Designation: E3399 – 23

Standard Test Method for Non-volatile Residue in Ethanol and Ethanol Solutions[1]

This standard is issued under the fixed designation E3399; 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 determination of the non-volatile residue content of ethanol and ethanol solutions at the time of test.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.2.1 The accepted SI unit of pressure is the Pascal (Pa); the accepted SI unit for temperature is degrees Celsius.

1.3 **Warning—**Mercury has been designated by many regulatory agencies as a hazardous substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.

1.4 *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.* For specific warning statements, see 6.4, 7.4, and 9.1.

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:*[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

E1 Specification for ASTM Liquid-in-Glass Thermometers

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

2.2 *U.S. Federal Standards*

United States Code of Federal Regulations, Title 27, Part 21 Formulas for Denatured Alcohol and Rum

United States Code of Federal Regulations, Title 27, Part 30.31 Determination of Proof

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *ethanol (ethyl alcohol, grain alcohol), n*—CH_3CH_2OH; can be produced chemically from ethylene or biologically from the fermentation of various sugars from carbohydrates found in agricultural crops and cellulosic residues from crops or wood.

3.1.2 *non-volatile residue, n*—the soluble, suspended, or particulate material remaining following the evaporation of a sample.

4. Summary of Test Method

4.1 When testing ethanol or ethanol solutions, a 100 mL ± 0.5 mL quantity of ethanol or ethanol solution is evaporated under controlled temperature conditions of minimum 100 °C, but not to exceed 180 °C.

5. Significance and Use

5.1 Manufacturers of ethanol are responsible for identifying and controlling impurities according to regulatory standards. Impurities in ethanol that are non-volatile are critical quality attributes for applications in the food, feed and pharmaceutical, personal care applications. Non-volatile residue is an attribute important to users of ethanol for these applications.

6. Apparatus/Materials

6.1 *Balance,* capable of weighing test specimens to the nearest 0.1 mg.

6.2 *Beakers,* of 100 mL capacity. Mark each beaker in the set, including the tare beaker, with an identifying number or letter.

[1] This test method is under the jurisdiction of ASTM Committee E48 on Bioenergy and Industrial Chemicals from Biomass and is the direct responsibility of Subcommittee E48.A0 on Product Specifications.

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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.

6.3 *Cooling Vessel*—A tightly covered vessel, such as a desiccator without desiccant, for cooling the beakers before weighing.

NOTE 1—The use of a desiccant could lead to erroneous results.

6.4 *Evaporation Bath*—(**Warning—**If a liquid-filled evaporation bath is used, care must be taken that the flash point of the liquid used is at least 30 °C higher than the highest bath temperature expected.) Either a solid metal block bath or a liquid bath, electrically heated. Temperature shall be maintained by means to ensure thermostatic controls or by refluxing liquids of suitable composition.

6.5 *Temperature Sensor,* liquid-in-glass thermometer conforming to the requirements in the specification(s) for ASTM 3C/IP73C, found in Specification E1, or another temperature measuring device or system, or both, of at least equivalent accuracy and precision over a temperature range from –5 °C to 400 °C.

6.6 *Graduated Cylinders,* with spout, capable of measuring 100 mL ± 0.5 mL.

6.7 *Handling Equipment,* forceps (stainless steel, spade ended) or tongs (stainless steel) for use in handling the beakers and conical jets, as required by this test method.

7. Procedure

7.1 Wash the beakers thoroughly in a mildly alkaline or neutral pH lab detergent cleaning solution.

7.1.1 The type of detergent and conditions for its use need to be established in each laboratory. The criterion for satisfactory cleaning shall be a matching of the quality of that obtained with chromic acid cleaning solutions on used beakers (fresh chromic acid, 6 h soaking period, rinsing with distilled water and drying). For this comparison visual appearance and mass loss on heating the glassware under test conditions may be used. Detergent cleaning avoids the potential hazards and inconveniences related to handling corrosive chromic acid solutions. The latter remains as the reference cleaning practice and as such may function as an alternate to the preferred procedure-cleaning with detergent solutions.

7.1.2 Remove the beakers from the cleaning solution by means of stainless steel forceps or tongs (see 6.7) and handle only with forceps or tongs thereafter. Wash the beakers thoroughly, first with tap water and then with distilled water, and dry in an oven at 150 °C for at least 1 h. Cool the beakers for at least 2 h in the cooling vessel placed in the vicinity of the balance. Heat the bath to the prescribed operating temperature.

7.2 Weigh the test beakers to the nearest 0.1 mg. Record the masses.

7.3 If suspended or settled solid matter is present, mix or shake the contents of the sample container thoroughly using an appropriate method (see Practice D4057).

7.4 Measure a 100 mL ± 0.5 mL test specimen in a graduated cylinder (see 6.6), and transfer it to a weighed beaker (see 6.2). Use one beaker for each test specimen to be tested and fill each beaker. Place the filled beakers in the evaporation bath, keeping the elapsed time between placing the first and last beaker in the bath to a minimum.

7.5 Allow the test specimen to evaporate to dryness under controlled temperature conditions of minimum 100 °C, but not to exceed 180 °C.

7.6 At the end of the evaporation period, transfer the beakers using forceps, tongs, or other suitable means from the evaporative bath to a drying oven. Dry beakers in 100 °C to 105 °C oven for 1 h ± 15 min.

7.7 At the end of the heating period, transfer the beakers using forceps, tongs, or other suitable means from the oven to the cooling vessel. Place the cooling vessel in the vicinity of the balance for at least 2 h. Weigh the beakers in accordance with 7.2. Record the masses.

8. Calculation

8.1 Calculate the non-volatile residue content of ethanol or ethanol solution as follows:

$$A = 1000(B - D) \quad (1)$$

where:

A = non-volatile residue content, mg/100 mL,
B = mass recorded in 7.7 for the sample beaker plus residue, g, and
D = mass recorded in 7.2 for the empty sample beaker, g.

9. Report

9.1 For ethanol or ethanol solutions with non-volatile residue contents ≥1 mg/100 mL, express the results to the nearest 1 mg/100 mL as non-volatile residue content by Test Method E3399. Round figures in accordance with Practice E29. For results <1 mg/100 mL, report as "<1 mg/100 mL."

10. Precision and Bias

10.1 A new interlaboratory study will be performed for this method per ASTM regulations.

10.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for this procedure, the bias has not been determined.

11. Keywords

11.1 ethanol; ethanol solutions; non-volatile residue