



National Standards Authority of Ireland

IRISH STANDARD

I.S. ENV 13999-4:2002

ICS 83.180

**ADHESIVES - SHORT TERM METHOD FOR
MEASURING THE EMISSION PROPERTIES
OF LOW-SOLVENT OR SOLVENT-FREE
ADHESIVES AFTER APPLICATION - PART 4:
DETERMINATION OF VOLATILE
DIISOCYANATES**

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Údarás um Chaighdeán Náisiúnta na hÉireann

ICS 83.180

English version

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

Adhésifs - Méthode de courte durée de mesurage des propriétés d'émission des adhésifs à faible teneur en solvants ou exempts de solvants après application - Partie 4: Dosage des diisocyanates volatils

Klebstoffe - Kurzzeit-Verfahren zum Messen der Emissionseigenschaften von lösemittelfreien oder lösemittelfreien Klebstoffen nach der Applikation - Teil 4: Bestimmung flüchtiger Diisocyanate

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Foreword

This document ENV 13999-4:2002 has been prepared by Technical Committee CEN/TC 193, "Adhesives", the secretariat of which is held by AENOR.

This document includes a Bibliography.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this European Prestandard : Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

ENV 13999-4:2002 (E)

1 Scope

This European Prestandard specifies a procedure for the determination of volatile organic isocyanates in the exhaust air of an emission test chamber after application of an adhesive.

The method is based on chemisorption of volatile isocyanates with 1-(2-methoxyphenyl)piperazine (in the following: 1-2MP) impregnated filters with subsequent desorption and liquid chromatographic analysis. The method permits measurement of a wide range of organic compounds containing isocyanate functional groups (NCO), including isocyanate monomers. For testing of adhesives emissions mainly toluene diisocyanate (TDI) and methylene bis (4-phenyl isocyanate) (4,4-diisocyanato-diphenylmethane, MDI) are of concern. The method as described in this Prestandard can be used for other isocyanates too, such as isophorone diisocyanate (IPDI) and 1,6-hexamethylene diisocyanate (HDI) - see ISO 16702. Isocyanate oligomers or prepolymers are not volatile enough to be detected in emission test chambers at room temperature.

2 Normative references

This European Prestandard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

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

ENV 13419-1, *Building products - Determination of the emission of volatile organic compounds - Part 1: Emission test chamber method.*

EN ISO 16017-1, *Indoor, ambient and workplace air - Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography - Part 1: Pumped sampling (ISO 16017-1:2000).*

prEN ISO 16017-2, *Indoor, ambient and workplace air - Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography - Part 2: Diffusive sampling (ISO/DIS 16017-2:1999).*

ISO 16702, *Workplace air quality - Determination of total isocyanate groups in air using 2-(1-methoxyphenyl) piperazine and liquid chromatography.*

3 Principle

An emission test chamber as described in ENV 13999-1 or ENV 13419-1 shall be used.

A sufficient volume of test chamber air is drawn with an appropriate flow through a glass fibre filter impregnated with 1-(2-methoxyphenyl)piperazine (1-2MP) reagent. Any volatile isocyanates present will react and form non-volatile isocyanate urea derivatives. Desorption is done with acetic anhydride and a 1-2MP solution in toluene. The resultant solution is concentrated and analysed by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection. Isocyanate-derived peaks are identified on the basis of their UV responses at more than one wavelength and/or by diode array detection (DAD) and comparison with a derivative standard. Quantification is by comparison with the relevant isocyanate monomer standard.

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