BS EN 1242:2013

BSI Standards Publication

Adhesives — Determination of isocyanate content

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National foreword

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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 - Determination of isocyanate content

Adhésifs - Détermination de la teneur en isocyanate

Klebstoffe - Bestimmung des Isocyanatgehaltes

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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of an identical text or by endorsement, at the latest by September 2013, and conflicting national standards shall be withdrawn at the latest by September 2013.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)

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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)*

3 Terms and definitions

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.

6 Apparatus

6.1 General

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.

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:

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

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.

8.2 Expression of result

Take as result the mean value in at least two determinations, rounded to the next whole number.

9 Precision

The maximum deviation of every single value from the mean value should comply with the figures of Table 1.

Table 1 — Precision

Isocyanate content (%)	**Maximum deviation of every single value from the mean value** (relative)
< 10	10 %
10 to 20	5 %
20 to 30	4 %
30 to 40	3 %
40 to 50	2 %

10 Test report

The test report shall include:

a) reference to this European Standard;

b) complete identification of the sample;

c) identification of the solvent used, if not toluene;

d) mass in grams of the test portion and the molarity of the dibutyl amine solution used;

e) the test results, the individual values and their mean;

f) any special features noted during the determination;

g) any operations not specified in this European Standard, or the document to which reference is made, and all incidents that may have affected the results;

h) date of test.

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