

---

---

**Binders for paints and varnishes —  
Determination of monomeric  
diisocyanates in isocyanate resins**

*Liants pour peintures et vernis — Détermination des diisocyanates  
monomères dans les résines isocyanates*

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

[ISO 10283:2007](https://standards.iteh.ai/catalog/standards/sist/5e7d0fa8-8a77-4cfe-853f-1f460ab45d03/iso-10283-2007)

<https://standards.iteh.ai/catalog/standards/sist/5e7d0fa8-8a77-4cfe-853f-1f460ab45d03/iso-10283-2007>



**PDF disclaimer**

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

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

ISO 10283:2007

<https://standards.iteh.ai/catalog/standards/sist/5e7d0fa8-8a77-4cfe-853f-1f460ab45d03/iso-10283-2007>



**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2007

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

Page

Foreword.....	iv
Introduction .....	v
1 Scope .....	1
2 Normative references .....	1
3 Terms and definitions.....	1
4 Principle .....	2
5 Reagents .....	2
6 Apparatus .....	2
7 Sampling.....	3
8 Procedure .....	3
8.1 Operating conditions.....	3
8.2 Column conditioning.....	8
8.3 Gas-chromatographic determination.....	8
9 Expression of results .....	9
9.1 Determination of calibration factor.....	9
9.2 Calculation of the monomeric diisocyanate content .....	9
10 Precision.....	9
10.1 Repeatability ( $r$ ).....	9
10.2 Reproducibility ( $R$ ).....	10
11 Test report .....	10

## 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10283 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 10, *Test methods for binders for paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 10283:1997), in which the normative references clause has been updated.

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

ISO 10283:2007  
<https://standards.iteh.ai/catalog/standards/sist/5e7d0fa8-8a77-4cfe-853f-1f460ab45d03/iso-10283-2007>

## Introduction

It is well-known fact that, due to the production methods used, all the commercial isocyanate resins named in this standard contain a certain amount of volatile monomeric isocyanates. This amount is generally less than 0,5 % relative to the resin as supplied. In view of the regulations relating to the handling of hazardous substances, it has become a matter of special concern that a generally accepted and applicable method of determination should be available. This standard is not intended to present a method suitable for the analytical determination of volatile isocyanates in any form and in any quantity. The standard specifies a method confined to determining the amounts of volatile isocyanates which occur in practice in isocyanate resins, namely about 0,1 % to 0,4 %. A further objective of the standard was to develop a method for determining with adequate accuracy as many as possible of the monomeric isocyanates which occur in isocyanate resins. It detects the principle isocyanates, namely toluene diisocyanate (TDI), hexamethylene diisocyanate (HDI), diphenylmethane diisocyanate (MDI) and isophorone diisocyanate (IPDI), and is a method considered by industry, authorities and institutes alike to be the state of the art.

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

ISO 10283:2007

<https://standards.iteh.ai/catalog/standards/sist/5e7d0fa8-8a77-4cfe-853f-1f460ab45d03/iso-10283-2007>

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

ISO 10283:2007

<https://standards.iteh.ai/catalog/standards/sist/5e7d0fa8-8a77-4cfe-853f-1f460ab45d03/iso-10283-2007>

# Binders for paints and varnishes — Determination of monomeric diisocyanates in isocyanate resins

## 1 Scope

This International Standard specifies a gas-chromatographic method for determining monomeric diisocyanates such as toluene diisocyanate<sup>1)</sup>, hexamethylene diisocyanate, isophorone diisocyanate<sup>2)</sup>, diphenylmethane diisocyanate<sup>3)</sup> and other diisocyanates in isocyanate resins as defined in Clause 3 and in solutions prepared from such resins, insofar as these are used in the formulation of paints and similar coating materials.

## 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 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, with or without solvent, based on aromatic, aliphatic or cycloaliphatic isocyanates containing isocyanate (NCO) groups

NOTE For the purposes of this International Standard, such isocyanate resins comprise:

- those which are manufactured from any diisocyanate, in particular toluene diisocyanate (TDI), hexamethylene diisocyanate (HDI), isophorone diisocyanate (IPDI) or diphenylmethane diisocyanate (MDI), and which contain urethane and/or biuret and/or isocyanurate groups;
- those which are prepared from mixtures of the isocyanate resins given above.

