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**Testing methods for
polychlorobiphenyl in industrial
water and wastewater**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Industrial Water Association (JIWA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS K 0093 : 2002** is replaced with this Standard.

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Testing methods for polychlorobiphenyl in industrial water and wastewater

1 Scope This Japanese Industrial Standard specifies the testing method for polychlorobiphenyl (hereafter referred to as “PCB”) in industrial water and wastewater, using gas chromatography or gas chromatography–mass spectrometry.

NOTES 1 The objects of this Standard are admixtures from monochlorobiphenyl to decachlorobiphenyl.

2 The gas chromatography is the method of determination for total PCB and the gas chromatography–mass spectrometry is the method of determination for PCB in every isomer of the same number of chlorine. When the quantities of total PCB are obtained by these two methods, in some cases, the determined values are not identical due to the characteristics of respective methods.

2 Normative references The standards given in attached table 1 contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) shall be applied.

3 Common items The common items are as follows:

- a) **General rules** General rules for chemical analysis shall be based on **JIS K 0050**.
- b) **Definitions** For the purposes of this Standard, the definitions given in **JIS K 0101**, **JIS K 0102**, **JIS K 0211** and **JIS K 0215** apply.
- c) **Gas chromatography** The common items for gas chromatography shall comply with **JIS K 0114**.
- d) **Gas chromatography–mass spectrometry** The common items for gas chromatography–mass spectrometry shall comply with **JIS K 0123**.
- e) **Determination range** The determination range shown in each testing method is expressed by mass (ng) of PCB in solvent to be injected into equipment used for each testing method.
- f) **Repeatability** The repeatability shall be represented by approximate value of coefficient of variation (%) obtained through repeated tests using standard solution.
- g) **Water** The water used in this Standard shall comply with A1 to A4 in **JIS K 0557**.
- h) **Reagents**
 - 1) Reagents specified in **JIS** without detrimental effects on test shall be used. If not specified in **JIS**, they shall be those without detrimental effects on test.
 - 2) The concentration of standard liquid is represented by mg/ml, µg/ml or ng/ml.

- 3) The concentration of the mixture solution of A and B is represented by C (a+b), which means that A and B are mixed in the ratio of *a* ml to *b* ml.
 - 4) Concentration shown in parentheses after the name of the standard solution is the exact concentration.
 - 5) The names of reagents are harmonized with the names specified in both the Chemicals Denomination Rules set by The Chemical Society of Japan and **JIS** reagents based on the Nomenclature of inorganic and organic chemistry set by International Union of Pure and Applied Chemistry (IUPAC).
 - 6) For the reagents (for testing) used for the preparation of standard solutions, the reference material of which the traceability is ensured as much as possible or the certified reference material shall be used. When they are not available, those of which the purity and uncertainty are clarified shall be used. They are indicated with the word "standard" in this Standard. Similar compounds may be contained as impurity in them in some cases, therefore it has to be ensured that those without detrimental effects on test shall be used.
 - 7) Precautions shall be taken against indoor pollution, inhalation by and attachment to human body, of reagents and wastewater, etc. Handling of reagents and wastewater, etc. shall be in accordance with related laws and regulations and full cautions shall be paid.
- i) **Glassware** General matters of glassware shall be as follows:
- 1) Glassware in accordance with **JIS R 3503** and **JIS R 3505** shall be used unless otherwise specified. Vaseline and the like shall not be used on the cock part of tube for column, separatory funnel and the like. For the cock part of separatory funnel and the like, the one of polytetrafluoroethylene may be used. When heating operation is necessary, borosilicate glass-1 specified in **JIS R 3503** shall be used.

Desiccant for desiccators shall be silica gel⁽¹⁾ unless otherwise specified.

