SLOVENSKI STANDARD
SIST EN 13999-3:2007

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SIST ENV 13999-3:2003

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

Klebstoffe - Kurzzeit-Verfahren zum Messen der Emissionseigenschaften von lösemittelarmen oder lösemittelfreien Klebstoffen nach der Applikation - Teil 3: Bestimmung flüchtiger Aldehyde

Adhésifs - Méthodes de mesurage rapide des caractéristiques émissives des adhésifs a teneur faible ou nulle en solvants apres application - Partie 3: Dosage des aldéhydes volatiles

Ta slovenski standard je istoveten z: EN 13999-3:2007

ICS:

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EUROPEAN STANDARD

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EUROPÄISCHE NORM

EN 13999-3

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English Version

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This European Standard was approved by CEN on 8 March 2007.

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 CEN Management Centre 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 CEN Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This document (EN 13999-3:2007) 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of an identical text or by endorsement, at the latest by October 2007, and conflicting national standards shall be withdrawn at the latest by October 2007.

This document supersedes ENV 13999-3:2002 which has been editorially revised.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, 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.

SAFETY STATEMENT

Persons using this European Standard should be familiar with the normal laboratory practice, if applicable. This European Standard 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 health and safety practices and to ensure compliance with any regulatory conditions.

1 Scope

This European Standard specifies a procedure for the determination of volatile aldehydes (especially formaldehyde and acetaldehyde) and other carbonyl compounds in the exhaust air of an emission test chamber after application of a low-solvent or solvent-free adhesive as defined in EN 923.

The method is based on chemosorption of volatile carbonyl compounds with 2,4-dinitrophenylhydrazine (in the following: DNPH) impregnated silica tubes or cartridges with subsequent solvent desorption, clean-up and liquid chromatographic analysis. The method permits measurement of several aldehydes including formaldehyde, acetaldehyde, propionaldehyde, butyraldehyde, valeraldehyde, isovaleraldehyde, hexanal, benzaldehyde, 2,5-dimethylbenzaldehyde, o-tolualdehyde, m-tolualdehyde, p-tolualdehyde, crotonaldehyde in the concentration range of approximately 10 µg/m³ to 1 mg/m³ (see ISO 16000-3).

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 1232, *Workplace atmospheres — Pumps for personal sampling of chemical agents — Requirements and test methods*

EN 13999-1, *Adhesives — Short term method for measuring the emission properties of low-solvent or solvent-free adhesives after application — Part 1: General procedure*

EN ISO 16000-9, *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 16000-3, *Indoor air — Part 3: Determination of formaldehyde and other carbonyl compounds — Active sampling method*

3 Principle

A sufficient volume of test chamber air is drawn with an appropriate flow through silicagel tubes or cartridges impregnated with 2,4-dinitrophenylhydrazine (DNPH) reagent. Any organic aldehydes and other carbonyl compounds present will react to form non-volatile dinitrophenylhydrazones. Desorption is done with acetonitrile.

The resultant solution is concentrated and analysed by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection. Dinitrophenylhydrazone peaks from formaldehyde, acetaldehyde and other aldehydes are identified on the basis of both their respective retention times and their UV responses at more than one wavelength and/or by diode array detection (DAD) and comparison with a derivative product (where available) or standard. Quantification is done by comparison with a relevant aldehyde or a dinitrophenylhydrazone standard.

4 Reagents

4.1 Acetonitrile

Acetonitrile of HPLC grade.

4.2 Standards

Commercially available DNPH-aldehyde derivatives (or their solution(s) in acetonitrile). If the DNPH-aldehyde derivatives are prepared in the laboratory, the respective reagents described in ISO 16000-3 shall be used.

5 Apparatus

5.1 Sampler

Commercially available tubes or cartridges filled with DNPH coated silica. If the samplers are prepared in the laboratory the respective reagents described in ISO 16000-3 shall be used. The ratio of the silica gel bed diameter to bed length shall not exceed 1:1. The capacity of the tube or cartridge for formaldehyde shall be at least 75 μg and adsorption efficiency at least 95 % at the sampling rate applied.

5.2 Sampling pump

The pump shall fulfil the requirements of EN 1232 or equivalent.

5.3 Tubing

Tubing of appropriate diameter to ensure a leak-proof fit to both pump and sample tube. PTFE tubes shall be used upstream of the sorbent to avoid loss of substance due to reaction with tubing walls.

5.4 Flow meter calibrator

Bubble meter or other appropriate suitable device for gas flow calibration.

5.5 High performance liquid chromatograph (HPLC)

A typical apparatus for HPLC, with ultraviolet (UV) detector.

5.6 Emission test chamber

An emission test chamber as described in EN 13999-1 or EN ISO 16000-9 shall be used.

6 Procedure

6.1 Test chamber air sampling

Open the sealed tubes or remove the caps from the sampler and assemble the sampling line. Always use a sampler with a sampling section and a control section, or use two samplers in series with the downstream sampler serving as a control section. Prepare the test chamber sampling port as described in EN 13999-1 or in EN ISO 16000-9.

If the sampling flow rate is determined with a calibrator, start the pump, note and record the sampling flow rate.

