BRITISH STANDARD

BS EN ISO
11909:2007

# Binders for paints and varnishes — Polyisocyanate resins— General methods of test

The European Standard EN ISO 11909:2007 has the status of a British Standard

ICS 87.060.20



BSI
British Standards

# National foreword

This British Standard was published by BSI. It is the UK implementation of EN ISO 11909:2007. It supersedes BS EN ISO 11909:1998 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee STI/3, Paints, media and related products.

A list of organizations represented on STI/3 can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

**Compliance with a British Standard cannot confer immunity from legal obligations.**

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 March 2007



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EUROPEAN STANDARD

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# EN ISO 11909

February 2007

ICS 87.060.20

Supersedes EN ISO 11909:1998

English Version

# Binders for paints and varnishes - Polyisocyanate resins - General methods of test (ISO 11909:2007)

Liants pour peintures et vernis - Résines de polyisocyanate - Méthodes générales d'essai (ISO 11909:2007)

Bindemittel für Beschichtungsstoffe - Isocyanatharze - Allgemeine Prüfverfahren (ISO 11909:2007)

This European Standard was approved by CEN on 6 February 2007.

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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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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN ISO 11909:2007: E

## Foreword

This document (EN ISO 11909:2007) has been prepared by Technical Committee ISO/TC 35 "Paints and varnishes" in collaboration with Technical Committee CEN/TC 139 "Paints and varnishes", the secretariat of which is held by DIN.

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 2007, and conflicting national standards shall be withdrawn at the latest by August 2007.

This document supersedes EN ISO 11909:1998.

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

### Endorsement notice

The text of ISO 11909:2007 has been approved by CEN as EN ISO 11909:2007 without any modifications.

INTERNATIONAL STANDARD

**ISO 11909**

Second edition
2007-02-01

# Binders for paints and varnishes — Polyisocyanate resins — General methods of test

*Liants pour peintures et vernis — Résines de polyisocyanate — Méthodes générales d'essai*

Reference number
ISO 11909:2007(E)

# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

ISO 11909 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*, in collaboration with CEN Technical Committee CEN/TC 139, *Paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 11909:1996), which has been editorially revised and the normative references updated.

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# Binders for paints and varnishes — Polyisocyanate resins — General methods of test

## 1 Scope

This International Standard details general test methods for polyisocyanate resins and solutions of polyisocyanate resins intended for use as binders in paints, varnishes and related products.

## 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.

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — One-mark pipettes*

ISO 1523, *Determination of flash point — Closed cup equilibrium method*

ISO 2811-1, *Paints and varnishes — Determination of density — Part 1: Pyknometer method*

ISO 2811-2, *Paints and varnishes — Determination of density — Part 2: Immersed body (plummet) method*

ISO 2811-3, *Paints and varnishes — Determination of density — Part 3: Oscillation method*

ISO 2811-4, *Paints and varnishes — Determination of density — Part 4: Pressure cup method*

ISO 3219, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate*

ISO 3251, *Paints, varnishes and plastics — Determination of non-volatile-matter content*

ISO 3679, *Determination of flash point — Rapid equilibrium closed cup method*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 4630-1, *Clear liquids — Estimation of colour by the Gardner colour scale — Part 1: Visual method*

ISO 4630-2, *Clear liquids — Estimation of colour by the Gardner colour scale — Part 2: Spectrophotometric method*

ISO 6271-1, *Clear liquids — Estimation of colour by the platinum-cobalt scale — Part 1: Visual method*

ISO 6271-2, *Clear liquids — Estimation of colour by the platinum-cobalt scale — Part 2*: *Spectrophotometric method*

ISO 10283, *Binders for paints and varnishes — Determination of monomeric diisocyanates in polyisocyanate resins*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

**3.1**
**isocyanate resin**
synthetic resin, containing free or blocked isocyanate groups, based on aromatic, aliphatic or cyclo-aliphatic isocyanates

[ISO 4618:2006]

## 4 Properties and test methods

Unless otherwise agreed, the properties to be measured and the test methods to be used shall be as given in Table 1.

**Table 1 — Properties and test methods**

| Property | Test method |
|---|---|
| Colour | ISO 6271-1 or ISO 6271-2 |
| | ISO 4630-1 or ISO 4630-2 |
| Viscosity | ISO 3219 |
| Non-volatile matter | ISO 3251, together with Table 2 below |
| Flashpoint | ISO 1523 or ISO 3679 |
| Density | ISO 2811-1 to 2811-4 |
| Isocyanate content | Annex A of this International Standard |
| Monomeric diisocyanate content | ISO 10283 |

**Table 2 — Test conditions for the determination of non-volatile matter**

| Resin basis [a] | Period of heating<br>h | Test temperature [b]<br>°C |
|---|---|---|
| HDI biuret | 1 | 80 |
| HDI cyanurate | 1 | 105 |
| TDI and MDI polyisocyanates, adducts and prepolymers | 1 | 125 |
| IPDI polyisocyanates | 1 | 150 |

[a] HDI = Hexamethylene diisocyanate
TDI = Tolylene diisocyanate
MDI = Diphenyl-4-methane-4,4′-diisocyanate
IPDI = Isophorone diisocyanate

[b] For binders dissolved in highly volatile solvents, a lower temperature may be used.

