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**Testing method for hexanedioic
acid bis (2-ethylhexyl) ester in
industrial water and wastewater**

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law:

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Contents

	Page
1 Scope	1
2 Normative references	1
3 Common matters	1
4 Sample	3
4.1 Sampling	3
4.2 Handling of sample	3
5 Pre-treatment of sample	3
5.1 Reagents	3
5.2 Implement	4
5.3 Operation	6
6 Gas chromatography mass spectrometry	8
6.1 Reagents	8
6.2 Apparatus	9
6.3 Preparatory operation	10
6.4 Operation	10
7 Marking of result.....	12
Attached Table 1 Normative references	13
Annex 1 (normative) Column chromatograph separation by using activated magnesium silicate.....	14
Annex 2 (informative) Method of confirmation for the lowest limit value of determination range.....	15
Annex 3 (informative) Calculation method for concentration by using sensitivity factor (<i>RF</i>)	16

Testing method for hexanedioic acid bis (2-ethylhexyl) ester in industrial water and wastewater

1 Scope This Japanese Industrial Standard specifies the testing method for hexanedioic acid bis (2-ethylhexyl) ester [adipic acid bis (2-ethylhexyl)] ($C_{22}H_{42}O_4$) (CAS No. 103-23-1)⁽¹⁾ in industrial water and factory wastewater.

Note ⁽¹⁾ The designation of di-2-ethylhexyl adipate, also, is used.

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

3 Common matters The common matters shall be as follows:

- a) **General rule** The general matters in common to chemical analysis shall be in accordance with **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 mass spectrometry** The general matters in common to gas chromatography mass spectrometry shall be in accordance with **JIS K 0123**.
- d) **Designation of compound** The designations of compounds, which are in accordance with the naming scheme of compound established by the Chemical Society of Japan [the naming scheme of organic chemistry by the International Union of Pure and Applied Chemistry (IUPAC)], shall be used, and the idiomatic name shall be described in parenthesis at the end.
- e) **Determination range** The determination range as shown in the testing method shall be expressed by the mass (pg) of target component in solvent introduced in a gas chromatograph mass spectrometer.
- f) **Analytical precision** The analytical precision shall be expressed with the coefficient of variation (%) which is obtained by repeated tests in the neighbourhood of the lowest limit of determination in the testing method⁽²⁾.

Note ⁽²⁾ Coefficient of variation (%) = $\frac{s}{\bar{x}} \times 100$

where, s : standard deviation

$\bar{x}$: mean value

- g) **Test atmosphere** Care shall be especially taken because the result will be considerably affected by contamination from reagent, organic solvent, implements, etc. and by contamination from the operation of the test and ambient air.
- h) **Water** The water to be used for blank test etc. shall be the water of A4 or A3 specified in **JIS K 0557**⁽³⁾.

Before using, take 1 L of water into a separatory funnel, add 50 g of sodium chloride in **5.1 a)** and 100 ml of hexane of **5.1 d)**, mix them with a vibrator approximately for 10 min, and leave them as they are. Carry out the operation of **5.3 b)** to **d)**, **j)** and **k)** on this solution [however, the addition of fluoranthene- d_{10} internal standard solution (10 $\mu\text{gC}_{16}\text{H}_{10}\text{-}d_{10}/\text{ml}$) is not to be carried out], take 1 μl of the concentrated solution with a microsyringe according to **6.4 a)**, introduce it into a gas chromatograph mass spectrometer and confirm the absence of peak at the position corresponding to the retention time of adipic acid bis (2-ethylhexyl)⁽⁴⁾.

Notes ⁽³⁾ For the water to be used for cleaning glassware, the water of A2 or A3 specified in **JIS K 0557** may be used.

⁽⁴⁾ When refinement is required, it shall be in accordance with clause 4 Remarks 6 of **JIS K 0557**. Or, the water of A4 or A3 shall be treated by activated carbon.

i) **Reagents** The common matters related to reagents shall be as follows:

- 1) The reagents, which are specified in **JIS** and do not hinder the test, shall be used. In the case of not being specified in **JIS**, the reagents that do not hinder the test shall be used.
- 2) The concentration of the reference solution shall be expressed by mg/ml or $\mu\text{g}/\text{ml}$.
- 3) The mixed solution of liquid reagents shall be expressed by reagent A-reagent B ($a+b$). This expression shall indicate that a ml of reagent A and b ml of reagent B are mixed.
- 4) The concentration indicated with a parenthesis at the end of designation of the reference solution means that it is the accurate concentration.
- 5) The designations of reagents have been made to conform to the naming scheme of compound established by the Chemical Society of Japan based on the naming scheme of inorganic chemistry and the naming method of organic chemistry by the International Union of Pure and Applied Chemistry (IUPAC) and the designation of reagent in **JIS** as much as possible.
- 6) For the compound to be used for preparation of reference solution, the one where its purity is known shall be used. It is described as "reference material" in this Standard. Similar compounds are contained as impurities in them in many cases, therefore those that do not hinder the test shall be used.
- 7) The handling of reagents, waste solution, etc. shall follow the related laws and ordinances and care shall be sufficiently taken.

j) **Glassware** The common matters to glassware shall be as follows:

- 1) If not especially specified, the glassware specified in **JIS R 3503** and **JIS R 3505** shall be used. When a heating operation is accompanied, the borosilicate glass-1 specified in **JIS R 3503** shall be used.

The desiccant to be used for a desiccator, if not especially specified, shall be silicagel⁽⁵⁾.

- 2) The glassware to be used for the test, which are previously washed with water, shall be washed with acetone (not less than a concentration of 300 in quality specified in **JIS K 8040**) after volatilizing acetone by leaving them as they are, heated at not less than 200 °C for not less than 2 h and left at the place being free from contamination as they are.

Note ⁽⁵⁾ Type A class 1 of the silicagel desiccants for packing specified in **JIS Z 0701** shall be used.

- k) **Working curve** The working curve shall be prepared within the determination range so that the determination range as shown in the testing method is divided into 4 to 6 steps and the reference solution is taken so as to correspond with the concentration at the time of introducing into the gas chromatograph mass spectrometer.

4 Sample

4.1 Sampling The sampling shall be as follows:

4.1.1 Implement The implement shall be as follows:

- a) **Sample vessel**, 1 000 ml of glass bottle with grounding stopper⁽⁶⁾

Note ⁽⁶⁾ The bottle having a screwed stopper with tetrafluoroethylene resin packing may be used instead of the grounding stopper.

4.1.2 Sampling operation The sampling operation shall be as follows:

- a) **Sampling of surface layer water** Take the sample according to 4.1.1 or 4.1.2 of **JIS K 0094**.
- b) **Sampling of water at each depth** Take the sample according to 4.1.4 of **JIS K 0094**. In addition to Bandone sampler, the one having the equivalent performance to this may be used.
- c) **Sampling from a piping apparatus** Take the sample according to 4.3 of **JIS K 0094**.

4.2 Handling of sample Carry out the test immediately after sampling. If not carried out immediately, preserve it in a dark place at 0 °C to 10 °C, and carry out the test as soon as possible.

5 Pre-treatment of sample After adding sodium chloride and adipic acid bis (2-ethylhexyl)-*d*₈ to the sample and mixing to extract by adding hexane, concentrate the extracted solution. After letting the extracted solution flow through the column chromatograph tube by using silicagel and separating to let obstructive materials flow out, elute the target material by using acetone-hexane eluant, concentrate it again, add fluoranthene-*d*₁₀ and make it a definite amount.

When an obstructive material does not exist, the operation of column chromatograph separation may be omitted.

5.1 Reagents The reagents shall be as follows: