

# JIS

## JAPANESE INDUSTRIAL STANDARD

### Testing methods for volatile organic compounds in industrial water and waste water

JIS K 0125—1995

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In the event of any doubt arising,  
the original Standard in Japanese is to be final authority.

## JAPANESE INDUSTRIAL STANDARD

J I S

Testing methods for volatile organic compounds K 0125-1995  
in industrial water and waste water

1. Scope This Japanese Industrial Standard specifies the testing methods for the volatile organic compounds, in industrial water and waste water, in which dichloromethane, dibromochloromethane, tetrachloromethane (carbon tetrachloride), trichloromethane (chloroform), tribromomethane (bromoform), bromodichloromethane, 1,2-dichloroethane, 1,1,1-trichloroethane, 1,1,2-trichloroethane, 1,1-dichloroethene (1,1-dichloroethylene), *cis*-1,2-dichloroethene (*cis*-1,2-dichloroethylene), *trans*-1,2-dichloroethene (*trans*-1,2-dichloroethylene), tetrachloroethene (tetrachloroethylene), trichloroethene (trichloroethylene), 1,2-dichloropropane, 1,3-dichloro-1-propene, 1,4-dichlorobenzene (*p*-dichlorobenzene), dimethylbenzene (xylene), benzene, and methylbenzene (toluene), are numbered.

Remarks 1. The summary of target materials classified by testing method is shown in Attached Table 1.

2. The standards cited in this Standard are listed in Attached Table 2.

2. Matters in common Matters in common shall be as follows:

- (1) General rules The general matters commonly applicable to chemical analysis shall follow JIS K 0050.
- (2) Gas chromatography The general matters commonly applicable to gas chromatography shall follow JIS K 0114.
- (3) Gas chromatography mass spectrometry The general matters commonly applicable to gas chromatography mass spectrometry shall follow JIS K 0123.
- (4) Definitions The definition of main terms used in this Standard shall follow JIS K 0101, JIS K 0102, JIS K 0211 and JIS K 0215.
- (5) Determination range The determination range of purge-trap-gas chromatography mass spectrometry, purge-trap-gas chromatography, and solvent extraction-gas chromatography shall be expressed by the mass (ng) of target component in a purge container or in solution (solvent) to be introduced into a gas chromatograph; and that of head space-gas chromatography mass spectrometry and head space-gas chromatography shall be expressed by the concentration ( $\mu\text{g/l}$ ) of target component in sample.
- (6) Repeatability of analysis The repeatability of analysis shall be indicated by the coefficient of variation (%) <sup>(1)</sup> which is obtained by means of repeatability test carried out within the determination range of each testing method.

Note <sup>(1)</sup> Coefficient of variation (%) =  $\frac{\sigma}{\bar{x}} \times 100$

where,  $\sigma$  : standard deviation

$\bar{x}$  : averaged value

- (7) Test environment When carrying out these tests, it shall preferably be the environment which is, after considering satisfactorily the pollution likely coming from surroundings, segregated from the testing room where such as organic solvent may be handled.

The temperature in a testing room shall preferably be kept at 20°C to 25°C.

- (8) Water Water to be used for reagent preparation and for blank test shall be either the water of A4 (or A3) specified in JIS K 0557 or the one refined to become equivalent to the above water, and prior to use, its acceptance or not shall be confirmed through the blank test carried out under each testing method <sup>(2)</sup>.

Note <sup>(2)</sup> When refinement is needed, the following shall be adopted.

Take 1 l to 3 l of water into an Erlenmeyer flask, heat it strong to boil, and continue boiling to make its volume about one third. Immediately transfer it to the place not influenced by contamination from surroundings, and allow it to be cooled (weak heating could not sufficiently eliminate volatile organic compounds).

(9) Reagents

- (a) When reagents are specified in corresponding Japanese Industrial Standard, employ the most superior grade one of that kind or having suitable quality grade; and when there is no corresponding Japanese Industrial Standard, employ the one having no hindrance for tests.
- (b) The mass concentration of reagent solution shall be generally indicated by g/l or mg/l (when compound is to be dissolved, use the mass of anhydrous one for calculation), and mol concentration shall be indicated by mol/l.

The concentration of reference solution, however, shall be indicated by the mass dissolved in 1 ml (mg/ml or µg/ml).

