JAPANESE INDUSTRIAL
JIS K 0125:1995
Testing methods for volatile organic compounds in industrial water and waste water

March, 2006

ERRATA

Page 22
5.2 (1), (f) Mixed reference solution of volatile organic compounds

Error: Place a little quantity of methanol in a 10 ml measuring flask,.....

Correct: Place a little quantity of methanol in a 50 ml measuring flask,.....

Remarks: This erratum is for correcting the first edition of this Standard.

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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.

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JAPANESE INDUSTRIAL STANDARD J I S

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

K 0125-1995

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 (μg/*l*) of target component in sample.

(6) Repeatability of analysis The repeatability of analysis shall be indicated by the coefficient of variation (%) (1) which is obtained by means of repeatability test carried out within the determination range of each testing method.

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

where, σ : standard deviation

$\bar{x}$: averaged value

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(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 (2).

Note (2) 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.

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(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 (1) with tetrafluoroethene resin film (or with the one with equivalent quality) (2). In advance, wash it with A2 or A3 water specified in JIS K 0557, heat it at 105 ± 2°C for about 3 h, and allow it to cool in a desiccator.

Notes (1) Tetrafluoroethene resin film shall be about 50 μm thick.

(2) 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 (3) (4), 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 (3) (4), 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 (3) (4), fill it with sample, and stopper it closely with no bubble inside.

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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.

Reamrks: 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.

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

Target materials	Determination range ng	Repeatability of analysis %
Dichloromethane (CH_2Cl_2)	0.5 to 250	10 to 20
Dibromochloromethane ($CHBr_2Cl$)	0.5 to 250	10 to 20
Tetrachloromethane (carbon tetrachloride) (CCl_4)	0.5 to 250	10 to 20
Trichloromethane (chloroform) ($CHCl_3$)	0.5 to 250	10 to 20
Tribromomethane (bromoform) ($CHBr_3$)	0.5 to 250	10 to 20
Bromodichloromethane ($CHBrCl_2$)	0.5 to 250	10 to 20
1,2-dichloroethane (CH_2ClCH_2Cl)	0.5 to 250	10 to 20
1,1,1-trichloroethane(CH_3CCl_3)	0.5 to 250	10 to 20
1,1,2-trichloroethane ($CHCl_2CH_2Cl$)	0.5 to 250	10 to 20
1,1-dichloroethene ($CCl_2=CH_2$)	0.5 to 250	10 to 20
cis-1,2-dichloroethene (*cis*-CHCl=CHCl)	0.5 to 250	10 to 20
trans-1,2-dichloroethene (*trans*-CHCl=CHCl)	0.5 to 250	10 to 20
Tetrachloroethene ($CCl_2=CCl_2$)	0.5 to 250	10 to 20
Trichloroethene($CHCl=CCl_2$)	0.5 to 250	10 to 20
1,2-dichloropropane ($CH_3CHClCH_2Cl$)	0.5 to 250	10 to 20
cis-1,3-dichloro-1-propene (*cis*-$ClCH=CHCH_2Cl$) (1)	0.5 to 250	10 to 20
trans-1,3-dichloro-1-propene (*trans*-$ClCH=CHCH_2Cl$) (1)	0.5 to 250	10 to 20
1,4-dichlorobenzene (*p*-dichlorobenzene) ($C_6H_4Cl_2$)	0.5 to 250	10 to 20
1,2-dimethylbenzene (*o*-xylene) [*o*-C_6H_4 $(CH_3)_2$] (2)	0.5 to 250	10 to 20
1,3-dimethylbenzene (*m*-xylene) [*m*-C_6H_4 $(CH_3)_2$] (2) 1,4-dimethylbenzene (*p*-xylene) [*p*-C_6H_4 $(CH_3)_2$] (2)	0.5 to 250	10 to 20
Benzene (C_6H_6)	0.5 to 250	10 to 20
Methylbenzene (toluene) ($C_6H_5CH_3$)	0.5 to 250	10 to 20

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

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Notes (1) When determining 1,3-dichloro-1-propene, respectively determine *cis*-form and *trans*-form, and then make them together for indication.

(2) When determining xylene, first determine total amount of 1,3-dimethylbenzene (*m*-xylene) and 1,4-dimethylbenzene (*p*-xylene), then determine 1,2 dimethylbenzene (*o*-xylene), and make them together for indication.