Note ⁽¹⁾ Silica gel type A grade 1 for packages specified in **JIS Z 0701** shall be used.
 - 2) Glassware used for testing shall be washed with hexane rinse water of **5.1 a)** in advance and cleaned with hexane (concentration 300 specified in **JIS K 8825**), and then left to volatilize hexane.
- j) **Working curves (gas chromatography-mass spectrometry)** For preparation of working curve, the standard solution shall be taken in amounts of 4 to 6 steps within the range specified in the test procedure which correspond to the PCB amounts at the time of introduction into the gas chromatograph-mass spectrometer.

4 Samples

4.1 Sampling

4.1.1 Sample container Glass bottle with a ground stopper, 1 000 ml in capacity⁽²⁾.

Note (2) Instead of a ground stopper, screw cap with liner of polytetrafluoroethylene may be used.

4.1.2 Sampling operation Sampling operation shall be as follows:

- a) **Sampling of surface water** Sample in accordance with 4.1.1 or 4.1.2 of JIS K 0094.
- b) **Sampling of water at various depths** Sample in accordance with 4.1.4 of JIS K 0094.
- c) **Sampling from piping** Sample in accordance with 4.3 of JIS K 0094.

4.2 Handling of sample Handling of the sample shall be as follows:

- a) The sample taken in accordance with 4.1.2 shall not be transferred to another vessel or a part of it taken out. All of the sampled volume shall be used for the test.
- b) The volume of the sample shall be determined by reducing the mass of container from the mass of container containing the sample or determined as the water volume required to fill up to the mark of the container, at the completion of test.
- c) Test shall be performed immediately after sampling. If this is not practicable, the sample shall be stored in a dark place at 0 °C to 10 °C and test shall be performed at earliest convenience. When storing in a cold place, it shall be ensured that the sample does not freeze.

5 Gas chromatography After extraction with hexane, dehydration and concentration of PCB in the sample, alkali decomposition shall be conducted. Extract again the decomposed solution with hexane and dehydrate and concentrate it. After column chromatographic separation using silica gel has been performed, the eluate shall be concentrated again to a specified volume. Inject a specified volume of this solution into gas chromatograph and determine by the method using electron capture detector (ECD).

Alkali decomposition may be omitted when there is little disturbance.

Determination range PCB: 0.5 ng or more Repeatability: 20 % (varies depending on equipment and measuring conditions)

5.1 Reagents Reagents shall comply with the following:

- a) **Hexane rinse water** Added with 100 ml of hexane specified in g) per 1 L of A4 (or A3) water specified in 3 g), shaken well and cleaned. This solution shall be used for preparation of reagents, test procedures, and blank tests.
- b) **Sulfuric acid** As specified in JIS K 8951.
- c) **Potassium hydroxide** As specified in JIS K 8574.
- d) **Sodium sulfate** That specified in JIS K 8987, heated for 60 min at about 600 °C, and allowed to cool in a desiccator.

- e) **Acetone** Acetone of concentration 300 or more as specified in **JIS K 8040**⁽³⁾.

Note ⁽³⁾ Before use, 50 ml of acetone shall be concentrated to approximately 1 ml in accordance with the operation in **5.3 d)** and for 1 µl to 10 µl of concentrated acetone confirm, in accordance with **5.4 c), d)** or **6.4 b)**, confirm that the peak value due to PCB will not be detected.

- f) **Ethanol** Concentration 300 or more specified in **JIS K 8093**⁽³⁾.

- g) **Hexane** Concentration 300 or more specified in **JIS K 8825**⁽⁴⁾.

Note ⁽⁴⁾ The operation described in Note ⁽³⁾ is carried out and it is confirmed beforehand that the peak value due to PCB will not be detected. Provided that, for hexane, 100 ml of it is used.

- h) **Potassium hydroxide-ethanol solution** Dissolve 70 g of potassium hydroxide into as small a quantity of hexane rinse water as possible and add ethanol to make approximately 1 L. Put the solution in a polyethylene bottle and leave it to stand for 2 or 3 days preventing contacting with carbon dioxide and the supernatant shall be transferred into alkali resistant vessel to store with soda-lime tube.

