



Designation: D3871 – 84 (Reapproved 2024)

Standard Test Method for Purgeable Organic Compounds in Water Using Headspace Sampling¹

This standard is issued under the fixed designation D3871; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of most purgeable organic compounds that boil below 200 °C and are less than 2 % soluble in water. It covers the low $\mu\text{g/L}$ to low mg/L concentration range (see Section 15 and Appendix X1).

1.2 This test method was developed for the analysis of drinking water. It is also applicable to many environmental and waste waters when validation, consisting of recovering known concentrations of compounds of interest added to representative matrices, is included.

1.3 Volatile organic compounds in water at concentrations above 1000 $\mu\text{g/L}$ may be determined by direct aqueous injection in accordance with Practice D2908.

1.4 It is the user's responsibility to assure the validity of the test method for untested matrices.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 8.5.5.1.

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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2. Referenced Documents

2.1 *ASTM Standards:*²

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2908 Practice for Measuring Volatile Organic Matter in Water by Aqueous-Injection Gas Chromatography

E355 Practice for Gas Chromatography Terms and Relationships

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129 and Practice E355.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *purgeable organic, n*—any organic material that is removed from aqueous solution under the purging conditions described in this test method (10.1.1).

4. Summary of Test Method

4.1 An inert gas is bubbled through the sample to purge volatile compounds from the aqueous phase. These compounds are then trapped in a column containing a suitable sorbent. After purging is complete, trapped components are thermally desorbed onto the head of a gas chromatographic column for separation and analysis. Measurement is accomplished with an appropriate detector.

5. Significance and Use

5.1 Purgeable organic compounds, including organohalides, have been identified as contaminants in raw and drinking water. These contaminants may be harmful to the environment and man. Dynamic headspace sampling is a generally applicable method for concentrating these components prior to gas

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

chromatographic analysis (1-5).³ This test method can be used to quantitatively determine purgeable organic compounds in raw source water, drinking water, and treated effluent water.

6. Interferences

6.1 Purgeable compounds that coelute with components of interest and respond to the detector will interfere with the chromatographic measurement. Likelihood of interference may be decreased by using dissimilar columns or a more selective detector for the chromatographic step.

7. Apparatus

7.1 *Purging Device*—Commercial devices are available for this analysis. Either commercial apparatus or the equipment described below may be used for this analysis. Devices used shall be capable of meeting the precision and bias statements given in 15.1.

7.1.1 *Glass Purging Device* having a capacity of 5 mL is shown in Fig. A1.1. Construction details are given in Annex A1. A glass frit is installed at the base of the sample reservoir to allow finely divided gas bubbles to pass through the aqueous sample while the sample is restrained above the frit. The sample reservoir is designed to provide maximum bubble contact time and efficient mixing.

7.1.2 Gaseous volumes above the sample reservoir are kept to a minimum to provide efficient transfer and yet large enough to allow sufficient space for foams to disperse. Inlet and exit ports are constructed from 6.4-mm (1/4-in.) outside diameter medium-wall tubing so that leak-free removable connections can be made using “finger-tight” compression fittings containing plastic ferrules. The optional foam trap is used to control occasional samples that foam excessively.

7.2 *Trap*—A short section of stainless steel or glass tubing is packed with a suitable sorbent. Traps should be conditioned before use (Section 11). While other trap designs and sorbent materials may be used (see Section 12), the trap and sorbent described here are recommended and were used to collect precision and bias data. If another trap design or sorbent material is used, these precision and bias statements should be verified. A suitable trap design is 150 mm long by 3.17 mm outside diameter (2.54 mm inside diameter). The front 100 mm is packed with 60 to 80 mesh 2,6-diphenyl-*p*-phenylene oxide followed by 50 mm of 35 to 60 mesh silica gel. One trap design is shown in Fig. A1.2, with details in Annex A1. The body assembly acts as a seal for the exit end of the trap. The modified stem assembly is used to seal the inlet end of the trap when it is not in use.

7.3 *Desorber* consists of a trap heater and an auxiliary carrier gas source to backflush the trap at elevated temperatures directly onto the gas-chromatographic column. Desorber 1 (Fig. A1.3 and Annex A1) is dedicated to one gas chromatograph, but Desorber 2 can be used as a universal desorber for many gas chromatographs with a septum-type liquid-inlet system.

7.3.1 *Desorber 1* is attached directly onto the gas-chromatograph liquid-inlet system after removing the septum nut, the septum, and the internal injector parts. The modified body assembly is screwed onto the inlet system using the PTFE gasket as a seal. A plug is attached to one of the stem assemblies.

7.3.1.1 The assembled parts, simply called “the plug,” are used to seal the desorber whenever the trap is removed to maintain the flow of carrier gas through the gas-chromatographic column.

7.3.1.2 The flow controller, PTFE tubing, and stem assembly are used to provide the trap-backflush flow. This entire assembly also provides gas flow to operate the purging device.

7.3.2 *Desorber 2* (Fig. A1.4 and Annex A1) may be attached to any gas chromatograph by piercing the gas-chromatographic liquid-inlet septum with the needle.

7.3.2.1 The desorber is assembled in accordance with Fig. A1.4 with internal volumes and dead-volume areas held to a minimum. The heat source is concentrated near the base of the desorber so that the internal seals of the body assembly do not become damaged by heat. The use of a detachable needle assembly from a microsyringe makes it easy to replace plugged or dulled needles.

7.3.2.2 The flow controller, PTFE tubing, and stem assembly are used to provide the trap-backflush flow. This entire assembly is also used to provide gas flow to operate the purging device.

7.4 *Gas Chromatograph* equipped with a suitable detector, such as flame ionization, electrolytic conductivity, microcoulometric (halide mode), flame photometric, electron capture, or mass spectrometer.

7.4.1 The gas chromatographic conditions described below are recommended and were used to obtain precision and bias data (Section 15). If other column conditions are used, the analyst must demonstrate that the precision and bias achieved are at least as good as that presented in Section 15.

7.4.2 *Column* is 2.4 m by 2.4 mm inside diameter stainless steel packed with a suitable packing. Glass or nickel columns may be required for certain applications. Helium carrier gas flow is 33 mL/min and a flame ionization detector is used.

7.4.3 *Chromatograph Oven* is held at room temperature during trap desorption, then rapidly heated to 60 °C and held for 4 min. Finally, the temperature is programmed to 170 °C at 8 °C/min and held for 12 min or until all compounds have eluted.

7.5 *Sampling Vials*, glass, 45 mL, sealed with PTFE-faced septa.⁴ Vial caps must be open-top screw caps to prevent vial breakage. The vials, septa, and caps are washed with detergent and hot water and rinsed with tap water and organic free water. The vials and septa are then heated to 105 °C for 1 h and allowed to cool to room temperature in a contaminant-free area. When cool, the vials are sealed with septa, PTFE side down, and screw capped. Aluminum foil disks may be placed

³ The boldface numbers in parentheses refer to a list of references at the end of this standard.

⁴ Pierce No. 13075 Screw Cap System Vials and 12722 Tuf-Bond Discs, Pierce Chemical Co., Rockford, IL, have been found satisfactory for this application.