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SIST EN 1242:2013

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English Version

Adhesives - Determination of isocyanate content

Adhésifs - Détermination de la teneur en isocyanate

Klebstoffe - Bestimmung des Isocyanatgehaltes

This European Standard was approved by CEN on 13 January 2013.

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-CENELEC 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-CENELEC 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

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 1242:2013) 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of an identical text or by endorsement, at the latest by September 2013, and conflicting national standards shall be withdrawn at the latest by September 2013.

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 1242:2005.

The main technical significant change is:

- Inclusion of ethanol in the list of reagents (Clause 5)

SAFETY PRECAUTIONS — Persons using this document should be familiar with the normal laboratory practice, if applicable. 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 to ensure compliance with any regulatory conditions.

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

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1 Scope

This European Standard specifies a method for the determination of the isocyanate content of adhesives, adhesive components and their basic constituents.

It is not applicable to products containing blocked isocyanate groups which can be liberated by the reagents used in this test method.

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 ISO 385, *Laboratory glassware — Burettes (ISO 385)*

EN ISO 648, *Laboratory glassware — Single-volume pipettes (ISO 648)*

EN ISO 1042, *Laboratory glassware — One-mark volumetric flasks (ISO 1042)*

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

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3 Terms and definitions

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For the purposes of this document, the terms and definitions given in EN 923:2005+A1:2008 and the following apply.

3.1 isocyanate content

percentage (mass/mass) of isocyanate groups (-NCO) in the product under test

4 Principle

A weighed quantity of product is converted in presence of an excess of dibutyl amine dissolved in toluene or another solvent forming urethanes. The unreacted dibutyl amine is backtitrated with hydrochloric acid, in the presence of a colour indicator or potentiometrically.

5 Reagents

5.1 Dibutyl amine, analytical grade [or freshly distilled].

NOTE As "dibutyl amine" either di-*n*-butylamine or di-isobutylamine can be used.

5.2 Toluene, dry, analytical grade.

NOTE Depending on the nature of the isocyanate other analytical grade solvents (e.g. dioxane, xylene, butane-2-one, ethyl acetate, chlorobenzene, dimethylformamide) or solvent mixtures can be used provided the product is dissolved completely without chemical reaction and it is ensured that equivalent results are obtained. Urethane prepolymers can be dissolved in dry toluene or xylene. When the specified conversion with dibutyl amine is completed propan-2-ol can be added before titration.

5.3 Acetone, dry, analytical grade.

5.4 Dibutyl amine 1 M concentration solution

Mix thoroughly 129 g (1 mol) of dibutyl amine (see 5.1) with dry toluene (see 5.2) and dilute with toluene to 1 000 ml in a measuring flask and store in a brown, glass stoppered bottle.

5.5 Dibutyl amine 0,2 M concentration solution

Mix thoroughly 25,8 g (0,2 mol) of dibutyl amine (see 5.1) with dry toluene (see 5.2) and dilute with toluene to 1 000 ml in a measuring flask and store in a brown, glass stoppered bottle.

The solutions (see 5.4 and 5.5) should be kept airtight and excluded from light. As the amine content decreases after several days of storage a blank test should be performed before each determination.

5.6 Bromophenol blue indicator solution: Dissolve 0,1 g of bromophenol blue (Reag.Ph.Eur. Indicator grade) and dilute with ethanol (see 5.9), analytical grade, to 100 ml.

NOTE Reag.Ph.Eur. = Reagent Pharmacopoeia European.

5.7 Methanol, analytical grade.

5.8 Hydrochloric acid 1,0 M/0,2 M aqueous solution, analytical grade.

5.9 Ethanol, analytical grade.

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6 Apparatus

6.1 General

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All volumetric glassware shall be class A, in accordance with EN ISO 385, EN ISO 648 or EN ISO 1042 as appropriate.

6.2 Analytical balance, with scale divisions of 0,1 mg.

6.3 Conical flasks, capacity 250 ml, fitted with ground glass stoppers.

6.4 Pipettes, one mark, capacity 25 ml, 50 ml or 100 ml.

6.5 Microburettes, capacity 25 ml, graduated in 0,01 ml divisions.

6.6 Device for potentiometric titration, fitted with glass electrode and a reference electrode.

6.7 Measuring, brown flasks, capacity 1 000 ml.

7 Procedure

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

Weigh, to the nearest 1 mg, in one of the flasks (see 6.3) a test portion, the mass of which depends on the expected isocyanate content.

For testing isocyanates (e.g. TDI, MDI or HDI) with high isocyanate contents weigh a test portion of 2 g.

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In testing isocyanates with low isocyanate contents (e.g. urethane prepolymers) estimate the mass of the test portion with the empirical formula:

$$\text{test portion in grams} = \frac{8,4}{\text{isocyanate content}} \quad (1)$$

and use for reacting instead of a 1,0 M concentration of dibutyl amine solution (see 5.4) a 0,2 M concentration of dibutyl amine solution (see 5.5) and for backtitration 0,2 M concentration of hydrochloric acid solution (see 5.8). The approximate isocyanate content is unknown, preliminary tests should be made.

After weighing close the flask with a ground stopper to prevent the isocyanate from evaporating and reacting with atmospheric moisture.

Add 20 ml of toluene (see 5.2) to the test portion and dissolve the isocyanate completely. If a polymer is insoluble, add 10 ml of dry, analytical grade acetone (see 5.3). Solutions may be aided by warming on a hotplate.

Using a pipette (see 6.4), add 25 ml of 1,0 M concentration of dibutyl amine solution (see 5.4) or 25 ml of 0,2 M concentration of dibutyl amine solution (see 5.5) depending on the isocyanate content of the product.

Fill up with dry toluene (see 5.2) to a total volume of 50 ml.

Swirl the flask (see 6.3) to start the reaction with the isocyanate. The reaction is complete when the liquid in the flask becomes clear, which takes about 2 min.

After the addition of 2 to 3 drops of bromophenol blue indicator solution (see 5.6) dilute the contents of the flask by slowly adding 100 ml of methanol (see 5.7) while the flask is being swirled.

Back-titrate the excess of dibutyl amine with 1,0 M (or 0,2 M) hydrochloric acid solution (see 5.8), which is added by the microburette (see 6.5). At the end point the colour of the indicator changes from blue to yellow via an intermediate greenish colour. Record the volume (V_1) used.

NOTE Optionally potentiometric titration can be used.

Repeat the procedure without a test portion as a blank test and record the volume (V_2) used.

8 Calculation and expression of the results

8.1 Calculation

Calculate, for each test, the isocyanate content (% NCO) by the following formula:

$$\% \text{ NCO} = 4,2 \times M \times \frac{(V_2 - V_1)}{m} \quad (2)$$

where:

V_1 is the volume, in millilitres, of hydrochloric acid (see 5.8) used for the test portion;

V_2 is the volume, in millilitres, of hydrochloric acid (see 5.8) used in the blank test;

M is the molarity of hydrochloric acid (see 5.8);

m is the mass, in grams, of the test portion.