- i) **Hexane-ethanol mixed solution (1+1)**

- j) **Trichlorobiphenyl (C₁₂H₇Cl₃) standard solution (0.1 mg/ml)** Dissolve 10 mg of trichlorobiphenyl for testing in a small amount of hexane and transfer the whole volume into a volumetric flask of 100 ml and add hexane up to the marked line⁽⁵⁾.

Note ⁽⁵⁾ Shall be stored in a dark place maintained at a temperature 5 °C or lower.

- k) **Tetrachlorobiphenyl (C₁₂H₆Cl₄) standard solution (0.1 mg/ml)** Dissolve 10 mg of tetrachlorobiphenyl for testing in a small amount of hexane and transfer the whole volume into a volumetric flask of 100 ml and add hexane up to the marked line⁽⁵⁾.

- l) **Pentachlorobiphenyl (C₁₂H₅Cl₅) standard solution (0.1 mg/ml)** Dissolve 10 mg of pentachlorobiphenyl for testing in a small amount of hexane and transfer the whole volume into a volumetric flask of 100 ml and add hexane up to the marked line⁽⁵⁾.

- m) **Hexachlorobiphenyl (C₁₂H₄Cl₆) standard solution (0.1 mg/ml)** Dissolve 10 mg of hexachlorobiphenyl for testing in a small amount of hexane and transfer the whole volume into a volumetric flask of 100 ml and add hexane up to the marked line⁽⁵⁾.

Information : Trichlorobiphenyl, tetrachlorobiphenyl, pentachlorobiphenyl and hexachlorobiphenyl for testing purpose are available on the market as KC-300, KC-400, KC-500 and KC-600 respectively.

- n) **PCB standard solution (20 µg/ml)** Put each 0.5 ml of trichlorobiphenyl (C₁₂H₇Cl₃) standard solution (0.1 mg/ml), tetrachlorobiphenyl (C₁₂H₆Cl₄) standard solution (0.1 mg/ml), pentachlorobiphenyl (C₁₂H₅Cl₅) standard solution (0.1 mg/ml) and hexachlorobiphenyl (C₁₂H₄Cl₆) standard solution (0.1 mg/ml) in a volumetric flask of 10 ml and add hexane up to the marked line. One millilitre of this solution contains 5 µg each of four kinds of PCBs for testing in **j)** to **m)**, thus total of 20 µg⁽⁵⁾.

- o) **PCB standard solution (1 µg/ml)** Put 0.5 ml of PCB standard solution (20 µg/ml) in a volumetric flask of 10 ml and add hexane up to the marked line. One millilitre of this solution contains 0.25 µg each of four kinds of PCBs for testing in **j)** to **m)**, thus total of 1 µg⁽⁵⁾.
- p) **Nitrogen** Nitrogen of Grade 2 specified in **JIS K 1107**.

