



**SLOVENSKI STANDARD**  
**SIST EN 1242:1999**  
**01-maj-1999**

---

Adhézifs - Détermination de la teneur en isocyanate

Adhesives - Determination of isocyanate content

Klebstoffe - Bestimmung des Isocyanatgehaltes

Adhésifs - Détermination de la teneur en isocyanate

**Ta slovenski standard je istoveten z: EN 1242:1998**

[SIST EN 1242:1999](https://standards.iteh.ai/catalog/standards/sist/fecabcd0-dc01-4d7c-88b5-b78fe2870b86/sist-en-1242-1999)

<https://standards.iteh.ai/catalog/standards/sist/fecabcd0-dc01-4d7c-88b5-b78fe2870b86/sist-en-1242-1999>

**ICS:**

83.180

Lepila

Adhesives

**SIST EN 1242:1999**

**en**

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

SIST EN 1242:1999

<https://standards.iteh.ai/catalog/standards/sist/fecabcd0-dc01-4d7c-88b5-b78fe2870b86/sist-en-1242-1999>

EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

EN 1242

February 1998

ICS 83.180

Descriptors: adhesives, components, chemical tests, determination of content, isocyanates, procedure

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 2 January 1998.

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.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

**iTeh STANDARD PREVIEW**  
(standards.iteh.ai)

SIST EN 1242:1999  
<https://standards.iteh.ai/catalog/standards/sist/fcabc0-dc01-4d7c-88b5-b78fe2870b86/sist-en-1242-1999>



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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Page 2  
EN 1242:1998

**Contents**

**Page**

Foreword		3
1	Scope	4
2	Normative references	4
3	Definitions	4
4	Principle	4
5	Safety	5
6	Reagents	5
7	Apparatus	6
8	Procedure	6
9	Calculation and expression of results	7
10	Precision	8
11	Test report	8

ITeK STANDARD PREVIEW  
(standards.iteh.ai)

SIST EN 1242:1999

<https://standards.iteh.ai/catalog/standards/sist/fecabcd0-dc01-4d7c-88b5-b78fe2870686/sist-en-1242-1999>

REPUBLIC OF ITALY  
MINISTERO DELLE ATTIVITÀ ECONOMICHE E DEL SUD  
DIPARTIMENTO REGIONALE DEL SUD  
SISTEMA REGIONALE DI STANDARDIZZAZIONE  
SISTEMA REGIONALE DI STANDARDIZZAZIONE  
SISTEMA REGIONALE DI STANDARDIZZAZIONE



**Foreword**

This European Standard 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 August 1998, and conflicting national standards shall be withdrawn at the latest by August 1998.

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

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

SIST EN 1242:1999

<https://standards.iteh.ai/catalog/standards/sist/fecabcd0-dc01-4d7c-88b5-b78fe2870b86/sist-en-1242-1999>

Page 4  
EN 1242:1998

## 1 Scope

This standard specifies a method for the determination of the isocyanate content of adhesives, adhesive components, their basic constituents surface protection systems, and related products containing free isocyanate groups. This method can also be used to determine the hydroxyl value and/or the hydroxyl content of surface protection systems of concrete.

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

This European Standard 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 Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

(standards.iteh.ai)

EN 923	Adhesives - Terms and Definitions
EN 1066	Adhesives - Sampling
EN 1067	Adhesives - Examination and preparation of samples for testing
EN 21512	Paints and varnishes - Sampling of products in liquid or paste form (ISO 1512:1991)
ISO 385-1	Laboratory glassware - Burettes - Part 1 : General requirements
ISO 648	Laboratory glassware - One-mark pipettes
ISO 1042	Laboratory glassware - One-mark volumetric flasks

## 3 Definition

For the purposes of this standard, the definitions in accordance with EN 923 and the following definition apply:

**isocyanate content:** The 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 an other solvent forming urethanes. The unreacted dibutyl amine is backtitrated

with hydrochloric acid, in the presence of a colour indicator or potentiometrically.

## 5 Safety

Persons using this standard shall be familiar with normal laboratory practice.

This standard does not purport to address all 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 European and national regulatory conditions.

## 6 Reagents

6.1 Dibutyl amine, analytical grade [or freshly distilled];

6.2 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.

6.3 Acetone, dry, analytical grade;

6.4 1 mole concentration of Dibutyl amine solution.

Mix thoroughly 129 g (1 mol) of dibutyl amine (see 6.1) with dry toluene (see 6.2) and dilute to 1000 ml in a brown, glass stoppered bottle.

6.5 0,2 mole concentration of Dibutyl amine solution

Mix thoroughly 25,8 g (0,2 mol) of dibutyl amine (see 6.1) with dry toluene (see 6.2) and dilute to 1000 ml in a brown, glass stoppered bottle.

NOTE: The solutions (see 6.4 and 6.5) 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.

6.6 Bromophenol blue (mass concentration  $\beta = 0,1 \%$ ) indicator solution: Dissolve 0,1 g of bromophenol blue and dilute with ethanol, analytical grade, to 100 ml;

6.7 Methanol, analytical grade;

Page 6  
EN 1242:1998

6.8 0,2 mole concentration/or 1 mole concentration aqueous hydrochloric acid, analytical grade.

## 7 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042 as appropriate.

7.1 Analytical balance, with scale divisions of 0,1 mg;

7.2 Conical flasks, capacity 250 ml, fitted with ground glass stoppers;

7.3 Pipettes, one mark, capacity 25 ml, 50 ml or 100 ml;

7.4 Microburettes, capacity 25 ml, graduated in 0,01 ml divisions;

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

## 8 Procedure iTeh STANDARD PREVIEW

Take a sample of the product to be tested in accordance with EN 1066. Examine and prepare this sample for testing in accordance with EN 1067. [SIST EN 1242:1999](https://standards.iteh.ai/catalog/standards/sist/fcabc0-dc01-4d7c-88b5-b70e287186/sist-en-1242-1999)

Weigh, to the nearest 1 mg, in one of the flasks (see 7.2) containing 25 ml of dry toluene (see 6.2) 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 2g.

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

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

and use for reacting instead of a 1 mole concentration of dibutyl amine solution (see 6.4) a 0,2 mole concentration of dibutyl amine solution (see 6.5) and for backtitration 0,2 mole concentration of hydrochloric acid solution (see 6.8).

NOTE 1: If 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 25 ml of toluene (see 6.2) to the test portion and dissolve the isocyanate completely. If a polymer is insoluble, add 10 ml of dry, analytical grade acetone (see 6.3).

NOTE 2: Solution may be aided by warming on a hotplate.

Using a pipette (see 7.3), add 25 ml of 1 mole concentration of dibutyl amine solution (see 6.4) or 25 ml of 0,2 mole concentration of dibutyl amine solution (see 6.5) depending on the isocyanate content of the product.

Swirl the flask (see 7.2) 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 1 % ethanolic bromophenol blue solution (see 6.6) dilute the contents of the flask by slowly adding 100 ml of methanol (see 6.7) while the flask is being swirled.

Back-titrate the excess of dibutyl amine with 1,0M (or 0,2M) aqueous hydrochloric acid (see 6.8), which is added by the microburette (see 7.4). At the endpoint the colour of the indicator changes from blue to yellow via an intermediate greenish colour. Record the volume ( $V_1$ ) used.

NOTE 3: Optionally potentiometric titration can be used.

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

## 9 Calculation and expression of the results

### 9.1 Calculation

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

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

where:

$V_1$  is the volume, in millilitres, of hydrochloric acid (see 6.8) used for the test portion;