



Standard Test Method for Permanganate Time of Tricresyl Phosphate¹

This standard is issued under the fixed designation D 1721; 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 approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the detection in tricresyl phosphate of the presence of impurities that reduce potassium permanganate.

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.* For specific hazard statements, see Section 6.

1.4 For hazard information and guidance, see the supplier's Material Safety Data Sheet.

2. Referenced Documents

2.1 *ASTM Standards:*²

D 1193 Specification for Reagent Water

3. Significance and Use

3.1 Impurities such as phenols, if present in tricresyl phosphate, will react with potassium permanganate, reducing it to manganese dioxide. In the permanganate test, the color of the test solution is observed at the end of a 30-min period, and if the pink color is still present, the sample is considered substantially free of oxidizable impurities.

3.2 The results of this measurement can be used for specification acceptance.

4. Apparatus

4.1 *Graduated Cylinders*, glass-stoppered, 100-mL capacity.

5. Reagents

5.1 *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.

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

5.3 *Potassium Permanganate Solution (0.316 g/L)*—Dissolve 0.316 g of potassium permanganate (KMnO_4) in water and dilute to 1 L with freshly boiled water (Note 1). The solution should be stored in brown bottles and be freshly prepared weekly as required.

NOTE 1—Clean glassware is essential to the stability of the KMnO_4 solution. Clean graduated cylinders and permanganate storage and handling equipment with concentrated hydrochloric acid (HCl, sp gr 1.19) to remove residual manganese oxide (MnO_2) which catalyzes reduction of KMnO_4 . Remove the acid with not less than ten rinsings with water.

6. Hazards

6.1 Tricresyl phosphate is hazardous through inhalation or skin absorption. Take care in handling the material. Avoid eye and skin contact and inhalation of vapors.

6.2 Ortho-isomer of tricresyl phosphate is considered toxic. Trace amounts may be present in tricresyl phosphate specimens.

7. Procedure

7.1 Weigh 10 ± 0.1 g of the sample to be tested into a 100-mL glass-stoppered graduated cylinder. To this, add 50 mL of the KMnO_4 solution. Vigorously shake the mixture for 2

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 D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

Current edition approved June 1, 2007. Published June 2007. Originally approved in 1960. Last previous edition approved in 2001 as D 1721 – 97 (2001).

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.

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.

min, noting the time when the shaking begins. Allow the test to stand for a period of 28 min following the shaking period and observe the color.

8. Report

8.1 If the color of the permanganate solution is still pink, report the sample as “passing.” If no pink color is present, report the sample as “not passing.”

9. Precision and Bias

9.1 *Precision*—No statement is made about the precision of assessing this test method, since the results of the test method are pass-fail.

9.2 *Bias*—Bias cannot be determined for this test method because there is no available material having an accepted reference value.

10. Keywords

10.1 permanganate time; tricresyl phosphate

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).