



Designation: D 4094 – 00

Standard Test Method for Acid Content of Ethylene-Acrylic Acid Copolymers¹

This standard is issued under the fixed designation D 4094; 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 approval. 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 acid content of ethylene-acrylic acid (EAA) copolymers containing 2.5 to 25 weight % of acrylic acid.

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

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 8.

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1898 Practice for Sampling of Plastics²

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI): The Modern Metric System⁴

3. Terminology

3.1 Units and symbols used in this test method are those recommended in IEEE/ASTM SI-10. Additional acronyms unique to this test method are defined in the text.

4. Summary of Test Method

4.1 In this test method, a weighed specimen is dissolved in a suitable hot solvent and titrated while hot with standard base to a visual equivalence point.

5. Significance and Use

5.1 EAA copolymers possess unusual chemical and physical properties because they contain free acid groups. Since polymer performance in end-use applications is a function of the amount of copolymerized acrylic acid, it is important that acid contents be determined quantitatively by a suitable method, such as that described herein.

6. Apparatus

6.1 *Balance*, analytical, accurate to 0.0001 g.

6.2 *Buret*, 25-mL, 0.1-mL subdivisions, Class A, polytetrafluoroethylene stopcock.

6.3 *Flask*, Erlenmeyer, 250-mL, female standard-taper joint, with condenser, reflux, matching male standard-taper joint.

6.4 *Stirrer/Hotplate*, magnetic.

6.5 *Stirrer*, magnetic.

6.6 *Stirring Bar*, magnetic, 40 mm (1.5 in.) long, polytetrafluoroethylene-encased.

7. Reagents and Materials

7.1 *Xylene*, reagent-grade.

7.2 *n-Butanol*, reagent-grade.

7.3 *Mixed Solvent*—Mix 3 volumes of xylene with 1 volume of *n*-butanol.

7.4 *Tetrabutylammonium Hydroxide (TBAH)*, solution in methanol or benzene, in 1 M concentration.

7.5 *Standard Base* (0.1 N)—Mix 1 volume of 1 M TBAH solution with 9 volumes of mixed solvent.

7.6 *Benzoic Acid*, primary standard.

7.7 *Thymol Blue (TB) Indicator (Formula Weight of 466.58)*—Prepare 0.5 % solution by weighing 0.125 g of reagent-grade TB (acid form) into a small beaker, adding 25 mL of mixed solvent, and adding 0.268 milliequivalents of 0.1 N base to form a clear, red-orange solution. Transfer to a glass dropping bottle.

8. Hazards

8.1 Solvents and titrants are malodorous and flammable and can cause burns to skin, eyes, and lungs. Wear proper body and eye protection when handling these materials and conduct all operations in a fume hood from which all possible sources of ignition have been removed.

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.11).

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² Discontinued. See 1998 *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 14.04.

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