Remarks 1. The contamination by surroundings likely affecting this testing method is thought to come from the apparatus for air conditioning, so scrupulous measures are needed to avoid the contamination, especially when adopted circulating method for air conditioning.

(1) Reagents Reagents shall be as follows:

(a) Water Follow 2. (8).

(b) Methanol Specified in JIS K 8891 (3).

(c) Dichloromethane reference solution (200 mg CH_2Cl_2/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely and measure its mass. Add promptly about 7.6 ml (4) of dichloromethane specified in JIS K 8161, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(d) Dibromochloromethane reference solution (200 mg $CHBr_2Cl$/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 4.2 ml (4) of dibromochloromethane, stopper it closely and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(e) Tetrachloromethane (carbon tetrachloride) reference solution (200 mg CCl_4/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 6.3 ml (4) of carbon tetrachloride specified in JIS K 8459, stopper it closely and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(f) Trichloromethane (chloroform) reference solution (200 mg $CHCl_3$/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 6.8 ml (4) of chloroform specified in JIS K 8322, stopper it closely and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(g) Tribromomethane (bromoform) reference solution (200 mg $CHBr_3$/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 4.1 ml (4) of tribromomethane (bromoform), stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

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(h) Bromodichloromethane reference solution (200 mg $CHBrCl_2$/ml)
Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 5.1 ml (4) of bromodichloromethane, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(i) 1,2-dichloroethane reference solution (200 mg CH_2ClCH_2Cl/ml)
Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 7.9 ml (4) of 1,2-dichloroethane specified in JIS K 8465, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(j) 1,1,1-trichloroethane reference solution (200 mg CH_3CCl_3/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 7.5 ml (4) of 1,1,1-trichloroethane, stopper it closely and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(k) 1,1,2-trichloroethene reference solution (200 mg $CHCl_2CH_2Cl$/ml)
Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 7 ml (4) of 1,1,2-trichloroethane, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(l) 1,1-dichloroethene reference solution (200 mg $CCl_2{=}CH_2$/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 8.3 ml (4) of 1,1-dichloroethene, stopper it closely and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(m) *cis*-1,2-dichloroethene reference solution (200 mg *cis*-CHCl=CHCl/ml)
Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 7.8 ml (4) of *cis*-1,2-dichloroethene, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(n) *trans*-1,2-dichloroethene reference solution (200 mg *trans*-CHCl=CHCl/ml)
Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 7.9 ml (4) of *trans*-1,2-dichloroethene, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

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(o) Tetrachloroethene reference solution (200 mg $CCl_2=CCl_2$/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 6.2 ml (4) of tetrachloroethene, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(p) Trichloroethene reference solution (200 mg $CHCl=CCl_2$/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 6.9 ml (4) of trichloroethylene specified in JIS K 8666, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(q) 1,2-dichloropropane reference solution (200 mg $CH_3CHClCH_2Cl$/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 8.7 ml (4) of 1,2-dichloropropane, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(r) *cis*-1,3-dichloro-1-propene reference solution (200 mg *cis*-$ClCH=CHCH_2Cl$/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 8.3 ml (4) of *cis*-1,3-dichloro-1-propene, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(s) *trans*-1,3-dichloro-1-propene reference solution (200 mg *trans*-$ClCH=CHCH_2Cl$/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 8.2 ml (4) of *trans*-1,3-dichloro-1-propene, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(t) 1,4-dichlorobenzene (*p*-dichlorobenzene) reference solution (200 mg $C_6H_4Cl_2$/ml) Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 6.9 ml (4) of 1,4-dichlorobenzene (*p*-dichlorobenzene), stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(u) 1,2-dimethylbenzene (*o*-xylene) reference solution [200 mg *o*-$C_6H_4(CH_3)_2$/ml] Place about 30 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 11.4 ml (4) of 1,2-dimethylbenzene (*o*-xylene), stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

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(v) 1,3-dimethylbenzene (*m*-xylene) reference solution [100 mg *m*-$C_6H_4(CH_3)_2$/ml] Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 5.8 ml (4) of 1,3-dimethylbenzene (*m*-xylene), stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(w) 1,4-dimethylbenzene (*p*-xylene) reference solution [100 mg *p*-$C_6H_4(CH_3)_2$/ml] Place about 40 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 5.9 ml (4) of 1,4-dimethylbenzene (*p*-xylene), stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(x) Benzene reference solution (200 mg C_6H_6/ml) Place about 30 ml of methanol into a 50 ml measuring flask, stopper it closely and measure its mass. Add promptly about 11.4 ml (4) of benzene specified in JIS K 8858, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(y) Methylbenzene (toluene) reference solution (200 mg $C_6H_5CH_3$/ml) Place about 30 ml of methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 11.6 ml (4) of toluene specified in JIS K 8680, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5).

