



Standard Test Method for Determination of Percent Hydroxyl on Cellulose Esters by Potentiometric Titration—Alternative Method¹

This standard is issued under the fixed designation D5897; 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 a procedure for determining the percent hydroxyl on cellulose esters by potentiometric titration. The typical range of percent hydroxyl measured is 0.7 to 10.0 %.

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

1.3 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D817 Test Methods of Testing Cellulose Acetate Propionate and Cellulose Acetate Butyrate

D871 Test Methods of Testing Cellulose Acetate

3. Summary of Test Method

3.1 The cellulose ester is dissolved in pyridine and the hydroxyl sites on the cellulose ester are acetylated with acetic anhydride in the presence of basic catalyst, 1-methylimidazole. The excess acetic anhydride is hydrolyzed and the resulting acetic acid is titrated with sodium hydroxide. An automatic titrator dispenses the titrant, potentiometrically determines the

endpoint, and calculates the percent hydroxyl on the cellulose ester based on a blank determination.

4. Significance and Use

4.1 This test method provides a simpler means for the determination of the hydroxyl content of cellulose esters than the preparation and measurement of the carbanilate derivative described in Test Methods **D817** and **D871**.

4.2 The hydroxyl content is an important indicator of solubility and reactivity.

5. Interferences

5.1 Undissolved ester may accumulate on the sides of the flask and on top of the stirring-star during dissolution, leading to low results. Gently swirling the solution during titration can reduce this problem.

5.2 The ground glass joints of the flask and the air condenser must always be rinsed into the flask with hydrolyzing solution at the point of hydrolysis and before titration. This will prevent erroneous results from material that may have refluxed into the joint.

6. Apparatus

6.1 *Titrator*, equipped with *Glass Electrode*.

6.2 *Heating/Stirring Module*, six-place.

6.3 *Heating/Stirring Block*, cut from polished-finish aluminum block to fit stirrer in 6.2 (see Fig. 1 for dimensions).

6.4 *Stirrer*, six place.

6.5 *Magnetic Stirrers*, size 25 mm and 50 mm.

6.6 *Stirring Bar*.

6.7 *Flask and Air Condenser*, (see Fig. 2 for dimensions).

6.8 *Bottle-Top Dispensers*, capable of dispensing 20 mL, 35 mL, and 50 mL, or equivalent.

6.9 *Analytical Balance*, capable of weighing 250 g to the fourth decimal place.

¹ This test method is under the jurisdiction of ASTM Committee **D01** on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee **D01.36** on Cellulose and Cellulose Derivatives.

Current edition approved June 1, 2013. Published June 2013. Originally approved in 1996. Last previous edition approved in 2007 as D5897 – 96 (2007). DOI: 10.1520/D5897-13.

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.