Standard Test Method for
Total Wax Content of Corrugated Paperboard[1]

This standard is issued under the fixed designation D 3344; 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.

ε[1] NOTE—Warning notes were placed in the text editorially in December 2000.

1. Scope

1.1 This test method covers the determination of the weight of wax that is present in a specimen of wax-treated corrugated paperboard. The test method is applicable to specimens that have been waxed by either impregnation (saturation) operations or coating operations, or combinations of such operations.

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.* For precautionary statement, see 5.4 and 7.2.

2. Terminology

2.1 *Definitions:*

2.1.1 *weight percent wax content*—the weight percent of wax present in and on corrugated board relative to the weight of unwaxed board substrate measured at 23°C (73°F) and 50 % relative humidity.

2.1.2 *weight of applied wax coating*—the weight of wax that has been applied to the corrugated board as a coating, expressed as weight per unit area, usually grams of coating per square metre or pounds of coating per thousand square feet of board covered.

NOTE 1—When it is known that a wax-coated specimen has no impregnating wax present, this extraction procedure is normally calculated to express the data as "weight of applied wax coating."

3. Summary of Test Method

3.1 The total quantity of wax associated with the corrugated board specimen is determined by extracting the wax from the board and evaporating the extract to dryness.

4. Significance and Use

4.1 Many of the functional properties of wax-treated corrugated paperboard and cartons are dependent on the amount of wax present.

4.2 In the case of wax-saturated, or wax-impregnated, paperboard the principal concern is with the weight of wax used relative to the weight of paperboard present, that is, the weight percent content or pickup. In some applications the saturating wax may be deposited in the three elements of the corrugated board in such a way as to individually control the amount in each element, that is, the medium and the two facings.

4.3 In the case of wax-coated corrugated paperboard the principal concern is the weight of wax on the board surface per unit area. The functional values of the wax coating as a barrier or a decorative coating are dependent, in part, on the amount of wax in the continuous surface layer, relative to the *area* covered. The weight of coating relative to the *weight* of substrate is not usually a concern with regard to product quality.

5. Apparatus

5.1 *Sample Trimming Equipment*—A suitable trimming board or template arrangement equipped with a razor edge knife for even cutting of specimens so that they have parallel sides and are of the right size. (A guillotine-type paper cutter is not recommended.)

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

5.3 *Beakers*, 1000-cm^3, Griffin-type.

5.4 *Solvent*[2]—Chlorinated hydrocarbon solvent, 1,1,1-trichloroethane. (**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.) The solvent used should be residue-free, and should be checked for a residue upon evaporation before using.

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

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

5.7 *Watch Glasses*.

5.8 *Steam Bath or Hot Plate in Hood*.

5.9 *Laboratory Hot Plate*.

5.10 *Analytical Balance* reading to the nearest 0.0001 g.

6. Test Specimen

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

6.2 From each sample unit, that is, each finished carton blank or paperboard sheet, cut representative specimens free of obvious defects. Each specimen should measure 100 by 100 mm, cut to the nearest 0.5 mm. Two specimens are required from each sample unit to be tested.

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

NOTE 3—Optionally, specimens of other dimensions may be used if required by sampling limitations. In such cases, calculations need to be appropriately adjusted.

7. Procedure

7.1 Weigh the two board specimens together to the nearest 1 mg and record the combined weight of sample in grams. Cut each specimen into small pieces, each measuring about 25 mm square, being careful to retain all of the trimmings, and being careful that no surface wax is lost during handling of the specimens. Place all pieces in a 1000-cm^3 beaker.

7.2 To the cut pieces, add 250 cm^3 of solvent 1,1,1-trichloroethane (**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.) and cover the beaker with a watch glass. Heat to 75°C (167°F) then maintain at 75°C 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-cm^3 beaker. Rinse the extraction beaker and the extracted paper chips with 50 cm^3 of hot solvent, filter this rinsing, and add it to the solvent in the tared beaker.

7.3 Repeat the solvent extraction using 250 cm^3 of fresh solvent, boiling for 1 h and rinsing with 30 cm^3 of hot solvent. Combine all extracts and rinsings in the same tared beaker.

7.4 Evaporate the combined solvent extracts on a steam bath 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 300°F to completely dissipate solvent vapors or moisture. Confirm that evaporation is complete when no solvent odor can be detected. Cool and reweigh the tared beaker. Record the weight of wax extracted, to the nearest 1 mg.

NOTE 4—If it is not possible to tare the 1000-cm^3 beaker because of space limitations on the analytical balance, evaporate most of the solvent in the 1000-cm^3 beaker and then quantitatively transfer the residue with rinsings to a smaller tared beaker, and continue the evaporation to dryness as described in 7.4.

NOTE 5—Use of a Soxhlet extraction technique may result in improved precision.

8. Calculations

8.1 Calculate the basis weight of waxed combined board at start, B_w, in grams per square metre, as follows:

$$B_w = (b/a) \times 10\ 000 \qquad (1)$$

where:
a = specimen area, total, cm^2, and
b = specimen weight, total, g.

8.2 Calculate the total wax content, T, in grams per square metre, as follows:

$$T = (c/a) \times 10\ 000 \qquad (2)$$

where:
c = weight of extracted wax, total, g

8.3 Calculate the basis weight of unwaxed board, B_u, in grams per square metre, as follows:

$$B_u = B_w - T \qquad (3)$$

8.4 Calculate the weight percent total wax content, W, based on unwaxed board, as follows:

$$W = (T/B_u) \times 100 \qquad (4)$$

9. Report

9.1 Report the completed test on the corrugated board as follows: total wax content, g/m^2, or total wax content, %.

10. Precision and Bias

10.1 *Precision*—The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.1.1 *Repeatability*—The difference between two 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:

5 % of the mean

10.1.2 *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:

12 % of the mean

NOTE 6—Precision was determined in an interlaboratory study on the following samples:

	Wax content, weight %
Fully wax saturated	42
Partially wax saturated, and both liners curtain coated, one side	12
Partially wax saturated, medium only	4

10.2 *Bias*—The procedure in this test method has no bias because the total wax content of corrugated paper and paperboard can be defined only in terms of a test method.

11. Keywords

11.1 corrugated paperboard; wax; wax extraction; wax treated board