5.2 Apparatus and instruments Apparatus and instruments shall be as follows:

- a) **Separating funnel** 200 ml to 2 000 ml in capacity. Before use, it has to be cleansed with hexane.
- b) **Concentrator** Rotary evaporator or Kuderna-Danish concentrator⁽⁶⁾.
Note ⁽⁶⁾ Those without capillary tubes.
- c) **Concentration flask** Egg plant shape flask for rotary evaporators and 500-ml concentrator flask with concentrator tube for Kuderna-Danish concentrator.
- d) **Flask** 200 ml in capacity with interchangeable ground-in stopper, for thermolysis.
- e) **Reflux condenser** Interchangeable Liebig condenser or tubular bulb condenser. 300 mm.
- f) **Micro syringe** Proper syringe of 1 µl to 10 µl, or automatic injector.
- g) **Column chromatograph tube** Column chromatograph tube shall be in accordance with the following:
 - 1) **Tube for column** Glass column of 10 mm inside diameter and of approximately 300 mm in length with a cock.
 - 2) **Column packing** Silica gel (powder) for PCB analysis of 150 µm to 250 µm in particle diameter, and allowed to cool in a desiccator after heated for 15 h or more at approximately 130 °C⁽⁷⁾.
Note ⁽⁷⁾ Two grammes of heat-dried silica gel is made to be a column chromatograph tube in accordance with **3)**, the operation applied correspondingly to **5.3 m)** to **n)** is carried out [provided that the operation of Note ⁽¹⁶⁾ is omitted.], 5 µL of it is injected into gas chromatograph by using a micro syringe and it is confirmed that the peak value will not be detected at the corresponding position to retention time of PCB.
 - 3) **Preparation of column chromatograph tube** At the bottom of tube, glass wool (cleaned with hexane and dried in advance) specified in **JIS K 8251** shall be filled in, bubble shall be removed by adding a small amount of hexane. Then, place 2 g of column packing in a beaker and make the packing slurry with addition of hexane, and in pour this slurry into column tube preventing bubbles from coming in. Also wash the column packing adhered to inside wall of a beaker with a small amount of hexane into the chromatograph tube. Then, add 1 g of sodium sulfate on the top of the column packing and adjust hexane to be a little bit over the sodium sulfate layer.
- h) **Cylindrical dropping funnel** 500 ml in capacity. For column chromatograph.

i) **Gas chromatograph** Shall comply with the following:

- 1) **Tube for column** Glass tube of 2 mm to 4 mm inside diameter and 1.5 m to 2.0 m in length.
- 2) **Column packing** That which is made by covering the carrier made of diatomaceous earth (grain size 150 μm to 180 μm), which has been treated with acid and then with silane, with 1.5 % to 5 % of silicone series stationary liquid. Any column packing having the separation performance equivalent to this may be used.

Information : Some of the column packing available on the market are coated with 1.5 % to 5 % of OV-1, OV-17, etc. as silicone stationary liquid on carries of Chromosorb W AW-DMCS, Uniport HP, Shimalite W AW-DMCS or those with equivalent performance (particle size is 150 μm to 180 μm each).

- 3) **Detector** Electron capture detector (ECD).
- 4) **Carrier gas** Helium [99.9 % (volume %) or more] or nitrogen specified in 5.1 p) purity.
- 5) **Flow rate of carrier gas** Flow rate shall be adjusted to 30 ml/min to 80 ml/min.
- 6) **Temperature of vaporizing chamber for sample** 200 °C to 250 °C.
- 7) **Temperature of column chamber** 180 °C to 250 °C.
- 8) **Temperature of detector chamber** 200 °C to 250 °C.

j) **Shaker**

5.3 Preparation operations Preparation operations shall be in accordance with the following:

- a) The sample taken in accordance with 4.1.2 shall be transferred into a separatory funnel of 2 000 ml. The inside wall of the sample container shall be washed well with 50 ml of hexane and the washing shall be poured into the separatory funnel⁽⁸⁾, and mixed well for about 10 min by means of shaker. After completion of this mixing, let it stand⁽⁹⁾.

Notes ⁽⁸⁾ When suspension in sample is in very high amount, there is a possibility that extraction is insufficient, so add 50 ml of acetone.

⁽⁹⁾ When emulsion is produced, add 5 to 7 drops of sulfuric acid and shake to mix.

- b) Transfer the separated water layer into another separatory funnel of 2 000 ml and transfer separated hexane solution into an Erlenmeyer flask. Add 50 ml of hexane to the water in the separatory funnel and mix it again for about 10 min by means of shaker and let it stand⁽⁹⁾. Discard the water layer and put the hexane layer into the above Erlenmeyer flask.
- c) Add approximately 10 g of sodium sulfate to the hexane and mix it by shaking lightly. After about 10 min of letting it stand dehydrate it⁽¹⁰⁾. Filter it with a filter paper Class 5 A (or Class 5 B)⁽¹¹⁾ and receive the filtrate into concentrator

flask. With a small volume of hexane, rinse the Erlenmeyer flask a few times and also the above filter paper and the sodium sulfate on the filter paper by the washing, and the washing shall be added to the concentrator flask.

