Designation: D612 – 88 (Reapproved 2017)

Standard Test Method for Carbonizable Substances in Paraffin Wax[1]

This standard is issued under the fixed designation D612; 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 U.S. Department of Defense.

1. Scope

1.1 This test method covers determination of carbonizable substances in paraffin wax. The test method is applicable to paraffin wax for pharmaceutical use, as defined by the U.S. National Formulary, with a melting point as determined in accordance with Test Method D87, between 117 °F and 149 °F (47 °C and 65 °C).

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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.* Specific warning statements are given in Annex A1.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D87 Test Method for Melting Point of Petroleum Wax (Cooling Curve)

D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 Five millilitres of melted wax are treated with 5 mL of concentrated, nitrogen-free sulfuric acid at 158 °F (70 °C). The color of the acid layer is compared with that of a colorimetric reference standard. If the color is not darker than the standard, the wax is reported as passing the test.

4. Significance and Use

4.1 This test method is a means for ascertaining whether pharmaceutical paraffin wax conforms to the standards for quality prescribed by the U.S. National Formulary.

5. Apparatus

5.1 *Test Tube,* as shown in Fig. 1, of heat-resistant glass (see Note 1) fitted with a well-ground glass stopper, the stopper and the tube bearing identical and indestructible numbers. The tube shall be 140 mm ± 2 mm in length and between 14.5 mm and 15.0 mm in outside diameter, and shall be calibrated at the 5 mL ± 0.2 mL and 10 mL ± 0.2 mL liquid levels. The capacity of the tube with stopper inserted shall be between 13.6 mL and 15.6 mL. A rolled edge may be provided for suspending the tube on the cover of the water bath.

NOTE 1—Borosilicate glass has been found satisfactory for this purpose.

5.2 *Water Bath,* suitable for immersing the test tube above the 10 mL line and equipped to maintain a temperature of 158 °F ± 1.0 °F (70 °C ± 0.5 °C). The bath shall be provided with a cover of any suitable material, with holes approximately 16 mm in diameter through which the test tubes may be suspended.

5.3 *Color Comparator,* of a suitable type for observing the color of the acid layer in comparison with the reference standard color solution. The size and shape of the comparator are optional, but the size and shape of the apertures shall conform to the dimensions prescribed in Fig. 1.

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,

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.10 on Properties of Petroleum Waxes and Alternative Wax-like Materials.

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[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.

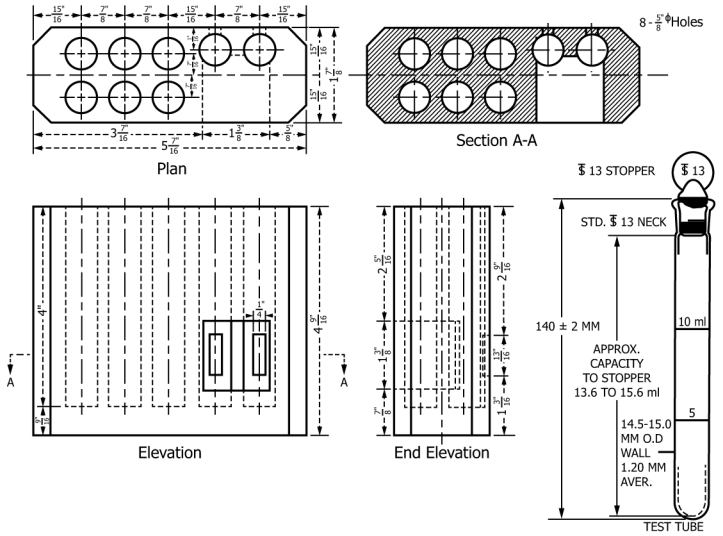

FIG. 1 Color Comparator for Carbonizable Substances in Liquid Petrolatum

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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water, such as reagent water corresponding to Specification D1193, Type III, or water of equal purity.

