INTERNATIONAL STANDARD

ISO 26603

Second edition
2017-03

Plastics — Aromatic isocyanates for use in the production of polyurethanes — Determination of total chlorine

Plastiques — Isocyanates aromatiques utilisés pour la production de polyuréthannes — Dosage du chlore total

Reference number
ISO 26603:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 26603:2008), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— a key has been added to Figure 1.

Introduction

Isocyanates are typically produced by phosgenation of an aromatic amine using chlorine-substituted benzenes (e.g. o-dichlorobenzene) as reaction solvents. ISO 15028 is used to determine the hydrolyzable chlorine content of the isocyanates. The test methods in this document are used to determine the total chlorine content of aromatic isocyanates. The difference between the total chlorine content and the hydrolyzable chlorine content is a measure of the reaction solvents left in the product, and therefore is a useful tool for assessing product quality.

Plastics — Aromatic isocyanates for use in the production of polyurethanes — Determination of total chlorine

SAFETY PRECAUTIONS — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, 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 regulatory requirements.

1 Scope

This document specifies the determination of the total chlorine content of aromatic isocyanates used in the preparation of polyurethanes. The difference between the total chlorine content and the hydrolyzable chlorine content (see ISO 15028) is a measure of the process solvents left in the product. Both test methods are applicable to a variety of organic compounds, including aliphatic isocyanates, but the amount of sample used might need to be adjusted. These test methods can be used for research or for quality control.

NOTE This document is technically equivalent to ASTM D4661–03.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3696, *Water for analytical laboratory use — Specification and test methods*

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

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second series*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
isocyanate
organic compound containing one or more NCO groups

3.2
polyurethane
polymer prepared by the reaction of an organic di- or polyisocyanate with compounds containing two or more hydroxyl groups

3.3
hydrolyzable chlorine
organic or inorganic chlorine compounds formed in the production of isocyanates that react with methanol under the conditions of ISO 15028 to liberate hydrogen chloride

3.4
total chlorine
inorganic and organically bound chlorine present in isocyanates that is converted to titratable chlorides under the combustion conditions of the test

4 Principle

In each test method, the organic matter in the sample is destroyed by combustion with oxygen, thus converting the organically combined chlorine to ionic chloride. The chloride is determined potentiometrically by titration with silver nitrate ($AgNO_3$) solution.

4.1 Test Method A

Combustion of the sample is done in a pressurized oxygen bomb.

4.2 Test Method B

Combustion is done at atmospheric pressure in a Schöniger oxygen flask.

NOTE For information on the Schöniger flask, see Reference [6].

5 Interferences

Thiocyanate, cyanide, sulphide, bromide, iodide or other substances capable of reacting with silver ion, as well as substances capable of reducing silver ion in acid solution, will interfere with the determination.

6 Sampling

Since organic isocyanates react with atmospheric moisture, take special precautions in sampling. Usual sampling methods, even when conducted rapidly, can expose the isocyanate to moisture and 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.

CAUTION — Provide adequate ventilation and wear protective gloves and eyeglasses.

7 Test Method A — Total chlorine by oxygen bomb

7.1 Reagents

7.1.1 Purity of reagents

Reagent-grade chemicals shall be used in all tests. 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. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of ISO 6353-2 and ISO 6353-3.

7.1.2 Purity of water

Unless otherwise indicated, references to water shall be understood to mean grade 2 water as defined by ISO 3696.

7.1.3 Ethyl alcohol, conforming to ISO 6353-2.

7.1.4 Nitric acid (diluted). While stirring vigorously, add 100 ml of nitric acid (HNO_3, specific gravity 1,42) to 100 ml of water cooled in an ice bath.

7.1.5 Oxygen, free of combustible materials and halogen compounds.

7.1.6 Silver nitrate, standard solution (0,01 M). Prepare a 0,01 M silver nitrate ($AgNO_3$) solution and check frequently enough to detect changes of 0,000 5 M, either gravimetrically or potentiometrically, using standard hydrochloric acid (HCl).

7.1.7 Sodium carbonate solution (50 g/l). Dissolve 135 g of sodium carbonate decahydrate ($Na_2CO_3 \cdot 10H_2O$) in water and dilute to 1 l.

7.2 Apparatus

7.2.1 Weighing bottle and balance, suitable for weighing a liquid sample by difference to the nearest 0,5 mg.

7.2.2 Oxygen bomb apparatus. A corrosion-resistant steel reactor capable of being pressurized to 40 atmospheres of pure oxygen, followed by electrical ignition of the sample by use of an internal fuse wire. The bomb shall be capable of withstanding the pressure build-up caused by the combustion of the sample. Parr Bomb No. 1108 is a suitable device (see Figure 1). Equivalent apparatus may be substituted with appropriate changes in the procedure.

7.2.3 Fuse wire, iron-nickel-chromium, No. 34 B and S gage.

7.2.4 Titrimeter, automatic (preferred) or manual, equipped with a silver/silver chloride electrode pair and a 10 ml capacity microburette.

7.2.5 Bubble counter, a 100 ml graduate and delivery tube, or a bent "L" glass tube connected to a piece of rubber tubing. The graduate is filled to the 50 ml mark with water to which 3 ml of 0,1 M $AgNO_3$ and 1 drop of concentrated nitric acid have been added. Any turbidity that develops indicates that HCl gas is being lost when venting the bomb.