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**Testing method for benzophenone
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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Testing method for benzophenone in industrial water and wastewater

1 Scope This Japanese Industrial Standard specifies the testing method for diphenylmethanone [benzophenone ($C_{13}H_{10}O$) (CAS No. 119-61-9)] in industrial water and wastewater from factory.

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 matter 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 International Union of Pure and Applied Chemistry (IUPAC)], shall be used, and the idiomatic name shall be described with a 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 by the coefficient of variation (%) which is obtained by the repeated tests at the neighbourhood of the lowest limit of determination in the testing method⁽¹⁾.

Note (1) Coefficient of variation (%) = $\frac{s}{\bar{x}} \times 100$

where, s : standard deviation

$\bar{x}$: mean value

- g) **Water** The water to be used for a blank test, etc. shall be the water of A4 or A3 specified in **JIS K 0557**⁽²⁾.

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 in **5.1 d**), shake to mix them with using a vibrator for approximately 10 min, and leave it as it is. Carry out the operation of **5.3 b**) to **d**), **j**) and **k**) [however, the addition of naphthalene- d_8 internal standard solution (10 $\mu\text{gC}_{10}\text{H}_8\text{-}d_8/\text{ml}$) in **5.1 i**) is not 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 that the peak does not exist at the position corresponding to the retention time of benzophenone⁽³⁾.

Notes (2) For the water to be used for cleaning glassware, the water of A2 or A3 specified in **JIS K 0557** may be used.

(3) 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 with activated carbon, etc.

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

- 1) The reagents, which are specified in **JIS** and do not hinder the test, shall be used. When not specified in **JIS**, the reagents without hindering the test shall be used.
- 2) The concentration of the reference solution shall be expressed by mg/ml or $\mu\text{g/ml}$.
- 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 conformed to the naming scheme of compound established by the Chemical Society of Japan based on the naming scheme of inorganic chemistry and the naming scheme 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 compounds to be used for preparation of reference solution, the concentration-known one shall be used. It is described as "reference material" in this Standard. The similar compounds are contained as impurities in them in many cases, therefore the one without hindering the test shall be used.
- 7) The handling of reagents, wastewater, etc. shall follow the related laws and ordinance, etc., and care shall be sufficiently taken.

i) **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⁽⁴⁾.

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

Note (4) Type A class 1 of the silicagel desiccant for packing specified in **JIS Z 0701** shall be used.

- j) **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 to 4 steps to 6 steps and the reference solution is taken so as to agree 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**, of 1 000 ml of glass bottle with grounding stopper⁽⁵⁾

Note ⁽⁵⁾ 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 sampled. If not carrying 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 benzophenone-*d*₁₀ to the sample and mixing up to extract by adding hexane, concentrate the extracted solution. After letting the concentrated solution flow through the column chromatograph tube used with silicagel and separating to let obstructive materials flow out, elute the target material with using acetone-hexane eluant, concentrate it again, add naphthalene-*d*₈ and make a definite volume.

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

5.1 Reagents The reagents shall be as follows:

- a) **Sodium chloride**, specified in JIS K 8150. After heating at approximately 600 °C for approximately 60 min, leave it cool in a desiccator as it is.
- b) **Sodium sulfate**, specified in JIS K 8987. After heating at approximately 600 °C for approximately 60 min, leave it cool in a desiccator as it is.

Information : For sodium sulfate, PCB • phthalic acid ester for test is on the market.