6.3 *Cobaltous Chloride Solution* (0.5 *N*)—Dissolve about 65 g of cobaltous chloride ($CoCl_2 \cdot 6H_2O$) in enough diluted hydrochloric acid (HCl, 1 + 39) (**Warning—**Hydrochloric acid (concentrated) causes burns, vapor extremely irritating. See A1.2) to make 1000 mL of solution. Transfer exactly 5 mL of this solution to a flask; add 15 mL of sodium hydroxide (NaOH, 1 + 5) (**Warning—**Sodium hydroxide, corrosive, can cause severe burns or blindness. Evolution of heat produces a violent reaction or eruption upon too rapid a mixture with water. See A1.4), and 5 mL of hydrogen peroxide. Boil for 10 min, cool, and add 2 g of potassium iodide (KI) and 20 mL of sulfuric acid (H_2SO_4, 1 + 4) (**Warning—**Sulfuric acid (concentrated) causes burns, vapor irritating, strong oxidizer. See A1.3). When the precipitate has dissolved, titrate the liberated iodine with 0.1 *N* sodium thiosulfate ($Na_2S_2O_3$) solution using starch solution as an indicator. Each millilitre of 0.1 *N* $Na_2S_2O_3$ solution consumed is equivalent to 0.023799 g of $CoCl_2 \cdot 6H_2O$. Adjust the final volume of $CoCl_2$ solution by the addition of diluted HCl (1 + 39) so that 1 mL contains 59.5 mg of $CoCl_2 \cdot 6H_2O$.

6.4 *Colorimetric Reference Standard Solution*—Prepare a reference standard pale amber solution for color comparison by mixing together 1.5 parts of $CoCl_2$ solution, 3.0 parts of $FeCl_3$ solution, and 0.5 parts of $CuSO_4$ solution. Measure 5 mL of this mixture into a test tube as specified in 3.1. This pale amber reference standard shall then be overlaid with 5 mL of white mineral oil.

6.5 *Cupric Sulfate Solution* (0.5 *N*)—Dissolve about 65 g of cupric sulfate ($CuSO_4 \cdot 5H_2O$) in enough diluted HCl (1 + 39) (**Warning—**Hydrochloric acid (concentrated) causes burns, vapor extremely irritating. See A1.2) to make 1000 mL of solution. Transfer exactly 10 mL of this solution to a flask, add 50 mL of water, 4 mL of acetic acid (**Warning—**Acetic acid (glacial) is corrosive, combustible, vapor irritating. See A1.5), and 3 g of KI. Allow the mixture to stand for 5 min, then titrate the liberated iodine with 0.1 *N* $Na_2S_2O_3$ solution, using starch solution as an indicator. Each millilitre of 0.1 *N* $Na_2S_2O_3$ solution is equivalent to 0.02497 g of $CuSO_4 \cdot 5H_2O$. Adjust the final volume of $CuSO_4$ solution by the addition of diluted HCl (1 + 39) so that 1 mL contains 62.4 mg of $CuSO_4 \cdot 5H_2O$.

6.6 *Ferric Chloride Solution* (0.5*N*)—Dissolve about 55 g of ferric chloride ($FeCl_3 \cdot 6H_2O$) in enough diluted HCl (**Warning—**Hydrochloric acid (concentrated) causes burns, vapor extremely irritating. See A1.2) (1 + 39) to make 1000 mL of solution. Transfer exactly 10 mL of the solution to a flask, add 5 mL of HCl (sp gr 1.19), 25 mL of water, and about 3 g of KI. Stopper and allow the mixture to stand for 5 min. Dilute the mixture with 50 mL of water, and titrate the liberated iodine with 0.1 *N* $Na_2S_2O_3$ solution, using starch solution as an indicator. Each millilitre of 0.1 *N* $Na_2S_2O_3$ solution is equivalent to 0.02703 g of $FeCl_3 \cdot 6H_2O$. Adjust the final volume of the $FeCl_3$ solution by the addition of diluted HCl (1 + 39) so that 1 mL contains 45.0 mg of $FeCl_3 \cdot 6H_2O$.

6.7 *Sulfuric Acid* (94.7 ± 0.2 %)—The sulfuric acid (H_2SO_4) (**Warning—**Sulfuric acid (concentrated) causes burns, vapor irritating, strong oxidizer. See A1.3) shall be nitrogen-free when analyzed in accordance with the following procedure: Dilute a small amount of the acid with an equal volume of water and superimpose 10 mL of the cooled liquid upon diphenylamine solution (1 g of diphenylamine in 100 mL

[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 *Annual 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.