BS EN 13999-1:2013

BSI Standards Publication

# Adhesives — Short term method for measuring the emission properties of low-solvent or solvent-free adhesives after application

## Part 1: General procedure

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## National foreword

This British Standard is the UK implementation of EN 13999-1:2013. It supersedes BS EN 13999-1:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/52, Adhesives.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

# Adhesives - Short term method for measuring the emission properties of low-solvent or solvent-free adhesives after application - Part 1: General procedure

Adhésifs - Méthode de mesurage rapide des caractéristiques émissives des adhésifs à teneur faible ou nulle en solvant après application - Partie 1 : Mode opératoire général

Klebstoffe - Kurzzeit-Verfahren zum Messen der Emissionseigenschaften von lösemittelarmen oder lösemittelfreien Klebstoffen nach der Applikation - Teil 1: Allgemeines Verfahren

This European Standard was approved by CEN on 19 October 2013.

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**CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels**

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# Foreword

This document (EN 13999-1:2013) has been prepared by Technical Committee CEN/TC 193 “Adhesives”, the secretariat of which is held by AENOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2014, and conflicting national standards shall be withdrawn at the latest by June 2014.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 13999-1:2006.

EN 13999-1:2013 includes the following significant technical changes with respect to EN 13999-1:2006:

a) In general, the text is adapted to the new CLP Regulation No 1272/2008 [11].

b) In 6.3, a new Note 2 is added, emphasizing the need that materials be tested under same conditions using the spreading techniques and the coat weights specific for each application.

c) In 7.5, the sampling time is changed (the sampling time at 24 h is eliminated, a sampling time at 72 h is added), and a note is added concerning the possibility of other sampling times, e.g. after 28 days.

d) “Specific cumulative emission” (previous 8.3.3) is eliminated.

e) The examples from Note to Annex A are eliminated.

EN 13999, under the general title *Adhesives — Short-term method for measuring the emission properties of low-solvent or solvent-free adhesives after application*, consists of the following parts:

— *Part 1: General procedure*

— *Part 2: Determination of volatile organic compounds*

— *Part 3: Determination of volatile aldehydes*

— *Part 4: Determination of volatile diisocyanates*

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# Introduction

Solvent-free and low-solvent adhesives may release considerable long-term emissions of volatile chemical compounds. Any documentation of emission characteristics requires a test method that includes all relevant and potentially hazardous substances.

It is essential that the test

- be reliable and reproducible,
- give results in a short time to be useful for decisions for development projects,
- verify that carcinogenic or sensitizing volatile substances are substantially absent,
- characterize the emission properties of the adhesive.

This can be achieved by sampling the atmosphere around the applied adhesive kept in an environmental test chamber at controlled ambient conditions.

# 1 Scope

This European Standard describes a conventional standard method for assessing potential emissions from adhesives after their application.

This European Standard applies only to “solvent-free” and “low-solvent” adhesives as they are defined in EN 923:2005+A1:2008. The adhesives shall be applicable at room temperature.

# 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 923:2005+A1:2008, *Adhesives — Terms and definitions*

EN 1067, *Adhesives — Examination and preparation of samples for testing*

EN 13999-2, *Adhesives — Short term method for measuring the emission properties of low-solvent or solvent-free adhesives after application — Part 2: Determination of volatile organic compounds*

EN 13999-3, *Adhesives — Short term method for measuring the emission properties of low-solvent or solvent-free adhesives after application — Part 3: Determination of volatile aldehydes*

EN 13999-4, *Adhesives — Short term method for measuring the emission properties of low-solvent or solvent-free adhesives after application — Part 4: Determination of volatile diisocyanates*

EN ISO 15605, *Adhesives — Sampling (ISO 15605)*

EN ISO 16000-9:2006, *Indoor air — Part 9: Determination of the emission of volatile organic compounds from building products and furnishing — Emission test chamber method (ISO 16000-9:2006)*

ISO 554, *Standard atmospheres for conditioning and/or testing — Specifications*

ISO 16000-6:2011, *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 terms and definitions given in EN 923:2005+A1:2008, EN ISO 16000-9:2006 and ISO 16000-6:2011 and the following apply.

### 3.1
**volatile organic compounds**
VOC
all volatile organic compounds eluting between and including n-hexane and n-hexadecane on a gas chromatographic column

Note 1 to entry: The measurement is carried out using a capillary column coated with 5 % phenyl / 95 % methyl-polysiloxane.

