

Chlorine, Retained, Tensile Loss: Multiple Sample Method

Developed in 1965 by AATCC Committee RR35; reaffirmed 1967, 1971, 1974, 1977, 1980, 1989, 1999; editorially revised and reaffirmed 1985, 1994, 2005; editorially revised 1992, 2004.

1. Purpose and Scope

1.1 This test method is a simplified procedure for testing multiple samples to determine the potential damage that may be caused by chlorine bleaching.

2. Principle

2.1 The samples are chlorine bleached in a domestic-type laundry machine, rinsed, dried and pressed between hot metal plates. The damaging action of the retained chlorine is calculated from the difference in tensile strength before and after pressing (see 10. 1).

3. Terminology

3.1 **retained chlorine**, *n.*—*in textiles bleached with chlorine-type bleaches*, available chlorine which remains in the material after washing and drying.

4. Safety Precautions

NOTE: These safety precautions are for information purposes only. The precautions are ancillary to the testing procedures and are not intended to be all inclusive. It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Manufacturers MUST be consulted for specific details such as material safety data sheets and other manufacturer's recommendations. All OSHA standards and rules must also be consulted and followed.

4.1 Good laboratory practices should be followed. Wear safety glasses in all laboratory areas.

4.2 Manufacturer's safety recommendations should be followed when operating laboratory testing equipment.

4.3 Use chemical goggles, rubber gloves and apron to protect skin and eyes during handling of sodium hypochlorite and sodium carbonate solutions.

4.4 An eyewash/safety shower should be located nearby for emergency use.

5. Apparatus and Reagents

5.1 Automatic agitator-type washing

machine having:

(a) Controlled wash-water and rinse-water temperature settings of $41 \pm 3^\circ\text{C}$ ($105 \pm 5^\circ\text{F}$).

(b) Normal agitator speed no slower than 47 cycles/min.

(c) Final spin speed (range 480-660 rpm).

(d) Final spin cycle time of 4-6 min.

5.2 Automatic tumble dryer having:

(a) Controlled exhaust temperature which cycles to 71°C (160°F) but does not exceed it.

(b) A cooling period while tumbling 5-10 min at the end of the drying cycle.

5.3 pH meter (see 10.2).

5.4 Scorch Tester (see 10.3).

5.5 Tensile Strength Tester (see 10.4).

5.6 Sodium hypochlorite solution, approx. 5% available chlorine (see 10.5).

5.7 Buffer solution (see 10.6).

5.8 Sodium tetraphosphate ($\text{Na}_6\text{P}_4\text{O}_{13}$).

5.9 Sodium carbonate.

5.10 Sodium bicarbonate.

5.11 Cotton cloth, kier-bleached, for ballast (see 7.2).

6. Preparation of Samples

6.1 Normally, only the warp strength is determined. Cut the sample approximately 35.6 cm (14 in.) in the warpwise direction and 20.3 cm (8 in.) in the fillingwise direction. (If testing filling specimens, reverse these figures.)

7. Procedure

7.1 Preparation of Washing Machine. If the washing machine has been used for anything other than chlorine bleaching, operate it through a full wash cycle with water, adding 0.15% sodium tetraphosphate at the beginning of the cycle. After machine is free of impurities, set the machine for the following conditions:

(a) Regular cycle.

(b) Low water level (see 10.7).

(c) Wash and rinse water at $41 \pm 3^\circ\text{C}$ ($105 \pm 5^\circ\text{F}$).

7.2 Chlorination. Permit wash water to enter machine to specified level. Add sufficient diluted sodium hypochlorite to wash water to obtain a bath liquor concentration of 0.10% by weight of available chlorine. Adjust the pH to 9.5 with sodium carbonate to raise the pH or sodium bicarbonate to lower it. Add all the test samples and sufficient untreated kier-boiled bleached cotton to make a 50:1 liquor-to-cloth ratio. Run machine through wash portion of cycle.

7.3 Rinsing. After the first load of wash water has been dumped and the machine has been refilled, add 10 mL of the buffer solution (see 10.6) for each gallon of water and continue operation through the remaining rinse cycles and the final spin cycle. If there is more than one deep rinse, the buffer is added only to the first one.

7.4 Drying and Conditioning. Place samples in a tumble dryer until just dry (do not press!). Transfer at once to a conditioned atmosphere at $65 \pm 2\%$ RH and $21 \pm 1^\circ\text{C}$ ($70 \pm 2^\circ\text{F}$) and allow to remain through the scorching step and tensile strength testing. The samples should be conditioned not less than 4 nor more than 24 h before scorching, and not less than 16 h before tensile testing.

7.5 Preparation of Specimens for Scorching. Prepare warp specimens by carefully cutting five strips approx. 35.6×3.2 cm (14.0×1.25 in.). Ravel the 3.2 cm (1.25 in.) strips to exactly 2.54 cm (1 in.) following ASTM Test Method D 5035, Breaking Force and Elongation of Textile Fabrics (Strip Test) (see 10.8). Then trim the 35.6 cm (14 in.) length of each strip to 30.5 cm (12 in.) From this set of five 2.54×30.5 cm (1×12 in.) strips, make two sets (from the same set of warp threads) of 2.54×15.2 cm (1×6 in.) raveled strip specimens by cutting the strips in two. Keep these sets separate; one set to be scorched, the other to be used as a control.

