



Standard Test Method for Silicon Content of Silicone Polymers and Silicone-Modified Alkyds by Atomic Absorption¹

This standard is issued under the fixed designation D 3733; 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 the determination of the silicon content of silicone polymers and silicone modified alkyds when present in the nonvolatile portion of polymers, resins, or liquid coatings to the extent of 1 % or more.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 1153 Specification for Methyl Isobutyl Ketone²
- D 1193 Specification for Reagent Water³
- D 2372 Practice for Separation of Vehicle from Solvent-Reducible Paints⁴
- D 2698 Test Method for Determination of the Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging⁴
- D 2832 Guide for Determining Volatile and Nonvolatile Content of Paint and Related Coatings⁴

3. Significance and Use

3.1 The silicon content of silicone modified alkyds has a direct relationship to the cost and performance characteristics, especially heat resistance of coatings prepared from them.

4. Summary of Test Method

4.1 The polymer solution or separated coating vehicle is diluted with methyl isobutyl ketone, and the silicon content is determined by atomic absorption spectroscopy.

5. Apparatus

5.1 *Atomic Absorption Spectrophotometer*, consisting of an atomizer and a single-slot burner; gas pressure-regulating and -metering devices for nitrous oxide (N₂O) and acetylene; a silicon hollow-cathode lamp with a regulated constant current supply; a monochromator and associated optics; a photosensitive detector connected to an electronic amplifier; and a read-out device.

5.2 *Centrifuge*.

5.3 *Pipets*, 5, 10, 15, and 20-mL capacity.

5.4 *Volumetric Flasks*, 25, 50, 100, and 1000-mL.

6. Reagents

6.1 *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.⁵ 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.

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

6.3 *Octaphenylcyclotetrasiloxane*.⁶

6.4 *Methyl Isobutyl Ketone* (MIBK), complying with Specification D 1153.

6.5 *Silicon, Standard Stock Solution* (100 μg/mL)—Weigh exactly 0.709 g of octaphenylcyclotetrasiloxane and quantitatively transfer to a 1-L flask. Dilute to volume with MIBK.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Material, and Applications and is the direct responsibility of Subcommittee D 01.33 on Polymers and Resins.

Current edition approved April 15, 1993. Published June 1993. Originally published as D 3733 – 78. Last previous edition D 3733 – 78 (1984)^{ε1}.

² *Annual Book of ASTM Standards*, Vol 06.04.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ *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.

⁶ Available as Standard Reference Material No. 1066a from Office of Standard Reference Materials, Room B-314, Chemistry Building, National Institute of Standards and Technology, Washington, DC 20234.