(z) Fluorobenzene solution Place about 40 ml of methanol in a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 1 ml (4) (6) of fluorobenzene, stopper it closely, and measure its mass. Then, add methanol up to the marked line. The concentration of this solution shall be calculated making use of mass difference before and after adding (5). This solution will give concentration of about 20 mg C_6H_5F/ml.

This solution can be used as internal standard substance after being diluted suitably.

(aa) Mixed reference solution of volatile organic compounds [{0.5 mg CH_2Cl_2, 0.5 mg $CHBr_2Cl$, 0.5 mg CCl_4, 0.5 mg $CHCl_3$, 0.5 mg $CHBr_3$, 0.5 mg $CHBrCl_2$, 0.5 mg CH_2ClCH_2Cl, 0.5 mg CH_3CCl_3, 0.5 mg $CHCl_2CH_2Cl$, 0.5 mg $CCl_2{=}CH_2$, 0.5 mg *cis*-CHCl=CHCl, 0.5 mg *trans*-CHCl=CHCl, 0.5 mg $CCl_2{=}CCl_2$, 0.5 mg $CHCl{=}CCl_2$, 0.5 mg $CH_3CHClCH_2Cl$, 0.5 mg *cis*-$ClCH{=}CHCH_2Cl$, 0.5 mg *trans*-$ClCH{=}CHCH_2Cl$, 0.5 mg $C_6H_4Cl_2$, 0.5 mg *o*-$C_6H_4(CH_3)_2$, 0.25 mg *m*-$C_6H_4(CH_3)_2$, 0.25 mg *p*-$C_6H_4(CH_3)_2$, 0.5 mg C_6H_6, 0.5 mg $C_6H_5CH_3$}/ml] (7) (8) Place about 100 ml of methanol in a 200 ml measuring flask, and add respectively 0.5 ml (4) of dichloromethane reference solution (200 mg CH_2Cl_2/ml), of dibromochloromethane reference solution (200 mg $CHBr_2Cl$/ml), of tetrachloromethane (carbon tetrachloride) reference solution (200 mg CCl_4/ml), of trichloromethane (chloroform) reference solution (200 mg

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$CHCl_3$/ml), of tribromomethane (bromoform) reference solution (200 mg $CHBr_3$/ml), of bromodichloromethane reference solution (200 mg $CHBrCl_2$/ml), of 1,2-dichloroethane reference solution (200 mg CH_2ClCH_2Cl/ml), of 1,1,1-trichloroethane reference solution (200 mg CH_3CCl_3/ml), of 1,1,2-trichloroethane reference solution (200 mg $CHCl_2CH_2Cl$/ml), of 1,1-dichloroethene reference solution (200 mg $CCl_2=CH_2$/ml), of *cis*-1,2-dichloroethene reference solution (200 mg *cis*-CHCl=CHCl/ml), of *trans*-1,2-dichloroethene reference solution (200 mg *trans*-CHCl=CHCl/ml), of tetrachloroethene reference solution (200 mg $CCl_2=CCl_2$/ml), of trichloroethene reference solution (200 mg $CHCl=CCl_2$/ml), of 1,2-dichloropropane reference solution (200 mg $CH_3CHClCH_2Cl$/ml), of *cis*-1,3-dichloro-1-propene reference solution (200 mg *cis*-$ClCH=CHCH_2Cl$/ml), of *trans*-1,3-dichloro-1-propene reference solution (200 mg *trans*-$ClCHCHCH_2Cl$/ml), of 1,4-dichlorobenzene (*p*-dichlorobenzene) reference solution (200 mg $C_6H_4Cl_2$/ml), of 1,2-dimethylbenzene (*o*-xylene) reference solution [200 mg *o*-$C_6H_4(CH_3)_2$/ml], of 1,3-dimethylbenzene (*m*-xylene) reference solution [100 mg *m*-$C_6H_4(CH_3)_2$/ml], of 1,4-dimethylbenzene (*p*-xylene) reference solution [100 mg *p*-$C_6H_4(CH_3)_2$/ml], of benzene reference solution (200 mg C_6H_6/ml), and of methylbenzene (toluene) reference solution (200 mg $C_6H_5CH_3$/ml), into the flask. Then add methanol up to the marked line ([5]). This reference solution shall be used when preparing a working curve.

