tr> <tr><td>Penta-chloride</td><td>0.2</td><td>0.5</td></tr> <tr><td>Hexa-chloride</td><td>0.2</td><td>0.4</td></tr> <tr><td>Hepta-chloride</td><td>0.3</td><td>0.7</td></tr> <tr><td>Octa-chloride</td><td>0.3</td><td>0.8</td></tr> <tr><td>Nona-chloride</td><td>0.6</td><td>2</td></tr> <tr><td>Deca-chloride</td><td>0.6</td><td>2</td></tr> <tr><td>PCB 77, 81</td><td>0.3, 0.3</td><td>0.8, 0.8</td></tr> <tr><td>PCB 105, 114</td><td>2, 0.3</td><td>4, 0.8</td></tr> <tr><td>PCB 118, 123</td><td>2, 0.3</td><td>6, 0.8</td></tr> <tr><td>PCB 126, 156</td><td>0.2, 2</td><td>0.6, 5</td></tr> <tr><td>PCB 157, 167</td><td>0.4, 0.2</td><td>2, 0.7</td></tr> <tr><td>PCB 169, 170</td><td>0.4, 2</td><td>2, 5</td></tr> <tr><td>PCB 180, 189</td><td>0.2, 0.4</td><td>0.4, 2</td></tr> </tbody> </table>	(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	(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
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Analytical Method for the FY2003 Monitoring Investigation (continued)

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(1) PCBs (continued)	<p><b>Wildlife</b></p> <pre> graph TD     A[Sample About 10 g] --&gt; B[Dehydration/ grinding Anhydrous Na2SO4]     B --&gt; C[Soxhlet extraction Dichloromethane 6 hrs]     D[Internal standard (cleanup spike)] --&gt; C     C --&gt; E[Dehydration]     E --&gt; F[Concentration 20 mL]     F --&gt; G[Take a portion]     G --&gt; H[Florisil column chromatography]     I[Florisil 10 g Washing: 20%-dichloromethane / hexane 50 mL Elution: 20%-dichloromethane / hexane 110 mL] --&gt; H     H --&gt; J[Concentration 100 µL]     J --&gt; K[Concentration 100 µL]     L[Internal standard (syringe spike)] --&gt; K     K --&gt; M[GC/HRMS]     </pre>	<p>GC/HRMS</p> <p>Resolution: 10,000</p> <p>Column: 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/Quantitation limit:</p> <p>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>0.69</td><td>2.1</td></tr> <tr><td>Di-chloride</td><td>2.5</td><td>7.5</td></tr> <tr><td>Tri-chloride</td><td>2</td><td>6</td></tr> <tr><td>Tetra-chloride</td><td>2.3</td><td>6.9</td></tr> <tr><td>Penta-chloride</td><td>1.9</td><td>5.7</td></tr> <tr><td>Hexa-chloride</td><td>1.1</td><td>3.3</td></tr> <tr><td>Hepta-chloride</td><td>1.6</td><td>4.8</td></tr> <tr><td>Octa-chloride</td><td>1.8</td><td>5.4</td></tr> <tr><td>Nona-chloride</td><td>1.3</td><td>3.9</td></tr> <tr><td>Deca-chloride</td><td>1.5</td><td>4.5</td></tr> <tr><td>PCB 77</td><td>0.69</td><td>2.1</td></tr> <tr><td>PCB 81</td><td>1.5</td><td>4.5</td></tr> <tr><td>PCB 105</td><td>2.2</td><td>6.6</td></tr> <tr><td>PCB 114</td><td>1.1</td><td>3.3</td></tr> <tr><td>PCB 118</td><td>3.7</td><td>11</td></tr> <tr><td>PCB 123</td><td>0.97</td><td>2.9</td></tr> <tr><td>PCB 126</td><td>0.96</td><td>2.9</td></tr> <tr><td>PCB 156</td><td>0.84</td><td>2.5</td></tr> <tr><td>PCB 157</td><td>1.2</td><td>3.6</td></tr> <tr><td>PCB 167</td><td>0.71</td><td>2.1</td></tr> <tr><td>PCB 169</td><td>1.4</td><td>4.2</td></tr> <tr><td>PCB 170</td><td>1.8</td><td>5.4</td></tr> <tr><td>PCB 180</td><td>1.5</td><td>4.5</td></tr> <tr><td>PCB 189</td><td>1.5</td><td>4.5</td></tr> </tbody> </table>	(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
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Analytical Method for the FY2003 Monitoring Investigation (continued)

