INTERNATIONAL STANDARD

ISO 20596-1

First edition
2018-06

Water quality — Determination of cyclic volatile methylsiloxanes in water —

Part 1: Method using purge and trap with gas chromatography-mass spectrometry (GC-MS)

Qualité de l'eau — Détermination des méthylsiloxanes cycliques volatiles dans l'eau —

Partie 1: Méthode par dégazage et piégeage avec chromatographie en phase gazeuse-spectrométrie de mass (GC-MS)

Reference number
ISO 20596-1:2018(E)

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Published in Switzerland

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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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This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods.*

A list of all parts in the ISO 20596 series can be found on the ISO website.

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Water quality — Determination of cyclic volatile methylsiloxanes in water —

Part 1: Method using purge and trap with gas chromatography-mass spectrometry (GC-MS)

WARNING — Persons using this document should be familiar with normal 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.

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

1 Scope

This document specifies a method for the quantitative determination of selected cyclic volatile methylsiloxanes (cVMS) in non-filtered water samples by purge and trap extraction with isotope dilution gas chromatography-mass spectrometry (GC-MS).

This method is applicable to the determination of individual cVMS, including:

— octamethylcyclotetrasiloxane (D4);

— decamethylcyclopentasiloxane (D5);

— dodecamethylcyclohexasiloxane (D6);

in surface water, ground water, and wastewater. It can be applied to samples within the concentration range of 0,01 µg/l to 1 µg/l of each of the target compounds. Depending on the matrix, the method may also be applicable to higher concentrations ranging from 1 µg/l to 100 µg/l after suitable dilution of the sample or reduction in sample size.

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 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation*

ISO 5667-4, *Water quality — Sampling — Part 4: Guidance on sampling from lakes, natural and man-made*

ISO 5667-6, *Water quality — Sampling — Part 6: Guidance on sampling of rivers and streams*

ISO 5667-10, *Water quality — Sampling — Part 10: Guidance on sampling of waste waters*

ISO 5667-11, *Water quality — Sampling — Part 11: Guidance on sampling of groundwaters*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

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3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Principle

Extraction of the analytes listed in Table 1 from the water sample by purge and trap extraction, solvent elution and determination by gas chromatography with mass spectrometric detection.

Table 1 — Analytes determinable by this method

Analyte	Formula	Abbreviation	CAS-RN[a]
Octamethylcyclotetrasiloxane	$C_8H_{24}O_4Si_4$	D4	556–67–2
Decamethylcyclopentasiloxane	$C_{10}H_{30}O_5Si_5$	D5	541–02–6
Dodecamethylcyclohexasiloxane	$C_{12}H_{36}O_6Si_6$	D6	540–97–6
[a] CAS-RN: Chemical Abstracts Services Registration Number.			

5 Interferences

WARNING — Silicone includes D4, D5 and D6, and is widely used in consumer products such as hair care products, cosmetics, hand lotions, and antiperspirant. As silicone is present in many consumer products, the user should take care not to use hand lotions or other possible sources of contamination before or during the sampling and analysis. Pay special attention to avoid any contamination.

5.1 General

Contamination introduced during the analytical procedure is monitored by the determination of blanks (see 9.3).

5.2 Interferences with sampling and extraction

Sampling containers shall consist of materials that do not change the composition of the sample during sample storage. All types of silicone polymer materials shall be avoided during sampling, sample storage and extraction. Sample containers shall be rinsed thoroughly with acetone (6.2) and *n*-hexane (6.3) prior to use. Sample containers shall be checked for possible background contamination before use when a new type of bottles is prepared.

5.3 Interferences with GC-MS

Silicones are also commonly found in parts and consumables associated with gas chromatography including septa for the vials and inlet. Additionally, GC columns are polydimethysiloxane based and when exposed to moisture and heat also contribute to background cVMS. Autosampler vial septa should be silicone free or thinly coated with PTFE (PTFE = polytetrafluoroethene) on the side exposed to the sample. The inlet septum should be replaced with a Merlin Microseal™[1)] to reduce background contamination from this source. Also any solvents should be dried prior to injection into the GC or care should be taken to use a solvent in which water is only soluble in the ppm levels.

1) Merlin Microseal is the trademark of a product. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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