INTERNATIONAL STANDARD

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Interior air of road vehicles —

Part 9:

Determination of the emissions of volatile organic compounds from vehicle interior parts — Large bag method

Air intérieur des véhicules routiers —

Partie 9: Détermination des émissions de composés organiques volatils des parties et matériaux intérieurs des véhicules — Méthode du grand sac . Cilox . Cilox







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Contents		
Fore	eword	iv
Intro	oduction	v
1	Scope	1
2	Normative references	1
3	Terms and definitions	
4	Principle	
5	Annaratus and materials	2
	Apparatus and materials 5.1 General 5.2 Large sampling bag 5.2.1 General 5.2.2 Material 5.2.3 Air tightness 5.2.4 Bag blank concentration 5.3 Purity of the filling gas 5.4 Thermostatic oven 5.5 Pumps 5.6 Integrating flow meter or gas meter	3 3 3 3 3
6	5.6 Integrating flow meter or gas meter Test conditions 6.1 General 6.2 Vehicle interior parts 6.3 Storage period and storage conditions 6.4 Heating temperature 6.5 Heating time 6.6 Gas amount to be filled in a sampling bag	4 5 5
7	Verification of test conditions 7.1 Monitoring of test conditions 7.2 Recovery rate	5 5
8	Test method 8.1 Test equipment 8.2 Preparation for testing 8.2.1 Eleated cleaning of sampling bag 8.2.2 Preparation of large sampling bags 8.3 Emission test	5 6 6 6
9	8.4 Gas sampling Analysis procedures 9.1 Analysis of VOC 9.2 Analysis of formaldehyde and other carbonyl compounds	
10	Calculation of sampling bag values	8
11	Test report	8
12	Quality assurance/quality control (QA/QC)	11
Ann	ex A (informative) Summary for heated cleaning process for large sampling bags	12
Ann	ex B (informative) Supplement on test report and recovery rate	13
	iography	15

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 6, *Indoor air*.

A list of all parts in the ISO 12219 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Volatile organic compounds (VOCs) are widely used in industry and can be emitted by many every-day products and materials. They have attracted much attention in recent years because of their impact on cabin air quality. After homes and workplaces, people spend a lot of time in their vehicles. It is important to determine the material emissions of interior parts and to reduce them to an acceptable level. Therefore, it is important to get comprehensive and reliable information about the types of organic compounds in the interior air of vehicles and also their concentrations.

This document outlines the sampling bag test method of volatile organic compounds (VOC), formaldehyde and other carbonyl compounds which diffuse from vehicle interior parts into the air inside road vehicles.

Measuring VOC from vehicle interior parts can be performed in several ways and the approach selected depends upon the desired outcome and the material type. For example, to obtain diffusion data from complete assemblies (e.g. instrument panel, seat, etc.), chambers/bags that have sufficient volume to house the complete assembly are employed. Meanwhile, to obtain diffusion data from representative samples of homogeneous vehicle interior materials, micro-scale chamber method can be chosen.

Each measurement method such as bag/micro-scale chamber/small-chamber sampling offers a complementary approach.

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Interior air of road vehicles —

Part 9:

Determination of the emissions of volatile organic compounds from vehicle interior parts — Large bag method

1 Scope

This document specifies a large bag sampling method for measuring volatile organic compounds (VOCs), formaldehyde and other carbonyl compounds which are emitted from vehicle interior parts into the air inside road vehicles. This method is intended for evaluation of large new vehicle interior parts, and complete assemblies. This is a screening method to compare similar car components under similar test conditions on a routine basis.

Evaluating VOC emissions of vehicle interior parts is an important aspect of the vehicle indoor air quality.

