

## Road vehicles—Interior parts and materials—

### Measurement methods of diffused volatile organic compounds (VOC)

#### 1 Scope

This Standard specifies the sampling bag test method for measuring volatile organic compounds (VOC), formaldehyde and other carbonyl compounds which diffuse from automobile interior parts into the air.

This Standard applies to interior parts such as seat, instrument panel and ceiling material.

#### 2 Normative reference

The following standards contain which, thorough reference in this Standard, constitute provisions of this Standard. The most recent editions of the standard indicated below shall be applied.

<b>ISO 16000-6</b>	Indoor air -- Part 6 : Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA sorbent, thermal desorption and gas chromatography MS/FID
<b>JIS A 1962</b>	Indoor air -- Determination of formaldehyde and other carbonyl compounds -- Active sampling method
<b>JIS A 1966</b>	Indoor air -- Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography -- Pumped sampling

#### 3 Terms and Definitions

For the purposes of this document, the following terms and definitions apply.

##### 3.1

##### **automobile interior part**

non-metallic part which is used inside of an automobile, including related materials such as adhesives and coating materials

##### 3.2

##### **test onset**

time point when a sampling bag is placed in a thermostatic oven

##### 3.3

##### **sampling bag**

resin bag having a capacity of 10 L used for measuring VOC, formaldehyde and other carbonyl compounds which diffuse from automobile interior parts

##### 3.4

##### **test concentration**

concentration of specific VOC, formaldehyde and other carbonyl compounds collected from a sampling bag which has been heated for a specified period of time with one or more test samples in it

##### 3.5

##### **blank concentration**

concentration of specific VOC, formaldehyde and other carbonyl compounds collected from a sampling bag

which has been heated for a specified period of time without any test sample

### 3.6

#### **recovery rate**

ratio of the total amount of VOC, formaldehyde and other carbonyl compounds collected from a sampling bag to the known total amount of VOC, formaldehyde and other carbonyl compounds supplied to the sampling bag

### 3.7

#### **sampling bag value**

concentration increment of a subject gas component due to the diffusion of VOC, formaldehyde and other carbonyl compounds from a test sample, multiplied by the total amount of the gas filled in the sampling bag

### 3.8

#### **test sample**

piece of material cut out from automobile interior parts which are to be tested

### 3.9

#### **volatile organic compound**

#### **VOC**

volatile organic compounds detected from a sampling bag as diffused from a test sample, excluding formaldehyde and other carbonyl compounds

**NOTE** VOC elements to be measured in this Standard are as follows. These elements are referred to generically as subject volatile organic compound (or subject VOC): toluene, xylene, ethyl benzene and styrene.

### 3.10

#### **total volatile organic compound**

#### **TVOC**

VOC in the range of n-hexane to n-hexadecane which are detected in a gas chromatographic analysis, as converted to toluene from the total sum of the peak areas of the gas chromatograph

**NOTE** When a gas chromatograph mass analyzer (CG/MS) is used, TVOC is represented by a VOC value which has been measured by means of a total ion mass detector (TIM) and calculated by a total ion chromatogram (TIC) (see **JIS K 0123**).

### 3.11

#### **formaldehyde and other carbonyl compound**

formaldehyde and other carbonyl compound (aldehyde and ketone) which diffuse from a test sample and detected from a sampling bag

**NOTE** The following materials are counted: formaldehyde and acetaldehyde.

## **4 Principle**

The test method specified in this Standard describes a procedure for calculating sampling bag values of VOC, formaldehyde and other carbonyl compounds which diffuse from automobile interior parts.

One or multiple test samples put in a sampling bag are heated at a specified temperature, and then the gas in the sampling bag is collected to measure the test concentrations. By comparing the test concentrations with the corresponding blank concentrations, the sampling bag values of VOC, formaldehyde and other carbonyl compounds diffusing from one test sample can be calculated (see clause **10**).

## **5 Test instruments and gases**

### **5.1 General**

Test instruments and gases necessary for determining the sampling bag values of VOC, formaldehyde and other carbonyl compounds diffusing from automobile interior parts are mainly as follows:

- sampling bag;
- nitrogen gas;
- thermostatic oven;
- pumps;
- integrating flowmeter;
- gas analyzer;
- sorbent tube.