# 4 Principle

The test determines the emissions of volatile organic substances from adhesive coatings. Very volatile and mainly particle bound compounds are not detected with the described procedures. The test is performed in an emission test chamber (Figure 1) at specified constant temperature, relative humidity, air mixing and air exchange.

A measurement of the concentrations of the compounds of interest in the air of the exhaust pipe is considered representative of the air in the whole test chamber.

Sampling and analysis of the non-polar and slightly polar volatile organic compounds, as defined in ISO 16000-6, shall be carried out as described in EN 13999-2.

Sampling and analysis of the volatile aldehydes shall be carried out as described in EN 13999-3. Sampling and analysis of the volatile isocyanates shall be carried out as described in EN 13999-4. The test includes:

— qualitative and quantitative determination of carcinogenic and sensitizing substances in an early stage after the application of the adhesive sample,

— determination of the VOC specific emission rate at two points of time.

NOTE There are specific applications for which an emission test cell (as described in EN ISO 16000-10 [9]) can be easier to handle than the emission test chamber, for instance when a sample is too small to be properly applied into the test chamber. In these cases, it is possible to apply the procedures as given in this European Standard with the exception that a test cell is used instead of the test chamber. However, as the air flow rate across the sample is higher than with the chamber method, the results can only be used with supporting correlation data.

## 5 Apparatus

### 5.1 General

The test apparatus shall be comprised of the following main components: support, air supply and mixing facilities, emission test chamber with monitoring and control systems, air sampling devices and devices for desorption and analysis.

### 5.2 Support

A clean stainless steel plate or a glass plate shall be used as a substrate for the adhesive to be applied.

### 5.3 Adhesive coating device

A device able to ensure a uniform coating of the adhesive to the support (5.2) shall be used.

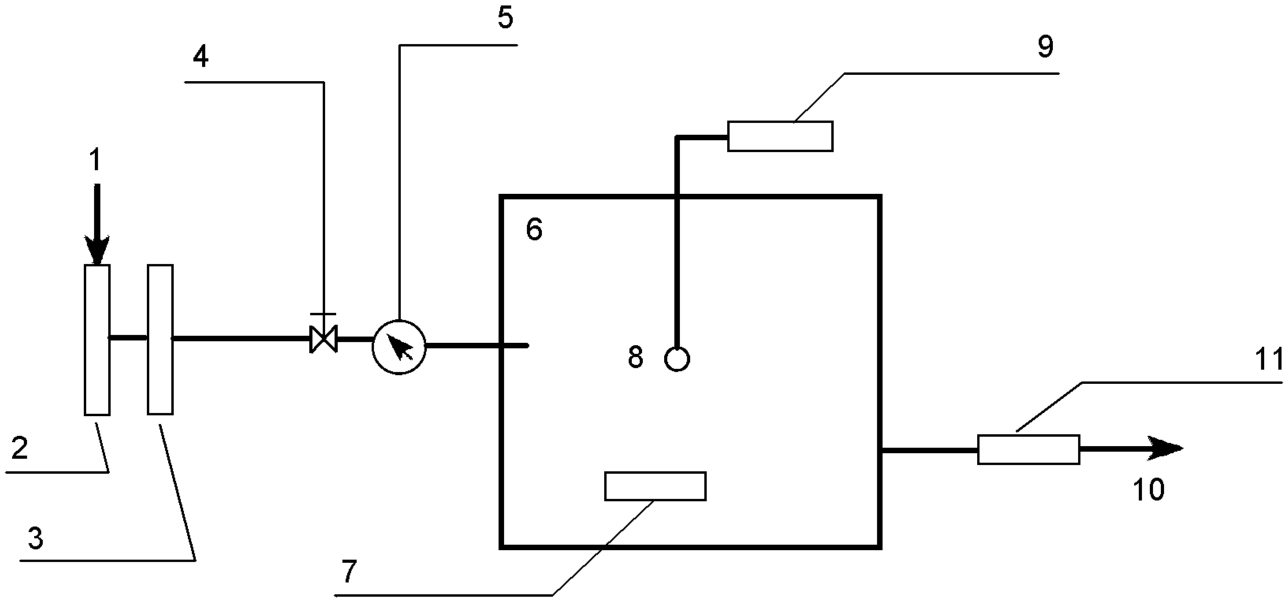
### 5.4 Emission test chamber

All the components of the chamber (see Figure 1) and connected pipes and tubes that will be in contact with the vapours shall be low emitting and low adsorbing and shall not contribute to the emission test chamber background concentration. Polished stainless steel or glass shall be used to build the chamber structure. The internal volume of the chamber shall not be less than 0,004 $m^3$, preferably from 0,1 $m^3$ to 1 $m^3$.

