Designation: D 565 – 99

An American National Standard

Standard Test Method for Carbonizable Substances in White Mineral Oil[1]

This standard is issued under the fixed designation D 565; 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.

1. Scope

1.1 This test method covers white mineral oil (Mineral Oil USP and Light Mineral Oil NF) to determine whether it conforms to the standard of quality required for pharmaceutical use as defined by the United States Pharmacopeia and the National Formulary, or the Food and Drug Administration.

1.2 The values stated in SI units are to be regarded as the standard.

1.2.1 The dimensions for the color comparator (see 5.3 and Fig. 1) are excepted for that part of the apparatus.

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 Notes 1-5.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4177 Pratice for Automatic Sampling of Petroleum and Petroleum Products[3]

2.2 *Official Compendia:*[4]

United States Pharmacopeia—Current Edition
Monograph on Mineral Oil
National Formulary—Current Edition
Monograph on Light Mineral Oil

2.3 *Government Document:*[5]

21CFR 172.878 Food and Drug Administration Title

3. Summary of Test Method

3.1 The mineral oil is treated with concentrated sulfuric acid (H_2SO_4) under prescribed conditions and the resulting color is compared with a reference standard to determine whether it passes or fails the test.

4. Significance and Use

4.1 This test method is a means for ascertaining whether pharmaceutical mineral oil conforms to the standards of the United States Pharmacopeia, the National Formulary, and the Food and Drug Administration.

5. Apparatus

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

5.2 *Water Bath*, suitable for immersing the test tube above the 10 mL line equipped to maintain a temperature of 100 ± 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 can 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, where such specifications are available.[6] 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.

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[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricantsand is the direct responsibility of Subcommittee D02.06on Analysis of Lubricants.

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

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[4] Available from U.S. Pharmacopeial Convention, 12601 Twinbrook Parkway, Rockville, MD 20852.

[5] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

[6] *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.

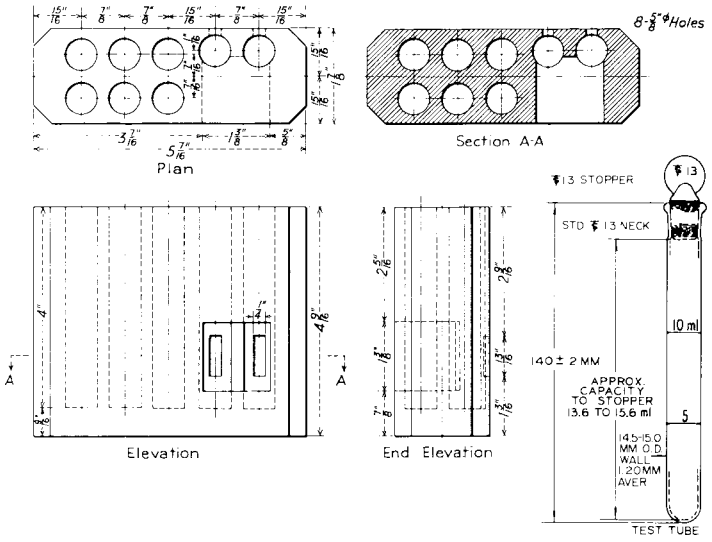

FIG. 1 Color Comparator for Carbonizable Substances in Liquid Petrolatum

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity conforming to Type III of Specification D 1193.

6.3 *Cobaltous Chloride Solution (0.25 M)*—Prepare a solution of hydrochloric acid (HCl) (**Warning**—Causes burns. Vapor extremely irritating.) by mixing 30 mL of concentrated HCl with 1170 mL of water. Slowly add the acid to the water. Dissolve 65 ± 1 g of cobaltous chloride hexahydrate ($CoCl_2 \cdot 6H_2O$) in the HCl solution to make 1000 mL of solution. Using a pipet, transfer 5 mL of this solution to a 250 mL iodine flask. Prepare a solution of sodium hydroxide (NaOH) (**Warning**—Corrosive. Can cause severe burns or blindness. Evolution of heat produces a violent reaction or eruption upon too rapid a mixture with water.) by mixing 5 g of NaOH with 20 mL of water. Add 15 mL of this NaOH solution to the iodine flask. Add 5 mL of hydrogen peroxide (H_2O_2) (3 % v/v). Boil for 10 ± 1 min, cool, and add 2 g of potassium iodide (KI). Prepare a solution of H_2SO_4 (**Warning**—Causes burns. Vapor extremely irritating. Strong oxidizer.) by mixing 6 mL of H_2SO_4 with 18 mL of water. Slowly add the acid to the water (see Note 1). Add 20 mL of this H_2SO_4 solution to the flask.) When the precipitate has dissolved, titrate the liberated iodine with 0.100 *M* sodium thiosulfate ($Na_2S_2O_3$) solution, using starch solution as an indicator. Each millilitre of $Na_2S_2O_3$ solution is equivalent to 0.0238 g of $CoCl_2 \cdot 6H_2O$. Adjust the final volume of $CoCl_2$ solution by the addition of HCl solution so that 1 mL contains 59.5 mg of $CoCl_2 \cdot 6H_2O$.

