



Designation: D7716 – 11a (Reapproved 2024)

Standard Test Method for Determination of Residual Methanol in Glycerin by Gas Chromatography¹

This standard is issued under the fixed designation D7716; 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 provides for the quantitative determination of residual methanol in glycerin by gas chromatography. The range of detection for residual methanol is 0.02 to 0.60 mass %.

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

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.*

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

D1193 Specification for Reagent Water

D7640 Specification for Engine Coolant Grade Glycerin

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E355 Practice for Gas Chromatography Terms and Relationships

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

¹ This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants and Related Fluids and is the direct responsibility of Subcommittee D15.04 on Chemical Properties.

Current edition approved Sept. 1, 2024. Published September 2024. Originally approved in 2011. Last previous edition approved in 2020 as D7716 – 11a (2020). DOI: 10.1520/D7716-11AR24.

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

3. Terminology

3.1 *Definitions*—This test method makes reference to many common gas chromatographic procedures, terms, and relationships. Detailed definitions can be found in Practices E355 and E594.

4. Summary of Test Method

4.1 The sample is analyzed by headspace gas chromatography. Calibration is achieved by the use of external standards of methanol in water.

5. Significance and Use

5.1 Methanol content reflects the quality of glycerin for use as an engine coolant. The current specification for the maximum methanol content is 0.1 % weight to weight (w/w).

6. Apparatus

6.1 *Chromatographic System*—See Practice E355 for specific designations and definitions. The gas chromatograph (GC) system shall be capable of operating at the conditions given in Table 1.

6.2 *Autosampler system*, Gerstel multipurpose sampler MPS-2³ or equivalent. This method can also be run manually.

6.3 *Column*, open tubular column with polyethylene glycol (PEG) bonded and cross-linked phase internal coating. The column should have an upper temperature limit of 260 °C. A column 30 m in length, with an internal diameter of 0.32 mm, and a 1.0 μ m film thickness has been found satisfactory. Any column with equivalent or better chromatographic efficiency and selectivity can be used.

6.4 *Electronic Data Acquisition System*—A computer capable of providing real-time graphic and digital presentation of the chromatographic data is recommended for use. Peak areas and retention times shall be measured by computer or electronic integration (integrator).

³ The sole source of supply of the apparatus known to the committee at this time is GERSTEL GmbH & Co.KG, Eberhard- Gerstel-Platz 1, 45473 Mülheim an der Ruhr, Germany. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.