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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Iron in Rosin Tall Oil Fatty Acids and Other Related Products¹

This standard is issued under the fixed designation D 1064; 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 These test methods cover colorimetric procedures for the determination of iron in rosin tall oil fatty acids and other related products. Both spectrophotometric and visual methods are covered.

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. Significance and Use

2.1 Iron is a possible contaminant in naval stores products, being introduced into these products during their production from various raw material sources. Gum rosin in particular is prone to iron contamination as the equipment used for its collection and processing is often made from iron containing metals.

2.2 Iron is a troublesome contaminant in rosin and fatty acids, as even trace quantities will catalyze the oxidation and a subsequent darkening of these products.

2.3 The test methods described in this standard were developed many years ago and to a large extent have been replaced by modern instrumental methods such as atomic absorbance and inductively coupled plasma spectroscopies. The test methods described in this standard are subject to interferences from other species in the sample being tested. However, the test methods described here are acceptable if approximate or trend values are required and if appropriate analytical instrumentation is not available.

3. Reagents

3.1 *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 Commit-

tee 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.

3.2 Unless otherwise indicated, references to water shall be understood to mean deionized or distilled water.

SPECTROPHOTOMETRIC METHOD

4. Summary of Test Method

4.1 Ferrous iron, in a dilute hydrochloric acid solution, forms a red-colored complex with 1,10-phenanthroline. The intensity of the color is measured at approximately 505 nm by means of a spectrophotometer.

5. Apparatus

5.1 *Photometer*—Any spectrophotometer or filter photometer that will measure accurately the transmittance of the solutions in the range from 500 to 520 nm.

5.2 *Dishes*, high-silica glass,³ silica, or porcelain, 50 and 100-mL capacity.

NOTE 1—Platinum or platinum-rhodium dishes are not recommended as they sometimes cause a color interference with the phenanthroline reagent.

5.3 *Watch Glasses*, to cover the dishes described in 5.2.

5.4 *Pipets*—One 100-mL, two 10-mL, three 5-mL, and two 2-mL pipets.

5.5 *Measuring Pipet*, Mohr-type, 10-mL.

5.6 *Volumetric Flasks*, 1-L and 50-mL capacities.

5.7 *Absorption Cells*, having a capacity of at least 25 mL.

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. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

³ The sole source of supply of the high-silica glass known to the committee at this time is Vycor. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.34 on Naval Stores.

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Smaller cells may be used when the color is developed in a flask or beaker.

6. Reagents

6.1 *Hydrochloric Acid* (1 + 19)—Dilute 1 volume of A.C.S. grade HCl (sp gr 1.18) with 19 volumes of water.

6.2 *Iron Solution, Standard* (1 mL = 0.1 mg Fe)—Dissolve 0.1000 g of pure iron wire (99.85 % iron) in 10 mL of H₂SO₄ (1 + 9) and 3 mL of HNO₃ (sp gr 1.42). Dilute with water to 1 L in a volumetric flask.

6.3 *Iron Solution, Standard* (1 mL = 0.01 mg Fe)—Pipet 100 mL of standard iron solution (1 mL = 0.1 mg Fe) into a 1-L volumetric flask and dilute to the mark with HCl (1 + 19).

6.4 *Hydroxylamine Hydrochloride Solution*—Dissolve 10 g of (c.p.) hydroxylamine hydrochloride in 190 mL of water. This reagent is stable at room temperature.

6.5 *Hydroquinone Solution*—Dissolve 2.5 g of hydroquinone, reagent or photographic grade, in 100 mL of HCl (1 + 200). Keep in a refrigerator at about 10°C when not in use.

6.6 *1,10-Phenanthroline Solution*—Dissolve 0.5 g of 1,10-phenanthroline in 500 mL of water.

6.7 *Sodium Acetate Solution*—Dissolve 100 g of A.C.S. grade NaC₂H₃O₂·3H₂O in 400 mL of water.

7. Preparation of Calibration Curve

7.1 Pipet 0.5, 1.0, 2.0, 3.0, 4.0, and 5.0-mL aliquots of standard iron solution (1 mL = 0.01 mg Fe) into absorption cells and dilute to 10 mL with HCl (1 + 19) measured with a Mohr-type pipet. Add 10 mL of HCl (1 + 19) to an additional absorption cell and carry through as a reagent blank.