NOTE 1—This freshly prepared H_2SO_4 solution will be hot. Allow to cool before continuing.

6.4 *Cupric Sulfate Solution (0.25 M)*—Prepare a solution of HCl (**Warning**—see 6.3) by mixing 30 mL of concentrated HCl with 1170 mL of water. Slowly add the acid to the water. Dissolve 65 ± 1 g of cupric sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) in enough HCl solution to make 1000 mL of solution. Using a pipet, transfet 10 mL of the solution to a 250-mL iodine flask, add 40 mL of water. Prepare a 6M acetic acid (CH_3COOH) (**Warning**— Corrosive. Combustible. Vapor irritating.) solution by mixing 353 mL of concentrated CH_3COOH with 1000 mL of water. Slowly add the acid to the water. Add 4 mL of 6M CH_3COOH, 3 g of Kl and 5 mL of HCl to the flask. Titrate the liberated iodine with 0.100 *M* $Na_2S_2O_3$ solution, using starch solution as an indicator. Each millilitre of $Na_2S_2O_3$ solution is equivalent to 0.0250 g of $CuSO_4 \cdot 5H_2O$. Adjust the final volume of the $CuSO_4$ solution by the addition of diluted HCl solution so that 1 mL contains 62.4 mg $CuSO_4 \cdot 5H_2O$.

6.5 *Ferric Chloride Solution (0.166 M)*—Prepare a solution of HCl (**Warning**—see 6.3) by mixing 30 mL of concentrated HCl with 1170 mL of water. Dissolve 55 ± 1 g of ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$) in enough HCl to make 1000 mL of solution. Using a pipet, transfer 10 mL of this solution into a 250-mL iodine flask, add 15 mL water, 3 g KI and 5 mL HCl, and allow the mixture to stand for 15 ± 1 min. Dilute with 100 mL of water and titrate the liberated iodine with 0.100 *M* $Na_2S_2O_3$ solution, using starch solution as an indicator. Each millilitre of $Na_2S_2O_3$ solution is equivalent to 0.0270 g of $FeCl_3 \cdot 6H_2O$. Adjust the final volume of $FeCl_3$ solution by the addition of the HCl solution so that 1 mL contains 45.0 mg of $FeCl_3 \cdot 6H_2O$.

6.6 The solutions prepared in 6.3-6.5 may be prepared in different quantities, provided the ratios of components are equivalent.

6.7 *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 the $FeCl_3$ solution and 0.5 parts of the $CuSO_4$ solution. Measure 5 mL of this mixture into a test tube as specified in 5.1. This pale amber reference standard shall then be overlaid with 5 mL of white mineral oil.

6.8 *Sulfuric Acid* (94.7 ± 0.2 %)—The H_2SO_4 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 of concentrated H_2SO_4). A blue color should not appear at the

zone of contact within 1 h. This test detects as little as 0.0002 % nitric acid (HNO_3).

7. Procedure

7.1 Clean a test tube with a chromic acid (H_2CrO_4) cleaning solution (**Warning**—Causes severe burns. A recognized carcinogen. Strong oxidizer.), or use a nonchromium containing, strongly oxidizing cleaning solution.

7.2 Fill the test tube to the 5 mL mark with H_2SO_4 (94.7 ± 0.2 %). Then add the oil to be tested to the 10-mL mark, insert the stopper loosely, and place the test tube in position in the water bath at 100 ± 0.5°C.

7.3 After the test tube has been in the water bath for 30 s, loosen the stopper sufficiently to release any pressure and reinsert, remove the test tube from the bath quickly, hold with a finger over the stopper, and give three vigorous, vertical shakes over an amplitude of about 5 in. (127 mm), shaking the test tube quickly and at a rate corresponding to 5 shakes/s. (A shaking machine may be employed provided the results that can be obtained agree with those obtained by the prescribed manual agitation.) Repeat every 30 s. Do not keep the test tube out of the bath longer than 3 s for each shaking period.

7.4 At the end of 10 min from the time the test tube was first placed in the bath, remove the test tube and allow to stand in the room for not less than 10 min nor more than 30 min. Observe and record any discoloration of the oil layer. Place the test tube in the color comparator, and compare the acid layer with 5 mL of the standard colorimetric solution and 5 mL of white mineral oil in a test tube that has been shaken vigorously for 10 s and allowed to stand just long enough for the contents to separate into two layers.

8. Interpretation of Results

8.1 White mineral oil shall be reported as passing the test only when the oil layer shows no change in color (see Note 2) and when the acid layer is not darker than the reference standard colorimetric solution.

NOTE 2—A bluish haze or a slight pink or yellow color in the oil layer should not be interpreted as a change in color.

8.2 If the oil layer is discolored or if the acid layer is darker than the reference standard colorimetric solution, white mineral oil shall be reported as not passing the test.

9. Precision and Bias

9.1 No statement is made about either the precision or bias of this test method since the result merely states whether there is conformance to the criteria for success specified in the procedure.

10. Keywords

10.1 carbonizable substances; mineral oil