### **5.2 Sampling bag**

#### **5.2.1 General**

General specification and requirements applicable to the sampling bags in this Standard shall be in accordance with 5.2.2, 5.2.3, 5.2.4 and 7.2.

#### **5.2.2 Material and capacity**

The sampling bag material should be fluorinated resin (e.g., vinyl fluoride (PVF), tetrafluoroethylene–hexafluoropropylene-copolymer), unless otherwise specified. The bag capacity shall be 10 L.

#### **5.2.3 Air tightness**

Sampling bags shall be sealed securely with a sealing material such as a tape or heat bonded so as to be isolated from the uncontrolled ambient air.

#### **5.2.4 Blank concentration**

Blank concentrations which are observed when a sampling bag is heated shall be as low as possible so that they do not adversely affect the test results.

### **5.3 Nitrogen gas**

VOC, formaldehyde and other carbonyl compound concentrations contained in the nitrogen gas to be filled in a sampling bag shall be as low as possible so that they do not adversely affect the test results.

### **5.4 Thermostatic oven**

Temperature shall be controlled in an oven which is capable of maintaining a constant and homogeneous temperature.

The thermostatic oven where a sampling bag is heated shall be capable of controlling temperatures within  $\pm 1,0$  °C.

### **5.5 Pumps**

Vacuum pumps or other apparatuses which can empty a sampling bag sufficiently shall be used.

### **5.6 Integrating flowmeter**

The volume of collected gases or other gases shall be measured with an integrating flowmeter.

**NOTE** Other equipment having equivalent or better performance may be used.

### **5.7 Gas analyzer**

VOC analysis shall be performed by means of a gas chromatograph with a hydrogen flame ionization detector

(CG/FID) or a gas chromatograph with a mass analyzer (CG/MS). A high-speed liquid chromatograph shall be used for the analysis of formaldehyde and other carbonyl compounds.

**NOTE** Gas analyzers are according to **JIS A 1962**, **JIS A 1966** and **ISO 16000-6**, but other equipment having equivalent or better performance may be used.

### **5.8 Sorbent tube**

Sorbent tubes such as Tenax-TA or Tenax-GR shall be used for collecting and analysis of VOC. DNPH cartridges shall be used for collecting and analysis of formaldehyde and other carbonyl compounds.

**NOTE** Sorbent tubes are according to **JIS A 1962**, **JIS A 1966** and **ISO 16000-6**.

## **6 Test conditions**

### **6.1 General**

Test conditions shall be in accordance with **6.2** to **6.6**. The test room shall be sufficiently ventilated so as to minimize the back ground effect.

### **6.2 Test sample size**

A test sample shall have an upper surface area of 100 cm<sup>2</sup> (e.g., 10 cm x 10 cm), in general. Thickness of the test sample is not specified.

**NOTE** Cut edges are left as they are without being sealed.

### **6.3 Curing conditions (Storage period and storage conditions)**

Storage period of test samples shall not be longer than 2 weeks after production. Each test sample shall be wrapped appropriately and stored so as not to be contaminated by chemical substances or affected by heat, humidity or other factors.

The storage period and storage conditions shall be reported. This requirement applies also to the case where such conditions are agreed upon between the parties concerned.

Details are specified in **Annex A** with respect to preparation and storage of test samples.

### **6.4 Heating temperature**

Sampling bags shall be heated at 65 °C.

**NOTE** The heating temperature may be specified between the parties concerned.

### **6.5 Heating time**

Sampling bags shall be heated for 2 h.

### **6.6 Gas amount to be filled in a sampling bag**

Amount of nitrogen gas to be filled in a sampling bag shall be 5 L.

## **7 Verification of test conditions**

### **7.1 Monitoring of test conditions**

Heating temperatures shall be monitored and recorded.

Temperature accuracy of measuring instruments shall be within  $\pm 0,5$  °C.

