



Standard Test Method for Peroxide Number of Petroleum Wax¹

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1. Scope

1.1 This test method covers the determination of the peroxide number of petroleum wax.

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.*

2. Terminology

2.1 *Definition:*

2.1.1 *peroxide number*—the milliequivalents of constituents per 1000 g of wax that will oxidize potassium iodide.

3. Summary of Test Method

3.1 A quantity of sample is dissolved in carbon tetrachloride and acidified with acetic acid solution. A solution of potassium iodide is added and, after a reaction period, the solution is titrated with sodium thiosulfate solution to the end point indicated by the color change of added starch solution.

4. Significance and Use

4.1 The magnitude of the peroxide number is an indication of the quantity of oxidizing constituents present. Deterioration of petroleum wax results in the formation of peroxides and other oxygen-carrying compounds. The peroxide number measures those compounds that will oxidize potassium iodide.

5. Apparatus

5.1 *Iodine Flask*, borosilicate glass, 250-mL capacity, glass-stoppered.

6. Reagents and Materials

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.² 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 distilled water or water of equal purity.

6.3 *Acetic Acid Solution*—Mix 4 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) with 996 mL of ACS special reagent grade glacial acetic acid (CH₃CO₂H). The acetic acid must pass the ½ h test for substances reducing K₂Cr₂O₇.

6.4 *Carbon Tetrachloride* (CCl₄).

6.5 *Potassium Dichromate, Standard Solution* (0.1 N)—Recrystallize potassium dichromate (K₂Cr₂O₇) twice from an aqueous solution and dry at about 325°F (164°C) to constant weight. Dissolve 2.452 g of the purified K₂Cr₂O₇ in water and dilute to 500 mL in a volumetric flask.

6.6 *Potassium Dichromate, Standard Solution* (0.01 N)—Dilute 100 mL of 0.1 N K₂Cr₂O₇ solution with water to 1000 mL in a volumetric flask.

6.7 *Potassium Iodide Solution*—Dissolve 120 g of potassium iodide (KI) in 100 mL of water. Discharge any color from this solution as follows: put 1 mL of KI solution, 50 mL of water, and 5 mL of starch solution in a 300-mL flask and blanket with nitrogen or carbon dioxide. If a blue color develops, add 0.005 N Na₂S₂O₃ solution from a microburet until color just disappears. Calculate and add sufficient Na₂S₂O₃ solution to the main KI solution to convert all free iodine to iodide. When starch solution is added to 1 mL of KI solution a blue color should not develop, but upon the addition of 1 drop of 0.01 N K₂Cr₂O₇ solution and 2 drops of concentrated hydrochloric acid (HCl, sp gr 1.19), a blue color should develop. Store this solution under chloroform by adding a few millilitres to the surface of the liquid.

6.8 *Sodium Thiosulfate, Standard Solution* (0.1 N)—Dissolve 12.5 g of sodium thiosulfate (Na₂S₂O₃·5H₂O) and 0.1 g of sodium carbonate (Na₂CO₃) in 500 mL of water. Let stand

¹ This test method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.10 on Properties of Petroleum Wax.

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² *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.

