



Designation: D6584 – 21

Standard Test Method for Determination of Total Monoglycerides, Total Diglycerides, Total Triglycerides, and Free and Total Glycerin in B-100 Biodiesel Methyl Esters by Gas Chromatography¹

This standard is issued under the fixed designation D6584; 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 quantitative determination of total monoglyceride, total diglyceride, total triglyceride, and free and total glycerin in B-100 methyl esters by gas chromatography. The range of quantitation for monoglyceride is 0.009 % to 0.77860 % by mass, for diglyceride is 0.092353 % to 0.54475 % by mass, and for triglyceride is 0.00092857 % to 1.3881 % by mass. The range of quantitation for free glycerin is 0.0005714 % to 0.019533 % by mass and for total glycerin from 0.0090714 % to 0.42767 % by mass. This procedure is not applicable to vegetable oil methyl esters obtained from lauric oils, such as coconut oil and palm kernel oil.

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

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

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 D02.04.0L on Gas Chromatography Methods.

Current edition approved Dec. 1, 2021. Published January 2022. Originally approved in 2000. Last previous edition approved in 2017 as D6584 – 17. DOI: 10.1520/D6584-21.

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.

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

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 Definitions:

3.1.1 This test method makes reference to common procedures, terms, and relationships. Detailed definitions of these can be found in Practices E355 and E594, and Terminology D4175. Several definitions for clarity are indicated below:

3.1.2 *biodiesel (B-100)*, *n*—fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats.

3.1.3 *bonded glycerin*, *n*—glycerin portion of the mono-, di-, and triglyceride molecules.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *total glycerin*, *n*—sum of free and bonded glycerin.

4. Summary of Test Method

4.1 The sample is analyzed by gas chromatography, after silylating with N-methyl-N-trimethylsilyltrifluoroacetamide (MSTFA). Calibration is achieved by the use of two internal standards and four reference materials. Mono-, di-, and triglyceride are determined by comparing to monoolein, diolein, and triolein standards respectively. Average conversion factors are applied to mono-, di-, and triglycerides to calculate the bonded glycerin content of the sample.

5. Significance and Use

5.1 Free and bonded glycerin content reflects the quality of biodiesel. A high content of free glycerin may cause problems during storage, or in the fuel system, due to separation of the glycerin. A high total glycerin content can lead to injector fouling and may also contribute to the formation of deposits at injection nozzles, pistons, and valves.

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