
**Plastics — Aromatic isocyanates for use
in the production of polyurethanes —
Determination of the isomer ratio in
toluenediisocyanate**

*Plastiques — Isocyanates aromatiques pour utilisation dans la
production de polyuréthanes — Détermination du rapport des
isomères dans le diisocyanate de toluène*

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 15064:2004

<https://standards.iteh.ai/catalog/standards/sist/6c5a459d-157a-44b6-bba2-bff339d01a33/iso-15064-2004>



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 15064:2004

<https://standards.iteh.ai/catalog/standards/sist/6c5a459d-157a-44b6-bba2-bff339d01a33/iso-15064-2004>

© ISO 2004

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
Web 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 Sampling	2
5 Principle	2
6 Reagents	2
7 Apparatus	3
8 Test conditions	3
9 Method A — Samples containing 5 % to 95 % of 2,6-TDI	3
10 Method B — Samples containing 0 % to 5 % of 2,6-TDI	6
Annex A (normative) Calibration curves	8

STANDARD PREVIEW
(standards.iteh.ai)

ISO 15064:2004

<https://standards.iteh.ai/catalog/standards/sist/6c5a459d-157a-44b6-bba2-bff339d01a33/iso-15064-2004>

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 15064 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[ISO 15064:2004](https://standards.iteh.ai/catalog/standards/sist/6c5a459d-157a-44b6-bba2-bff339d01a33/iso-15064-2004)

<https://standards.iteh.ai/catalog/standards/sist/6c5a459d-157a-44b6-bba2-bff339d01a33/iso-15064-2004>

Introduction

No International Standard for the determination of isomer contents of toluenediisocyanate (TDI) has been published. This International Standard is based on infrared spectroscopy and consists of two test methods that can be used to determine the 2,4- and 2,6-isomer contents of commercially important toluenediisocyanates over a broad range of isomer concentrations. It is important to know the isomer content of TDI since the isomers have different reaction rates, and the processing and properties of polyurethane systems, especially flexible polyurethane foams, may be significantly affected. This method is based on work done in the Bayer Central Analytical Department (Leverkusen) in the early 1960s, and was originally published as part of ASTM D 1638. It is now designated ASTM D 4660, *Standard Test Methods for Polyurethane Raw Materials: Determination of the Isomer Content of Toluenediisocyanate*.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[ISO 15064:2004](https://standards.iteh.ai/catalog/standards/sist/6c5a459d-157a-44b6-bba2-bff339d01a33/iso-15064-2004)

<https://standards.iteh.ai/catalog/standards/sist/6c5a459d-157a-44b6-bba2-bff339d01a33/iso-15064-2004>

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

ISO 15064:2004

<https://standards.iteh.ai/catalog/standards/sist/6c5a459d-157a-44b6-bba2-bff339d01a33/iso-15064-2004>

Plastics — Aromatic isocyanates for use in the production of polyurethanes — Determination of the isomer ratio in toluenediisocyanate

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions prior to use.

1 Scope

This International Standard specifies methods for the measurement of toluene-2,6-diisocyanate in mixtures of the 2,4- and 2,6-isomers. Two methods based on infrared spectroscopy are required to give accurate results over a broad range of isomer concentrations. Method A is applicable to TDI samples containing 5 % to 95 % of the 2,6-isomer. Method B is applicable to TDI samples containing 0 % to 5 % of the 2,6-isomer. Both methods are based on the quantitative measurement of absorption bands arising from out-of-plane C–H deformation vibrations of the aromatic ring at 810 cm^{-1} and 782 cm^{-1} ($12,3\text{ }\mu\text{m}$ and $13,8\text{ }\mu\text{m}$).

(standards.iteh.ai)

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 648, *Laboratory glassware — One-mark pipettes*

ISO 4787, *Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

3 Terms and definitions

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

3.1

isomer

a compound having the same percentage composition and molecular mass as another compound, but differing in chemical structure and properties

3.2

isomer content

the amount of an isomer expressed as a mass percentage of the total isomer amount

3.3

TDI

toluenediisocyanate

3.4

polyurethane

a polymer prepared by the reaction of an organic di- or polyisocyanate with compounds containing two or more hydroxyl groups

4 Sampling

Since organic isocyanates react with atmospheric moisture, take special precautions in sampling. Usual sampling methods (for example, sampling an open drum with a thief), even when conducted rapidly, can cause contamination of the sample with insoluble ureas; therefore, blanket the sample with a dry inert gas (e.g. nitrogen, argon or dried air) at all times.

WARNING — Organic isocyanates are hazardous when absorbed through the skin, or when the vapours are breathed in. Provide adequate ventilation and wear protective gloves and eyeglasses.

