



Designation: D 1396 – 92 (Reapproved 1998)

Standard Test Methods for Chemical Analysis of Poly(Vinyl Butyral)¹

This standard is issued under the fixed designation D 1396; 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 methods cover procedures for the determination of poly(vinyl alcohol), poly(vinyl acetate), and butyraldehyde in poly(vinyl butyral).

1.2 The procedures appear in the following order:

	Sections
Poly(vinyl alcohol)	4
Poly(vinyl acetate)	5
Butyraldehyde	6

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:*

D 1193 Specification for Reagent Water²

3. Reagents

3.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall 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.

3.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

Current edition approved May 15, 1992. Published July 1992. Originally published as D 1396 – 56 T. Last previous edition D 1396 – 73 (1987)^{ε1}.

² *Annual Book of ASTM Standards*, Vol 11.01.

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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

4. Poly(Vinyl Alcohol)

4.1 *Reagents:*

4.1.1 *Ethylene Dichloride*, technical grade.

4.1.2 *Phenolphthalein Indicator Solution* (10 g/L)—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %), methanol, or isopropanol.

4.1.3 *Potassium Hydroxide, Standard Alcoholic Solution* (0.5 N)—Dissolve 33 g of KOH in methanol and dilute to 1 L. Standardize against potassium acid phthalate using phenolphthalein indicator solution.

4.1.4 *Pyridine Acetic Anhydride Reagent*—Mix slowly 1000 mL of pyridine and 87 mL of acetic anhydride. Make only about a week's supply, and keep it in a brown bottle.

4.2 *Procedure:*

4.2.1 Transfer 2.2 g of the dry sample to a clean, dry, 500-mL glass-stoppered flask. Add 25.0 mL of pyridine-acetic anhydride reagent. Insert the stopper, and heat the flask on a sand or steam bath below the boiling point for 5½ h. Swirl gently until the sample is completely dissolved. Vent the flask occasionally during the first part of the heating period to prevent the stopper from blowing out.

4.2.2 At the end of the 5½-h period, add 25 mL of ethylene dichloride and shake well. Add 100 mL of water, and shake vigorously immediately after adding the water. Let the flask stand for ½ h.

4.2.3 Add a few drops of phenolphthalein solution and titrate with 0.5 N alcoholic KOH solution. Shake vigorously during the titration.

4.2.4 *Blank*—Run a blank determination on the reagents, following the same procedure as for the sample.

4.3 *Calculation*—Calculate the percentage of poly(vinyl alcohol) as follows:

$$\text{Poly(vinyl alcohol), \%} = [(B - V) N \times 4.4] / S \quad (1)$$

where:

B = KOH solution required for titration of the blank, mL,

V = KOH solution required for titration of the sample, mL,

N = normality of the KOH solution, and

S = specimen weight, g.

5. Poly(Vinyl Acetate)

5.1 *Apparatus:*