# Annex A
(normative)

# Determination of isocyanate content (percentage by mass of isocyanate groups)

## A.1 Principle

The polyisocyanate resin is reacted with excess dibutylamine. The excess dibutylamine is then titrated with hydrochloric acid, either using bromophenol blue as the indicator or potentiometrically.

## A.2 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water of at least grade 3 purity as defined in ISO 3696.

**A.2.1 Dibutylamine**, solutions containing about 2 mol/l and about 0,2 mol/l, respectively.

To prepare the approximately 2 mol/l solution, dissolve 65 g of water-free distilled dibutylamine (boiling point 157 °C to 162 °C at 1,033 kPa) in toluene (A.2.2) in a 250 ml one-mark volumetric flask, make up to the mark with the same toluene and mix well. Standardize this solution by titrating a 20 ml portion with 1 mol/l hydrochloric acid (A.2.3).

Prepare the approximately 0,2 mol/l solution in analogous fashion, starting with 6,5 g of dibutylamine. Standardize this solution by titrating a 20 ml portion with 0,1 mol/l hydrochloric acid (A.2.3).

**A.2.2 Toluene**, previously dried over calcium chloride and filtered.

**A.2.3 Hydrochloric acid**, $c$(HCl) = 1 mol/l or 0,1 mol/l.

**A.2.4 Ethanol**, water-free.

**A.2.5 Bromophenol blue**, solution.

Triturate 1 g of bromophenol blue in a mortar with 1,5 ml of sodium hydroxide solution, $c$(NaOH) = 1 mol/l, and dissolve in a mixture of 20 ml of ethanol (A.2.4) and 10 ml of water.

## A.3 Apparatus

Ordinary laboratory apparatus and glassware, complying with the requirements of ISO 385 and ISO 648, together with the following:

**A.3.1 Conical flasks**, capacity 250 ml and 500 ml, with ground-glass stoppers.

**A.3.2 Potentionmetric titration apparatus**, fitted with a glass electrode and a reference electrode (for use with highly coloured resins — see Clause A.5).

## A.4 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

## A.5 Procedure

Carry out the determination in duplicate.

By reference to Table A.1, select the appropriate mass of test portion. If the approximate isocyanate content is not known, carry out a preliminary determination using a test portion of 3,5 g.

Weigh, to the nearest 1 mg (or 0,1 mg — see below), the appropriate mass of test portion into a 500 ml conical flask and dissolve it in 25 ml of toluene (A.2.2), if necessary with slight heating. After cooling to room temperature, pipette 20 ml of the appropriate dibutylamine solution (A.2.1) into the flask. Close the flask and allow to stand for 15 min, swirling occasionally. Dilute with 150 ml of ethanol (A.2.4), add a few drops of bromophenol blue solution (A.2.5) and titrate with the appropriate hydrochloric acid (A.2.3) until the colour changes to yellow. If separation occurs during the titration, add further ethanol.

**Table A.1 — Mass of test portion and permitted difference between results**

| **Isocyanate content**<br>% (by mass) | **Maximum mass of test portion**<br>g | **Permitted difference between individual values and mean value**<br>% (absolute) |
|---|---|---|
| below 1 | 25 | 0,15 |
| 1 to 10 | 12 | |
| above 10 to 20 | 6 | |
| above 20 to 25 | 5 | |
| above 25 to 30 | 4 | 0,2 |
| above 30 to 40 | 3,5 | |
| above 40 to 50 | 3 | |

If 0,1 mol/l hydrochloric acid is used, the test portion shall be weighed to the nearest 0,1 mg, its mass shall be about one-tenth that in Table A.1 and the 0,2 ml/l dibutylamine solution shall be used.

In the case of highly coloured resins, titrate potentiometrically.

## A.6 Expression of results

Calculate the isocyanate content, IC, expressed as a percentage by mass, using the following equation:

$$\mathrm{IC} = \frac{(V_1 - V_2) \times c}{m} \times 4,2$$

where

$V_1$ is the volume, in millilitres, of hydrochloric acid required for the standardization of the dibutylamine solution;

$V_2$ is the volume, in millilitres, of hydrochloric acid required for the determination;

$c$ is the actual concentration, in moles per litre, of the hydrochloric acid used;

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

## A.7 Precision

NOTE The precision data were obtained with methanol as solvent.

The repeatability $r$ and the reproducibility $R$ depend on the product tested.

| | **Repeatability** | **Reproducibility** |
|---|---|---|
| | $r$ | $R$ |
| IPDI trimer<br>NCO content about 12 % (by mass) | 0,11 | 0,34 |
| HDI biuret<br>NCO content about 16 % (by mass) | 0,36 | 0,50 |
| TDI adduct<br>NCO content about 13 % (by mass) | 0,19 | 0,27 |
| MDI prepolymer<br>NCO content about 7 % (by mass) | 0,55 | 0,67 |

## A.8 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this International Standard (ISO 11909:2007);

c) the result of the test, as indicated in Clause A.6;

d) any deviation from the test method specified;

e) the date of the test.

# Bibliography

[1] ISO 4618:2006, *Paints and varnishes — Terms and definitions*

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