



Designation: D 4530 – 00

An American National Standard

Standard Test Method for Determination of Carbon Residue (Micro Method)¹

This standard is issued under the fixed designation D 4530; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the amount of carbon residue (see Note 1) formed after evaporation and pyrolysis of petroleum materials under certain conditions and is intended to provide some indication of the relative coke forming tendency of such materials.

1.2 The test results are equivalent to the Conradson Carbon Residue test (see Test Method D 189).

NOTE 1—This procedure is a modification of the original method and apparatus for carbon residue of petroleum materials, where it has been demonstrated that thermogravimetry is another applicable technique.² However, it is the responsibility of the operator to establish operating conditions to obtain equivalent results when using thermogravimetry.

1.3 This test method is applicable to petroleum products that partially decompose on distillation at atmospheric pressure and was tested for carbon residue values of 0.10 to 30 % (m/m). Samples expected to be below 0.10 weight % (m/m) residue should be distilled to remove 90 % (V/V) of the flask charge (see Section 9). The 10 % bottoms remaining is then tested for carbon residue by this test method.

1.4 Ash-forming constituents, as defined by Test Method D 482, or non-volatile additives present in the sample will add to the carbon residue value and be included as part of the total carbon residue value reported.

1.5 Also in diesel fuel, the presence of alkyl nitrates, such as amyl nitrate, hexyl nitrate, or octyl nitrate, causes a higher carbon residue value than observed in untreated fuel, which may lead to erroneous conclusions as to the coke-forming propensity of the fuel. The presence of alkyl nitrate in the fuel may be detected by Test Method D 4046.

1.6 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.7 *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 appro-*

priate safety practices and to determine the applicability of regulatory limitations prior to use. For specific hazard statements see 8.2.3 and 8.4.

2. Referenced Documents

2.1 ASTM Standards:

D 189 Test Method for Conradson Carbon Residue of Petroleum Products³

D 482 Test Method for Ash from Petroleum Products³

D 4046 Test Method for Alkyl Nitrate in Diesel Fuels by Spectrophotometry⁴

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁴

D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products⁴

E 1 Specification for ASTM Thermometers⁵

E 133 Specification for Distillation Equipment⁶

2.2 IP Standard:

Specification for IP Standard Thermometers⁷

3. Terminology

3.1 Definitions:

3.1.1 *carbon residue, n— in petroleum products*, the part remaining after a sample has been subjected to thermal decomposition.

3.1.1.1 *Discussion*—The amount of residue is dependent on the test conditions of evaporation and pyrolysis. The term may be misleading here in that the residue may contain other than carbon decomposition products. However, the term is retained due to its wide common usage.

4. Summary of Test Method

4.1 A weighed quantity of sample is placed in a glass vial and heated to 500°C under an inert (nitrogen) atmosphere in a controlled manner for a specific time. The sample undergoes coking reactions, and volatiles formed are swept away by the

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

Current edition approved May 10, 2000. Published August 2000. Originally published as D 4530 – 85. Last previous edition D 4530 – 85.

² See *Fuel*, Vol 63, July 1984, pp. 931–934.

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ *Annual Book of ASTM Standards*, Vol 05.02.

⁵ *Annual Book of ASTM Standards*, Vol 14.03.

⁶ *Annual Book of ASTM Standards*, Vol 14.04.

⁷ *Methods for Analysis and Testing*, Institute of Petroleum Standard Methods for Petroleum and Its Products, Part I, Vol 2. Available from Institute of Petroleum, 61 New Cavendish St., London, W1M 8AR, UK.