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<p>(1) PCBs (continued)</p>	<p><b>Air</b> Sampled by high-volume air sampler (HV) with quartz-fiber-filter (QFF), polyurethane form (PUF) and active carbon felt (ACF) sorbent media.</p> <p>Internal standard (cleanup spike)</p> <p>Internal standard (cleanup spike)</p> <p>Internal standard (cleanup spike)</p> <p>Internal standard (sampling spike)</p> <p>Internal standard (cleanup spike)</p> <p>Internal standard (cleanup spike)</p> <p>Soxhlet extraction</p> <p>Soxhlet extraction</p> <p>Soxhlet extraction</p> <p>Acetone 2 hrs Toluene 16 hrs</p> <p>Acetone 16 hrs</p> <p>Acetone 2 hrs Toluene 16 hrs</p> <p>Dehydration / Concentration</p> <p>Concentration</p> <p>Dehydration / Concentration</p> <p>20 mL Constant volume</p> <p>10 mL</p> <p>20 mL Constant volume</p> <p>Hexane redissolution</p> <p>Hexane 50 mL, twice</p> <p>Washing</p> <p>Dehydration / concentration</p> <p>20 mL Constant volume</p> <p>Take a portion / Concentration</p> <p>20 mL</p> <p>Multilayer silica gel column cleanup</p> <p>Silica gel (0.9 g) 10%-AgNO<sub>3</sub> / silica gel (3 g) Silica gel (0.9 g) 22%-H<sub>2</sub>SO<sub>4</sub> / silica gel (3 g) 44%-H<sub>2</sub>SO<sub>4</sub> / silica gel (5 g) Silica gel (0.9 g) 2%-KOH / silica gel (1 g) Silica gel (0.9 g) Washing: hexane 70 mL Elution: hexane 100 mL</p> <p>Concentration</p> <p>100 µL</p> <p>Internal standard (syringe spike)</p> <p>Concentration</p> <p>100 µL</p> <p>GC/HRMS</p>	<p>GC/HRMS</p> <p>Resolution: 10,000</p> <p>Column: 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/Quantitation limit:</p> <table border="1"> <thead> <tr> <th data-bbox="1098 551 1203 577">Air (pg/m<sup>3</sup>)</th> <th data-bbox="1267 584 1353 611">Detection limit</th> <th data-bbox="1369 584 1453 611">Quantitation limit</th> </tr> </thead> <tbody> <tr> <td data-bbox="1129 611 1171 638">(1)</td> <td></td> <td></td> </tr> <tr> <td data-bbox="1098 638 1230 665">Mono-chloride</td> <td data-bbox="1283 638 1353 665">0.041</td> <td data-bbox="1385 638 1437 665">0.12</td> </tr> <tr> <td data-bbox="1098 665 1230 692">Di-chloride</td> <td data-bbox="1283 665 1353 692">0.33</td> <td data-bbox="1385 665 1437 692">1.0</td> </tr> <tr> <td data-bbox="1098 692 1230 719">Tri-chloride</td> <td data-bbox="1283 692 1353 719">1.1</td> <td data-bbox="1385 692 1437 719">3.2</td> </tr> <tr> <td data-bbox="1098 719 1230 745">Tetra-chloride</td> <td data-bbox="1283 719 1353 745">0.58</td> <td data-bbox="1385 719 1437 745">1.7</td> </tr> <tr> <td data-bbox="1098 745 1230 772">Penta-chloride</td> <td data-bbox="1283 745 1353 