This document is complementary to existing standards and provides test laboratories and the manufacturing industry with a cost-effective evaluation of vehicle interior parts. This method is only applicable to newly manufactured vehicle parts. This method is applicable to all types of vehicles, and vehicle products which are used as parts in the interior of vehicles.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16000-3, Indoor air — Part 3: Determination of formaldehyde and other carbonyl compounds in indoor air and test chamber air — Active sampling method

ISO 16000-6, 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 using MS or MS-FID

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

3.1

vehicle interior part

part which is used in the interior of a vehicle including related materials such as adhesives and coating materials

[SOURCE: ISO 12219-2:2012, 3.1]

3.2

sampling bag value

concentration increment of a subject gas component due to the diffusion of VOCs, formaldehyde and other carbonyl compounds from a *vehicle interior part* (3.1), multiplied by the total amount of the gas filled in the sampling bag

[SOURCE: ISO 12219-2:2012, 3.2 modified — "vehicle interior part" replaces "test specimen".]

3.3

volatile organic compound

VOC

organic compound eluting between and including n-hexane and n-hexadecane on a gas chromatographic column specified as a 5 % phenyl 95 % methyl polysiloxane capillary column

3.4

total volatile organic compound TVOC

sum of volatile organic compounds, sampled on Tenax TA®, which elute between and including n-hexane and n-hexadecane on a non-polar capillary column, detected with a flame ionization detector (TVOC-FID) or mass spectrometric detector (TVOC-MS), and quantified by converting the total area of the chromatogram in that analytical window to a nominal mass using the chromatographic response factor for toluene (toluene equivalents)

[SOURCE: ISO 16000-6:2011, 3.4, modified —Note has been deleted.]

4 Principle

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

Vehicle interior parts put in a sampling bag are heated at a specified temperature, and then a fraction of the gas in the sampling bag is collected to measure the test concentrations. By comparing the test concentrations with the corresponding bag blank concentrations, the sampling bag values of VOC, formaldehyde and other carbonyl compounds diffusing from vehicle interior parts can be calculated (see <u>Clause 10</u>).

The analytical part of the overall measurement procedure is based on the use of sorbent tubes with subsequent thermal desorption and gas chromatographic analysis for VOCs (according to ISO 16000-6) and the use of 2,4-dinitrophenylhydrazine (DNPH) sorbent tubes, followed by high performance liquid chromatography (HPLC) analysis with ultraviolet absorption for the determination of formaldehyde and other carbonyl compounds (according to ISO 16000-3).

The specified analytical procedure is valid for the determination of volatile organic compounds (VOCs) ranging in concentration from sub- μ g/m³ to several mg/m³. The method is applicable to the measurement of non-polar and slightly polar VOCs ranging in volatility from n-C₆ to n-C₁₆.

The specified analytical procedure for formaldehyde and other carbonyl compounds is valid for the determination of carbonyls within the concentration range of approximately 1 $\mu g/m^3$ to 1 mg/m^3 .

5 Apparatus and materials

5.1 General

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

- sampling bag;
- nitrogen gas or air (filling gas);

- thermostatic oven;
- pumps;
- integrating flow meter;
- analytical equipment is described in ISO 16000-3 and ISO 16000-6.

5.2 Large sampling bag

5.2.1 General

Large sampling bags used in this document shall be in accordance with 5.2.2, 5.2.3, 5.2.4 and 7.2

They shall be low-emitting, low-permeable and low-sorption plastic bags having a capacity of 20 l to 2 000 l used for measuring VOC, formaldehyde and other carbonyl compounds which can emit from vehicle interior parts. The size of the bag depends on the size of the part and is to be agreed upon between the parties.

5.2.2 Material

Though no specification is set for the material of a sampling bag, the recommended materials are fluorinated resin [vinyl fluoride (PVF), polyvinylidene fluoride (PVDF), 4 ethylene fluoride - 6 propylene fluoride copolymer (FEP)], ethylene-vinyl alcohol copolymer (EVOH). Large sample bags made of just a single layer of polyethylene terephthalate (PET) or polyolefins (PEHD) can have high permeation through the bag material. Total film thickness should be above 20 microns.

5.2.3 Air tightness

The large sampling bag shall be sealed airtight using a sealing material such as a tape that will not affect the test or by the heat seal method so that the gases inside are not mixed with any uncontrolled outside air.

5.2.4 Bag blank concentration

The bag blank concentration generated by heating a large sampling bag filled with only nitrogen or air shall be at such a low level as to not affect the test. Each target VOC, collecting 1 l on the sampling tube, shall have a value less than 20 ng collected on each tube and for formaldehyde and/or any other carbonyl compound, collecting 3 l on the DNPH cartridge, shall have a value less than 200 ng collected on each cartridge.