The emission test chamber shall be airtight in order to avoid uncontrolled air exchange with external air. The emission test chamber shall be operated slightly above atmospheric pressure to avoid any influence from the laboratory atmosphere. The emission test chamber is considered sufficiently airtight if at least one of the following requirements is fulfilled:

— air leakage is less than 0,5 % of the chamber volume per minute at an overpressure of 1 000 Pa;

— air leakage is less than 5 % of the supply air flow rate.



**Key**

| | |
|---|---|
| 1 | air inlet |
| 2 | air filter |
| 3 | air conditioning system unit |
| 4 | air flow regulator |
| 5 | air flow meter |
| 6 | test chamber |
| 7 | device to circulate air and control of air velocity |
| 8 | temperature and humidity sensors |
| 9 | monitoring system for temperature and humidity |
| 10 | exhaust outlet |
| 11 | manifold for air sampling |

**Figure 1 — Example of an emission test chamber**

## 5.5 Air supply and mixing facilities

The emission test chamber shall have facilities (e.g. electronic mass flow-meter controllers) capable of continuously controlling the air exchange rate at a fixed value with an accuracy of ± 3 %. The supply and exhaust air system shall be designed to ensure proper mixing of the air inside the chamber and to keep the air velocity at the surface of the test specimen in the range from 0,1 m/s to 0,3 m/s. The supply air shall be very clean and pre-filtered. The TVOC background concentration shall be lower than 20 µg/m$^3$. The background concentration of any single target VOC shall be lower than 2 µg/m$^3$. The control of background concentration shall be carried out with the empty support (5.2) inside the test chamber.

A four-step filter is suggested:

a) filtration membrane particle size (maximum 0,5 mg/m$^3$ breakthrough);

b) activated charcoal (maximum 0,003 mg/m$^3$ breakthrough);

c) high efficiency compressed air dryer (dew point −40 °C at least);

d) high filtration grade (< 0,5 mg/m$^3$ particles breakthrough at 21 °C).

### 5.6 Air sampling devices

The sampling devices as described in EN 13999-2, EN 13999-3 and EN 13999-4 shall take the sample air directly from the exhaust air. The devices shall be connected as close as possible to the test chamber and maintain the same temperature. The sum of sampling air flows should be smaller than 80 % of the inlet air flow to the emission test chamber to avoid under pressure.

Placing the whole apparatus in a room conditioned at (23 ± 1) °C is recommended.

### 5.7 Devices for desorption and analysis

Devices for desorption and analysis shall be used as described in EN 13999-2, EN 13999-3 and EN 13999-4.

## 6 Adhesive sample

### 6.1 General

Take the adhesive sample to be tested in accordance with EN ISO 15605. Examine and prepare this sample for testing in accordance with EN 1067.

### 6.2 Pre-conditioning

Pre-conditioning of the adhesive sample at an operation temperature of (23 ± 1) °C shall be performed at least 24 h before the test is started. To avoid loss of material, the adhesive sample shall be kept in a sealed container.

### 6.3 Preparation of a test specimen

Test specimens shall be prepared not later than 8 weeks after the manufacture of the adhesive sample. The adhesive sample shall be unpacked in the testing laboratory only immediately before starting the test. Unless otherwise specified choose a support with a loading factor (the ratio between the area of application and the chamber volume) of 0,40 $m^2/m^3$ ± 0,02 $m^2/m^3$. The loading factor shall be recorded in the test report. Different adhesives designed for the same application shall be tested at the same loading factor.

NOTE 1 This loading factor corresponds to a totally covered floor in a residence with 2,5 m high walls.

Mechanically stir the sample until homogeneous. Weigh the support to ± 0,1 g. Spread an adequate amount of the adhesive to be tested by a suitable coating device following the manufacturer's instructions until the total surface of the support is covered by a bubble free adhesive coating of uniform surface structure and equal coat weight. Take the coat weight as recommended by the manufacturer. If a range of coat weights is recommended by the manufacturer, the highest coat weight shall be taken.

NOTE 2 The coat weight of an adhesive after application depends on the type of adhesive and its use, surface structure and formulation. To obtain comparable results, it is thus essential that materials be tested under the same conditions using the spreading techniques and the coat weights recommended by the manufacturer, specific for each application. As an example, coat weights for floor-covering and wood-parquet adhesives are usually in the range of (300 ± 50) $g/m^2$.