Notes ⁽¹⁰⁾ As for alternative dehydration method, there is a method to freeze water by keeping extracted solution in a dark place at -20°C . This method should be applied when concentration operation would not be performed on the same day.

⁽¹¹⁾ Filter papers shall be rinsed in advance with hexane before use.

- d) Using a concentrator and heating⁽¹²⁾ in a water bath of approximately 40°C temperature, concentrate the hexane until it becomes about 5 ml on a water bath⁽¹³⁾.

Notes ⁽¹²⁾ When a rotary evaporator is used, concentrate in a water-bath of about 40°C under decompression method. Since evaporation-drying may occur, caution shall be needed. When a Kuderna-Danish type concentrator is used, concentration will be performed by heating at 75°C or lower under ambient pressure instead of the decompression method. After completion of concentration, remove the snyder-column together with the concentration part from the apparatus. Rinse the snyder-column with small volume of hexane and cool the concentrator with a snyder-column attached.

⁽¹³⁾ When operations of **e)** and successive ones cannot be performed immediately, store the solution in a dark place at -20°C or lower.

- e) Transfer the whole concentrated solution to a flask and the inside wall of the concentrator flask in which concentrated solution has been contained shall be rinsed with 25 ml of potassium hydroxide–ethanol solution twice, and the washing shall be put into the flask.
- f) After mounting a reflux condenser on the flask, heat the flask on a boiling water bath for about 1 h and let it stand until it cools down to about 50°C .
- g) Add 100 ml of hexane to this solution and after shaking it moderately to mix, let it stand until it cools down to room temperature. Transfer this solution into a separatory funnel of 200 ml and rinse the inside wall of the flask with a small volume of hexane–ethanol mixture (1+1) 2 or 3 times to add the washing to this separatory funnel.
- h) Add 25 ml of hexane rinse water to this 200 ml separatory funnel and let it stand after mixing by shaking⁽¹⁴⁾. Transfer the water layer to another 200 ml separatory funnel and transfer the hexane layer to an Erlenmeyer flask. Add 50 ml of hexane to the water layer transferred in a 200-ml separatory funnel and mix it by shaking to left it stand. Discard the water layer and add the hexane layer to the above hexane layer.

Note ⁽¹⁴⁾ When emulsion is produced, add 5 ml to 7 ml of ethanol and shake gently to mix.

- i) Add 100 ml of hexane rinse water to this hexane solution, and mix by shaking vigorously and rinse it. Repeat further this rinsing operation twice.
- j) Carry out the operations of **c)** and **d)** on the hexane solution rinsed.

- k) Pour the concentrated hexane solution gently into the top of column chromatograph tube. The container in which this concentrated hexane solution has been contained shall be rinsed a few times with about 1 ml of hexane and this washing also shall be poured gently into the top of column chromatograph tube.
- l) Further, after rinsing the inside wall of the upper portion of the column chromatograph tube with a small volume of hexane, open the cock at the lower portion of column chromatograph tube and lower the liquid level of hexane to the top surface of sodium sulfate.
- m) Mount a cylindrical dropping funnel on the top of column chromatograph tube and let hexane of 500 ml run down at a rate of 1 drop per second⁽¹⁵⁾, and collect the eluate of the PCB fraction⁽¹⁶⁾.

Notes ⁽¹⁵⁾ If necessary, it may be pressurized using nitrogen.

⁽¹⁶⁾ Eluate of fraction containing all PCBs and not organic chlorinated compounds except PCB and [1,1'-(2,2'-dichlorobinylidene) bis (4-chlorobenzen)] (DDE). Elution range may vary heavily depending on slight difference of PCB content in the sample and activity of packing (silica-gel) in the chromatograph tube and eluate fraction shall be confirmed using the standard solution of PCB in advance.