Users should be aware that TVOC bag blank values from bags vary significantly depending on material, age, cleaning procedure and analytical process. Report specific bag cleaning parameters and all analytical conditions and calculation methods in order to interpret TVOC report values.

5.3 Purity of the filling gas

For the nitrogen gas or air filled in a large sampling bag, use a high purity gas containing as few impurities as possible so that the concentration of any contained VOC, formaldehyde and other carbonyl compounds will not affect the diffusion test. It is recommended to use nitrogen gas of a purity of 99,999 % or more.

5.4 Thermostatic oven

To control the temperature, use a thermostatic chamber capable of uniform temperature control.

The thermostatic chamber into which a sampling bag is set shall be capable of controlling the temperature distribution accuracy of \pm 2 °C.

5.5 Pumps

Use a vacuum pump or equipment that can draw the gas sufficiently from a sampling bag.

5.6 Integrating flow meter or gas meter

The volume of sampled gases or other gases shall be measured and adjusted to standard conditions (23 °C and 101,3 kPa) with an integrating flow meter or a gas meter ($V \pm 0.1$ l, where V is volume).

6 Test conditions

6.1 General

Test conditions shall be in accordance with <u>6.2</u> to <u>6.6</u>. The test environment shall be sufficiently ventilated to minimize the background effect.

6.2 Vehicle interior parts

The parts to be tested shall be complete, uncut parts. If there is any cutting, sectioning or processing of the part before the test, it shall be indicated on the report, as it can affect the results.

6.3 Storage period and storage conditions

The product to be tested shall be new parts manufactured, packaged, and handled by ordinary means.

The parts to be tested shall be packaged on the same day as manufacturing step.

Before storing or transporting a vehicle interior part, package it individually in an appropriate material such as an aluminium foil and put the package in a polyethylene film bag so that it is not contaminated with chemicals and affected by heat or moisture during the storage and transport.

The vehicle interior parts shall be stored one by one in an appropriate package so that they are not contaminated with chemicals and affected by heat, humidity, or other conditions.

The storage period of a vehicle interior part shall not exceed 2 weeks after it is manufactured, unless a different storage period is agreed prior to testing.

When it is stored in the test laboratory until the measurement starts, the part shall be kept in sealed state with the packaging materials above during the storage period to prevent any degradation.

The vehicle interior parts are removed from the packaging 1 week before the test.

The protectors shall be removed in the case of components, if applicable.

Store vehicle interior parts at 23 °C \pm 2 °C and nominally 50 % RH. In order to prevent contamination of the test specimens with hydrocarbons, attention shall be paid to fresh air exchange and air flow in the storage room.

The parts shall be stored individually with sufficient space between them so that they are not contaminated with chemicals and affected by heat, humidity, or other condition.

Ensure that all surfaces of the test specimen can be ventilated without obstruction and that the parts are not placed on their visible sides. Flat-spread materials in particular (e.g. leather, fabrics, foils, plastics plates) shall be placed on a rack or grating.

Deviations from the preliminary storage procedure described shall be documented in precise detail in the test report.

See ISO 12219-8 for additional storage best practices.

The storage period and condition of each vehicle interior part is a report item, which is also applied when the period and condition are agreed between the test requestor and test laboratory parties.

6.4 Heating temperature

The temperature of sampling bag heating shall be (65 ± 2) °C. The temperature is controlled by the general oven sensor (see Figure 2).

A different heating temperature may be agreed between the test requestor and test laboratory parties.

6.5 Heating time

Large sampling bags shall be heated for 4 h ± 5 min.

A different heating time may be agreed upon between the delivery/acceptance parties (see Figure 2).

6.6 Gas amount to be filled in a sampling bag

The amount of nitrogen gas or air to be filled in a sampling bag shall be (50 ± 5) % of the bag size.

A different gas charge amount may be agreed upon between the delivery/acceptance parties.

7 Verification of test conditions

7.1 Monitoring of test conditions

Monitor and record the heating temperature (e.g. the temperature inside the oven). The temperature accuracy of the measurement instrument shall be within \pm 0,5 °C of the specified temperature.

