



JAPANESE
INDUSTRIAL
STANDARD

Translated and Published by
Japanese Standards Association

JIS K 0450-10-10 : 2006

(JIWA/JSA)

**Testing method for bisphenol A in
industrial water and waste water**

ICS 13.060.50

Reference number : JIS K 0450-10-10 : 2006 (E)

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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 0450-10-10:2000** is replaced with this Standard.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

JIS K 0450 consists of the following parts.

JIS K 0450-10-10 *Testing method for bisphenol A in industrial water and waste water*

JIS K 0450-20-10 *Testing method for alkyl phenols in industrial water and wastewater*

JIS K 0450-30-10 *Testing method for phthalic acid esters in industrial water and wastewater*

JIS K 0450-40-10 *Testing method for hexanedioic acid bis(2-ethylhexyl) ester in industrial water and wastewater*

JIS K 0450-50-10 *Testing method for benzophenone in industrial water and wastewater*

Date of Establishment: 2000-11-20

Date of Revision: 2006-03-25

Date of Public Notice in Official Gazette: 2006-03-27

Investigated by: Japanese Industrial Standards Committee

Standards Board

Technical Committee on Environment and Recycling Policy

JIS K 0450-10-10:2006, First English edition published in 2008-03

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

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Testing method for bisphenol A in industrial water and waste water

1 Scope This Japanese Industrial Standard specifies the testing method for 4,4'-(1-methylethylidene) bisphenol [bisphenol A] ($C_{15}H_{16}O_2$) (CAS No. 80-05-7) in industrial water and waste water.

2 Normative references The standards listed in attached table 1 contain provisions which, through reference in this text, constitute provisions of this part of **JIS K 0450**. The most recent editions of these standards (including amendments) shall be applied.

3 Common matters Common matters shall be as follows.

- a) **General rules** The general matters common to chemical analysis shall be in accordance with **JIS K 0050**.
- b) **Terms and definitions** For the purposes of this part of **JIS K 0450**, the definitions given in **JIS K 0101**, **JIS K 0102**, **JIS K 0211** and **JIS K 0215** apply.
- c) **Gas chromatography/mass spectrometry method** The general matters common to gas chromatography/mass spectrometry shall be in accordance with **JIS K 0123**.
- d) **Name of compound** For the name of compound, the nomenclature of compounds [based on the nomenclature of organic chemistry of International Union of Pure and Applied Chemistry (IUPAC)] established by the Chemical Society of Japan shall be used, and the common name thereof shall be suffixed in parentheses.
- e) **Water** The water to be used in this Standard shall be A1 to A4 water specified in **JIS K 0557**.
- f) **Reagents**
 - 1) The reagents, which are specified in **JIS** and do not hinder the test, shall be used. When there are no specifications in **JIS**, those which do not hinder the test shall be used.
 - 2) The concentration of standard solution shall be expressed in mg/ml, μ g/ml or ng/ml.
 - 3) The concentration of the mixed solution of A and B shall be expressed by C(a+b). This expression indicates that A and B are mixed in the rate of a ml and b ml.
 - 4) The concentration indicated in parentheses behind the name of standard solution means that it is a correct concentration.
 - 5) The designations of reagents shall basically conform to the nomenclature of compounds established by the Chemical Society of Japan based on the nomenclature of inorganic chemistry and the nomenclature of organic chemistry of the International Union of Pure and Applied Chemistry (IUPAC) and the designations of **JIS** reagents.

- 6) The standard substances or the approved standard substances of which the traceability is secured to a maximum extent shall be used for the reagents for the preparation of standard solution. When they are not available, those of which purity and uncertainty are clarified shall be used. In this Standard, they are described as “standard products”. Some of them may include the resembling compounds as impurities, so those which do not hinder the test shall be used.
 - 7) Care shall be taken of the room pollution, the inhalation and adherence to human body due to reagents and waste liquids. For handling reagents and wastewater, care shall be sufficiently taken to ensure that the applicable laws and regulations shall be observed.
- g) **Glasswares** The common matters of glasswares shall be as follows.
- 1) Unless otherwise specified, the glasswares specified in **JIS R 3503** and **JIS R 3505** shall be used. However, for the cock parts of the separatory funnel and the like, those made of tetrafluoroethylene may be used. When heating operation is accompanied, the borosilicate glass-1 specified in **JIS R 3503** shall be used.

For the desiccant to be used in a desiccator, unless otherwise specified, silica gel⁽¹⁾ shall be used.