(ab) Mixed reference solution of volatile organic compounds [{50 μg CH_2Cl_2, 50 μg $CHBr_2Cl$, 50 μg CCl_4, 50 μg $CHCl_3$, 50 μg $CHBr_3$, 50 μg $CHBrCl_2$, 50 μg CH_2ClCH_2Cl, 50 μg CH_3CCl_3, 50 μg $CHCl_2CH_2Cl$, 50 μg $CCl_2=CH_2$, 50 μg *cis*-CHCl=CHCl, 50 μg *trans*-CHCl=CHCl, 50 μg $CCl_2=CCl_2$, 50 μg $CHCl=CCl_2$, 50 μg $CH_3CHClCH_2Cl$, 50 μg *cis*-$ClCH=CHCH_2Cl$, 50 μg *trans*-$ClCH=CHCH_2Cl$, 50 μg $C_6H_4Cl_2$, 50 μg *o*-$C_6H_4(CH_3)_2$, 25 μg *m*-$C_6H_4(CH_3)_2$, 25 μg *p*-$C_6H_4(CH_3)_2$, 50 μg C_6H_6, 50 μg $C_6H_5CH_3$}/ml] Place a little quantity of methanol into a 10 ml measuring flask, add 1 ml of mixed reference solution of volatile organic compounds which has been prepared at (aa) into the flask, and further add methanol up to the marked line ([5]). This reference solution shall be used when the preparatory operation at (3) is carried out.

(ac) Helium Helium (with purity of 99.9999 vol% or higher).

(ad) Nitrogen High purity nitrogen of class 1 specified in JIS K 1107.

Notes ([3]) Prior to use, methanol shall be confirmed to be harmless for measurement by the injection of methanol according to the procedures of blank test in (4). After breaking the seal, if it is kept in a laboratory, it may be polluted by air in the laboratory, therefore it shall be stored in the place free from any contamination.

([4]) Take the specified volume of solution (calculate it by the formula of mass/density), which corresponds to mass of compound, using a transfer pipet or microsyringe.

When it is prepared by diluting reference solution, employ a transfer pipet.

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(5) Prepare it when it is used. When it is handled as follows and stored in a dark and cold place, it can be stored for 1 month to 3 months. The solution on the market is also serviceable.

Storing method of reference solution Freeze immediately the solution, which has been prepared, with liquefied nitrogen, transfer it into an ampule under the condition frozen with either liquefied nitrogen or freezing mixture of acetone, dry ice and the like, and store it after stoppering by fusing.

(6) Instead, 4-bromofluorobenzene (C_6H_4BrF) may be used for internal standard substance. In this case, prepare this as follows:

Preparation of 4-bromofluorobenzene solution Place about 40 ml methanol into a 50 ml measuring flask, stopper it closely, and measure its mass. Add promptly about 0.7 ml of 4-bromofluorobezene, stopper it closely, and measure its mass. Then, add methanol up to the marked line. Calculate the concentration of this solution making use of the difference of mass before and after adding. The concentration of this solution is about 20 mg C_6H_4BrF/ml.

Either when the solution is put into sample as internal standard substance or when it is used for sensitivity control of a gas chromatograph mass spectrometer in Remarks 2., use the solution prepared according to Note (16).

(7) When mixed reference solution of volatile organic compound is needed, the solution with known concentration on the market is available.

(8) When respective material is to be tested alone, the reference solution for required item shall be prepared according to (aa) or (ab).

(2) Implements and apparatus Implements and apparatus shall be as follows:

(2.1) Gastight syringe Capable of taking 5 ml to 25 ml (9).

(2.2) Microsyringe Capable of taking 1 μl to 100 μl (10).

(2.3) Bubbler Device making minute bubbles when purge gas is let go through sample.

(2.4) Purge·trap device Satisfying the following conditions:

(a) Purging container Glass-made container either capable of pouring 0.5 ml to 25 ml sample into or with an inlet for sample introducing. Prior to use, clean with water, heat at 105 ± 2°C for about 3 h, and allow it to cool in a desiccator.

