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STANDARD

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**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 11909:1996(E)

Foreword

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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.

International Standard ISO 11909 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

Annex A forms an integral part of this International Standard.

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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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 842:1984, *Raw materials for paints and varnishes — Sampling*.

ISO 1523:1983, *Paints, varnishes, petroleum and related products — Determination of flashpoint — Closed cup equilibrium method*.

ISO 2811:1974, *Paints and varnishes — Determination of density*.

ISO 3219:1993, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate*.

ISO 3251:1993, *Paints and varnishes — Determination of non-volatile matter of paints, varnishes and binders for paints and varnishes*.

ISO 3679:1983, *Paints, varnishes, petroleum and related products — Determination of flashpoint — Rapid equilibrium method*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 4630:1981, *Binders for paints and varnishes — Estimation of colour of clear liquids by the Gardner colour scale*.

ISO 6271:1981, *Clear liquids — Estimation of colour by the platinum-cobalt scale*.

ISO 10283:—¹⁾, *Binders for paints and varnishes — Determination of monomeric diisocyanates in polyisocyanate resins*.

3 Definition

For the purposes of this International Standard, the following definition applies.

1) To be published.

3.1 polyisocyanate resin: Synthetic resin containing reactive isocyanate groups and based on aromatic, aliphatic or cycloaliphatic isocyanates.

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 (Platinum-cobalt scale) or ISO 4630 (Gardner colour scale)
Viscosity	ISO 3219
Non-volatile matter	ISO 3251, together with table 2 below
Flashpoint	ISO 1523 or ISO 3679
Density	ISO 2811
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 ¹⁾	Period of heating	Test temperature ²⁾
	h	°C
HDI biuret	1	80
HDI cyanurate	1	105
TDI and MDI polyisocyanates, adducts and prepolymers	1	125
IPDI polyisocyanates	1	150

1) HDI = Hexamethylene diisocyanate
 TDI = Toluene diisocyanate
 MDI = Diphenyl-4-methane-4,4'-diisocyanate
 IPDI = Isophorone diisocyanate

2) 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 (see 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 (see A.2.3).

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

A.2.3 Hydrochloric acid, $c(\text{HCl}) = 1 \text{ mol/l}$ 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, $c(\text{NaOH}) = 1 \text{ mol/l}$, 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 385-1 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 Potentiometric titration apparatus, fitted with a glass electrode and a reference electrode (for use with highly coloured resins — see A.5).

A.4 Sampling

Take a representative sample of the product to be tested, as described in ISO 842.

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 % (m/m)	Maximum mass of test portion g	Permitted difference between individual values and mean value % (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 mol/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:

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

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 1 The precision data were obtained with methanol as solvent.

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

	Repeatability (r)	Reproducibility (R)
HDI trimer NCO content about 12 % (m/m)	0,11	0,34
HDI biuret NCO content about 16 % (m/m)	0,36	0,50
TDI adduct NCO content about 13 % (m/m)	0,19	0,27
MDI prepolymer NCO content about 7 % (m/m)	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);
- 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.