Note ⁽¹⁾ The desiccant of Type A Class 1 silica gel for packing specified in **JIS Z 0701** shall be used.
 - 2) The glasswares to be used in the test shall be washed with water before using, and then furthermore washed with acetone (of the quality of concentration 300 or more, specified in **JIS K 8040**), and acetone shall be volatilized by leaving it as it is. Afterward, they shall be heated about at 200 °C about for 2 h and be allowed to cool at a pollution-free place.
- h) **Working curve** For the preparation of the working curve, the determination range shown in the test method shall be divided into four stages to six stages, and the standard solution shall be taken so that the amount of bisphenol A on introduction to the gas chromatograph mass spectrometer shall coincide with respective amount. The working curve shall be prepared within the determination range.

4 Sample

4.1 Sampling

4.1.1 Sample container For the sample container, a 1 000 ml glass bottle⁽²⁾ with grounding stopper shall be used.

Note ⁽²⁾ A screw cap (with a tetrafluoroethylene packing) may be used instead of a grounding stopper.

4.1.2 Sampling operation The sampling operation shall be as follows.

- a) **Sampling of surface water** In accordance with 4.1.1 or 4.1.2 of **JIS K 0094**.

- b) **Sampling of water at each depth** In accordance with **4.1.4** of **JIS K 0094**.
- c) **Sampling from piping apparatus** In accordance with **4.3** of **JIS K 0094**.

4.2 Handling of sample The test shall be carried out immediately after sampling. When it is unable, it shall be preserved at a dark place of 0 °C to 10 °C and be tested as quickly as possible. When it is preserved at a cool place, it shall be made so as not to be frozen.

5 Pretreatment of sample For the pretreatment of sample, the solvent extraction method or the solid phase extraction method shall be applied. After the analyte is concentrated, the column chromatographic separation and derivatization shall be carried out. For the sample containing suspended matters, the solid phase extraction method may influence extraction and therefore, care shall be taken.

When plenty of suspended matters are contained in the sample, carry out operations of NOTE 2.

5.1 Solvent extraction method Adjust the pH value to about 3 by adding hydrochloric acid in the sample, add sodium chloride, add bisphenol A-*d*₁₆ as the internal standard material, extract by using dichloromethane, then dehydrate and concentrate the extracted solution⁽³⁾.

Note ⁽³⁾ In accordance with **3 f) 7)**.

5.1.1 Reagents The reagents shall be as follows.

- a) **Water** The water of A4 (or A3) in **3 e)** shall be used⁽⁴⁾ for the preparation of reagents, operations and the blank test.

Note ⁽⁴⁾ Use the water immediately after purified. When the confirmation of water quality is required, carry out the operation of Annex 1 (normative) before using, and confirm that the peak is not found at the position corresponding to the retention time of bisphenol A. When the purification is additionally required, treat by the method according to Remarks 6 in clause 4 of **JIS K 0557** or treat the water of A4 or A3 with activated carbon and the like.

- b) **Hydrochloric acid (1 mol/L)** Prepare by using the hydrochloric acid specified in **JIS K 8180**.
- c) **Sodium chloride** Heat the sodium chloride specified in **JIS K 8150** at about 600 °C for about 60 min, and then allow it to cool in a desiccator.
- d) **Sodium sulfate** Heat the sodium sulfate specified in **JIS K 8987** at about 600 °C for about 60 min, and then allow it to cool in a desiccator.
- e) **Acetone** Of 300 or more in concentration specified in **JIS K 8040**⁽⁵⁾.

Note ⁽⁵⁾ Before using, carry out the operation of **5.1.3 d)** and **e)** by using 30 ml and the operation of derivatization of **5.3.3 g)** and **h)** by using the concentrated solution obtained. Next, carry out the operation of **6.3 c)** by using this solution and confirm that the peak is not found at the position corresponding to the retention time of bisphenol-A.

- f) **Dichloromethane** Of 300 or more in concentration specified in **JIS K 8117**⁽⁶⁾.

Note ⁽⁶⁾ Before using, carry out the operation of **5.1.3 d)** and **e)** by using 120 ml and the operation of derivatization of **5.3.3 g)** and **h)** by using the concentrated solution obtained. Next, carry out the operation of **6.3 c)** by using this solution and confirm that the peak is not found at the position corresponding to the retention time of bisphenol-A.

- g) **Bisphenol A-*d*₁₆ internal standard solution (10 µg/ml–acetone)** Take 0.100 g of bisphenol A-*d*₁₆ (C₁₅H₁₆O₂-*d*₁₆) standard product and transfer it to a 100 ml volumetric flask and add acetone up to the marked line. Take 1 ml of this solution and transfer it to a 100 ml volumetric flask and add acetone up to the marked line⁽⁷⁾.

Note ⁽⁷⁾ Preserve it at a dark place of 5 °C or lower.

- h) **Bisphenol A-*d*₁₆ internal standard solution (1 µg/ml–acetone)** Take 1 ml of bisphenol A-*d*₁₆ internal standard solution (10 µg/ml–acetone) and transfer it to a 10 ml volumetric flask and add acetone up to the marked line⁽⁷⁾.