7.2 Recovery rate

Recovery rate is defined as the percentage 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.

Use a standard gas and others containing the applicable component at levels similar to the expected vehicle interior part measurement concentrations to measure the recovery rate of applicable VOC, formaldehyde or other carbonyl compounds. The sampling bag shall be able to achieve an average recovery rate of 70 % or more for each reported target VOC or 60 % or more for formaldehyde or other reported carbonyl compounds. Follow the same process to recover the standard gases as described in the vehicle interior part test conditions. The recovery shall be conducted after installation of the sample system and after major maintenance, more often if agreed to by the test requester.

It is 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 emitted 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 introduced to the sample bags for additional recovery tests (see B.3).

8 Test method

8.1 Test equipment

The test equipment arrangement is illustrated in Figure 1.

8.2 Preparation for testing

8.2.1 Heated cleaning of sampling bag

Apply a heated cleaning process (see Annex A) to the large sampling bags before starting the test.

The heating temperature, operation method and number of repetitions shall be agreed upon between the delivery/acceptance parties before the test in consideration of the type and size of the large sampling bags.

8.2.2 Preparation of large sampling bags

Cut off one end of a cleaned sampling bag and put in the vehicle interior parts. Vehicle interior parts are put in the bag at the same direction as in the cabin. Related sub-assemblies, which are to be tested together, should be spaced out within the bag. Because fluctuation of the VOC concentration occurs.

Then seal the bag by a sealing material (tape) or heat-sealing method. Charge the sampling bag with nitrogen gas or air and discharge the gas using a pump.

After that, charge the sampling bag with a certain amount (see <u>6.6</u>) of nitrogen gas or air. Perform the same steps to another equivalent and empty sampling bag (with no vehicle interior part) as the bag blank.

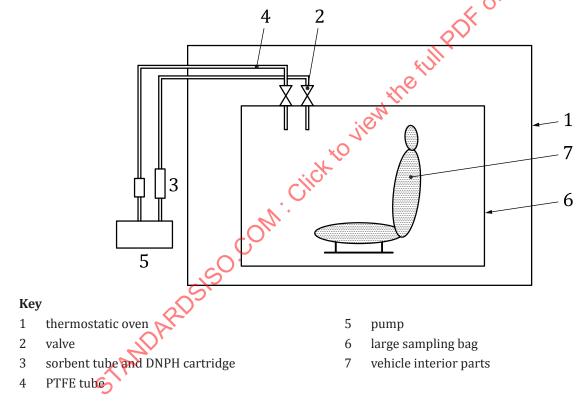
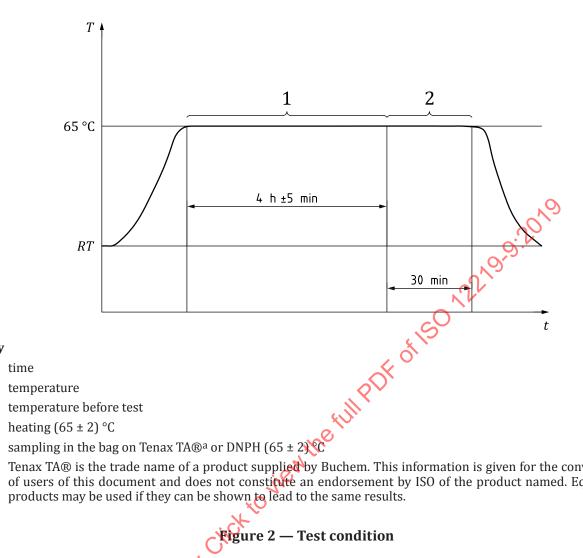


Figure 1 — Outline of test equipment arrangement (sampling line instead of PTFE tube)

8.3 Emission test

Place the sampling bag containing the vehicle interior parts into the thermostatic oven set to a specified temperature (see 6.4) and take out the end of the polytetrafluoroethylene tube attached to the sleeve from the opening of the oven.

Also place the bag blank in the thermostatic oven and apply heat for a certain time (see Figure 2).