7.2 Develop color in the solutions as described in 8.4.

7.3 Measure the transmittance or absorbance of the solution at 505 nm with the spectrophotometer adjusted to read 100 % transmittance or zero percent absorbance for the reagent blank.

7.4 Using semilogarithmic paper, plot the percentage transmittance of the solutions against milligrams of iron present. Or, plot the percentage absorbance on rectilinear graph paper against the milligrams of iron present.

8. Procedure

8.1 Weigh 5.00 g of the sample into a high-silica glass, silica, or porcelain dish. If the iron content is less than 5 ppm, use a weight of sample such that the solution on which the spectrophotometer reading is made will contain from 0.05 to 0.050 mg of iron.

8.2 Place the dish in an electrically heated muffle furnace and raise the temperature slowly until the sample is completely charred. Then raise the temperature to 500°C and maintain at 500 to 550°C until the sample is greyish white. This usually required 4 to 6 h, but no harm will be done if the sample is allowed to remain in the muffle overnight. Remove the dish from the muffle furnace, cool to room temperature, and add 5 mL of HCl (1 + 1) in such a manner that any ash on the sides of the dish is washed to the bottom. Cover the dish with a watch glass and heat just to boiling.

8.3 Transfer the sample to a 50-mL volumetric flask, using water to wash the last trace from the dish and watch glass. Dilute to the mark with water and mix thoroughly.

8.4 Pipet 10 mL of the solution into an absorption cell (Note

2). When the iron content is too high to be read from the calibration curve, take a smaller aliquot and dilute to 10 mL with water. Add 2 mL of hydroxylamine hydrochloride or hydroquinone solution, 5 mL of 1,10-phenanthroline solution, and 5 mL of sodium acetate solution, using volumetric pipets. Mix thoroughly after each addition.

NOTE 2—When 25-mL absorption cells are not available for the spectrophotometer, develop color as described in 7.4, but in a flask, beaker, or large test tube, and transfer an aliquot of the solution to a suitable absorption cell.

8.5 *Blank*—Run a blank along with the sample to be sure none of the reagents have become contaminated (Note 3) and for a reference solution against which the transmittance of the sample is measured. Measure the transmittance of the blank with the spectrophotometer adjusted to read 100 % transmittance, or zero percent absorbance at a wavelength of 505 nm when the absorption cell contains water.

NOTE 3—When the reagents contain more than 0.002 or 0.003 mg of iron, prepare new reagents and look for the source of contamination.

8.6 Measure the transmittance of the sample solution at 505 nm with the spectrophotometer adjusted to read 100 % for the blank. Read the iron content of the sample solution in milligrams from the calibration curve.

8.7 *Calculation*—Calculate the iron content of the sample in parts per million as follows:

$$\text{Iron, ppm} = (A/B) \times 1000 \quad (1)$$

where:

A = milligrams of iron found, and

B = grams of sample represented in the aliquot used.

8.8 *Check Determination*—Make a single determination daily using 1 to 5 mL of standard iron solution (1 mL = 0.01 mg Fe) and proceeding as described in 7.1 and 7.2. If the determined and known values do not agree within the limits of experimental error (± 1 % transmittance) repeat the test. If the second results do not agree, prepare a new standard solution. If determined and known values do not check after preparing a new standard, investigate for errors in technique and reagents, and if none can be found, prepare a new calibration curve. This should not be necessary, and be sure to thoroughly investigate all other factors before doing this.

VISUAL METHOD

9. Summary of Test Method

9.1 Ferrous iron, in a dilute hydrochloric acid solution, forms a red-colored complex with 1,10-phenanthroline. The intensity of the color is compared visually in matched Nessler tubes against reference color standards containing known amounts of iron, in which color has been developed in the same manner.

10. Apparatus

10.1 *Nessler Tubes*—For visual comparison of colors, matched 50-mL Nessler tubes shall be used.

11. Reagents

11.1 See Section 3.