- (c) The concentration of liquid reagent shall be indicated by mixing ratio [reagent ( $a + b$ )] with water (or other liquid reagents). This formula means that  $a$  ml of reagent is mixed with  $b$  ml of water (or other liquid reagents).
- (d) The names of reagents are coordinated with those mentioned in the nomenclature of compounds specified by the Chemical Society of Japan and in JIS reagents, which are based on the nomenclature system for inorganic compounds and for organic compounds prescribed by the International Union of Pure and Applied Chemistry (IUPAC).
- (e) The standard material for volatile organic compounds to be used for preparation of reference solution often contains impure compounds similar to them, therefore, it shall be important to choose the reagent giving no hindrance to tests.
- (f) When handling reagents, waste solution and the like, sufficient attention shall be paid while observing related laws, rules and the like.

- (10) Glassware Employ principally the glassware which have been specified in JIS R 3503 and JIS R 3505.

When heating operation is needed, they shall be those made with borosilicate glass-1 specified in JIS R 3503.

- (11) Working curve Working curve shall be prepared as follows; divide the determination range which has been shown in testing method, into from 4 steps to 6 steps, take reference solutions to agree with these steps, and prepare the curve plotting in the determination range.

Alternatively, following the internal standard method specified in 8.3.2 (1) of JIS K 0123, prepare it on the base of the ratio between internal standard substance with definite concentration and these standard materials.

Working curve shall be newly prepared for every test, and when many samples are continuously tested according to the same testing method, the values used for indication shall be suitably confirmed using reference solution in the middle of the test.

- (12) Note, remarks, figure and table Note, remarks, figure and table are orderly numbered in each clause.

### 3. Sample

#### 3.1 Sampling Sampling shall be as follows:

- (1) Implements Implements shall be as follows:

- (a) Container for sample A 40 ml to 250 ml glass-made container with a screwed glass lid, the lid of which is lined <sup>(1)</sup> with tetrafluoroethylene resin film (or with the one with equivalent quality) <sup>(2)</sup>. In advance, wash it with A2 or A3 water specified in JIS K 0557, heat it at  $105 \pm 2^\circ\text{C}$  for about 3 h, and allow it to cool in a desiccator.

Notes <sup>(1)</sup> Tetrafluoroethylene resin film shall be about 50  $\mu\text{m}$  thick.

<sup>(2)</sup> In case of a purge-trap apparatus employing a vial, the vial for a head-space [vial specified in 5.2 (2) (2.1)] may be used.

- (2) Sampling procedures Sampling procedures shall be as follows:

- (a) Sampling of surface water Take sample according to 4.1.1 or 4.1.2 of JIS K 0094, put it into a sample container so as not to make bubbling <sup>(3)</sup> <sup>(4)</sup>, fill it with sample, and stopper it closely with no bubble inside.
- (b) Sampling water from each depth Take water according to 4.1.4 of JIS K 0094, put it into a sample container so as not to make bubbling <sup>(3)</sup> <sup>(4)</sup>, fill it with sample and stopper it closely with no bubble inside.
- (c) Sampling from piping system Take sample according to 4.3 of JIS K 0094, put it into a sample container so as not to make bubbling <sup>(3)</sup> <sup>(4)</sup>, fill it with sample, and stopper it closely with no bubble inside.

Notes (3) When oxidizing material, such as residual chlorine, coexists in sample, add L (+) – ascorbic acid specified in JIS K 9502 by 10 mg to 20 mg per 1 mg of residual chlorine for making reduction, and stopper it closely after filling.

(4) When the sample for the test of trihalomethanes [dibromochloromethane, trichloromethane (chloroform), tribromomethane (bromoform), and bromodichloromethane] coexists oxidizing material such as residual chlorine, carry out the procedures shown in Note (3), add hydrochloric acid (1 + 11) (prepare using hydrochloric acid specified in JIS K 8180) to make its pH about 2 or less, and stopper it closely after filling.

Remarks: The coexistence of oxidizing material like residual chlorine in sample secularly generates not only trihalomethanes but also by-products (chlorinated hydrocarbons) by procedure of disinfection, which results in fluctuation of the concentration of volatile organic compounds in sample, therefore its reduction is indispensable.

3.2 Handling of sample Test immediately after sampling. When immediate test is impeded, store it in a dark place at 4°C or lower without freezing, and carry out the test as soon as possible (5).

Note (5) While sampling and storing sample, volatile organic compounds may change its concentration owing to dissipation, evaporation and the like, so that full care shall be taken. Even when the concentration of volatile organic compound in sample is low, storing sample in a dark place sometimes results in sudden decrease of its concentration, where the stability of volatile organic compounds depends on the type of material.

4. Expresison of results On the expression of results, the testing method employed shall be clearly marked.

5. Testing methods As for testing methods, any method out of purge-trap-gas chromatography mass spectrometry, head space-gas chromatography mass spectrometry, purge-trap-gas chromatography, head space-gas chromatography, and solvent extraction-gas chromatography, shall be applied to.

