Designation: D 590 – 93 (Reapproved 2002)

An American National Standard

Standard Test Method for Petroleum Wax in Paper[1]

This standard is issued under the fixed designation D 590; 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 determines quantitatively the total amount of petroleum wax in wax-impregnated papers, including papers referred to as "dry waxed" or simply "waxed" papers, and paper products made from such papers.

1.2 This test method is not intended for use with wax-sized papers, because these generally contain such quantities of rosin or other materials soluble under the conditions of this test method, or both, that the accuracy for quantitatively measuring wax is seriously impaired.

1.3 This test method does not differentiate between "surface wax" and "total wax." It measures the total amount of wax materials extracted under the conditions specified.

1.4 *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 precautionary statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, or Related Products[2]

D 685 Practice for Conditioning Paper and Paper Products for Testing[2]

D 1968 Terminology Relating to Paper and Paper Products[2]

E 122 Practice for Calculating Sample Size to Estimate, with a Specified Tolerable Error, the Average for a Characteristic of a Lot or Process[3]

2.2 *TAPPI Standard:*

T 208 Test method for moisture in paper by toluene distillation[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1986 or the *Dictionary of Paper.*[5]

4. Summary of Test Method

4.1 The wax is extracted from a test specimen using a Soxhlet-type extraction assembly and toluene as the extraction solvent. The solvent is evaporated, and the extracted wax or the extracted specimen, or both, are dried at 105°C and then weighed. The percentage of wax present is calculated based on the dry weight of the extracted wax or the dry weight of the extracted specimen and either the dry sample weight or a conditioned sample weight basis, as agreed upon between the users of this test method.

5. Significance and Use

5.1 The percentage of wax present in wax-impregnated papers may be an indicator of the degree to which these papers resist penetration of aqueous liquids.

6. Apparatus

6.1 *Extraction Assembly*—Either of the following styles of extraction assembly are suitable for performing this test method.

6.1.1 *Soxhlet Extraction Apparatus*—Consisting of the widely recognized Soxhlet glassware assembly, including a Soxhlet extraction tube, pure cellulose extraction thimble, condenser, solvent reservoir, and in addition, an electric heating mantle, and variable power source. Such equipment is in general use and available from numerous suppliers of laboratory glassware and apparatus.

6.1.2 *High-Efficiency Extraction Assembly*—One such assembly found satisfactory is the Soxtec System HT®.[6] Other similar high-efficiency extraction assemblies may also be suitable and may be used as described in 11.6 and 11.7.

---

[1] This test method is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Test Methods.

Current edition approved Sept. 15, 1993. Published November 1993. Originally published as D 590 – 40 T. Last previous edition D 590 – 87.

[2] *Annual Book of ASTM Standards,* Vol 15.09.

[3] *Annual Book of ASTM Standards,* Vol 14.02.

[4] Available from Technical Association of the Pulp and Paper Industry, Technology Park/Atlanta, PO Box 105113, Atlanta, GA 30348.

[5] Formerly published by American Paper and Pulp Assoc. (currently API), New York, NY.

[6] The Soxtec System HT® is manufactured by Tecator, Hogansas, Sweden, and may be purchased in the United States through Fisher Scientific.

Operating instructions for a high-efficiency extraction assembly will be received with the equipment and should be followed, consistent with 11.6 and 11.7.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specification of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[7] Other grades may be used, provided that it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

7.2 *Toluene*, ACS grade.

8. Hazards

8.1 In addition to other precautions, all use of toluene including the extraction procedure itself and evaporation of residual solvent should be done in an approved hood. The oven used for drying of the final extract should be approved for use with solvent vapors.

9. Sampling

9.1 *Acceptance Sampling*—Acceptance sampling shall be done in accordance with Practice D 585.

9.2 *Sampling for Other Purposes*—The sampling and the number of test specimens depends on the purpose of the testing. Practice E 122 is recommended.

10. Conditioning

10.1 Conditioning of the sample prior to testing as specified in Practice D 685 is not a requirement of this test method. The rate at which equilibrium moisture content is achieved is greatly reduced for wax-impregnated papers, in comparison with those that have not been wax treated.

10.2 For referee purposes or other needs where an accurate or reproducible moisture level in the sample is required, one of the following procedures is to be followed:

10.2.1 The parties involved in the testing must agree how long the test units must be conditioned as required in Method D 585 prior to weighing the test specimen. Results are then reported on a "conditioned sample" weight basis.

10.2.2 The parties involved must agree to determine moisture content in the sample as described in T208. Results are then reported on a "dry sample" weight basis.

10.3 For the purposes of this test method, moisture levels in the sample may not be determined by oven moisture as low-molecular wax components may volatilize and cause results for moisture to be inaccurate.

11. Procedure

11.1 Weigh out to the nearest 0.2 mg at least two representative 0.5-g test specimens from each test unit. Sample size may be varied as necessary to provide at least about 0.1 g of extracted wax.

11.2 In cases where the level of wax is to be reported on an area basis, cut the test specimen, preferably with a cutting die of dimensions known to the nearest 0.01 in. (0.25 mm) or measure the dimensions of the specimen taken for testing to that accuracy prior to weighing. Where the dimensions are to be measured, the sample must be cut with a device that provides corners that are square (90°) so that the accurate area of the sample may be determined by simple geometric principles.

