

Fluorine Content of Carpet Fibers

Developed in 2000 by AATCC Committee RA57; editorially revised and reaffirmed 2001; reaffirmed 2002; editorially revised 2005.

1. Purpose and Scope

1.1 This method is for determining the amount of fluorocarbon soil-resist agent present on carpet fibers by measurement of elemental fluorine on the pile fiber. It is useful for measurement of fluorocarbon present on carpet pile fiber as tested within the practical range of 100-1000 µg/g (ppm). It may also be used for yarns treated with fluorocarbon anti-soiling agents.

2. Principle

2.1 A weighed fiber specimen is burned in an oxygen combustion flask and the released hydrogen fluoride is absorbed into a sodium hydroxide solution. The fluorine content is measured as dissolved sodium fluoride at a constant pH and ionic strength using a fluoride ion activity electrode and a specific ion meter.

3. Terminology

3.1 **fluorine content, n**—in textile floor coverings, the ratio of the weight of total elemental fluorine to the total weight of carpet fibers.

3.2 **soil resist agent, n**—a material applied to, or incorporated in, carpet face fiber which retards and/or limits the build-up of soil.

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 All chemicals should be handled with care. Use chemical goggles or face shield, impervious gloves and an impervious apron during the dispensing and mixing of the fluoride and sodium hydroxide standards. The standards should be made and dispensed in a hood.

4.3 An eyewash/safety shower should be located nearby and a self-contained breathing apparatus should be readily available for emergency use.

4.4 Exercise care in cutting the specimens with the scalpel or razor blade; protective gloves may provide additional protection.

4.5 Oxygen supply pressure from the pressure regulator should not exceed 14 kPa (2 psi).

4.6 Do not operate the test equipment until the manufacturer's operating instructions have been read and understood. It is the responsibility of whoever operates the test equipment to conform to the manufacturer's directions for safe operation.

4.7 Exposure to chemicals used in this procedure must be controlled at or below levels set by governmental authorities (e.g., Occupational Safety and Health Administration's [OSHA] permissible exposure limits [PEL] as found in 29 CFR 1910.1000 of January 1, 1989). Fluoride salts and sodium hydroxides are listed with OSHA permissible exposure limits [PEL]. In addition, the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) comprised of time weighted averages (TLV-TWA), short term exposure limits (TLV-STEL) and ceiling limits (TLV-C) are recommended as a general guide for air contaminant exposure which should be met (see 12.1).

5. Apparatus, Reagents, Materials

5.1 1000 mL combustion flask with ball stopper (with platinum wire hook) and clamp (one each per specimen; reusable).

5.2 Platinum carrier basket (one per sample; reusable).

5.3 Infrared ignition cabinet.

5.4 Black paper specimen wrappers (one per sample).

5.5 Specific ion meter.

5.6 Fluoride ion selective electrode.

5.7 Reference electrode.

5.8 0.001N Sodium Hydroxide (NaOH).

5.9 TISAB (Total Ionic Strength Adjustment Buffer with CDTA (1,2-cyclohexanediaminetetraacetic acid) or formulated for fluorine analysis to prevent aluminum interference) (see 12.2).

5.10 Re-pipette or bottle-top dispenser (optional).

5.11 Sodium Fluoride (NaF) standard (see Appendix A).

5.12 Mechanical shaker (optional).

6. Calibration

6.1 Calibrate the specific ion meter using the procedures described in the operation manuals for the fluoride ion activity electrode and the specific ion meter. Calibrate the voltage output on the specific ion meter to read 40.0 for every 1 µg/g (ppm) fluoride in the standard solution (see 12.3, 12.4). Calibration should be done on a daily or every shift basis.

7. Sampling

7.1 Prepare the yarn sample by removing the tufts from the carpet sample with a scalpel or razor blade. Cut the tufts as close to the primary backing as possible having a stubble not greater than 1 mm. Twenty to 40 tufts are required for a single sample, depending on carpet construction. Place the tufts in a paper envelope and identify by test number.

7.1.1 *Sampling Option #1.* To determine an overall average fluorine content of carpet fibers, take samples from across the entire width of the carpet to compensate for possible variability in application and combine the tufts in a single envelope.

7.1.2 *Sampling Option #2.* To determine the variability of fluorine content across a carpet sample, take multiple samples from various areas of the carpet and analyze each set of carpet tufts separately (see 12.5).

8. Procedure

8.1 Weigh samples using tared weighing pans. Weigh between 0.110 g and 0.150 g of randomly selected tufts from each sample and record the weight. Fold all of the weighed tufts into the black paper sample holder and place the paper holder into the platinum basket. Suspend the basket from the platinum wire hook on the flask stopper.

8.2 Number each combustion flask and plastic cup.

8.3 Pipette 20.0 mL of the 0.001N sodium hydroxide solution into the oxygen combustion flask (see 12.6).

8.4 Turn on the oxygen supply. Place the flexible plastic tube into the oxygen combustion flask. Turn on the pressure regulator and allow oxygen to fill the flask for about 5-20 s. Turn off the oxygen flow (see 12.7).

8.5 Immediately place the flask stopper with the platinum basket suspended from the hook on the flask and clamp in place. Do not allow the sample basket to become wet or the sample will not ignite.

8.6 Place the flask in the infrared ignition cabinet. Adjust the flask level so that the test specimen in the suspended platinum basket is in the center of the infrared light beam and the tab on the black paper exposed to the infrared light. Turn on the infrared light until the sample ignites. Turn off the infrared light once the sample ignites. Allow the flame to burn out. Do not open the cabinet until the flaming has completely ceased. Confirm that the test specimen has burned completely. If any portion of the sample remains unburned, abort the test. Remove the flask from the ignition cabinet using a flask grip or gloves, as the flask will be hot.

