



Designation: E 1616 – 94

Standard Test Method for Analysis of Acetic Anhydride Using Gas Chromatography¹

This standard is issued under the fixed designation E 1616; 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 describes the determination of assay and impurities in acetic anhydride by gas chromatography. The acetic anhydride should be at least 95 % pure.

1.2 *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.* For specific hazards statements see Section 9.

2. Referenced Documents

2.1 ASTM Standards:

E 355 Practice for Gas Chromatography Terms and Relationships²

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Practice E 355.

4. Summary of Test Method

4.1 The sample is injected onto a gas chromatographic column. The components are separated as they pass through the column with helium carrier gas, their presence in the effluent is detected by a flame ionization detector, and recorded as a chromatogram. The concentrations of sample components are calculated as weight percentages using area normalization.

5. Significance and Use

5.1 This test method provides for the determination of assay and impurities in acetic anhydride. Acetic anhydride is used as an intermediate in many chemical processes. Its relative concentration affects the efficiency of these processes. This test method provides a test procedure for assay and impurity specification acceptance as well as manufacturing control.

6. Interferences

6.1 This test method describes the chromatographic separation of possible impurities in acetic anhydride. Other impurities could coelute with the known components and cause an

interference. Therefore, an efficient gas chromatographic column in good condition should be used in order to ensure the necessary chromatographic separations and proper peak shapes so that good quantitative data may be obtained.

6.2 This analysis is based upon the assumption that all components are eluted from the gas chromatographic column.

7. Apparatus

7.1 *Gas Chromatograph*—Any gas chromatograph equipped with a flame ionization detector and a split injection system for use with capillary columns that can be operated at the conditions given in Table 1.

7.2 *Column*—The column must give satisfactory resolution and proper peak shapes for the components listed in Fig. 1. Table 1 contains a description of a column that has been found satisfactory.

7.3 *Recorder/Integrator*—Electronic integration is recommended for this analysis.

7.4 *Syringe*, 10-μL capacity.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.³ 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.

8.2 *High-Purity Acetic Anhydride (99.8 % or Greater Purity)*—It is important that the acetic anhydride used to prepare calibration standards be as pure as possible. If the acetic anhydride specified by this test method is not suitable for standard preparation, it should be redistilled prior to use or another source found. Note that American Chemical Society reagent grade acetic anhydride may not be sufficiently pure for this procedure. Its titration assay will include other anhydrides present as well as acetic anhydride.

8.3 *Carrier Gas*—Chromatographic-grade helium.

¹ This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.02 on Product Standards.

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² *Annual Book of ASTM Standards*, Vol 14.02.

³ *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.