11.3 The test specimen must provide ready and immediate surface access to the solvent. The test specimen may be folded loosely, or "pleated." It must not be cut into smaller pieces, as these tend to adhere to one another and prevent complete extraction.

11.4 Place the test specimen into the extraction device. Add solvent to the extraction reservoir.

11.5 If appropriate for the equipment being used, obtain a tare weight on the portion of the apparatus that will contain the extracted wax upon completion of the test.

11.6 Operate the extraction equipment for a sufficient time period to completely extract all of the wax present. In the absence of prior knowledge about the sample, 10 to 15 cycles of the Soxhlet equipment is appropriate. Fifteen-minutes extract and fifteen minutes rinse have been found sufficient for some samples using the Soxtec System HT®.

11.7 For samples whose extraction properties are unknown, remove the test specimen after 8 to 10 cycles or more for the Soxhlet equipment, or 10 min or more using the Soxtec System HT® and determine the sample air dry weight. Repeat after five cycles or a predetermined additional extraction period. Continue extraction until no further weight loss is found. For the Soxtec System HT® equipment, trials may also be required to determine the proper rinse time following extraction.

11.8 When extraction is complete, dry the extracted wax or the specimen from which the wax has been extracted, or both, for 2 h at 105°C, after first removing all but the last traces of solvent on a steam bath or by air drying. Use a suitable explosion-proof oven.

12. Calculation

12.1 Calculation or results may be done in several ways, based upon agreement between the users of this test method.

12.1.1 Calculation based on weight of wax extracted:

$$\% \text{ wax} = \frac{\text{weight of wax extracted (g)} \times 100}{\text{weight of original specimen (g)}} \quad (1)$$

12.1.2 Calculation based on weight of test specimen after extraction:

$$\% \text{ wax} = \frac{(\text{weight of original specimen (g)} - \text{specimen extracted weight (g)}) \times 100}{\text{weight of original specimen (g)}} \quad (2)$$

[7] *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

12.1.3 "Weight of original specimen" is the weight determined on the agreed basis in accordance with 9.2. Where it has been agreed to determine wax on a "dry basis" in accordance with 10.2.2, the specimen weight (see 11.1) must be corrected for the moisture determined to be present (see section 10.2.2) as follows:

$$\text{weight of original specimen ("dry basis")} = \text{specimen weight (g) (see 11.2)} \times \frac{(100.00 - \%\ \text{moisture})}{100} \quad (3)$$

12.1.4 Where it has been agreed to report wax in terms of weight per area, divide the weight of wax extracted (g) by the area of the original specimen (see 11.2). Convert to agreed units of weight per unit area using appropriate conversion factors. Where grams of wax per square metre of sample are required, for example,

$$\text{wax, g/m}^2 = \frac{\text{weight of wax extracted (g)} \times 10\,000}{\text{specimen area (cm}^2\text{)}} \quad (4)$$

13. Report

13.1 Report the following information:

13.1.1 Average percentage of wax present on calculation basis (see 10.2.1 or 10.2.2) agreed upon between the buyer and the seller,

13.1.2 Wax content per unit area, if required, in units as agreed upon between the buyer and the seller, and

13.1.3 Calculation basis used (see 10.2).

14. Precision and Bias

14.1 *Precision*—Based on limited information from one laboratory, the repeatability standard deviations and the 95 % repeatability limits for this test method using the equipment specified in 6.1.2 are approximately 0.8 and 2.2 % respectively, for waxed paper and paper products containing wax in the range from 15 to 30 %. Precision using the equipment specified in 6.1.1, that for the purposes of this test method is considered to be equivalent, is expected to be similar. Work to develop additional repeatability and reproducibility values is in progress.

14.2 *Bias*—The procedure in this test method has no bias because the percentage of wax extracted from a specimen is defined in terms of the procedure, solvent, and calculations specified in this test method.

14.2.1 Limited data indicate that there is no bias between the solvent used in the previous edition of this test method, 1,1,1-trichloroethane, and that used in the current edition, toluene, when the procedures specified in 6.1.2 are followed.

14.2.2 There is no available data to show the bias, if any, between the Soxhlet-type extraction procedures specified in 6.1.1 or 6.1.2 of this test method and the open-dish extraction specified in the previous edition of this test method when all procedures employ the same solvent. However, bias in this case is believed to be small or nonexistent.

14.2.3 Because of environmental and safety concerns associated with the use of both the previous solvent, 1,1,1-trichloroethane, and the previous procedure that required the boiling of a test specimen in an open dish of solvent without condensation of solvent vapor, no work to determine any bias between this test method and previous editions is planned.

15. Keywords

15.1 dry waxed paper; extraction; paper; paper products; waxed paper

ANNEX

(Mandatory Information)

A1. TEST METHOD D590 REVISION

A1.1 This version of Test Method D 590 replaces the 1,1,1-trichloroethane solvent with toluene, and eliminates the practice of extraction in an open vessel, replacing it with extraction in an enclosed system such as a Soxhlet extraction system or a Soxtec System HT®.