Designation: D3522 – 86 (Reapproved 2007)

Standard Test Method for Applied Coating Wax and Impregnating (Saturating) Wax in Corrugated Board Facing[1]

This standard is issued under the fixed designation D3522; 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 the determination of the weight of wax that has been applied for coating of corrugated board, and the amount of impregnating (saturating) wax in the same facing.

1.2 This test method is especially applicable to board that has a coated surface and also contains wax saturation within the facing structure.

NOTE 1—The amount of surface wax on board that may or may not contain impregnating (saturating) wax within its structure may be determined alternatively by Test Method D3521. If it is known that the specimen has coating wax only, with no internal impregnating wax, the total coating wax applied may be determined by Test Method D3344.

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 7.1.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Product

D3344 Test Method for Total Wax Content of Corrugated Paperboard

D3521 Test Method for Surface Wax Coating On Corrugated Board

3. Terminology

3.1 *Definitions:*

3.1.1 *weight percent impregnating wax*—the weight percent of wax in the facing relative to the weight of unwaxed facing measured at 23°C (73°F) and 50 % relative humidity.

3.1.2 *weight of applied coating wax*—the weight of applied coating per unit area of board, usually grams per square metre or pounds per thousand square feet of board covered.

4. Summary of Test Method

4.1 The coated facing is peeled from the medium and then split into two layers, one bearing the coating on waxed fibers and one containing the waxed fibers only. The layers are extracted separately, collecting both fibers and wax, leading to a calculation of the applied surface coating wax and the amount of impregnating wax.

5. Significance and Use

5.1 The resistance of corrugated fiberboard shipping containers to damage by moisture is improved by wax treatment, and a common practice involves a light wax saturation applied to the medium and facings, followed by a curtain coating or roll coating operation applying wax to the surface. The functional performance of the board is dependent upon the amount of wax deposited in each operation.

5.1.1 For the wax impregnation within the facing, the principal concern is with the weight of wax used relative to the weight of paperboard present, that is, the weight percent loading or pickup. This method measures that loading and assumes that the loading is distributed uniformly throughout the facing. However, the method does not provide a measure of the weight of impregnating wax per unit area, since not all of the facing fibers are utilized in the testing.

5.1.2 For the wax coating the principal concern is in the weight of wax present on the surface per unit area. This method measures the amount of material applied, and assumes that the major portion of molten coating applied will congeal and remain on the surface, without undue migration into the fibrous structure of the medium.

NOTE 2—In a typical curtain coating application, a portion of the coating will partially migrate into and become embedded in the fibers of the facing to the extent of about 10 % of the coating applied.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.10.0A on Physical/Chemical Properties.

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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.

5.1.3 These procedures involve extractions of designated sections of the paperboard and determination of the extractable material reported as wax. This assumes that the nonwax extractables in the paperboard substrate are in negligible amount (for example, about 0.2 weight %).

NOTE 3—If the application of a correction for nonwax extractables is desired, a suitable unwaxed board specimen may be extracted and appropriate calculations made.

6. Apparatus

6.1 *Sample-Trimming Equipment*—A suitable trimming board or template arrangement equipped with a razor edge knife for even cutting of specimens to required size, with parallel sides. (A guillotine-type paper cutter is not recommended.)

6.2 *Measuring Rule*, steel edged, for measuring the size of specimen to within 0.5 mm.

6.3 *Beakers*, 1000-ml, Griffin type.

6.4 *Steel Screen*, 325-mesh, approximately 150 mm in diameter, to fit into funnel.[3]

6.5 *Glass Funnel*, approximately 100 mm in diameter.

6.6 *Watch Glasses*.

6.7 *Steam Bath* or *Hot Plate* in hood.

6.8 *Hot Plate*.

7. Reagents and Materials

7.1 *Solvent*, chlorinated hydrocarbon, 1,1,1-trichloroethane (**Warning**—May cause irritation.). The solvent used should be residue-free, and should be checked for residue upon evaporation before using.[4]

8. Sampling and Test Specimens

8.1 From each test unit, obtained in accordance with Practice D585, that is, each finished carton blank or paperboard sheet, cut representative specimens that are free of obvious defects. Each specimen should measure 100 by 100 mm, cut to the nearest 0.5 mm. Record the area of the surface to be tested, and whether it is an inside or outside facing. Duplicate specimens are required from each sample unit.

NOTE 4—The operator may be required to increase the replication and treatment of specimens to obtain a better estimate of "average" wax loading (*1*) if the waxing is at an extremely low loading level, or (*2*) if the wax loading shows obvious wide variations in distribution over the board area.

NOTE 5—Optionally, specimens of other dimensions may be used if required by sampling limitations. In such cases, calculations need to be appropriately adjusted.

8.2 Condition all boards at 23°C (73°F) and 50 % relative humidity for a minimum of 48 h before beginning test procedure.

