



Standard Test Method for Polyhydric Alcohols in Alkyd Resins¹

This standard is issued under the fixed designation D 2998; 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.

ε¹ NOTE—Editorial changes were made throughout in November 1996.

1. Scope

1.1 This test method covers the qualitative and quantitative determination of the polyols in alkyl resins, including resin- and polymer-modified alkyds. Quantities as low as 0.5 % may be detected and measured.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses 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.* For a specific hazard statement, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 260 Practice for Packed Column Gas Chromatography³

3. Summary of Test Method

3.1 The resin sample is dried, subjected to aminolysis to release the polyols, treated directly to form the trimethylsilyl (TMS) ether derivatives of the polyhydric alcohols which are separated by gas-liquid chromatography. An internal standard is used for quantitative purposes. The description of apparatus and the recommended operating conditions are presented as a guide to a satisfactory analysis as all suitable variations in temperatures, column lengths, substrates, liquid phases, programming rates, etc., have not been determined.

4. Significance and Use

4.1 This test method is designed to identify polyhydric alcohols and to quantify amounts of polyols contained in alkyl resins. Polyols are significant ingredients in alkyl resins and impact on such important physical parameters of alkyd paints as weatherability, flexibility, moisture resistance, and adhesion.

Use of this test method will provide a means whereby the relative applicability of the alkyd resin to the particular end use may be estimated by the buyer and the seller.

5. Apparatus

5.1 *Linear Programmed Temperature Gas Chromatograph*—Any instrument with a thermal conductivity detector and with programming features should be suitable if adaptable to the following recommended operating conditions:

Detector cell temperature, °C	300
Detector cell current, mA	160
Injection port temperature, °C	300
Helium flow at exit, cm ³ /min	80
Programmed temperature details:	
Approximate column heating rate, °C/min	6
Starting column temperature, °C	100
Finishing column temperature, °C	300

NOTE 1—Flame ionization detectors are not recommended for this test as the reaction medium can contaminate the flame jet.

5.2 *Column*—A 16-ft (4.9-m) length of ¼-in. (6.4-mm) outside diameter copper tubing packed with 20 weight percent of silicone grease on 60 to 80-mesh white flux-calcined diatomaceous earth,⁴ and conditioned at 315°C until “bleeding” reaches a minimum.

5.3 *Column and Instrument Performance*—Note the separation of closely adjacent polyol peaks and express as peak resolution *R* as follows:

$$R = 2Y/(A + B) \quad (1)$$

where:

Y = distance between maxima of closely adjacent peaks,

A = base width of first peak *A*, and

B = base width of peak *B*.

NOTE 2—If the peak resolution is equal to or greater than 1.0, the column and instrument are in satisfactory condition.

5.4 *Syringe*, having a fixed needle, 50-μL capacity.

5.5 *Erlenmeyer Flask*, 125-mL, with standard joint and water-cooled condenser to fit.

5.6 *Micro Test Tube* or screw cap vial.

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

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

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

⁴ The sole source of supply of the flux-calcined diatomaceous earth, Chromosorb W known to the committee at this time is Celite Corp., P. O. Box 5108, Denver, CO 80217-5108. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.