772">0.11</td> <td data-bbox="1385 745 1437 772">0.32</td> </tr> <tr> <td data-bbox="1098 772 1230 799">Hexa-chloride</td> <td data-bbox="1283 772 1353 799">0.029</td> <td data-bbox="1385 772 1437 799">0.086</td> </tr> <tr> <td data-bbox="1098 799 1230 826">Hepta-chloride</td> <td data-bbox="1283 799 1353 826">0.01</td> <td data-bbox="1385 799 1437 826">0.03</td> </tr> <tr> <td data-bbox="1098 826 1230 853">Octa-chloride</td> <td data-bbox="1283 826 1353 853">0.019</td> <td data-bbox="1385 826 1437 853">0.057</td> </tr> <tr> <td data-bbox="1098 853 1230 880">Nona-chloride</td> <td data-bbox="1283 853 1353 880">0.013</td> <td data-bbox="1385 853 1437 880">0.039</td> </tr> <tr> <td data-bbox="1098 880 1230 907">Deca-chloride</td> <td data-bbox="1283 880 1353 907">0.0057</td> <td data-bbox="1385 880 1437 907">0.017</td> </tr> <tr> <td data-bbox="1129 907 1203 934">PCB 77</td> <td data-bbox="1283 907 1353 934">0.0043</td> <td data-bbox="1385 907 1437 934">0.013</td> </tr> <tr> <td data-bbox="1129 934 1203 960">PCB 81</td> <td data-bbox="1283 934 1353 960">0.0051</td> <td data-bbox="1385 934 1437 960">0.015</td> </tr> <tr> <td data-bbox="1129 960 1203 987">PCB 105</td> <td data-bbox="1283 960 1353 987">0.0072</td> <td data-bbox="1385 960 1437 987">0.022</td> </tr> <tr> <td data-bbox="1129 987 1203 1014">PCB 114</td> <td data-bbox="1283 987 1353 1014">0.0082</td> <td data-bbox="1385 987 1437 1014">0.025</td> </tr> <tr> <td data-bbox="1129 1014 1203 1041">PCB 118</td> <td data-bbox="1283 1014 1353 1041">0.0050</td> <td data-bbox="1385 1014 1437 1041">0.015</td> </tr> <tr> <td data-bbox="1129 1041 1203 1068">PCB 123</td> <td data-bbox="1283 1041 1353 1068">0.0052</td> <td data-bbox="1385 1041 1437 1068">0.016</td> </tr> <tr> <td data-bbox="1129 1068 1203 1095">PCB 126</td> <td data-bbox="1283 1068 1353 1095">0.0089</td> <td data-bbox="1385 1068 1437 1095">0.027</td> </tr> <tr> <td data-bbox="1129 1095 1203 1122">PCB 156</td> <td data-bbox="1283 1095 1353 1122">0.0083</td> <td data-bbox="1385 1095 1437 1122">0.025</td> </tr> <tr> <td data-bbox="1129 1122 1203 1149">PCB 157</td> <td data-bbox="1283 1122 1353 1149">0.0077</td> <td data-bbox="1385 1122 1437 1149">0.023</td> </tr> <tr> <td data-bbox="1129 1149 1203 1176">PCB 167</td> <td data-bbox="1283 1149 1353 1176">0.007</td> <td data-bbox="1385 1149 1437 1176">0.021</td> </tr> <tr> <td 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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
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Analytical Method for the FY2003 Monitoring Investigation (continued)

