SLOVENSKI STANDARD
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Determination of the silanol group content on the surface of fumed silica - Reaction gas chromatographic method (ISO 23157:2021)

Bestimmung des Gehaltes an Silanolgruppen auf der Oberfläche von gefällter Kieselsäure - Gaschromatographisches Verfahren (ISO 23157:2021)

Ta slovenski standard je istoveten z: **prEN ISO 23157**

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Determination of the silanol group content on the surface of fumed silica — Reaction gas chromatographic method

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Foreword

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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).

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For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs and extenders*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Fumed silica (pyrogenic silicon dioxide) has a relatively high concentration of silanol groups formed during high-temperature flame hydrolysis and the following cooling process. This specified property enables fumed silica to be widely used as a reinforcing filler, thickener, thixotropy-increasing and anti-sagging additive in numerous industries, for example silicone rubber, coating materials, adhesives and sealants.

The determination of the silanol group content on the surface of fumed silica is essential for both manufacturers and users to develop a high-performance, surface modified fumed silica and improve existing products. Further, it also facilitates the communication among interested parties.

In practice, methods of titration, thermogravimetry (TG), infrared spectroscopy (IR) and reaction gas chromatography can be a choice for the determination of the silanol group content on the surface of fumed silica. Among these methods, the method of reaction gas chromatography is preferred by users due to the advantage of higher sensitivity and better reproducibility.

This document provides a detailed procedure for how to conduct the testing of the silanol group content on the surface of fumed silica by means of the reaction gas chromatographic method.

Determination of the silanol group content on the surface of fumed silica — Reaction gas chromatographic method

SAFETY STATEMENT — Persons using this document should be familiar with usual laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine applicability of any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance to this document be carried out by suitably qualified staff.

1 Scope

This document specifies a method for the determination of the silanol group content on the surface of fumed silica by reaction gas chromatographic method.

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 18451-1, *Pigments, dyestuffs and extenders — Terminology — Part 1: General terms*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18451-1 and the following apply.

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

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

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

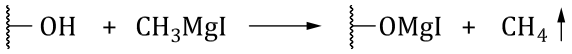
3.1
fumed silica
amorphous silica produced from silicon halides by high-temperature flame hydrolysis

[SOURCE: ISO 18473-3:2018, 3.1]

3.2
silanol group
hydroxyl group on the surface of fumed silica

4 Principle

A suitable amount of the dried fumed silica sample reacts with the Grignard reagent dispersed in toluene in an airtight reaction bottle to produce methane, which is injected into a gas chromatograph for separation, and further detected by a flame ionization detector (FID). The amount of methane produced is directly proportional to the silanol group content on the surface of fumed silica and is quantified from the peak areas using the external standard method. Then, the silanol group content in the sample is calculated according to the following reaction.



5 Reagents

5.1 General

During the analysis, use only reagents of recognized analytical grade. Other grades may also be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Information about the major reagents is given in Table 1.

Table 1 — Information about major reagents

Reagent	CAS No.[a]
Toluene	108–88–3
Methyl magnesium iodide[b]	917–64–6
Methane	74–82–8
Diethyl ether	60–29–7

[a] Chemical Abstracts System Registry Number.

[b] Dissolved in diethyl ether.

5.2 Toluene

Toluene (AR, purity ≥ 99,9 %, see Table 1) is used as a dispersion agent to promote the contact and reaction between the fumed silica sample and the Grignard reagent. Trace water is removed with calcium hydroxide before use.

5.3 Grignard reagent

The Grignard reagent is a solution of 3,0 mol/l methyl magnesium iodide (see Table 1), which reacts with hydroxyl to produce methane.

5.4 Reaction solution

Dilute the Grignard reagent (5.3) 10 times with toluene (5.2) (volume ratio) as a reaction solution. The reaction solution shall be used immediately after dilution. A volumetric flask or another clean glass container may be used for dilution. Ensure that the samples to be tested and the calibration substances react with the identical reaction solution. Carry out the injection with the syringe immediately.

5.5 Carrier gas and diluent gas

Use nitrogen of 99,999 % or higher purity. High-purity helium may also be used.

5.6 Auxiliary gases

Use hydrogen of high purity and air for the flame ionization detector.

Suitable filters shall be installed in the gas chromatograph connection pipes to adsorb residual impurities.

5.7 Calibration substance

Use methane (see Table 1, purity ≥ 99,9 %) for the calibration. For a representative chromatogram of methane, see Annex A.