7.6 Preparation of Scorch Tester. Prepare Scorch Tester in advance so that the heating plates of the instrument maintain a temperature of $185 \pm 1^\circ\text{C}$ ($365 \pm 2^\circ\text{F}$). If necessary, to avoid air currents, enclose instrument in a cabinet. Make sure the two heating plates are clean and in good adjustment (uniformly in contact at all points) (see 10.9). Scorch each strip of one set separately (one at a time) by placing the 2.54×15.2 cm (1×6 in.) specimen in the Scorch Tester with the long direction of the raveled strip perpendicular to the long direction of the heating plates so that it is scorched across the center of the raveled strip. Scorch the strips for 30 s. Check reading of the thermometers frequently during use. Condition the specimens again, this time conditioning at least 16 h before making tensile strength measurements.

7.7 Tensile Strength. Make tensile strength tests on the unscorched and scorched specimens and record the individual values. From these, calculate the average tensile strength for each set.

8. Calculation of Results and Report

8.1 Use the formula:

$$(T_c - T_{cs})/T_c \times 100$$

= % loss in tensile strength due to damage caused by retained chlorine

where:

T_c = average tensile strength of chlorinated specimens, unscorched, and

T_{cs} = average tensile strength of chlorinated specimens, scorched.

8.2 Report percent loss in tensile strength for *warp* due to damage caused by retained chlorine.

8.2.1 If filling strength loss is determined report percent loss in tensile strength for *filling*.

9. Precision and Bias

9.1 *Precision*. The standard deviation obtained in developing this method was $\sigma = 5.98$.

The confidence interval based on 5 specimens is C.I. ± 5.2 . This confidence interval is around the percent loss in tensile strength.

9.2 *Bias*. The retained chlorine by tensile loss, multiple sample method, can be defined only in terms of a test method. There is no independent method for determining the true value. As a means of estimating this property, the method has no known bias.

10. Notes

10.1 Where it is suspected that either fabric or finish is susceptible to damage on heating, this susceptibility can be determined by running a distilled-water control, that is, the fabric is run through the entire procedure, but using distilled water in place of the hypochlorite solution. The loss in strength due to heat alone is calculated by the equation:

$$(T_w - T_{ws})/T_w \times 100$$

= Percent tensile strength loss due to scorching alone

where:

T_w = average tensile strength of the water-treated specimens, unscorched, and

T_{ws} = average tensile strength of the water-treated specimens, scorched.

If this loss is appreciable, there is some question as to whether the chlorine damage test is applicable. It is usually not necessary to determine the effect of wet chlorination on the fabric, as this factor is cancelled out in the calculation for chlorine damage. However, where it is desirable to determine the effect of the wet chlorination step, this may be calculated as follows:

$$(T_w - T_c)/T_w \times 100$$

= Percent tensile strength loss due to wet chlorination

where:

T_w = average tensile strength of the water-treated specimens, unscorched (handled as above) and

T_c = average tensile strength of the original fabric chlorinated and unscorched.

10.2 Any standard laboratory pH meter having suitable provision for high pH readings. Colorimetric methods cannot be used with NaOCl.

10.3 Available from SDL Atlas L.L.C., 1813A Associate Lane, Charlotte NC 28217; tel: 704/329-0911; fax: 704/329-0914; e-mail: info@sdlatlas.com.

10.4 Example: A Scott tensile strength tester having a suitable breaking-strength range (see ASTM D 76 and ASTM D 5035).

10.5 For determining the percent available chlorine, pipette a 1.00-mL portion of approx. 5% sodium hypochlorite solution into an Erlenmeyer flask and dilute to 100 mL with distilled water. Add 6 mL of 12% KI followed by 20 mL of 6N H_2SO_4 . Titrate with 0.1N sodium thiosulfate solution and calculate as follows:

Percent available chlorine

$$= \frac{\text{mL Na thiosulfate} \times 0.1N \times 0.0355}{1 \text{ mL} \times \text{sp. grav. Na hypochlorite}} \times 100$$

10.6 Buffer Solution: Prepare a solution containing 290 g of sodium tetrphosphate and 93 g of monosodium phosphate ($NaH_2PO_4 \cdot H_2O$) in sufficient water to make 1000 mL of solution.

10.7 Determine water volume at low-water level and use in calculating liquor-to-bath ratio.

10.8 Available from ASTM, 100 Barr Harbor Dr., West Conshohocken PA 19428; tel: 610/832-9500; fax: 610/832-9555.

10.9 The test block should exert a force of 255 g (9 oz), or 9 g/cm² (2 oz/in²). It can be calibrated by attaching a spring balance to the upper plate with a small thread sling and determine the force required to just separate the plates. If readjustment is necessary, follow instructions supplied by the manufacturer. Make sure that the two heating plates are clean and adjusted to give uniform contact at all points.