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Lepila - Hitra metoda za merjenje emisijskih lastnosti lepil z malo topila ali brez njega po uporabi - 1. del: Splošni postopek

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

Klebstoffe - Kurzzeit-Verfahren zum Messen der Emissionseigenschaften von lösemittelarmen oder lösemittelfreien Klebstoffen nach der Applikation - Teil 1: Allgemeines Verfahren
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Adhésifs - Méthode de mesurage rapide des caractéristiques émissives des adhésifs à teneur faible ou nulle en solvant après application - Partie 1 : Mode opératoire général
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English Version

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Emissionseigenschaften von lösemittelfreien oder
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Allgemeines Verfahren

This European Standard was approved by CEN on 1 August 2006.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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Foreword

This document (EN 13999-1:2006) 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of an identical text or by endorsement, at the latest by February 2007, and conflicting national standards shall be withdrawn at the latest by February 2007.

This document supersedes ENV 13999-1:2001.

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

Safety statement

Persons using this document should be familiar with the principles of normal laboratory. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and ensure compliance with any regulatory conditions.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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 which includes all relevant and potentially hazardous substances.

The test shall:

- be reliable and reproducible;
- give results in a short time to be useful for decisions for development projects;
- verify that carcinogenic or sensitising volatile substances are absent;
- characterise 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.

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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. The adhesives shall be applicable at room temperature.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN 923:2005, *Adhesives — Terms and definitions*

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

EN ISO 15605, *Adhesives — Sampling (ISO 15605:2000)*

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)*

ENV 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*

ENV 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*

ENV 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*

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

ISO 16000-6:2004, *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/FID*

3 Terms and definitions

For the purposes of this European Standard the terms and definitions given in EN 923:2005, EN ISO 16000-9:2006, ISO 16000-6:2004 and the following apply.

3.1

specific cumulative emission (SCE)

total volatile organic compounds per unit of test area, emitted in a time interval between 24 h and 240 h after the start of the test

3.2

volatile organic compounds (VOC)

organic compounds whose boiling point ranges from (50 °C to 100 °C) to (240 °C to 260 °C) and generally has a saturation pressure of 25 °C greater than 10² kPa

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 ENV 13999-2.

Sampling and analysis of the volatile aldehydes shall be carried out as described in ENV 13999-3.

Sampling and analysis of the volatile isocyanates shall be carried out as described in ENV 13999-4.

The test includes:

- qualitative and quantitative determination of carcinogenic and sensitising substances in an early stage after the application of the adhesive sample;
- determination of the VOC specific emission rate at three points of time, a determination of specific cumulative VOC emission in the interval between 24 h and 240 h.

NOTE There are specific applications for which an emission test cell (as described in EN ISO 16000-10) may be easier to handle than the emission 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. But, as the air flow rate across the sample is higher than with the chamber method, the results may only be used with supporting correlation data.

5 Apparatus

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.

5.1 Support

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

5.2 Adhesive coating device

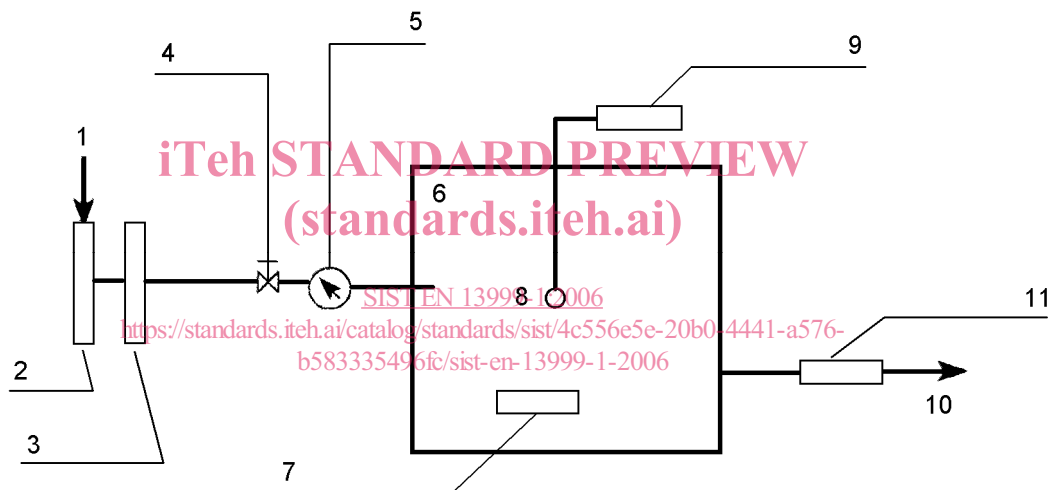
A device able to ensure a uniform coating of the adhesive to the support (5.1) shall be used.

5.3 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 m³, preferably from 0,1 m³ to 1 m³.

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 $1/1\,000$ of the chamber volume per minute at an overpressure of 1 000 Pa;
- air leakage in operating condition is less than 1% of the supply airflow 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

5.4 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 $\pm 3\%$. 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, not containing any volatile compounds at a level higher than the required test chamber background concentrations of 10 µg/m³ (total VOC) and 2 µg/m³ for any single component. The control of background concentration shall be carried out with the empty support (5.1) inside the test chamber.

NOTE A four-step filter is suggested:

- a) filtration membrane particle size (maximum 0,5 mg/m³ breakthrough);
- b) activated charcoal (maximum 0,003 mg/m³ breakthrough);
- c) high efficiency compressed air dryer (dew point -40 °C at least) ;
- d) high filtration grade (< 0,5 mg/m³ particles breakthrough at 21 °C).

5.5 Air sampling devices

The sampling devices as described in Parts 2, 3 and 4 of this European Standard 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 shall be smaller than the supply air flow to the emission test chamber.

NOTE Placing the whole apparatus in a room conditioned at (23 ± 2) °C is recommended.

5.6 Devices for desorption and analysis

Devices for desorption and analysis shall be used as described in Parts 2, 3 and 4 of this European Standard.

6 Adhesive sample

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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 ± 2) °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 m²/m³ ± 0,02 m²/m³. 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 (0) 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 using the spreading techniques and coat weights recommended by the manufacturer.

Weigh the plate again to $\pm 0,1$ g and adjust the adhesive weight to $\pm 5\%$ 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

Clean and condition the test chamber by flushing with clean air at the test temperature for at least 24 h and check for contamination before any test as described in EN ISO 16000-9.

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 ± 2 °C and ± 5 % RH. The supply air flow shall be adjusted to an air exchange rate of (0,5 \pm 0,025) times per h.

Unless otherwise specified in this 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:

24 h;

48 h;

240 h.

Analyse the VOC in the 24 h, the 48 h and the 240 h air samples. Take the 24 h air samples for determination of carcinogenic and sensitising substances.

Sample the test chamber air and determine the VOC in the time intervals (24 \pm 1) h, (48 \pm 1) h and (240 \pm 1) h after starting the test in compliance with Part 2 of this European Standard.

Sample from test chamber air and determine aldehydes in the time interval (26 \pm 1) h after starting the test in compliance with Part 3 of this European Standard.