9. Procedure

9.1 Take each duplicate specimen and separate the coated facing from the combined board as follows:

9.1.1 Pry the facing loose from the medium and peel it back to remove it. This is accomplished conveniently by working from the four corners of the specimen to achieve rather complete removal of the facing. Some thin layers of fibers of the original facing will be left adhering to the glue line and medium and may be ignored. (Set aside or discard the remaining part of the combined board, that is, the medium and second facing.)

9.1.2 Take the removed facing and carefully peel or split it into two sections: (*1*) a layer that represents the *complete* area of the coated surface, together with a thin layer of adhering fiber, and (*2*) the layers representing the main thickness of the wax-saturated facing below the coating. The delaminating operation may involve several peelings of layers to collect the appropriate separate sections.

9.1.3 Combine the appropriate similar sectionings from the duplicate specimens, and cut the larger pieces with scissors into small chips about 25 mm square, being careful to retain all cuttings without loss. Weigh separately to the nearest 1 mg the two accumulated sections—(*1*) the coated section and (*2*) the impregnated section.

9.2 Extract each facing section separately as follows:

9.2.1 Place the section pieces in a 1000-ml beaker. Add 250 ml of the solvent 1,1,1-trichloroethane and cover the beaker with a watch glass. Heat to 75°C (167°F) for 1 h on a steam bath or hot plate in a hood. Pour off the solvent, passing it through the stainless steel screen in the funnel to remove fibers, and collect the solvent in a clean, tared 1000-ml beaker. Rinse the extraction beaker and the extracted paper chips with 50 ml of hot solvent, filter this rinsing, and add it to the solvent in the tared beaker.

9.2.2 Repeat the solvent extraction using 250 ml of fresh solvent, boiling for 1 h and rinsing with 30 ml of hot solvent. All extracts and rinsings are combined in the same tared beaker.

9.2.3 Collect all of the extracted paper chips and fibers remaining in the beaker and on the screen, from the extraction of the coated section, and place the fibers in a 250-ml beaker, allow to air-dry for 30 min, dry in an oven at 100°C for 60 min and then equilibrate at 23°C (73°F) and 50 % relative humidity for 24 h. Weigh to the nearest 1 mg and record as fibers from the coated section. (Discard fibers from impregnated section.)

9.2.4 Evaporate the combined solvent extracts from each section on a steam bath in a hood, or optionally, overnight in the air current of a hood. Evaporation may be hastened by use of a stream of nitrogen. For the final stages of evaporation, place the beaker on a hot plate at about 150°C (about 300°F) to dissipate solvent vapors or moisture completely. Confirm that evaporation is complete when no solvent odor can be detected. Cool and weigh the tared beaker. Record the weight of wax extracted to the nearest 1 mg.

10. Calculation

10.1 Calculate the percent of impregnated wax in the facing, *a*, as follows:

[3] A suitable stainless steel screen, made of Type 304 steel, at 325 mesh (12.7 count per millimetre), using wire diameter 0.0355 mm (0.0014 in.) with openings 0.043 mm (0.0017 in.) may be obtained from Newark Wire Cloth Co., 351 Verona Ave., Newark, NJ 07104.

[4] A suitable solvent is Inhibisol (brand), obtainable from Amerace-Esna Corp., Chemical Specialities Div., Tenafly, NJ.

$$a = \frac{c}{b-c} \times 100 \quad (1)$$

where:

b = weight of sample impregnated section, waxed fibrous layers before extraction, g, and

c = weight of wax extracted from impregnated section, g.

10.2 Calculate the coating wax weight, grams per square metre, i, as follows:

$$i = \frac{f - \left(\frac{h \times a}{100}\right)}{d} \quad (2)$$

where:

a = percent of impregnated wax in facing

d = specimen area, total of coated surface, m^2,

f = weight of wax extracted from coated section, g, and

h = weight of extracted fibers from coated section, g.

11. Report

11.1 Report which facing of the corrugated board was tested and report the completed test on the facing as follows:

11.1.1 Impregnated wax in facing, weight percent.

11.1.2 Applied coating wax weight on facing, grams per square metre.

12. Precision and Bias

12.1 The precision of the method as obtained by statistical examination of interlaboratory test results is as follows:

12.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

18 % of the mean for impregnating wax content in facing

5 % of the mean for applied coating wax on facing

12.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

20 % of the mean for impregnating wax content in facing

6 % of the mean for applied coating wax on facing

NOTE 6—The precision data were obtained on two samples having a surface wax coating of 35 to 40 g/m^2 and an impregnating wax content of 5 % and 12 %. It is anticipated that precision would be considerably impaired at very low percentages of impregnating wax.

12.2 *Bias*—The procedure for measuring applied coating and impregnating wax in corrugated board facing has no bias because the value can be defined only in terms of a test method.

13. Keywords

13.1 coating; corrugated; impregnating; wax

ANNEX

(Mandatory Information)

A1. PRECAUTIONARY STATEMENT—1,1,1-TRICHLOROETHANE

A1.1 **Warning**—May cause irritation.

Avoid contact with the eyes, skin, and clothing.

Use only with adequate ventilation.

Avoid prolonged breathing of vapor or spray mist.

Avoid prolonged or repeated contact with skin.

Do not take internally.