### **7.2 Recovery rate**

Recovery rates of the subject VOC, formaldehyde and other carbonyl compounds shall be measured using corresponding standard gases. Sampling bags shall provide average recovery rates better than 60 % for formaldehyde and 70 % for toluene.

**NOTE** It will be difficult to satisfy the minimum accuracy requirements for the test if there is a sink effect or leakage and if the calibration accuracy is insufficient. Sink effect and absorption characteristics are closely related with the kinds of diffused VOC, formaldehyde and other carbonyl compounds. In order to identify their effects, VOC, formaldehyde and other carbonyl compounds with different molecular mass or polarity may be subjected to additional recovery tests (see **Annex B**).

## **8 Test method**

### **8.1 Test equipment**

The test equipment arrangement is outlined in **Figure 1**.

### **8.2 Preparation for testing**

#### **8.2.1 Cleaning of sampling bags**

Sampling bags shall be cleaned prior to testing as follows:

- connect a teflon tube to the sampling bag sleeve;
- fill the bag with nitrogen gas, then empty the bag using a pump;
- repeated the filling and emptying operation three times.

**NOTE** Sampling bags may be heated beforehand in order to lower the blank concentrations.

#### **8.2.2 Preparation of sampling bags**

Cut an end of a sampling bag after cleaning, and put a or multiple test samples in it. Fold the cut end of the sampling bag and securely seal the portion using a sealing material (e.g., tape) or heat bond the portion. Fill the sampling bag with nitrogen gas, and then empty the bag. Fill the bag again with a certain amount (5 L) of nitrogen gas. The same sequence of operation shall be applied to a sampling bag without a test sample to make it a blank sampling bag.

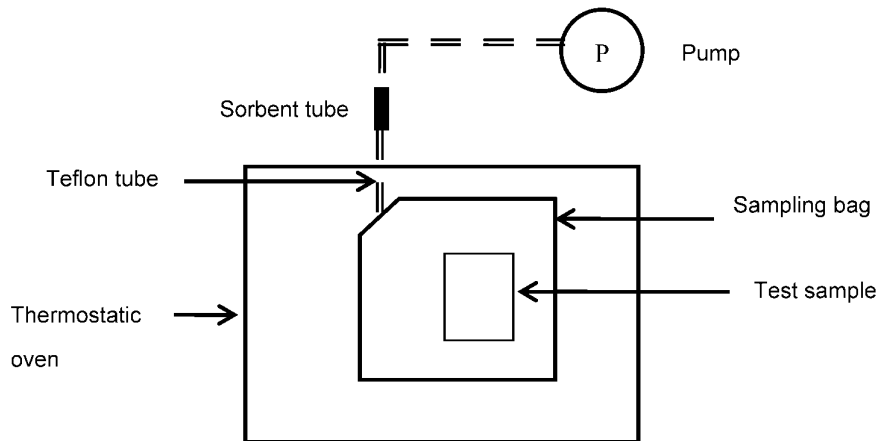
### **8.3 Diffusion test**

Put the sampling bag containing one or multiple test samples in a thermostatic oven kept at a specified temperature (see **6.4**), and take the teflon tube connected to the sleeve outside of the oven through the opening. The blank sampling bag shall also be put in the oven. Both sampling bags shall be heated for a specified period of time (see **6.5**).

### **8.4 Gas collection**

Confirm that the temperature in the oven is in a steady state condition after heating for a specified period of time (see **6.5**), and then connect a sorbent tube specified in **5.8** for collecting the gases in the sampling bag. Gases shall also be collected from the blank sampling bag at the same time.

The collected gas volumes shall be 1 L for VOC and 3 L for formaldehyde and other carbonyl compounds, after temperature calibration. Additional collection of VOC may be performed as required.



**Figure 1 – Outline of test equipment arrangement**

## 9 Analysis procedures

### 9.1 Analysis of VOC

Connect the VOC sorbent tube (see 5.8) to the thermal desorption system and desorb VOC by heating. Analyze VOC in accordance with **JIS A 1966** and **ISO 16000-6**. Determine the test concentration and the blank concentration of VOC.

