

Standard Test Method for Permanganate Time of Tricresyl Phosphate¹

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1. Scope

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

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 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.

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

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

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). 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 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.”

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.

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

³ “*Reagent Chemicals, American Chemical Society Specifications*,” Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see “*Analar Standards for Laboratory U.K., Chemicals*,” BDH Ltd., Poole, Dorset, U.K., and the “*United States Pharmacopeia, and National Formulary*,” U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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

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