Connect the sampler to the chamber sampling port, note and record the time the sampler was connected. Never use more than 50 % of the test chamber supply air flow rate as the combined sampling flow rates. At the end of the sampling period disconnect the sampler from the chamber sampling port, note and record the time of disconnection, repeat the sampling flow determination, turn off the pump.

Disconnect the sampler or the samplers from the sampling line and seal both ends using inert caps.

NOTE   The recommended sampling flow rate is in the range of 0,2 l/min. The recommended air volume is 24 l.

6.2   Storage of loaded samplers

Store each sampler so that it is isolated from other samples and from sources for volatile aldehydes, protected from light. If not analysed on the same day that the sample is collected, store the samplers at a temperature below 10 °C. The time between sampling and analysis shall be as short as possible and shall not exceed 14 days.

6.3   Blanks

Take at least one blank sample which is a sampler equivalent to the ones used for sampling. Subject the blank samples to the same handling procedure in the laboratory as the actual samples except for the actual period of sampling, i.e. repeat the sampling procedure up to the point of actual sample collection. Do not perform sampling but repeat normal post-sampling procedure for the sampling tubes. Mark, store and analyse blank samples in sequence with the actual samples. For blank control of the test chamber see EN 13999-1.

6.4   Analysis

6.4.1   Cleaning of glassware

Before use, clean all glassware to remove any residual grease or chemicals by soaking overnight in laboratory detergent solution and then rinsing thoroughly with water. Alternatively, use a laboratory washing machine.

6.4.2   Sample desorption (cartridges)

Desorb samples, blanks and standards by slowly passing a small amount of acetonitrile through the cartridges as described in ISO 16000-3.

6.4.3   Sample desorption (tubes)

Desorb samples, blanks and standards by separately emptying the content of the sections of the tube, adding a small amount of acetonitrile as described in ISO 16000-3 and letting it stand at room temperature in the dark for at least 30 min with occasional agitation.

6.4.4   Standard solutions

Prepare standard solutions in the analytical range of interest from commercially available DNPH-aldehyde derivatives (or their solution(s) in acetonitrile), or from self-produced DNPH-aldehyde derivatives as described in ISO 16000-3. Store all standard solutions in tightly capped containers in a refrigerator and keep protected from light. Allow them to equilibrate to room temperature before use. They should be replaced after four weeks.

6.4.5   Calibration of liquid chromatograph

Calibrate the system by injecting a known fixed volume (e.g. 25 µl) of at least five standard solutions covering the analytical range of interest into the liquid chromatograph using UV detection as described below. A standardised injection technique is required to obtain reproducible peak heights/areas. Prepare a calibration graph of UV response versus analyte concentration in the standard solutions as described in ISO 16000-3.

Once linear response (correlation coefficient of at least 0,999 for response versus concentration) has been documented, an intermediate concentration standard near the anticipated levels of component, but at least ten times the detection limit, should be chosen for daily calibration.

The day to day response of the analytical system for the various components shall be registered on a control card or in a corresponding electronic data system and should be within 20 %. If greater variability is observed the system shall be checked, re-calibrated and fresh standards shall be prepared.

6.4.6   Analysis of desorbed sample solutions

Inject a known fixed volume (25 µl) of the desorbed sample solution into the liquid chromatograph. Determine the UV response at the retention times being specific for the respective DNPH-aldehyde derivatives and read the concentration of the analyte in the sample from the calibration graph. Analyse the sample blank in the same way.

NOTE   A variety of chromatographic conditions can be used for the analysis of volatile aldehydes in solution. The choice will depend largely on the nature of interfering compounds, which may affect the chromatographic analysis. Typical conditions are as follows:

- column dimensions 250 mm length $\times$ 4,6 mm internal diameter, two columns in series;

- column packing C18;

- mobile phase acetonitrile / water, linear gradient;

- flow rate 1 ml/min;

- UV detector 360 nm and/or diode array detector.

7   Identification and calculation

7.1   Identification aldehydes

For identification of the aldehydes, analyse the samples with the UV detector operating at one wavelength and scan full UV spectra for all detected compounds. Alternatively, operation at two wavelengths may be used. A positive identification can be assumed if both the UV spectrum in the chromatogram and a standard spectrum of a DNPH-aldehyde derivative match to a high degree and if the retention time corresponds to the retention time of the DNPH-aldehyde derivative standard compound.

7.2   Concentration of analytes in the sampled air

Calculate the volume, V_S, in litres, of each air sample. Calculate the quantity q_{sample}, in µg, of any aldehyde in the sample tubes, by comparison with standard solutions as described in ISO 16000-3. Calculate the concentration C_m, in µg/m^3, of any aldehyde in the sampled chamber air correcting for blanks as follows:

$$C_m = (q_{sample} - q_{blank}) \times 1\ 000 / V_S \tag{1}$$

where

C_m is the concentration of analyte in the air sample, in µg/m³;

q_{sample} is the total quantity of analyte in the sample (all sections/tubes/cartridges in series), in µg;

q_{blank} is the quantity of analyte in the blank, in µg;

V_S is the volume of air sampled, in litres.