- i) **Nitrogen** Nitrogen Class 2, specified in **JIS K 1107**.

NOTE 1 For the bisphenol A deuteride, there are types of which the number of deuterium is 16, 6 and 8, and any type may be used. However, for the one of which the number of deuterium is 16, the deuterium in hydroxyl radical disappears due to the substitution with TMS.

5.1.2 Implements The implements shall be as follows.

- a) **Separatory funnel** 1 000 ml to 2 000 ml
- b) **Graduated test tube with grounding stopper** 10 ml to 20 ml having the graduation at 0.5 ml and 1 ml
- c) **Erlenmeyer flask with grounding stopper** 100 ml to 500 ml
- d) **Microsyringe** 100 µl to 200 µl
- e) **Concentrator** Rotary evaporator or Kuderna-Danish concentrator⁽⁸⁾

Note ⁽⁸⁾ Those without capillary.

- f) **Flask for concentrator** An egg-plant type flask when a rotary evaporator is used, and a concentration flask with concentration tube when a Kuderna-Danish concentrator is used. The flask shall be the one of 100 ml to 500 ml with interchangeable ground joint and be capable of connecting with a concentrator.

- g) **Shaker**

5.1.3 Operation The operation shall be as follows.

- a) Take an adequate amount (for example, 1 L) of sample⁽⁹⁾ taken in **4.1.2** in a separatory funnel, adjust the PH at about 3 by adding hydrochloric acid (1 mol/L), and add 30 g of sodium chloride per 1 L of liquid amount⁽¹⁰⁾. Then add 100 µl of bisphenol A-*d*₁₆ internal standard solution (1 µg/ml–acetone) with a microsyringe, add 50 ml of dichloromethane per 1 L of liquid amount, shake to mix it by using a shaker for about 10 min, and leave it as it is.

Notes ⁽⁹⁾ When plenty of suspended matters are contained in the sample, carry out operations of NOTE 2.

⁽¹⁰⁾ When the sample is sea water, do not add sodium chloride.

- b) Transfer the dichloromethane layer to an Erlenmeyer flask with grounding stopper. Add 50 ml of dichloromethane per 1 L of liquid amount in the water layer of separatory funnel, shake to mix it by using a shaker for about 10 min, and leave it as it is. Join the dichloromethane layer obtained together to the previous Erlenmeyer flask with grounding stopper.
- c) Add 20 g to 30 g⁽¹¹⁾ of sodium sulfate in 100 ml of dichloromethane solution in the Erlenmeyer flask with grounding stopper, shake to mix it lightly, leave it for about 10 min as it is, and dehydrate it⁽¹²⁾. Filter it by using the filter paper of Class 5 A (or Class 5 B)⁽¹³⁾ and receive the filtrate in a flask for concentrator. Wash the Erlenmeyer flask with grounding stopper in which the dichloromethane solution had been contained twice or thrice by using a small amount of dichloromethane. Furthermore, wash the previous filter paper and sodium sulfate on filter paper with this washings and join the filtrate together to the flask for concentrator.

Notes ⁽¹¹⁾ When emulsion is produced, it is required to add it more excessively.

⁽¹²⁾ For the dehydration operation, in addition to this method, there is a method that the dichloromethane solution is preserved at a dark place at -20 °C and separated by freezing water. This method should be used when the concentration operation is not carried out on that day.

⁽¹³⁾ The filter paper shall be washed with dichloromethane preliminarily.

- d) Concentrate the dichloromethane solution by using a concentrator as heating it in a water bath at about 40 °C⁽¹⁴⁾ until it becomes about 5 ml.

Note ⁽¹⁴⁾ When a rotary evaporator is used, concentrate it in a water bath at about 40 °C under reducing pressure, and take care so as not to be exsiccated. When a Kuderna-Danish concentrator is used, concentrate it by heating at 75 °C or lower under atmospheric pressure, not the reducing pressure system. After completing the concentration, detach the Snyder column attached to the concentration part from the apparatus, wash it by adding a small amount of dichloromethane from the upper part of Snyder column and allow it to cool as the Snyder column is attached as it is.

- e) Transfer this concentrated solution to a graduated test tube with grounding stopper. Wash the flask for concentrator used for concentration with 2 ml to 3 ml of dichloromethane and join its washings together in the graduated test tube with grounding stopper. Spray nitrogen gently as heating it in a water bath at about 40 °C, and concentrate it until it becomes about 0.5 ml⁽¹⁵⁾ ⁽¹⁶⁾ ⁽¹⁷⁾.

Notes ⁽¹⁵⁾ When the operation of **6.3** is not carried out immediately, preserve this concentrated solution at a dark place at -20 °C.

⁽¹⁶⁾ When the column chromatographic separation is omitted, move to the operation of **5.3.3 f**).