(b) Thermostatic device for a purging container Capable of keeping a purging container at constant temperature from 20°C to 40°C.

(c) Tube for trapping Quartz tube, stainless steel tube, or stainless steel tube lined with inert material, measuring 0.5 mm to 5 mm in inside diameter and 50 mm to 300 mm in length.

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(d) Packing of tube for trapping 2,6-diphenyl-1,4-diphenoxide polymer (177 μm to 250 μm or 250 μm to 500 μm in grain size), silica gel (250 μm to 500 μm in grain size) and active charcoal (250 μm to 500 μm in grain size), or the one equal to these.

Informative reference 1. 2,6-diphenyl-1,4-diphenoxide polymer is on the market with a trade name as Tenax GC or Tenax TA.

(e) Trap tube Fill the packing of tube for trapping into the tube for trapping ([11]), and prior to use, heat it at regeneration temperature for 30 min to 60 min ([12]) while helium is flowing in the tube at the rate of 20 ml/min to 40 ml/min.

(f) Heating device of trap tube Capable of keeping a trap tube at 20°C to 40°C during purging, of raising it up to about 180°C to 280°C within one minute when volatile organic compound, which has been trapped in a trap tube, is to be desorbed through heating, and further of keeping that temperature for at least about 4 min.

(g) Purge gas Employ helium specified in (1) (ac) or nitrogen in (1) (ad) ([13]). Control its flow rate to the specified value in the range from 20 ml/min to 60 ml/min when using.

(h) Device for cooling and condensing ([14]) Quartz glass tube or capillary column with 0.32 mm to 0.53 mm in inside diameter, and capable of cooling it at − 30°C or lower when condensing and of heating up to the temperature of a column chamber or around 200°C within one minute when desorbing.

(2.5) Gas chromatograph mass spectrometer

(2.5.1) Gas chromatograph Satisfying the following conditions:

(a) Pipe for capillary column ([15]) Pipe made of quartz glass, hard glass, or stainless steel pipe lined with inert material, measuring 0.2 mm to 0.32 mm in inside diameter and about 25 m to 60 m in length.

(b) Capillary column ([15]). Pipe for capillary column of which inside wall has been coated with phenylmethylpolysiloxane (or dimethylpolysiloxane) by 0.1 μm to 3 μm in thickness. Otherwise, the one with equal separative performance to this.

Informative reference 2. The capillary column with 0.2 mm to 0.32 mm in inside diameter, for this test, is on the market with a trade name as AQUATIC, DB-624, Halomatics 624, Vocol and the like.

(c) Carrier gas Employ helium specified in (1) (ac) ([13]). Control its linear speed in the range from 20 cm/s to 40 cm/s when using.

(d) Temperature of column chamber With temperature control accuracy to the nearest 0.5°C or less in the range of 35°C to 230°C, and capable of temperature raising (for instance, capable of keeping at 40°C for about 1 min, and of raising up to 230°C at the rate of 2°C to 10°C per minute).

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(e) Temperature of interface part From 150°C to 280°C

(2.5.2) Mass spectrometer Satisfying the following conditions:

(a) Ionization method Electron-ionization method (EI method)

(b) Detection method Capable of selected ion monitoring method (SIM), and of controlling its sensitivity under the specified determination range. Otherwise, the one capable of performing the same method.

(c) Temperature of ion generating source The optimum condition for the apparatus.

(d) Accelerating voltage of electrons 70 V

Notes (9) High concentration of volatile organic compound in sample causes itself to be adsorbed in the syringe which results in its contamination affecting next sample. In this case, wash the syringe 3 times promptly with methanol, then 3 times with water in 2. (8), and 3 times with the sample to be tested hereafter.

It is preferable to get ready three gastight syringes, that is, for blank test, for low concentration measuring (about 10 μg/*l* or less concentration of volatile organic compound), and for high concentration measuring.

(10) It is preferable to get ready 3 microsyringes, belonging to the same lot, which are for blank test, for low concentration measuring, and for high concentration measuring.

(11) Usually, 2,6-diphenyl-1,4-diphenoxide polymer is alone used, but instead, may be also usable combinedly with silica gel, active charcoal, or silica gel plus active charcoal. It is necessary in advance to confirm that target volatile organic compound is quantitatively adsorbed or desorbed. When silica gel is employed, the procedure for removing moisture is indispensable.