5.1 Purge-trap-gas chromatography mass spectrometry This method shall be applied as simultaneous determination (or individual determination) when volatile organic compounds listed in Table 5.1 are tested.

By means of aerating inert gas into sample, turn volatile organic compounds into gas phase to capture it in a trap tube, heat the trap tube to desorb the volatile organic compounds, allow it to cool and condense (cryofocus) in a cooling-and-condensing apparatus and introduce it into a gas chromatograph mass spectrometer. Otherwise, after trapping in a trap tube, heat on the trap tube to desorb volatile organic compounds, and introduce them into a gas chromatograph mass spectrometer. Then, detect them with a selected ion detecting method (SIM) or other equivalent methods, measure chromatogram of each selected ion, and obtain the concentration of volatile organic compounds. In this case, determination range and the repeatability of analysis shall be as shown in Table 5.1.

Table 5.1. Summary of target materials, its determination range, and repeatability of analysis

Target materials	Determination range ng	Repeatability of analysis %
Dichloromethane ( $\text{CH}_2\text{Cl}_2$ )	0.5 to 250	10 to 20
Dibromochloromethane ( $\text{CHBr}_2\text{Cl}$ )	0.5 to 250	10 to 20
Tetrachloromethane (carbon tetrachloride) ( $\text{CCl}_4$ )	0.5 to 250	10 to 20
Trichloromethane (chloroform) ( $\text{CHCl}_3$ )	0.5 to 250	10 to 20
Tribromomethane (bromoform) ( $\text{CHBr}_3$ )	0.5 to 250	10 to 20
Bromodichloromethane ( $\text{CHBrCl}_2$ )	0.5 to 250	10 to 20
1,2-dichloroethane ( $\text{CH}_2\text{ClCH}_2\text{Cl}$ )	0.5 to 250	10 to 20
1,1,1-trichloroethane ( $\text{CH}_3\text{CCl}_3$ )	0.5 to 250	10 to 20
1,1,2-trichloroethane ( $\text{CHCl}_2\text{CH}_2\text{Cl}$ )	0.5 to 250	10 to 20
1,1-dichloroethene ( $\text{CCl}_2=\text{CH}_2$ )	0.5 to 250	10 to 20
<i>cis</i> -1,2-dichloroethene ( <i>cis</i> - $\text{CHCl}=\text{CHCl}$ )	0.5 to 250	10 to 20
<i>trans</i> -1,2-dichloroethene ( <i>trans</i> - $\text{CHCl}=\text{CHCl}$ )	0.5 to 250	10 to 20
Tetrachloroethene ( $\text{CCl}_2=\text{CCl}_2$ )	0.5 to 250	10 to 20
Trichloroethene ( $\text{CHCl}=\text{CCl}_2$ )	0.5 to 250	10 to 20
1,2-dichloropropane ( $\text{CH}_3\text{CHClCH}_2\text{Cl}$ )	0.5 to 250	10 to 20
<i>cis</i> -1,3-dichloro-1-propene ( <i>cis</i> - $\text{ClCH}=\text{CHCH}_2\text{Cl}$ ) <sup>(1)</sup>	0.5 to 250	10 to 20
<i>trans</i> -1,3-dichloro-1-propene ( <i>trans</i> - $\text{ClCH}=\text{CHCH}_2\text{Cl}$ ) <sup>(1)</sup>	0.5 to 250	10 to 20
1,4-dichlorobenzene ( <i>p</i> -dichlorobenzene) ( $\text{C}_6\text{H}_4\text{Cl}_2$ )	0.5 to 250	10 to 20
1,2-dimethylbenzene ( <i>o</i> -xylene) [ <i>o</i> - $\text{C}_6\text{H}_4$ ( $\text{CH}_3$ ) <sub>2</sub> ] <sup>(2)</sup>	0.5 to 250	10 to 20
1,3-dimethylbenzene ( <i>m</i> -xylene) [ <i>m</i> - $\text{C}_6\text{H}_4$ ( $\text{CH}_3$ ) <sub>2</sub> ] <sup>(2)</sup>	0.5 to 250	10 to 20
1,4-dimethylbenzene ( <i>p</i> -xylene) [ <i>p</i> - $\text{C}_6\text{H}_4$ ( $\text{CH}_3$ ) <sub>2</sub> ] <sup>(2)</sup>	0.5 to 250	10 to 20
Benzene ( $\text{C}_6\text{H}_6$ )	0.5 to 250	10 to 20
Methylbenzene (toluene) ( $\text{C}_6\text{H}_5\text{CH}_3$ )	0.5 to 250	10 to 20

(In each case, the determination range differs depending on the type of apparatus and measuring condition.)