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



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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 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.