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Analytical Method for the FY2003 Monitoring Investigation (continued)

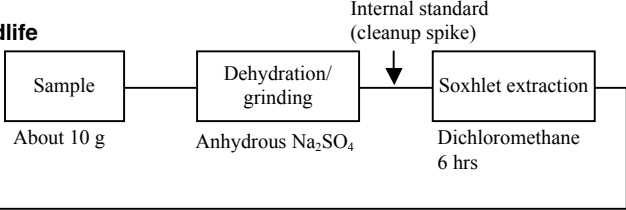
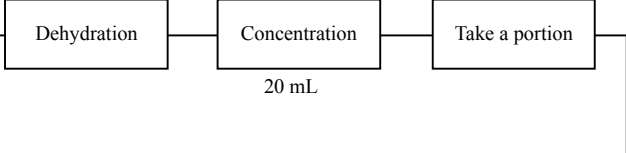
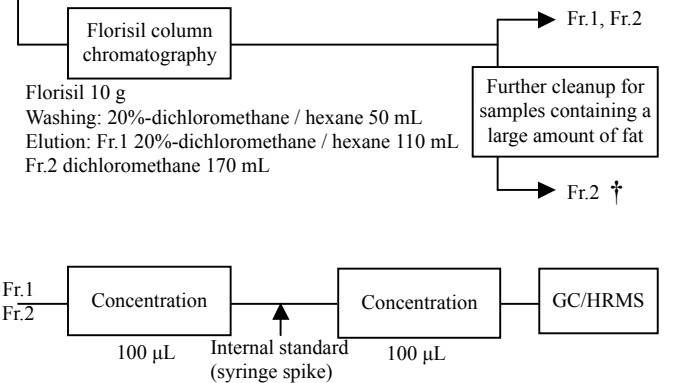
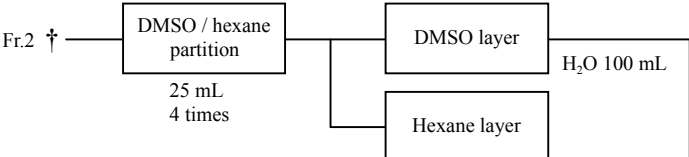
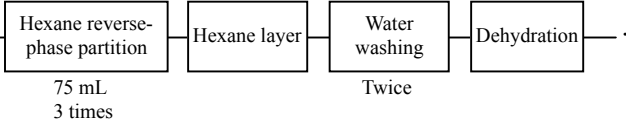
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Analytical Method for the FY2003 Monitoring Investigation (continued)

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<p>(7) Toxaphene                      (7.1) Parlar-26                      (7.2) Parlar-50                      (7.3) Parlar-62</p>	<p><b>Surface water</b></p> <p><b>Bottom sediment</b></p>	<p>GC/NICI-MS                      Column: RH12                      Column length: 60 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></th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr> <td>(7.1)</td> <td>20</td> <td>40</td> </tr> <tr> <td>(7.2)</td> <td>30</td> <td>70</td> </tr> <tr> <td>(7.3)</td> <td>90</td> <td>300</td> </tr> </tbody> </table> <p>Bottom sediment (pg/g-dry)</p> <table border="1"> <thead> <tr> <th></th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr> <td>(7.1)</td> <td>30</td> <td>90</td> </tr> <tr> <td>(7.2)</td> <td>50</td> <td>200</td> </tr> <tr> <td>(7.3)</td> <td>2000</td> <td>4000</td> </tr> </tbody> </table>		Detection limit	Quantitation limit	(7.1)	20	40	(7.2)	30	70	(7.3)	90	300		Detection limit	Quantitation limit	(7.1)	30	90	(7.2)	50	200	(7.3)	2000	4000
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Analytical Method for the FY2003 Monitoring Investigation (continued)

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(2) HCB		Other than toxaphene																																																
(3) Drins (3.1) Aldrin (3.2) Dieldrin (3.3) Endrin	<p><b>Wildlife</b></p> 	<p>GC/HRMS Column: DB-17HT Column length: 30 m Column I.D.: 0.32 mm Film thickness: 0.15 µm</p>																																																
(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		<p>Toxaphene GC/NICI-MS Column: HT8 Column length: 60 m Column I.D.: 0.25 mm Film thickness: 0.15 µm</p>																																																
(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		<p>Detection/Quantitation limit:</p> <p>Wildlife (pg/g-wet)</p> <table border="1" data-bbox="1102 763 1453 1379"> <thead> <tr> <th></th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr><td>(2)</td><td>7.5</td><td>23</td></tr> <tr><td>(3.1)</td><td>0.84</td><td>2.5</td></tr> <tr><td>(3.2)</td><td>1.6</td><td>4.8</td></tr> <tr><td>(3.3)</td><td>1.6</td><td>4.8</td></tr> <tr><td>(4.1)</td><td>3.5</td><td>11</td></tr> <tr><td>(4.2)</td><td>1.9</td><td>5.7</td></tr> <tr><td>(4.3)</td><td>3.3</td><td>9.9</td></tr> <tr><td>(4.4)</td><td>0.97</td><td>2.9</td></tr> <tr><td>(4.5)</td><td>1.2</td><td>3.6</td></tr> <tr><td>(4.6)</td><td>2.0</td><td>6.0</td></tr> <tr><td>(5.1)</td><td>2.4</td><td>7.2</td></tr> <tr><td>(5.2)</td><td>1.3</td><td>3.9</td></tr> <tr><td>(5.3)</td><td>1.2</td><td>3.6</td></tr> <tr><td>(5.4)</td><td>1.6</td><td>4.8</td></tr> <tr><td>(5.5)</td><td>2.8</td><td>8.4</td></tr> </tbody> </table>		Detection limit	Quantitation limit	(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
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(6) Heptachlors (6.1) Heptachlor (6.2) <i>trans</i> -Heptachlor epoxide (6.3) <i>cis</i> -Heptachlor epoxide	<p>Fr.1: HCB, DDTs, chlordanes, aldrin, heptachlor, HCHs, <i>trans</i>-heptachlor epoxide, mirex, toxaphene</p> <p>Note: GC/NICI-MS method is applied for toxaphene.</p> <p>Fr.2: endrin, dieldrin, <i>cis</i>-heptachlor epoxide</p>																																																	
(7) Toxaphene (7.1) Parlar-26 (7.2) Parlar-50 (7.3) Parlar-62	<p>Further cleanup of the Fr.2 is to be conducted by the following procedure for samples containing a large amount of fat</p> 																																																	
(8) Mirex																																																		
(9) HCHs (9.1) $\alpha$ -HCH (9.2) $\beta$ -HCH (9.3) $\gamma$ -HCH (9.4) $\delta$ -HCH																																																		

