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Standard Test Method for Permanganate Time of Acetone and Methanol[1]

This standard is issued under the fixed designation D 1363; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the detection in acetone and methanol 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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 329 Specification for Acetone[2]
D 1152 Specification for Methanol (Methyl Alcohol)[2]
D 1193 Specification for Reagent Water[3]
D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)[2]
E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[4]
E 346 Test Method for Analysis of Methanol[4]

3. Summary of Test Method

3.1 Substances reacting with potassium permanganate in neutral solutions reduce it to manganese dioxide which colors the solution yellow. In the permanganate test the time required for the color of the test solution to change to that of a standard solution is measured. The color of the test solution changes from pink-orange to yellow-orange.

4. Significance and Use

4.1 The permanganate time can be used to judge the presence of oxidizable materials that may be associated with manufacture or contamination during distribution and to assess compliance with a specification.

4.2 Many chemical processes that use acetone or methanol, or both, involve catalyst, metals, or ligand complexes that are sensitive to oxidation. Since oxidizable contaminants may affect the efficiency of these processes, this test method provides a comparative test for manufacturing control and assessing compliance with a specification.

5. Apparatus

5.1 *Color Comparison Tubes*—Matched 50-mL, tall form Nessler tubes, provided with ground on, optically clear, glass caps.

5.2 *Constant-Temperature Bath*, capable of maintaining a temperature of 15.0 ± 0.5°C or of 25.0 ± 0.5°C. It is important that the constant-temperature bath be protected from direct light. If a glass constant-temperature bath is employed, it should be wrapped or coated with an opaque material.

5.3 *Pipet*, capable of delivering 2.0 mL of solution.

5.4 *Interval Timer and Clock*, capable of measuring a time interval of 120 min or more. An alarm arrangement may be desirable.

6. Reagents

6.1 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 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D 1193.

6.3 *Potassium Permanganate Solution* (0.200 g/L)—Dissolve 0.200 g of potassium permanganate ($KMnO_4$) and dilute to 1 L with freshly boiled water. Clean glassware is essential to the stability of this solution. The solution should be stored in brown bottles and be prepared every week needed.

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

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[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 15.05.

[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.

6.4 *Cobaltous Chloride-Platinum Cobalt Standard Solution*—Weigh on analytical balance 175 mg of cobaltous chloride ($CoCl_2 \cdot 6H_2O$) and add 21.4 mL of 500 Pt-Co standard (Note 1). Transfer to a 50-mL volumetric flask, dilute to the mark, and mix thoroughly. This standard solution represents the color of the end point to which the sample solution fades in the $KMnO_4$ test. The solution is stable and should be kept in a 50-mL glass-stoppered Nessler tube exactly the same as those in which the test is run.

NOTE 1—The preparation of the 500 Pt-Co standard is covered in Test Method D 1209.

7. Procedure

7.1 Employ the following time and temperature conditions during the test:

	Temperature of Test, °C	Permanganate Time, min
Acetone	25	30
Methanol	15	50

NOTE 2—It is advisable to check specifications D329, D1152 and Test Method E 346 for possible changes in acetone and methanol specifications and permanganate time test conditions.

NOTE 3—Clean test cylinders and permanganate storage and handling equipment with concentrated hydrochloric acid (HCl, sp gr 1.19) to remove residual manganese dioxide (MnO_2) which catalyzes reduction of $KMnO_4$. Remove the acid with not less than ten rinsings with reagent water.

7.2 Fill a 50-mL Nessler tube beyond the mark with the sample under test and place in the constant-temperature bath. Maintain the water level in the bath approximately 25 mm (1 in.) below the top of the tube. When the specimen has reached the specified temperature, bring the level to the 50-mL mark. With a pipet, add 2 mL of the $KMnO_4$ solution. Stopper the tube, invert once to mix the contents, return to the bath and note the time. At the end of the minimum time specified for the material being tested, remove the tube and compare it to the color standard by viewing downward through the tube against a white background from which diffused white light is reflected.

8. Report

8.1 Report the following information:

8.1.1 If the residual pink color of the specimen is greater than the standard, report the permanganate time as "greater than *X* minutes." If the residual pink color of the specimen is equal to that of the standard, report the permanganate time as "*X* minutes." If the residual pink color of the specimen is less than the standard, report as "less than *X* minutes," where "*X* minutes" is the minimum time specified for the material being tested (see 7.1).

8.1.2 An estimate of the actual permanganate time may be made by closely observing the sample and reporting to the nearest minute the time when the color of the specimen matches that of the standard. Duplicate determinations that agree within 3.0 % are suitable for averaging (see Section 9).

9. Precision and Bias [6]

9.1 *Precision*—These precision statements are based on an interlaboratory study in which three samples of methanol having average permanganate times of 60, 73, and 95 min were each analyzed by fourteen different laboratories in duplicate on two different days. In this interlaboratory study, the within-laboratory coefficient of variation was found to be 2.35 % with 40 df and the between-laboratories coefficient of variation 8.20 % with 12 df. Based upon these coefficients calculated in accordance with Practice E 180, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.2 *Repeatability*—Two results, each the mean of duplicates, obtained by the same operator on different days should be considered suspect if they differ by more than 6.7 %.

9.3 *Reproducibility*—Two results, each the mean of duplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 25.2 %.

9.4 *Bias*—Bias cannot be determined because there is no available material having an accepted reference value.

10. Keywords

10.1 acetone; methanol; permanganate time

[6] Supporting data are available from ASTM Headquarters. Request RR:D01-1033.

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