



Designation: E222 – 17

Standard Test Methods for Hydroxyl Groups Using Acetic Anhydride Acetylation¹

This standard is issued under the fixed designation E222; 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 These test methods cover the determination of hydroxyl groups attached to primary and secondary carbon atoms in aliphatic and alicyclic compounds and phenols.

1.2 Three test methods are given as follows:

	Sections
Test Method A (Pressure Bottle Method)	8 – 14
Test Method B (Reflux Method)	16 – 22
Test Method C (Perchloric Acid Catalyzed Method)	24 – 30

1.2.1 Test Method A is recommended for general use. Test Method B is included to give a standard procedure for the method that has been used widely. Test Method C is recommended when the results are required in a minimum period of time or where ambient temperature for the reaction is desired.

1.2.2 The results obtained using Test Methods A and B will be essentially the same, but the results obtained using Test Method C will be higher (up to approximately 4 % relative) than those obtained using the other two methods.

1.2.3 Statements on precision are included with each test method. The precision of Test Methods A and C is consistent over a wide range of hydroxyl content (tested over hydroxyl number range of 250 to 1600), whereas Test Method B is less precise at the higher hydroxyl content level than it is at the lower hydroxyl content level. In general, Test Method A is approximately two-fold as precise as Test Method C. Test Method B has approximately the same precision as Test Method C at the lower hydroxyl content level but poorer precision at the higher hydroxyl content level.

1.2.4 The interferences are essentially the same for the three methods. Some compounds can be analyzed using Test Methods A or B but not using Test Method C because of interfering reactions of the strong acid catalyst with the compound being analyzed or the acetate product formed in the determination. However, because of its increased reactivity, Test Method C is applicable for determination of some compounds, particularly sterically hindered secondary alcohols, which react too slowly or not at all in Test Methods A and B.

NOTE 1—Other methods for determination of hydroxyl groups are given in Test Methods [D1957](#), [D2195](#), [E326](#), [E335](#), and [E567](#).

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

1.4 Review the current appropriate Safety Data Sheets (SDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

1.5 *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 7.

1.6 *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

[D1193 Specification for Reagent Water](#)

[D1957 Test Method for Hydroxyl Value of Fatty Oils and Acids](#) (Withdrawn 2007)³

[D2195 Test Methods for Pentaerythritol](#) (Withdrawn 2011)³

[D6809 Guide for Quality Control and Quality Assurance Procedures for Aromatic Hydrocarbons and Related Materials](#)

[E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals](#) (Withdrawn 2009)³

[E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis](#)

[E203 Test Method for Water Using Volumetric Karl Fischer Titration](#)

¹ These test methods are under the jurisdiction of ASTM Committee [D16](#) on Aromatic, Industrial, Specialty and Related Chemicals and are the direct responsibility of Subcommittee [D16.15](#) on Industrial and Specialty General Standards.

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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.

³ The last approved version of this historical standard is referenced on www.astm.org.

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