Analytical Method for the FY2003 Monitoring Investigation (continued)

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

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

Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks																					
<p>(10) Organotin compounds</p> <p>(10.1) TBT (10.2) DBT (10.3) TPT (10.4) DPT (10.5) MPT</p>	<p><b>Bottom sediment</b></p> <p>Add surrogate mixture 0.1 µg/mL (except MPT-d: 0.5 µg/mL) 100 µL</p> <pre> graph TD     Start[Sample 2g] --&gt; Add[Add surrogate mixture 0.1 µg/mL (except MPT-d: 0.5 µg/mL) 100 µL]     Add --&gt; E1[Extraction 1M HCl methanol / ethyl acetate (1:1) 10 mL Shaking 20 min]     E1 --&gt; C1[Centrifuging 2500 rpm 20 min]     C1 --&gt; TL[Top liquid layer]     C1 --&gt; R[Residue]     R --&gt; E2[Extraction 1M HCl methanol / ethyl acetate (1:1) 10 mL Shaking 20 min]     E2 --&gt; SLS[Solid-liquid separation]     SLS --&gt; TL     SLS --&gt; R2[Residue]     TL --&gt; C2[Concentration Rotary evaporator, until about 5 mL]     C2 --&gt; D[Derivatization Acetic acid - sodium acetate buffer solution (pH 5) 20 mL 2% NaBEt4 2 mL Shaking 10 min]     D --&gt; E3[Extraction Hexane 5 mL x 2 times]     E3 --&gt; C3[Centrifuging 2500 rpm 5 min]     C3 --&gt; Deh[Dehydration Anhydrous Na2SO4]     Deh --&gt; C4[Concentration Nitrogen gas blow until 1 mL]     R2 --&gt; C5[Concentration Nitrogen gas blow until 0.2 mL]     C4 --&gt; SPK[Sep-Pak Florisil cartridge Elution: 5% diethyl ether / hexane 6 mL]     C5 --&gt; SPK     SPK --&gt; C6[Concentration Nitrogen gas blow until 0.2 mL]     C6 --&gt; IS[Internal standard 1 µg/mL 20 µL]     IS --&gt; GC[GC/MS-SIM]     </pre>	<p>GC/MS (QP-MS)</p> <p>Column: DB-5MS Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 µm</p> <p>Detection/Quantitation limit:</p> <table border="1"> <thead> <tr> <th colspan="3">Bottom sediment (ng/g-dry)</th> </tr> <tr> <th></th> <th>Detection limit</th> <th>Quantitation limit</th> </tr> </thead> <tbody> <tr> <td>(10.1)</td> <td>0.4</td> <td>1.2</td> </tr> <tr> <td>(10.2)</td> <td>0.4</td> <td>1.2</td> </tr> <tr> <td>(10.3)</td> <td>0.09</td> <td>0.28</td> </tr> <tr> <td>(10.4)</td> <td>0.06</td> <td>0.16</td> </tr> <tr> <td>(10.5)</td> <td>0.8</td> <td>2.4</td> </tr> </tbody> </table>	Bottom sediment (ng/g-dry)				Detection limit	Quantitation limit	(10.1)	0.4	1.2	(10.2)	0.4	1.2	(10.3)	0.09	0.28	(10.4)	0.06	0.16	(10.5)	0.8	2.4
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Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks																		
<p>(10) Organotin compounds (continued)</p> <p>(10.1) TBT (10.2) DBT (10.3) TPT (10.4) DPT (10.5) MPT</p>	<p><b>Wildlife</b></p>	<p>GC/MS</p> <p>Column: DB-5MS Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 <math>\mu</math>m</p> <p>Detection/Quantitation limit:</p> <p>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>(10.1)</td> <td>1</td> <td>3</td> </tr> <tr> <td>(10.2)</td> <td>1</td> <td>3</td> </tr> <tr> <td>(10.3)</td> <td>0.5</td> <td>1.5</td> </tr> <tr> <td>(10.4)</td> <td>0.5</td> <td>1.5</td> </tr> <tr> <td>(10.5)</td> <td>5</td> <td>15</td> </tr> </tbody> </table>		Detection limit	Quantitation limit	(10.1)	1	3	(10.2)	1	3	(10.3)	0.5	1.5	(10.4)	0.5	1.5	(10.5)	5	15
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<p>(11) Tetrabromo bisphenol A</p>	<p><b>Bottom sediment</b></p>	<p>GC/MS-SIM</p> <p>Column: SGE BPX-5 Column length: 30 m Column I.D.: 0.25 mm Film thickness: 0.25 <math>\mu</math>m</p> <p>Detection/Quantitation limit:</p> <p>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>(11)</td> <td>5.5</td> <td>18</td> </tr> </tbody> </table>		Detection limit	Quantitation limit	(11)	5.5	18												
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Analytical Method for the FY2003 Monitoring Investigation (continued)