### 9.2 Analysis of formaldehyde and other carbonyl compounds

Dissolve and desorb the DNPH derivatives of the carbonyl compounds in the DNPH cartridge using acetonitrile. Analyze formaldehyde and other carbonyl compounds in accordance with **JIS A 1962**. Determine the test concentrations and the blank concentrations of formaldehyde and other carbonyl compounds.

## 10 Calculation of sampling bag values

Determine the sampling bag value of a gas component diffused from a test sample as follows:

$$W = \frac{(C_s - C_b) \times V_s}{u}$$

where

- $C_s$  is test concentration ( $\mu\text{g}/\text{m}^3$ );
- $C_b$  is blank concentration ( $\mu\text{g}/\text{m}^3$ );
- $V_s$  is amount of gas filled in the sampling bag ( $\text{m}^3$ );
- $W$  is sampling bag value ( $\mu\text{g}$ );
- $u$  is number of test samples.

## 11 Test report

Test report shall contain the following items, in general (see **Annex B**):

- a) Testing organization:
  - Name and address;
  - Name of the person responsible for the test.
- b) Automobile interior part tested:

- Kind or type (part name, if possible);
  - Selection process of parts to be tested (e.g., Sampling method);
  - Other information on the automobile interior part (production date, batch number, arrival date at the testing organization, date of unpacking, storage conditions, date and time when the test samples were prepared for testing, etc.).
- c) Test results:**
- Sampling bag values of the subject VOC, formaldehyde and other carbonyl compounds or TVOC.
- d) Test conditions:**
- Sampling bag conditions (temperature, time, nitrogen gas volume);
  - Upper surface area of the test sample;
  - Information regarding collection of the subject VOC, formaldehyde and other carbonyl compounds (sorbent tubes used, collected gas volumes, etc.).
- e) Measuring instrument:**
- Information regarding the instruments used and the handling in terms of sampling bags, sealing materials, thermostatic ovens, pumps, gas analyzers, etc.
- f) Quality control / assurance:**
- Blank concentrations of the subject VOC, formaldehyde and other carbonyl compounds;
  - Recovery rates used for evaluating the sink effect of the subject VOC, formaldehyde and other carbonyl compounds;
  - Number of measurements;
  - Accuracies of temperature measurement.

An example of the test report format is shown in **Table 1**.

**Table 1 – Example of test report format**

**a) Testing organization**

Name:	XXXXX Corporation
Address:	A-B, CC, DD-ku, Tokyo
Responsible person:	Ichiro Kikaku

**b) Automobile interior part tested**

Kind or type (Part name):	Seat
Product number:	AB1234
Sampling method:	A piece of 10 cm x 10 cm has been cut out from the part
Shape of the sample:	Thickness 3 mm, mass 12 g
Production date:	2007.03.13
Arrival date:	2007.03.15
Date of unpacking:	2003.03.16
Date and time when the test samples were prepared for testing:	2007.03.16, 15:00
Test date:	2007.03.16
Remarks:	

**c) Test results**

<u>Gas component</u>	<u>Sampling bag values (µg)</u>
Formaldehyde	N.D. (≤0.xyz)
Acetaldehyde	1,0
Toluene	2,0
o-, m-, p- xylene	3,0
Ethyl benzene	4,0
Styrene	5,0
TVOC	100

**d) Test conditions**

Heating temperature: 65 °C

Heating period: 2 h

Nitrogen gas volume: 5 L

Upper surface area of the test sample: 100 cm<sup>2</sup>

Collecting conditions of VOC

Sorbent tube: Tenax-TA

Collected air volume: 1 L×2

Collecting conditions of formaldehyde and other carbonyl compounds

Sorbent tube: DNPH

Collected air volume: 3 L

Ambient air temperature: 23 °C

Ambient air humidity : 55 % (RH)

Remarks :

**e) Measuring instrument**

Thermostatic oven: Type AAA manufactured by BBB Corp.

Sealing material: Teflon sealing tape

Heat bond: Not used

Air collecting equipment: Sampling pump manufactured by CCC Corp.