1) The term “toluene diisocyanate” is used here and in the following text for 4-methyl-1,3-phenylene diisocyanate (2,4-toluene diisocyanate) and 2-methyl-1,3-phenylene diisocyanate (2,6-toluene diisocyanate).

2) The term “isophorone diisocyanate” is used here and in the following text for 2-isocyanatomethyl-3,5,5-trimethylcyclohexylisocyanate. The stereoisomers are identified at the appropriate points in the text by (I) and (II).

3) The term “diphenylmethane diisocyanate” is used here and in the following text for 4,4-diisocyanatodiphenylmethane, 2,4-diisocyanatodiphenylmethane and 2,2-diisocyanatodiphenylmethane.

## 4 Principle

The content of monomeric diisocyanate in isocyanate resins is determined by gas chromatography, using tetradecane or, in the case of diisocyanates of low volatility, anthracene as the internal standard.

## 5 Reagents

During the analysis, use only reagents of recognized analytical grade.

**5.1 Ethyl acetate**, anhydrous (dried with 0,5 nm molecular sieve) and ethanol-free (ethanol content < 200 ppm).

**5.2 Tetradecane or anthracene.**

**5.3 Toluene diisocyanate** (isomeric mixture).

**5.4 Hexamethylene diisocyanate.**

**5.5 Isophorone diisocyanate** (isomeric mixture).

**5.6 Diphenylmethane diisocyanate.**

**5.7 Solution of internal standard.**

Weigh approximately 1,4 g of tetradecane or anthracene to the nearest 0,1 mg into a 1 000 ml volumetric flask and make up to the mark with ethyl acetate (5.1).

**5.8 Reference solution of monomeric diisocyanate.**

Weigh approximately 1,4 g of the relevant monomeric diisocyanate to the nearest 0,1 mg into a 1 000 ml volumetric flask and make up to the mark with ethyl acetate (5.1).

Protect the monomeric diisocyanate reference solutions from air and moisture.

NOTE If stored properly, they will remain stable for about two weeks.

**5.9 Calibration solution.**

Pipette 10 ml of the internal-standard solution (5.7) and 10 ml of the reference solution (5.8) into a sample bottle or conical flask (see 6.2). Using the 25 ml measuring cylinder, add 15 ml of ethyl acetate and mix.

NOTE Instead of preparing a calibration solution, the internal standard and the monomeric diisocyanate can be weighed directly with 40 ml of ethyl acetate into a 50 ml sample bottle fitted with a septum seal (dried free of water). Steps 5.7 and 5.8 are then no longer necessary.

## 6 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

**6.1 Analytical balance.**

**6.2 Conical flask**, of capacity 50 ml, fitted with a ground-glass stopper, or **sample bottle** of capacity 50 ml, fitted with a septum seal.

**6.3 One-mark pipette**, of capacity 10 ml.



**6.4 Measuring cylinder**, of capacity 25 ml.

**6.5 One-mark volumetric flask**, of capacity 1 000 ml.

**6.6 Sample-injection syringe**, of capacity 2 µl or 10 µl.

**6.7 Gas-chromatography**, with an exchangeable glass sample-evaporation tube, a flame ionization detector and an integrator.

## 7 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528. Store the sample in a cool, dry place and in the dark.

Under unfavourable storage conditions, reactions take place, particularly at elevated temperatures, which alter the monomeric isocyanate content of some isocyanate resins. In order to prevent these reactions as far as possible, samples must be stored in cool, dark conditions. However, it is then necessary to readjust the samples to room temperature before opening the containers so that ingressing atmospheric moisture cannot condense and thus change the monomeric isocyanate content. If there is any doubt, discard reference materials or samples which have been stored for prolonged periods.

## 8 Procedure

### 8.1 Operating conditions

The test conditions given in the examples are recommended as being suitable. Columns and test/operating conditions giving equivalent or superior performance may also be used.

The temperatures specified for the injector and the column depend on the thermal stability of the polyisocyanate resin under test. The monomeric diisocyanate content of many polyisocyanate resins, e.g. those with a biuret structure, may be altered at elevated temperatures. In such cases, the temperatures specified in the examples shall be used. The glass sample-evaporation tube shall be cleaned or changed as necessary, but at least at the start of each day's work.