



Designation: D1093 – 11 (Reapproved 2017)

## Standard Test Method for Acidity of Hydrocarbon Liquids and Their Distillation Residues<sup>1</sup>

This standard is issued under the fixed designation D1093; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

### 1. Scope

1.1 This test method covers the qualitative determination of the acidity of hydrocarbon liquids and their distillation residues. (**Warning**—Many hydrocarbon liquids are extremely flammable. Harmful if inhaled. Hydrocarbon liquid vapors can cause a flash fire.)

1.2 If desired to determine the basicity of a hydrocarbon liquid, proceed in accordance with 9.2 or 9.3, but substitute 3 drops of phenolphthalein indicator solution for the methyl orange indicator. A pink or red color in the aqueous solution when phenolphthalein is used indicates basicity.

1.3 The results obtained by this test method are qualitative expressions. However, for the preparation of reagents and in the procedure, acceptable SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety problems, 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.*

1.5 *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:<sup>2</sup>

D86 Test Method for Distillation of Petroleum Products and

Liquid Fuels at Atmospheric Pressure

D91 Test Method for Precipitation Number of Lubricating Oils

D850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials

D1078 Test Method for Distillation Range of Volatile Organic Liquids

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

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *acidity, n*—the quality, state or degree of being acid.

3.1.1.1 *Discussion*—In this test method, the criterion for acidity is a pink or red color when methyl orange indicator is used.

3.1.2 *basicity, n*—the quality, state or degree of being basic.

3.1.2.1 *Discussion*—In this test method, the criterion for basicity is a pink or red color when phenolphthalein indicator is used.

3.1.3 *distillation residue, n*—that portion of the sample remaining after distillation using specified procedures.

### 4. Summary of Test Method

4.1 The sample of distillation residue or hydrocarbon liquid is shaken with water and the aqueous layer tested for acidity to methyl orange indicator.

4.2 The aqueous layer can also be tested for basicity using phenolphthalein indicator.

### 5. Significance and Use

5.1 Some petroleum products are treated with mineral acid as part of the refining procedure. Obviously, any residual mineral acid in a petroleum product is undesirable. The absence of a positive indication in the test for acidity of the distillation residue or aqueous extract of a hydrocarbon liquid is an assurance of the care used in refining the fuel or solvent.

<sup>1</sup> 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 D02.06 on Analysis of Liquid Fuels and Lubricants.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## 6. Apparatus

6.1 *Centrifuge Tube*, cone-shaped, 100 mL capacity; calibration not necessary.

6.2 *Centrifuge*, capable of swinging two centrifuge tubes at 1500 r/min.

NOTE 1—Apparatus used for Test Method D91 is satisfactory.

## 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>3</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1 Commercially prepared solutions may be used when they meet or exceed the specifications set in 7.1.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV Specifications D1193.

7.3 *Ethanol (95 %)* (**Warning**—Flammable, Vapor Harmful, if denatured it cannot be made non-toxic. Either pure grain or denatured with 5 % methanol.

7.4 *Methyl Orange Indicator Solution* (1 g/L)—Prepare a methyl orange indicator aqueous solution equal to a concentration of 1 g/L.

7.4.1 One way to do this is to dissolve 1.0 g  $\pm$  0.1 g of methyl orange in water and dilute to 1 L.

7.5 *Phenolphthalein Indicator Solution*—Dissolve 1.0 g  $\pm$  0.1 g of phenolphthalein in 100 mL of alcohol. Add sodium hydroxide solution (NaOH, 0.8 g/L) dropwise to develop a very faint pink color.

## 8. Sampling

8.1 Using Practice D4057 (manual sampling) or Practice D4177 (automatic sampling) as a guideline, ensure that a sample representative of the material to be tested is obtained.

## 9. Procedure

9.1 All distillation flasks and glassware used in this test shall be clean and dry. There shall be no contaminant present that could affect the acidic or basic nature of the material under test.

<sup>3</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

9.2 *Distillation Residue*—After measuring the percent residue collected from the distillation by Test Method D86, Test Method D850, or Test Method D1078 as required, extract the residue with water as described below. Use either the graduated cylinder containing the residue from the distillation procedure directly or another appropriately sized container, such as a test tube, especially if the volume of water plus residue would make an extraction in the original graduated cylinder impractical. If an alternate container is used, ensure the cooled residue is transferred with minimal delay. Add a volume of water equal to about three times the volume of residue, stopper, and shake the container vigorously for at least 30 s. Ensure there is ample head space in the stoppered container to allow adequate mixing. Allow the liquids to separate and pipet the clean aqueous layer into a second clean small container, such as a test tube. Add 1 drop of methyl orange indicator solution to the container, disperse or mix the indicator solution with the aqueous layer, then observe the color of the aqueous layer.

9.3 *Hydrocarbon Liquid*—Place 50 mL of the sample, 15 mL of water, and 3 drops of methyl orange indicator solution in a clean centrifuge tube. Stopper the tube with a clean stopper (**Warning**—During shaking, vent the centrifuge tube at frequent intervals, especially at the initial 5 s of shaking so that any build-up of vapors or pressure can be released), shake vigorously for 30 s, and centrifuge at 1500 r/min for 10 min. Remove the centrifuge tube and observe the color of the aqueous layer.

9.4 For the determination of basicity use 3 drops of phenolphthalein indicator instead of methyl orange in either 9.2 or 9.3.

## 10. Report

10.1 If the methyl orange indicator is used, report the material as acidic if a pink to red color is observed.

10.2 If the phenolphthalein indicator is used, report the material as basic if a pink to red color appears.

10.3 If neither indicator shows the color changes specified, the material can be reported neutral.

## 11. Precision and Bias

11.1 In case of pass-fail data, or other qualitative tests, no generally accepted method for determining precision or bias is currently available.

## 12. Keywords

12.1 distillation residue acidity; distillation residue basicity; hydrocarbon acidity; hydrocarbon basicity

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