5 Principle

5.1 Method A

In method A (5 % to 95 % 2,6-isomer), the infrared spectrum of a cyclohexane solution of the sample is recorded in the 770 cm^{-1} to 840 cm^{-1} ($12\text{ }\mu\text{m}$ to $13\text{ }\mu\text{m}$) region. The absorbance ratio of the 810 cm^{-1} and 782 cm^{-1} bands is measured and converted to % toluene-2,6-diisocyanate from a previously established calibration curve.

5.2 Method B

ISO 15064:2004
<https://standards.iteh.ai/catalog/standards/sist/6c5a459d-157a-44b6-bba2-bff339d01a33/iso-15064-2004>

In Method B (0 % to 5 % 2,6-isomer), the absorbance of the 782 cm^{-1} band is measured from an infrared spectrum of an undiluted sample and then converted to % 2,6-isomer from a previously established calibration curve.

6 Reagents

Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that the reagents shall conform to the specifications of ISO 6353-2. Other grades may be used, provided that it is first determined that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.1 Cyclohexane, distilled and stored over silica gel to remove traces of moisture.

6.2 Diisocyanate standards.

Pure samples of 2,4-TDI and 2,6-TDI are required for calibration. The following criteria can be used to judge purity:

for 2,4-TDI: freezing point = $22,0\text{ }^{\circ}\text{C}$; $n_{\text{D}}^{20} = 1,567\ 81$; $\rho_4^{20} = 1,218\ 6$

for 2,6-TDI: freezing point = $18,2\text{ }^{\circ}\text{C}$; $n_{\text{D}}^{20} = 1,571\ 11$; $\rho_4^{20} = 1,227\ 0$.

The diisocyanates can be prepared by phosgenating the corresponding pure amines and vacuum-distilling the products. Since these diisocyanates will react with moisture and may discolour in the presence of air, store them under dry nitrogen.

7 Apparatus

7.1 Spectrophotometer: any single- or double-beam recording infrared spectrophotometer, or FTIR spectrophotometer, accurate to 0,2 % transmission and capable of resolving the two peaks of the 2,4-isomer doublet at 810 cm^{-1} .

7.2 Sealed sodium chloride (NaCl) liquid absorption cells, with 0,2 mm (method A) and 0,1 mm (method B) path lengths. The actual path lengths of the cells shall be known to $\pm 0,002\text{ mm}$.

7.3 Glassware: 25 ml glass-stoppered volumetric flasks, 0,80 ml pipette and an all-glass syringe conforming to ISO 648 or ISO 4787.

7.4 Laboratory balance, capable of weighing to $\pm 0,1\text{ mg}$.

8 Test conditions

Since isocyanates react with moisture, keep laboratory humidity low, preferably under 50 % relative humidity. Thoroughly dry all laboratory ware. Store cells (7.2) in a dessicator and use thin rubber or plastic gloves while handling.

9 Method A — Samples containing 5 % to 95 % of 2,6-TDI

9.1 Calibration

9.1.1 Weigh the amounts of pure 2,4- and 2,6-TDI given in Table 1, 2 or 3 as appropriate for the calibration range desired. Calibration over a narrow range gives more accurate results than a wide-range calibration.

9.1.2 Prepare standard solutions and calibration curves as follows:

9.1.2.1 Using a pipette, transfer 0,80 ml (0,98 g) of each standard isomer mixture into dry, 25 ml glass-stoppered volumetric flasks (see 7.3). Dilute each to volume with cyclohexane (6.1) and mix thoroughly.

9.1.2.2 Fill two 0,2 mm sealed, liquid absorption cells (one for a single-beam instrument) with cyclohexane and record the spectrum from 770 cm^{-1} to 840 cm^{-1} ($12\text{ }\mu\text{m}$ to $13\text{ }\mu\text{m}$). Refill the sample cell with a solution from 9.1.2.1 and record the spectrum, superimposing it over the previously recorded solvent spectrum. Repeat the process for each solution in 9.1.2.1.

9.1.2.3 Using the solvent spectrum as the baseline, measure the absorbance of each standard solution at 810 cm^{-1} (2,4-TDI) and 782 cm^{-1} (2,6-TDI) and calculate the $810/782\text{ cm}^{-1}$ absorbance ratio. See Figure A.4 in Annex A for an illustrative spectrum. Construct a calibration curve (see Figure A.1) by plotting absorbance ratio (ordinate) versus mass ratio of 2,4- to 2,6-TDI (abscissa).

9.1.2.4 For convenience in narrow-range calibrations, the absorbance ratio may be plotted against the concentration (expressed in mass %) of each isomer (see Figure A.2 and Figure A.3). This allows direct determination of composition without equations. However, the relationship is not linear and the shape of the calibration curve must be carefully determined.