(12) Besides above procedures, the trap tube shall be aerated with helium at the rate of 20 ml/min to 40 ml/min under regeneration temperature (about 180°C to 280°C) for about 10 min, prior to every measurement of sample.

(13) If target material is detected in purge gas or carrier gas, they are required to be purified through the purifying tube packed with molecular sieve, active charcoal, silica gel, or the like.

(14) Alternatively, it is called a cryofocus device.

When volatile organic compound, adsorbed in a trap tube, is to be heated for desorbing, the apparatus equipped with a capillary column, measuring 0.25 mm in inside diameter, shall need this device to make the adsorbing band of volatile organic compound be narrow; if the inside diameter is 0.32 mm or larger, the device is not needed necessarily.

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(15) Besides the above mentioned, the column measuring 0.53 mm or more in inside diameter can be used, and in this case, the conditions required by a gas chromatograph mass spectrometer follow those in Remarks 4.

Remarks 2. With employing either 4-bromofluorobenzene solution in Note (6) or respective volatile organic compounds, a gas chromatograph mass spectrometer shall be operated as shown in (4), and then, controlled to make its sensitivity be detectable 0.5 ng of the above material.

3. Because the optimum condition of each process is influenced by the type and amount of adsorbent or others, the conditions, by which sufficient recovery is expected, shall be previously obtained.

4. The capillary column measuring 0.53 mm to 0.75 mm in inside diameter and 30 m to 120 m in length can be also used. The conditions of a gas chromatograph mass spectrometer, which are to be adopted in this case, shall be shown in the following.

In this case, the cooling-and-condensing apparatus can be omitted.

1) Purge·trap device Follow from (2.4) (a) to (g).

2) Gas chromatograph mass spectrometer Follow (2.5). The pipe for capillary, interface (separator), and eliminating device of carrier gas, however, shall conform to the following.

(a) Pipe for capillary column Pipe made of quartz glass, hard glass, or stainless steel pipe lined with inert material, measuring 0.53 mm to 0.75 mm in inside diameter and 30 m to 120 m in length.

(b) Interface (separator) Connecting a gas chromatograph with mass spectrometer, separately eliminating almost all carrier gas, and capable of controlling the temperature of sample introducing part.

(c) Eliminating device of carrier gas (*)

Note (*) In this case, carrier gas shall be evacuated by means of an interface, so the vacuum pump with a rather large capacity is preferably installed. By this installation, the sensitivity may be a little lowerwed.

5. The schematic diagram of joining purging part, trapping part, cooling-and-condensing device, and gas chromatograph mass spectrometer are shown in Fig. 5.1.

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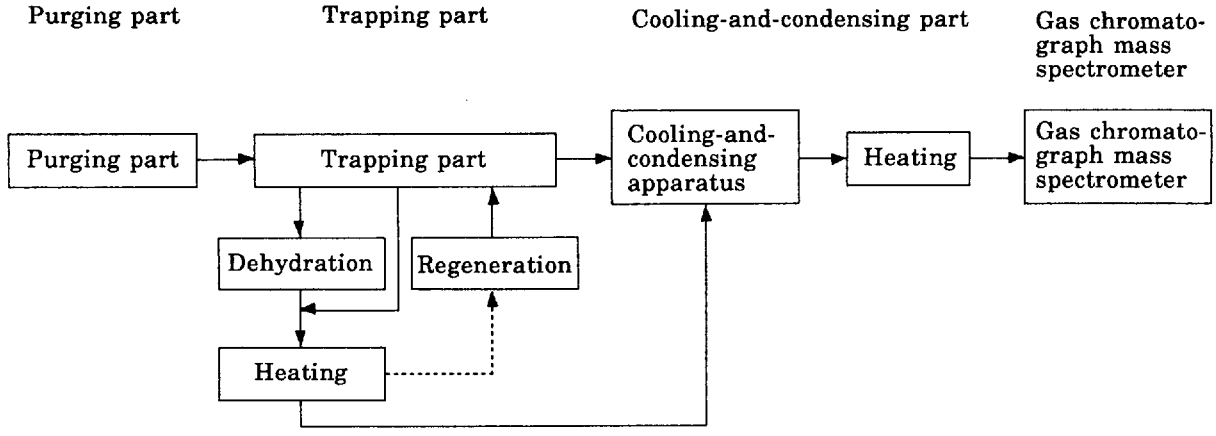
Fig. 5.1. Schematic diagram of joining



(3) Preparatory operation Preparatory operation shall be as follows:

(a) Control the flow rate of purge gas to be 20 ml/min to 40 ml/min, and displace the air in purging container with purge gas.

