



Standard Test Method for Identification of Carboxylic Acids in Alkyd Resins¹

This standard is issued under the fixed designation D 2455; 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 determination of the carboxylic acids in alkyd resins, including resin-modified alkyds. It may be used for analyzing polyesters but additional peaks may appear from monomers such as styrene.

1.2 The constituents of three resin samples have been correctly identified in collaborative work by three laboratories. It is apparent that quantities as low as 1 % can readily be detected.

1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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

D 2245 Test Method for Identification of Oils and Oil Acids in Solvent-Reducible Paints³

3. Summary of Test Method

3.1 The resin specimen is subjected to rapid transesterification with lithium methoxide to form methyl esters that are separated by programmed temperature gas-liquid chromatography on polar and nonpolar columns, then identified by their retention relative to a standard. If the presence of maleic or fumaric acids is indicated, an alternative transesterification with boron trifluoride is necessary.

4. Significance and Use

4.1 The presence of carboxylic acids in alkyd resins has a direct effect on the crosslinking capability of the resin. This test

method helps the user determine the reactivity of the resin.

5. Apparatus

5.1 *Linear Programmed Temperature Gas Chromatograph*—Any instrument with programming features should be suitable if equipped with a hot-wire type of detector and adaptable to the following operating conditions:

Detector cell temperature, °C	300
Detector cell current, mA	150
Injection port temperature, °C	330
Helium flow at exit, cm ³ /min	85
Programmed temperature details:	
Approximate column heating rate, °/min	4.0
Starting column temperature, °C:	
Polyester-Carbowax 20M	125
Silicone grease, DC-11	75
Finishing column temperature, °C:	
Polyester-Carbowax 20M	225
Silicone grease, DC-11	250

5.2 Columns:

5.2.1 *Polar*—Bend a 6-ft (1.8-m) length of ¼-in. (6.4-mm) copper tubing into a U-shape and fill one side with 20 % polyethylene glycol 20M⁴ on acid-washed 60 to 80-mesh white, flux-calcined diatomite, (USP code S1A)⁵ and fill the other side with 20 % diethylene glycol succinate on the same solid support. Maintain constant vibration during the filling and add small amounts of the packing material alternately to each side. Condition at 225°C until “bleeding” reaches a minimum. Mount this column so that the sample passes first through the polyester section. Two 3-ft (0.9-m) sections, packed separately and joined together may be used if preferred.

5.2.2 *Nonpolar*—Pack a 6-ft (1.8-m) length of ¼-in. (6.4-mm) copper tubing with 20 % silicone grease on acid-washed 60 to 80-mesh Chromosorb W.⁵ Condition at 250°C until “bleeding” reaches a minimum.

5.3 *Syringe*, having a fixed needle, 10-μL capacity.

5.4 *Separatory Funnel*, 250 mL.

⁴ The sole source of supply of the polyethylene glycol, Carbowax 20M known to the committee at this time is Union Carbide Corp., 39 Old Ridgebury Rd., Danbury, CT 06817. 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.

⁵ The sole source of supply of white, flux-calcined diatomite, 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.

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 06.01.