8.7 Shake the flask.

8.7.1 If a mechanical shaker is used, place the flask into the holder and secure into position. Turn on the mechanical shaker for 1.5-2 min. Remove the flask.

8.7.2 If shaking is done by hand, shake vigorously for a minimum of 1 min.

8.8 Open the flask and pour all of the sodium hydroxide solution into a 100 mL plastic cup labeled with the test number.

8.9 Dispense 20.0 mL of TISAB into the empty flask. When dispensing, lift the flask stopper as little as possible to prevent any loss of gases. Replace the stopper and clamp in place. Repeat step 8.7 above. Pour all of the TISAB solution into the cup containing the sodium hydroxide solution.

8.10 Remove the platinum basket from the solution using tweezers. Cover the cup and allow the mixture to cool by sitting at room temperature for a minimum of 30 min.

8.11 Place the fluoride ion activity and reference electrodes into the cup containing the cooled test solution from the flask. Stir slowly and allow the meter to stabilize. Read and record the meter reading alongside the sample weight (see 12.8).

8.12 Discard the solutions in the plastic cups and to prevent cross-contamination, do not reuse the cups. Wash the platinum baskets and glassware, rinse with deionized or distilled water, and dry prior to using in a new test.

9. Calculation

9.1 Calculate the results as follows (if calibrated according to 6.1):

Fluorine content on carpet fiber $\mu\text{g/g}$ (ppm) =

$$\frac{\left[\begin{array}{c} \text{Meter reading (millivolt)} \\ \text{from step 8.11 above} \end{array} \right]}{\left[\text{sample weight (grams)} \right]}$$

9.1.1 Measured levels of less than 100 $\mu\text{g/g}$ (ppm) fluorine are considered as background levels and below the detection limit of this method.

10. Precision & Bias

10.1 *Interlaboratory test data.* An interlaboratory study was conducted in 1998 utilizing eight laboratories and two nylon face fiber carpets. Each laboratory made five determinations from each specimen using a single operator.

10.2 *Components of variance.* The components of variance shown as variances and the square root of the variances (to show appropriate units of measure) are listed in Tables I and II.

10.3 *Precision parameters.* For the components of variance reported in Table I, two averages of observed values should be considered significantly different at the 95% probability level if the difference equals or exceeds the critical differences listed in Table II.

10.3.1 Because only two carpets were used in this evaluation, users of the test method are advised to apply statistical caution in comparison of other carpets.

10.4 *Bias.* Fluorine content can be defined only in terms of a test method. While there are other independent means to determine fluorine content, any bias of this method in comparison to them have not been established.

11. Report

11.1 Report carpet description and date of testing.

11.2 Report any variation from procedure.

11.3 Report test result from 9.1.

12. Notes

12.1 Available from Publications Office, ACGIH, Kemper Woods Center, 1330 Kemper Meadow Dr., Cincinnati OH 45240; tel: 513/742-2020.

12.2 Total Ionic Adjustment Buffer (TISAB) is available from most laboratory supply companies. The buffer solution contains the following: sodium citrate, sodium hydroxide, acetic acid and water.

12.3 These calibrations of the electrode and meter simplify the calculations of fluorine content on carpet fibers. This 40:1 ratio accounts for the dilution of the hydrogen fluoride gas in the 40 mL of sodium hydroxide and TISAB solution.

12.4 In keeping with standard laboratory practices, two-point calibrations across the range to be tested give better precision and accuracy in determining fluoride content of solutions. For example, a two-point calibration may use 1 $\mu\text{g/g}$ (ppm) and 3 $\mu\text{g/g}$ (ppm) fluoride standards for 40.0 and 120.0 voltage output meter readings, respectively.

12.5 In order to prevent confusion, a sample numbering system should be established wherein all items to be used with a specific test specimen are numbered the same; i.e., paper envelopes, plastic cups, combustion flasks, etc.

12.6 For convenience in processing large numbers of samples, re-pipette dispensers may be used for the stock solutions of sodium hydroxide and TISAB buffer. If so, they should be checked for calibration daily.

12.7 The pressure from the pressure regulator should not exceed 14 kPa (2 psi). Exces-

**Table I—Components of Variance for Fluorine Content
(Expressed as Variances and Standard Deviations)^a**

Component	Variance	Standard Deviation $\mu\text{g/g}$ (ppm)
Specimens within materials and laboratories	630.0	25.1
Laboratories	378.0	19.4
Interaction: Materials times laboratories	59.6	7.7

^aSquare roots of the components of variance are reported to express the variability in the appropriate units of measure rather than as the squares of those units of measure.

**Table II—Critical Differences for the Conditions Noted
95% Probability Level, Single Material Comparisons $\mu\text{g/g}$ (ppm) Fluorine**

Number of Test Results in each Average	Within-Laboratory Precision		Between-Laboratory Precision	
	Standard Error	Critical Difference	Standard Error	Critical Difference
1	25.1	70	31.7	88
2	17.8	49	26.3	73
3	14.5	40	24.2	67
4	12.6	35	23.1	64
5	11.2	31	22.4	62
7	9.5	27	21.6	61
9	8.9	23	21.2	59

NOTE 1—The tabulated values of the critical differences should be considered to be a general statement, particularly with respect to between-laboratory precision.