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Designation: D 1119 - 9600

Standard Test Method for Percent Ash Content of Engine Coolants and Antirusts¹

This standard is issued under the fixed designation D 1119; 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 ash content after ignition of commercial engine coolants and antirusts, as packaged or after use.

1.2 Some commercial antirusts are solid products. This procedure applies only to liquid products.

1.2 This test method is designed to aid in identifying types of coolants and antirusts. While the ash is primarily due to the inorganic inhibitors used, it is not a measure of the total inhibitor concentration because of the loss of organic inhibitors.

1.3 An ash content over that stated in Specification D 3306 may indicate a salt-based product.

1.4 The

<u>1.3 The</u> values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only. $\frac{1.5}{1.5}$ standard.

<u>1.4</u> 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. A specific precautionary statement is given in Note 1.

2. Referenced Documents

2.1 ASTM Standards:

¹ This test method is under the jurisdiction of ASTM Committee D=15 on Engine Coolants and is the direct responsibility of Subcommittee D15.04 on Chemical Properties. Current edition approved—July_Sept. 10, <u>1996</u>. 2000. Published—September <u>1996</u>. December 2000. Originally published as D 1119 – 50. Last previous edition D 1119 – 85 (1990). D 1119 – 96.

D 1176 Test Method for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes² D 3306 Specification for Ethylene Glycol Base Engine Coolant for Automobile and Light Duty Service²

3. Terminology

3.1 Definitions of Terms Specific to This Standard: Definition:

3.1.1 ash content, %, <u>n</u>—the weight components of the solid residual of an ignited engine coolant or antirust sample compared to the original weight of the sample. not removed by ignition at 750°C.

4. Summary of Test Method

4.1 To determine the ash-value, <u>content</u>, the antirust or coolant is weighed, evaporated or charred, and finally ignited at a strong heat.

5. Significance and Use

5.1 This test method is designed to aid in identifying types of coolants and antirusts. While the ash is primarily due to the inorganic inhibitors used, it is not a measure of the total inhibitor concentration because of the loss of organic inhibitors. 5.2 An ash content greater than that stated in Specification D 3306 may indicate a salt-based product.

6. Apparatus

56.1 Crucible, high-silica glass, porcelain, or Pt crucible, platinum of 50-mL capacity, equipped with cover.

67. Procedure

67.1 Ignite the crucible and cover over a Meker burner for 5 min and allow to cool to room temperature in a desiccator; then weigh to the nearest 0.1 mg.

NOTE 1-Caution: Warning: When handling a hot crucible use proper caution and safety equipment such as safety glasses, gloves, tongs, etc.

<u>67.2</u> Weigh 20 \pm 0.1 g of well-mixed antirust or coolant (Note 2 and Note 3), including all phases, into the crucible. <u>Sample</u> in accordance with Test Method D 1176. Record the weight of the sample to the nearest 0.1 mg.

NOTE 2—For obtaining 2—The weight of sample taken is suitable for most commercial products. If there is reason to believe that the sample, reference should ash content may be high, the sample size may be decreased to Test Method D 1176. 5 or 10 ± 0.1 g.

7.3 Place the covered crucible in a cool small sand bath in a vented hood. Raise the temperature by any convenient means until the liquid is evaporated. If the sample ignites, remove the heat source until the burning ceases; then reapply the heat.

NOTE 3—The weight of sample taken is suitable for most commercial products. If there is reason to believe that the ash content may be high, the sample size may be decreased to 5 or 10 g.

6.3 Place the covered crucible in a cool small sand bath in a vented hood. Raise the temperature by any convenient means until the liquid is distilled. If the sample ignites, remove the heat source until the burning ceases; then reapply the heat.

Note 4 <u>3</u> Caution: Many types of coolant and antirust have tendencies to spatter upon heating, with consequent loss of sample and erroneous results. All precautions consistent with good laboratory practice should be followed.

Note-5-4-Warning: Avoid inhalation of the fumes.

 $6\underline{7}.4$ When the <u>liquid has been distilled or burned off and the</u> crucible appears dry, transfer the crucible to a support over a Meker-type <u>burner</u>, increase the flame height, and <u>ignite</u> <u>burner</u>. Ignite strongly at red heat until all carbonaceous material disappears from both crucible and cover. The crucible cover may be removed and carefully ignited, directly if desired, taking care to avoid losing any material deposited on the underside of the cover. Place the crucible and cover, with contents, in a desiccator to cool. When they have cooled to room temperature, weigh the crucible, cover, and contents (ash) to the nearest 0.1 mg. Proceed to 7.6.

<u>7.5</u> If desired, muffle furnace ignition may be used in place of the Meker-type burner. If a muffle furnace is used, proceed as specified in <u>6.1</u> <u>7.1</u> and <u>6 7</u>.3, and then complete the procedure as follows: When the <u>liquid has been distilled or burned off and the</u> crucible appears dry, <u>transfer place</u> the crucible <u>sample</u> and <u>lid to cover</u>, with contents, in a muffle furnace. (To prevent losses from spattering due to rapid burning or volatilization of <u>volatile</u> constituents, the muffle furnace <u>should shall</u> be below 200°C when this transfer is made.) Raise the temperature at the maximum rate to between 750 and 900°C and ignite for 1 h at this temperature. Place the crucible and cover, with contents, in a desiccator to cool. When they have cooled to room temperature, weigh the crucible, cover, and contents (ash) to the nearest 0.1 mg. Then proceed as specified in 6.6.

6.6 Repeat mg.

7.6 Repeat the ignition and cooling until constant weight is obtained.

78. Calculation

78.1 Calculate ash, %, as follows:

² Annual Book of ASTM Standards, Vol 15.05.

ash = $\frac{A - B}{C - B} \times 100$

where:

- A = mass of crucible, cover, and ash,
- B = mass of crucible and cover, and

C = mass of crucible, cover, and sample.

8. Precision and Bias

8.1 The reproducibility for engine coolants should be within ± 20 % of the mean. These precision limits may apply to some types of antirusts.

9. Precision and Bias

<u>9.1 The reproducibility for engine coolants should be within ± 20 % of the mean. These precision limits may apply to some</u> types of antirusts.

10. Keywords

910.1 ash; engine coolant

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(1)