



Standard Test Method for Determination of Free and Total Glycerin in Biodiesel Blends by Anion Exchange Chromatography¹

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1. Scope

1.1 This test method covers and describes an anion exchange chromatography procedure for determining free and total glycerin content of biodiesel (B100) and blends (B0 to B20) with diesel fuel oils defined by Specification [D975](#) Grades 1-D, 2-D, and low sulfur 1-D and 2-D and Specification [D6751](#) (for B100 feedstocks). It is intended for the analysis of biodiesel and blend samples containing between 0.5 to 50 mg/kg glycerin.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

[D975](#) Specification for Diesel Fuel Oils

[D1193](#) Specification for Reagent Water

[D4057](#) Practice for Manual Sampling of Petroleum and Petroleum Products

[D4177](#) Practice for Automatic Sampling of Petroleum and Petroleum Products

[D6299](#) Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

[D6751](#) Specification for Biodiesel Fuel Blend Stock (B100) for Middle Distillate Fuels

[D6792](#) Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories

¹ This test method is under the jurisdiction of ASTM Committee [D02](#) on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee [D02.04.0C](#) on Liquid Chromatography.

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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.

[E177](#) Practice for Use of the Terms Precision and Bias in ASTM Test Methods

[E691](#) Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 Definitions:

3.1.1 *free glycerin, n*—measure of the amount of glycerin remaining in the fuel.

3.1.2 *total glycerin, n*—sum of the free glycerin and the glycerin portion of any unreacted or partially reacted oil or fat.

4. Summary of Test Method

4.1 *Free Glycerin*—A small volume of an extract of the blend sample is directly injected into an ion chromatograph consisting of appropriate ion exchange columns and into an electrochemical detector. Glycerin is separated based on its affinity for ion exchange sites of the resin with respect to the resin's affinity for the eluent. An electrochemical detector is employed for detection of glycerin. Glycerin is quantified by peak area based on an external calibration curve, and is reported as $\mu\text{g/g}$ (mg/kg), or may be converted to wt%. Calibration standards are prepared from commercially available glycerin (99+% purity) in an aqueous solution.

4.2 *Total Glycerin*—A small volume extract of a saponified blend sample is directly injected into an ion chromatograph consisting of appropriate ion exchange columns and into an electrochemical detector. Glycerin is separated based on its affinity for ion exchange sites of the resin with respect to the resin's affinity for the eluent. An electrochemical detector is employed for detection of glycerin. Glycerin is quantified by peak area based on an external calibration curve, and is reported as $\mu\text{g/g}$ (mg/kg), or may be converted to wt%. Calibration standards are prepared from commercially available glycerin (99+% purity) in an aqueous solution.

5. Significance and Use

5.1 Petroleum-based diesel may be blended with biodiesel. High levels of free glycerin in biodiesel can cause injector deposits ("gel effect"), as well as clogging fuel systems. High levels of unreacted glycerides can cause injector deposits and can adversely affect cold weather operation and filter plugging.