Weigh the plate again to ± 0,1 g and adjust the adhesive weight to ± 5 % of the target coat weight by adding more or by removing parts of the sample. Record coat weight and loading factor in the test report.

The whole procedure shall not take longer than 5 min.

# 7 Procedure

## 7.1 Number of determinations

Sampling at the chamber outlet and analyses shall be made in duplicate. Both individual values and the mean value shall be given in the test report.

## 7.2 Preconditioning of the test chamber

The test chamber apparatus, including supply air, shall be checked for background contamination prior to carrying out a new materials emission test. The TVOC background concentration shall be lower than 20 µg/m$^3$. The background concentration of any single target VOC shall be lower than 2 µg/m$^3$.

## 7.3 Operation of the test chamber

The test chamber shall be operated at standard climate conditions as described in ISO 554 (23 °C, 50 % RH). The temperature of the chamber and the humidity of the supply air shall be monitored continuously and controlled within ± 1 °C and ± 5 % RH. The supply air flow shall be adjusted to an air exchange rate of (0,5 ± 0,025) times per hour.

Unless otherwise specified in this European Standard, the specifications of EN ISO 16000-9 for the operation of a test chamber apply.

## 7.4 Starting of the test

Place the prepared test specimen (6.3) into the centre of the chamber. Consider this instant as time 0 (zero) for all the time measurements throughout the test.

## 7.5 Sampling and analysis of emitted substances

Perform air sampling according to the following scheduling:

- 72 h;
- 240 h.

Analyse the VOC in the 72 h, and the 240 h air samples. Take the 72 h air samples for determination of carcinogenic and sensitizing substances.

Sample the test chamber air and determine the VOC in the time intervals (72 ± 1) h and (240 ± 1) h after starting the test in compliance with EN 13999-2.

Sample from test chamber air and determine aldehydes in the time interval (74 ± 1) h after starting the test in compliance with EN 13999-3.

Sample from test chamber air and determine diisocyanates in the time interval (76 ± 1) h after starting the test in compliance with EN 13999-4 if the adhesive is polyurethane resin based. Testing in line with EN 13999-4 is not necessary for other products.

If a multisampling port is available, sampling of VOC, aldehydes and diisocyanates can be performed in parallel, starting at (72 ± 1) h.

NOTE Other sampling times (e.g. after 28 days) can be useful for special purposes such as the determination of emission decay curves or long term emissions or for compliance with the Construction Products Regulation (EU) No. 305/2011 [12].

# 8 Expression of results

## 8.1 Calculations

Calculate the chamber air concentrations and the specific emission rates ($SERa$) from the results obtained according to EN 13999-2, EN 13999-3 and EN 13999-4 by Formula (1) and Formula (2):

Chamber air concentration = (Mass in air sample/Volume collected) (1)

where

the mass shall be expressed in µg and the volume in $m^3$.

Specific emission rate = [(Chamber air concentration × air exchange flow)/test area] (2)

where

chamber air concentration shall be expressed in µg/ $m^3$, air exchange flow in $m^3$/h, and the test area in $m^2$.

## 8.2 Carcinogenic and sensitizing compounds concentration

Review the results of sampling and analysis according to 7.5 for carcinogenic and sensitizing compounds.

Report the concentration (µg/$m^3$) and specific emission rate [µg/($m^2$ × h)] and the nature of any detected carcinogenic and sensitizing compound if exceeding a concentration of 5 µg/$m^3$.

The compounds to be taken into consideration are defined in Annex A according to the Regulation (EC) No 1272/2008 [11] and further adaptations to technical progress.

The procedures for identification and quantification depend on the appropriate analytical technique and are described in EN 13999-2, EN 13999-3 and EN 13999-4 respectively.

## 8.3 Volatile organic compounds

### 8.3.1 General

Express the results of VOC as specific emission rate or chamber air concentration.