Gas analyzers

Formaldehyde and other carbonyl compounds

High-speed liquid chromatograph: DDDD

Detector: EEEE

VOC (Toluen, xylene, other TVOC)

Thermal desorption system: FFFF

Gas chromatograph – Mass analyzer: GGGG

**f) Quality control / assurance**

<u>Test item</u>	<u>Test concentration (µg/m<sup>3</sup>)</u>	<u>Blank concentration (µg/m<sup>3</sup>)</u>
Formaldehyde	N.D.(≤000)	N.D.(≤000)
Acetaldehyde	×	N.D.(≤000)
Toluene	××	N.D.(≤000)



o-, m-, p- xylene	xxx	N.D.(≤000)
Ethyl benzene	yyy	N.D.(≤000)
Styrene	zzz	N.D.(≤000)
TVOC	●●●●	●●●
Number of measurements:	1	
Recovery rate:	85 % (Toluene)	
Temperature accuracy:	± 0,5 °C	

**g) Data analysis**

Sampling bag values,  $W$ , of the test sample are calculated by the following equation.

$$W = \frac{(C_s - C_b) \times V_s}{u}$$

where

- $C_s$  is test concentration of VOC, formaldehyde and acetaldehyde diffused from the test samples under heating at a specified temperature for a specified period of time;
- $C_b$  is blank concentration of the sampling bag which was heated, without any test sample, under the same heating condition;
- $V_s$  is the gas amount filled in the sampling bag ( $m^3$ );
- $u$  is number of test samples.

**h) Remarks**

None.

## Annex A (informative)

### General procedures for preparation and storage of test samples

#### A.1 Introduction

This annex describes general procedures for preparation and storage of test samples, but does not constitute a part of normative requirements. The procedures should be reviewed and revised, if necessary, according to new knowledge and experiences to be obtained as well as to the progress of international evaluation of the procedures based on such technical findings in the future.

#### A.2 Sampling of products and transportation and storage of test samples

##### A.2.1 General

In order to determine sampling bag values of VOC and aldehydes diffusing from automobile interior parts using sampling bags, the subject products shall be handled properly before and during the test. This procedure applies only to automobile interior parts which are new and to be used in the automobile assembly.

##### A.2.2 Sampling method of products

Products to be subjected to testing shall be manufactured, wrapped and handled in the normal way. The test samples cut out from the products which have been randomly selected from the subject products shall be wrapped immediately and transported to the testing organization as soon as possible.

##### A.2.3 Wrapping and transportation of test samples

Test samples shall be protected from chemical contamination or effects by heat or humidity. Each test sample shall be wrapped with aluminum foil (with the glossy surface outside) or aluminum processed wrapping material and encapsulated in a polyethylene bag or in a sheet lined with transparent polyvinyl fluoride film.

**NOTE** The diffusion characteristic of the test samples may be affected by the transportation conditions. Special attention needs to be paid to the temperature effect.

##### A.2.4 Storage of test samples until testing

Diffusion tests on the test samples shall be initiated upon arrival at the testing organization. In case the test samples are stored in the testing organization until the onset of the testing, they shall be securely sealed with the aforementioned material (see **A.2.3**) in order to avoid any degradation during the storage period (generally, two weeks).

#### A.3 Miscellaneous

Preparation and storage of test samples shall be in accordance with the aforementioned procedures, however, the parties concerned may establish other procedures taking test sample material or manufacturing process into consideration.

## Annex B (informative)

### Additional information on test report and recovery rate

#### **B.1 Introduction**

This annex describes additional information on test report and recovery rate, but does not constitute a part of normative requirements. The information should be reviewed and revised, if necessary, according to new knowledge and experiences to be obtained as well as to the progress of international evaluation of such information based on technical findings in the future.

#### **B.2 Test report and recovery rate**

The test report and recovery rate shall be in accordance with the normative requirements in this Standard, however, this Standard does not impede establishment of other processes or procedures to be agreed upon between parties concerned.

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#### **Related standards:**

- |                   |  |
|-------------------|--|
| <b>JIS K 0123</b> | General rules for gas chromatography/mass spectrometry |
| <b>JIS Z 8703</b> | Standard atmospheric conditions for testing            |