(b) Control the flow rate of purge gas to be 20 ml/min to 40 ml/min, heat a trapping tube to as near the allowable high limit temperature as possible using a heating apparatus for trapping tube, and keep that temperature for at least 30 min.

(c) Control the flow rate of carrier gas to be 20 cm/s to 40 cm/s linear speed, and raise the temperature of a column chamber (for instance, first keep it at 40°C for 1 min, and then, raise it to 230°C with the rate of 2°C to 10°C per minute).

(d) Inject a decided quantity of water (decided quantity from 0.5 ml to 25 ml, for instance, 5 ml) into a purging container using a gastight syringe (9). Next, with a microsyringe (10), inject 1 µl of mixed reference solution of volatile organic compound prepared in (1) (ab) and 1 µl (about 20 ng) of fluorobenzene solution (A) (16) (17) (18) as internal standard substance, into the purging container.

(e) Carry out the procedures from (4) (d) to (h), and confirm the positions showing the retention time of volatile organic compound and fluorobenzene.

Notes (16) Preparation of fluorobenzene solution (A) Put 0.1 ml of fluorobenzene solution specified in (1) (z) into a 100 ml measuring flask in which about 70 ml of methanol has been previously placed, and add methanol up to the marked line. The concentration of this solution becomes about 20 µg C_6H_5F/ml.

(17) The internal standard solution employed at test operation shall be the same one.

When the concentration of target component is high, control suitably the concentration of fluorobenzene solution (A) according to Note (16), and use it.

(18) When operation is carried out according to 8.3.2 (2) of JIS K 0123, addition of internal standard substance is not needed.

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Remarks 6. This preparatory operation is carried out as a pre-stage of the operation in (4), but when a lot of samples are to be tested continuously, this may be omitted after second trial.

(4) Operation Operation shall be as follows:

(a) Carry out the operation of (3) (a).

(b) Carry out the operation of (3) (b). The retention time of a trapping tube using a heating device for the trapping tube shall be about 10 min.

(c) Inject a suitable quantity of sample (decided quantity from 0.5 ml to 25 ml, for instance, 5 ml) ([19]), which has been taken at 3., into a purging container using a gastight syringe ([9]). Then, with a micro-syringe ([10]), inject 1 μl (about 20 ng) of fluorobenzene solution (A) ([16]) ([17]) ([18]) as internal standard substance and 1 μl of methanol into the purging container.

(d) Place the purging container in a thermostat for a purging container, and make the temperature of sample constant (for instance, 20°C, or 40°C or less).

(e) Confirm the temperature of a trapping tube to be near room temperature, and while the solution of (c) is being purged with purging gas trap volatile organic compound, which has been purged, into the trapping tube ([20]) ([21]).

(f) Freeze beforehand a cooling-and-condensing apparatus (for instance, down to – 50°C or – 120°C), raise abruptly the temperature of heating apparatus for a trapping tube within 1 min (for instance, 180°C or 280°C), aerate carrier gas for about 4 min to desorb volatile organic compound from a trapping tube, and make it be adsorbed into a cooling-and-condensing apparatus ([22]).

(g) Heat the cooling-and-condensing apparatus ([23]), and introduce volatile organic compound into a gas chromatograph mass spectrometer with carrier gas.

(h) Establish selected ion peculiar to the volatile organic compound and fluorobenzene ([24]), measure with the selected-ion detecting method or other equivalent method, and record the selected-ion chromatogram.

(i) Confirm that the chromatogram coincides with the retention time of volatile organic compound and fluorobenzene, which have been recorded in (3), and read respectively the indicated values ([25]) on the position corresponding to the retention time.

(j) Providing against the following operation, carry out the procedures (a) and (b), and regenerate a trapping tube.

(k) In terms of a blank test, take the same quantity of water as sample, and carry out the procedures from (c) to (h). If peaks are found at the positions where correspond to the retention time of volatile organic compound and fluorobenzene which have been previously recorded in (3), and if its indicated value ([25]) is not less than the lower limit of determination value, the operation shall be tried again ([26]) ([27]). Providing against the following operation, carry out the procedures (a) and (b), and regenerate a trapping tube.

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