



Designation: D4526 – 20

Standard Practice for Determination of Volatiles in Polymers by Static Headspace Gas Chromatography¹

This standard is issued under the fixed designation D4526; 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 Headspace gas chromatography (GC) involves the determination of volatile components in a polymer solution by gas chromatography of a vapor phase in thermal equilibrium with the sample matrix. Volatiles in finely ground insoluble polymers can also be determined with and without an extracting solvent.

1.2 This practice provides two procedures:

1.2.1 *Procedure A*—Automatic headspace analysis.

1.2.2 *Procedure B*—Manual injection headspace analysis.

1.3 *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 Section 7.*

NOTE 1—There is no known ISO equivalent to this standard.

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D883 Terminology Relating to Plastics

D4322 Test Method for Residual Acrylonitrile Monomer Styrene-Acrylonitrile Copolymers and Nitrile Rubber by Headspace Gas Chromatography (Withdrawn 2010)³

E260 Practice for Packed Column Gas Chromatography

¹ This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

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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.

³ The last approved version of this historical standard is referenced on www.astm.org.

E355 Practice for Gas Chromatography Terms and Relationships

E594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography

3. Terminology

3.1 *General*—The units, symbols, and abbreviations used in this test method are in accordance with Terminology D883 and Practice IEEE/ASTM SI-10.

4. Significance and Use

4.1 There are various reasons why one would measure the level of unreacted or residual monomer, water, or other volatile components in a polymer sample.

4.2 Volatiles of interest are often at trace concentrations. Headspace analysis is suited for determination of these trace components which often cannot be determined by conventional gas chromatography because of sample decomposition or interferences.

4.3 For polymer analysis, sample treatment for headspace analysis is simpler than conventional gas chromatography, where additional precipitation steps are required to prevent polymer contamination of the chromatographic column.

4.4 This headspace practice will be able to determine qualitatively any component with sufficient vapor pressure. It is capable of yielding semiquantitative results and can be used for relative comparisons between samples.

5. Apparatus

5.1 *Gas Chromatograph*, equipped with an appropriate detector and backflush valve.

5.1.1 For Procedure A, an *Automated Headspace Sampler*, including backflush capability, thermostated sample tray, and associated accessories fulfill these requirements while providing for automatic sequential sampling of headspace vapors.

5.1.2 Procedure B requires the following additional equipment:

5.1.2.1 *Constant-Temperature Bath*, capable of maintaining $90 \pm 1^\circ\text{C}$.

5.1.2.2 *Gas-Tight Gas Chromatographic Syringes*, which can be heated to 90°C for sampling and injection.

*A Summary of Changes section appears at the end of this standard