Substance	Analytical Method/Flow Chart	Remarks						
<p>(11) Tetrabromo bisphenol A (continued)</p>	<p><b>Wildlife</b></p> <p>Internal standard (cleanup spike) <sup>13</sup>C<sub>12</sub>-TBBPA</p> <p>Sample 10 g</p> <p>Homogenization Methanol 50 mL x 2 times</p> <p>Hexane washing</p> <p>Extraction Dichloromethane 50 mL x 2 times</p> <p>Dehydration / Concentration Until 20 mL</p> <p>Collect a portion</p> <p>Concentration to dryness</p> <p>Derivatization 1M KOH / EtOH 0.5 mL; diethyl sulfate 0.2mL</p> <p>alkali decomposition 70°C, 1 hr</p> <p>Extraction Hexane 1 mL x 2 times</p> <p>Dehydration / Concentration</p> <p>Multilayer silica gel column cleanup</p> <p>Silica gel (0.5 g) 10%-AgNO<sub>3</sub> / silica gel (2 g) Silica gel (0.5 g) 22%-H<sub>2</sub>SO<sub>4</sub> / silica gel (3 g) 44%-H<sub>2</sub>SO<sub>4</sub> / silica gel (5 g) Silica gel (0.5 g) 2%-KOH / silica gel (1 g) Silica gel (0.5 g) Washing: 10%-dichloromethane / hexane 100 mL Pre-posting: 10%-dichloromethane / hexane 50 mL Elution: 50%-dichloromethane / hexane 100 mL</p> <p>Concentration 50 µL</p> <p>Internal standard (syringe spike) <sup>13</sup>C<sub>12</sub>-2,2',3,4,4',6-HxBDE (#139)</p> <p>Concentration 50 µL</p> <p>GC/HRMS</p>	<p>GC/HRMS</p> <p>Column: DB-5MS Column length: 60 m Column I.D.: 0.32 mm Film thickness: 0.25 µm</p> <p>or</p> <p>Column: DB-17HT Column length: 60 m Column I.D.: 0.32 mm Film thickness: 0.15 µm</p> <p>Detection/Quantitation limit:</p> <p>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>(11)</td> <td>0.030</td> <td>0.090</td> </tr> </tbody> </table>		Detection limit	Quantitation limit	(11)	0.030	0.090
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