### 8.3.2 Determination of the specific emission rate at the sampling times

Calculate the total VOC area specific emission rate at 72 h, 240 h or 672 h (see NOTE at 7.5) from the test beginning with Formula (3):

$$SERa\ (t) = C_{voc}\ (t)\frac{F_{cham}}{A_{samp}} \tag{3}$$

where

| | |
|---|---|
| $SERa\ (t)$ | is the area specific emission rate at the time *t* expressed in µg/($m^2$ × h); |
| $C_{voc}\ (t)$ | is the concentration of total VOC in the chamber at time *t* expressed in µg/$m^3$; |
| $F_{cham}$ | is the air change flow expressed in $m^3$/h; |
| $A_{samp}$ | is the area of the sample expressed in $m^2$. |

### 8.3.3 Determination of the empirical parameters *a* and *b*

**Table 1 — Example illustrating the tabulation of all the total VOC concentration data**

| **Sample**<br>$i$ | **Sample time**<br>h<br>$t_i$ | **Total VOC**<br>µg/m³<br>$C_{\mathrm{voc(i)}}$ | **Log₁₀ (t)**<br>$Lt_i$ | **Log₁₀ (Total VOC)**<br>$Lc_i$ |
|---|---|---|---|---|
| 1 | 72 | | | |
| 2 | 240 | | | |
| … | | | | |
| $N$ | 672 | | | |

$i$ is the sample number.

$t_i$ is the sampling time of the *sample* $i$ expressed in hours.

$C_{voc(i)}$ is the total VOC concentration of the *sample* $i$ expressed in micrograms per cubic metre.

$Lt_i$ is the base 10 logarithm of the sampling time of the *sample* $i$ (considered as adimensional value; see NOTE below).

$Lc_i$ is the base 10 logarithm of the total VOC concentration of the *sample* $i$ (considered as adimensional value; see NOTE below).

$N$ is the number of data pairs available.

The logarithmic data pairs $Lt_i$ and $Lc_i$, when reported on an x-y plot, tend to dispose near a straight line.

Calculate the intercept and the slope of the least square fitting line of the logarithmic data pairs with Formula (4) and Formula (5):

$$q = \frac{\sum Lt_i^2 \cdot \sum Lc_i - \left(\sum Lt_i \cdot \sum (Lt_i \cdot Lc_i)\right)}{N \cdot \sum Lt_i^2 - \left(\sum Lt_i\right)^2} \qquad \left(\sum = \sum_{i=1}^{N}\right) \tag{4}$$

$$m = \frac{N \cdot \sum (Lt_i \cdot Lc_i) - \left(\sum Lt_i \cdot \sum Lc_i\right)}{N \cdot \sum Lt_i^2 - \left(\sum Lt_i\right)^2} \tag{5}$$

where

$q$ is the intercept of the fitting line (considered as adimensional value; see NOTE below);

$m$ is the slope of the fitting line (considered as adimensional value; see NOTE below);

$Lt_i$ is the base 10 logarithm of the sampling time of the *sample* $i$ (considered as adimensional value; see NOTE below);

$Lc_i$ is the base 10 logarithm of the total VOC concentration of the *sample* $i$ (considered as adimensional value; see NOTE below).

Alternatively, it is possible to use a least square linear fitting routine that can be found in most commercial statistical and data analysis software to assign the base 10 logarithm of the time (the $Lt_i$ values) to the independent variable (the x-axis) and the base 10 logarithm of the total VOC concentrations (the $Lc_i$ values) to the dependent variable (the y-axis).

Notice that, due to the decrease of the total VOC values (and the related logarithms) in time, the slope of the resulting fitting line shall be negative.

Calculate the $a$ and $b$ parameters with Formula (6) and Formula (7), using the intercept $q$ and the slope $m$ determined with the linear least square fitting:

$$a = 10^q \tag{6}$$

$$b = m \tag{7}$$

NOTE The variables and the parameters that appear in the empirical equation and in the argument of logarithms are considered as adimensionalized (divided by their measure unit):

— $C_{voc}$ [adimensional] = $C_{voc}$ / µg/m$^3$;

— $t$ [adimensional] = $t$ / $h$.

To simplify the formulae, this formal operation and the complementary one are considered implicit.

## 9 Test report

The test report shall include:

a) reference to this European Standard;

b) volume of the test chamber;

c) complete identification of the adhesive being tested, in particular the type, name, designation, manufacturer, lot number, date of supply, and mass of the test sample;

d) method of application of the adhesive and the coat weight according to 6.3;

e) loading factor;

f) test results expressed according to Clause 8;

g) modification of the procedure described in this European Standard and any circumstances which may have affected the results;

h) precise designation of the test laboratory and the test person;

i) date of test.

# Annex A
(normative)

## Carcinogenic and sensitizing substances

All the substances classified with at least one of the following risk indication:

a) H350, H350i, H340, H360F, H360D (CMR Cat.1A or Cat.1B),

b) H351, H341, H361D (CMR Cat. 2),

c) H334,

according to the Regulation (EC) No 1272/2008 [11] and further adaptations to technical progress shall be included in the review as described in 8.2.

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