- n) Transfer this eluate into a concentrator flask and concentrate to 5 ml or less in volume⁽¹²⁾, and add hexane to be 5 ml⁽¹³⁾.
- o) As a blank test, put hexane rinse water of the same volume as the sample into a 2 000 ml separatory funnel and carry out the operations in **a)** to **n)**.

NOTES 1 For sample with little disturbance, alkali decomposition in **e)** to **j)** may be omitted.

- 2 When sample contains much fat (biological greases) and the residue remains in hexane layer without being decomposed even after operations specified in **e)** to **j)**, the eluate obtained from column chromatographic separation described in **k)** to **m)** will contain grease. Due to this residue, disturbance peak may arise on chromatogram for PCB measurement by a gas chromatograph, and in this case, on succession to the operation in **j)**, the operation of column chromatographic separation by using active magnesium silicate as specified in Annex 1 (normative) shall be applied to separate grease.

5.4 Operation Operation shall be in accordance with the following:

- a) Inject 5 µl of PCB standard solution (1 µg/ml) figures 1 and 2 into the gas chromatograph by means of a micro syringe.
- b) Allocate numbers (hereafter referred to as “peak number”) based on figures 1 and 2 to the peaks of chromatogram obtained, and read the height of each peak (mm). After *K*-value⁽¹⁸⁾ of each peak shall be calculated by the following equation from the height (*H*₁) and *CB*₀ (%) (table 1)⁽¹⁷⁾ that corresponds to the peak number of the peak.

$$K = \frac{CB_0(\%)}{H_1}$$

Note ⁽¹⁷⁾ As the relative sensitivity of electron capture detector will vary considerably depending on the conditions, draw a working curve based on peak numbers in figures 1 and 2, or rate of peak content in table 1 for PCB standard solution, and determine the range of linearity in advance.

Table 1 Example of CB_0 (%)

a) When OV-1 is used as a coating of column packing

Peak number	1	2	3	4	5	6	7	8	9	10
$CB_0(\%)$	1.67	5.78	2.68	7.57	5.23	7.88	4.83	3.30	10.68	2.37

Peak number	11	12	13	14	15	16*	17	18	19	20
$CB_0(\%)$	5.70	3.16	4.20	1.24	6.44	6.16	1.68	4.45	3.45	3.15

Peak number	21	22	23	24	25	26	Σ
$CB_0(\%)$	3.47	1.27	1.54	0.29	0.71	0.21	99.11

Note * The peak number 16 may be split into 16 [$CB_0(\%) = 2.16$] and 16' [$CB_0(\%) = 4.00$] depending on conditions.

b) When OV-17 is used as a coating of column packing

Peak number	1	2	3	4	5	6	7	8	9	10
$CB_0(\%)$	1.69	6.00	3.17	6.60	2.74	1.35	8.62	4.86	2.54	2.09

Peak number	11	12	13	14	15	16	17	18	19	20
$CB_0(\%)$	8.65	7.05	0.99	3.18	5.42	6.35	4.28	4.00	4.75	2.82

Peak number	21	22	23	24	25	26	27	28	29	Σ
$CB_0(\%)$	0.23	2.26	1.57	3.30	0.08	2.95	0.28	0.71	0.15	98.68

Note ⁽¹⁸⁾ K -value may vary depending on operation conditions of gas-chromatograph such as contamination on linear source, etc. Therefore, K -value shall be calculated by gas-chromatograph using the standard solution of PCB measured under the same condition as in measurement of the sample.

- A proper volume of 1 μ l to 10 μ l (for example, 5 μ l) of hexane solution obtained in **5.3 n**) shall be injected into gas chromatograph with a micro syringe.
- Each peak on the chromatogram obtained shall be given in peak number of the peak at the corresponding position on the chromatogram for PCB standard solution.
- Then, read the heights of each peak (in mm) and calculate $CB_2(\%)$ from the following equation based on the height (H_2) and K -value for the peak number of the peak.

$$CB_2(\%) = K \times H_2$$