Key

- t
- T

RT

- 1
- 2
- Tenax TA® is the trade name of a product supplied by Buchem. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent

8.4 **Gas sampling**

After the heating time has elapsed (see 6.5) the dead volume in the sample tubing shall be purged with the gas from the bag sample before connecting the sorbent tubes and sampling.

The minimum gas volume to be sampled from the bag sample should be 1 l for VOCs and 3 l for carbonyls (on DNPH cartridges).

Sampling lines shall be as short as possible and may be heated to ensure against condensation.

Further recommendation on sampling volumes for VOCs can be found in ISO 16000-6. The limited 3 l sampling volume can compromise the detection limits for formaldehyde as reported in ISO 16000-3.

The collection of duplicate VOC samples is recommended to provide a backup.

The specific length of the sampling tube may be agreed upon between the delivery/acceptance parties.

Analysis procedures

9.1 Analysis of VOC

Analyse VOCs in accordance with ISO 16000-6. Determine the test concentration and the bag blank concentration of VOCs.

9.2 Analysis of formaldehyde and other carbonyl compounds

Analyse formaldehyde and other carbonyl compounds in accordance with ISO 16000-3. Determine the test concentrations and the bag blank concentrations of formaldehyde and other carbonyl compounds.

10 Calculation of sampling bag values

Determine the sampling bag value from a vehicle interior part using Formula (1).

$$m = (\rho_{\rm s} - \rho_{\rm b}) \times V_{\rm s} \tag{1}$$

where

m is the sampling bag value (µg);

 $\rho_{\rm S}$ is the test concentration (µg/m³);

 $\rho_{\rm b}$ is the bag blank concentration ($\mu g/m^3$);

 $V_{\rm S}$ is the volume of gas introduced to the bag (m³).

11 Test report

Generally, the report should contain the following contents (refer to Table 1 and Annex B).

- a) Test laboratory
 - Name and address of test laboratory
 - Name of responsible tester
 - Date of test
- b) Information on vehicle interior parts
 - Type of vehicle interior parts (and product name if available)
 - Background of vehicle interior parts (such as manufactured year/month/day, batch number, date of arrival at test laboratory, date/time of unpacking, storage conditions, and date/time of part preparation)
- c) Result
 - Sampling bag value of applicable VOC, formaldehyde and other carbonyl compounds or TVOC
- d) Test conditions
 - Sampling bag conditions (such as temperature, time, amount of nitrogen or air, bag material, and bag capacity)
 - Outline dimensions of vehicle interior part
 - Information on gas sampling of applicable VOC, formaldehyde and other carbonyl compounds (such as sampling tube in use and gas sampling amount)
- e) Measurement instruments
 - Information on instruments and methods in use (such as sampling bag, sealing material, thermostatic oven, pump, and analyser)

- f) Data on quality control/quality assurance
 - Bag blank concentrations of applicable VOC, formaldehyde and other carbonyl compounds
 - Recovery rate data for evaluating the sink effect of applicable VOC, formaldehyde and other carbonyl compounds
 - Number of measurements
 - Accuracy of measurement

An example report form is indicated in <u>Table 1</u>.

Table 1 — Report form example

a) Test laboratory

Test laboratory name	XXXX, Co., Ltd.
Address	XXXX, Tokyo, Japan
Responsible tester	Ichiro Kikaku 🔿

b) Type of product

Type of product (product name)	Front seat RH		Product code AB1234	
Part sampling method	Fill this column if the	ill this column if the part is cut or manufactured.		
Part shape	Enter the shape information such as thickness, mass, and outline dimensions.			
Day/month/year of manufacturing	Date of arrival at test laboratory	Date/time of unpacking	Date/time of part preparation	
DD/MM/YYYY	DD/MM/YYYY	DD/MM/YYYY, HH o'clock	DD/MM/YYYY, HH o'clock	
Date of test DD/MM/YYYY	Others	_		

c) Test result

Test item	Sampling bag value (µg)	
Formaldehyde	N.D.(<0,000)	
Acetaldehyde	1,0	
Toluene	2,0	
o-, m-, p-xylene	3,0	
Ethyl benzene	4,0	
Styrene	5,0	
<i>p</i> -dichlorobenzene	1,0	
TVOC (informative)	100	

d) Test conditions

Heating temperature (°C) Heating time (h)		Amount of nitrogen gas or air (l)	Outline dimensions of part (cm)	
65	4,0	000 (nitrogen gas) 00 × 00 × 00		
VOC sampling conditions		Sampling conditions for formaldehyde and other carbonyl compounds		
Sampling tube	Sampled gas amount (l)	Sampling tube	Sampled gas amount (1)	
Tenax TA®	1 l × 2 tubes	DNPH	3	
Test site temperature (°C) Test site humidity (% RH)		Bag material	Bag capacity (1)	
23 50		PVF	300	
Special notes				
_			120	

e) Measurement instruments

Thermostatic chamber		XXX made by YYY
Sealing material		Polytetrafluoroethylene seal tape
Heat seal		- "X
Gas sampling device		Sampling pump made by ZZZ
	Formaldehyde and other carbonyl compounds	High performance liquid chromatographs
Analyser	Analyser VOC (toluene, xylene, etc., TVOC)	Detector
Analysei		Heating introduction device
		Gas chromatograph - mass spectrometer

f) Quality control/quality assurance

Toot itom	Test concentration	Bag blank concentration	
Test item	$(\mu g/m^3)$	(µg/m³)	
Formaldehyde	N.D. (<000)	N.D. (<000)	
Acetaldehyde	×	N.D. (<000)	
Toluene	× ×	N.D. (<000)	
o-, m-, p-xylene	× × ×	N.D. (<000)	
Ethyl benzene	Δ	N.D. (<000)	
Styrene	× × ×	N.D. (<000)	
<i>p</i> -dichlorobenzene	p -dichlorobenzene $\Delta\Delta$ N.D. (<000)		
TVOC (informative)	••••	•••	
Number of measurements	Recovery rate data	Accuracy of temperature	
1	70 % (formaldehyde)	±1 °C	
1	70 % (acetaldehyde)	±1 °C Accuracy of volume	

1	85 % (toluene)	±1 °C	
1	80 % (o-, m-, p-xylene)	±1 °C	
1	80 % (ethyl benzene)	±1 °C	
1	80 % (styrene)	±1 °C	
1	80 % (p-dichlorobenzene)	±1 °C	

g) Data analysis

Data analysis	The sampling bag value from a vehicle interior parts (<i>m</i>) is calculated by the for-
	mula below. The test concentration ps indicates the concentration of VOC, formaldehyde or other carbonyd company and a pritted from the vehicle interior parts beat 2014 a certain term.
	carbonyl compounds emitted from the vehicle interior parts heated at a certain temperature for a certain length of time, and ρ_b indicates the bag blank concentration of the sampling bag with no vehicle interior parts that is heated under the same
	conditions. V_s indicates the gas charge amount (m ³). $m = (\rho_s - \rho_b) \times V_s$

h) Remark

Nothing particular.

12 Quality assurance/quality control (QA/QC)

If this bag method is to be used for estimating the contribution of a product to atmospheric concentrations of organic vapours into vehicle cabin air experimentally, follow the analytical quality assurance plans given in relevant standards (e.g. ISO 16000-9).

An appropriate level of quality control shall be employed following ISO 16000-3 and ISO 16000-6 including:

- Field blanks are prepared according to <u>Clause 8</u>.
- The field blank level is acceptable if artefact peaks are no greater than 10 % of the typical areas of the analytes of interest.
- Desorption efficiency of VOCs/carbonyls should be checked according to ISO 16000-3 and ISO 16000-6.
- The collection efficiency can be controlled by using back-up tubes or taking samples of different sampling volumes less than the safe sampling volume.
- Repeatability of the air sampling and analytical method shall be determined. A relative standard deviation \leq 15 % (ISO 16000-3 and ISO 16000-6) from the duplicate measurements should be reached.

NOTE The repeatability of the emission test will be influenced by the homogenity of the material under test.

- The recovery of n-C6 to n-C16 hydrocarbons shall be 95 % (ISO 16000-6).
- Documentation illustrating traceable calibrations for temperature, humidity, and flow measurements.