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Test Method for Free and Hydrolyzed Formaldehyde: Water Extraction

1. Purpose and Scope

1.1 This test method is applicable to wettable (non-hydrophobic) textile fabrics that may contain free or hydrolyzed formaldehyde, particularly fabrics finished with chemicals containing formaldehyde. It provides a water extraction process and a subsequent colorimetric analysis of the amount of free and hydrolyzed formaldehyde extracted.

2. Principle

2.1 A weighed fabric specimen is submerged in a flask filled with deionized water. The sealed or capped flask is placed in a water bath at a controlled temperature for a specified length of time. The amount of formaldehyde extracted into the water is then determined colorimetrically.

3. Terminology

3.1 **free formaldehyde, n.**—formaldehyde that is not bonded to a finish or a fabric. In this form, formaldehyde can be readily extracted from the fabric by immersion in water.

3.2 **hydrolyzed formaldehyde, n.**—although originally part of larger chemical structure, such as a crosslinking reactant, the formaldehyde is cleaved from the larger molecule and dissolved into the water.

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 practice should be followed. Wear safety glasses in all laboratory areas.

4.2 When handling glacial acetic acid to prepare Nash reagent, use chemical goggles or face shield, impervious gloves and an impervious apron during preparation. Concentrated acids should be handled only in an adequately ventilated laboratory hood. CAUTION: Always add

acid to water.

4.3 Formaldehyde is a carcinogen. Use in an adequately ventilated laboratory hood. Avoid inhalation or skin contact. Use chemical goggles or face shield, impervious gloves and an impervious apron when working with formaldehyde (see 8.1 and 9.1).

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

4.5 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; see www.osha.gov for latest version). 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 14.3).

5. Uses and Limitations

5.1 The procedure is intended for use in the range of free and hydrolyzed formaldehyde on non-hydrophobic fabric from 20 µg/g up to about 3500 µg/g. This test method approximates hydrolyzed formaldehyde and closely simulates normal user conditions. However, TM206 is an aqueous immersion method and fabrics with a propensity for bleeding could be a concern; if the dye has an absorbance in the range of 412nm, it could interfere with the spectrophotometric measurement. It is not intended to approximate formaldehyde released under hot storage conditions.

5.2 Alternatively, AATCC TM112, Test Method for Formaldehyde Release from Fabric: Sealed Jar (see 14.1), determines free and hydrolyzed formaldehyde and additional formaldehyde as generated under more severe conditions than this test method. The appropriate test should be chosen based on that distinction (see 14.2).

6. Apparatus, Reagents, Materials

6.1 250-mL Erlenmeyer flasks with stoppers.

6.2 Weighing balance, with a minimum

sensitivity of 0.01 g.

6.3 Thermostatically controlled water bath: $40 \pm 1^\circ\text{C}$ ($105 \pm 2^\circ\text{F}$).

6.4 Nash reagent prepared from ammonium acetate, acetic acid and acetylacetone and water (see 7.1).

6.5 Formaldehyde solution, approximately 37% (see 4.3).

6.6 Volumetric flasks, 50, 250, 500 and 1000-mL, Class A.

6.7 Volumetric pipettes, 1, 2, 3, 5, 10, 15, 20, 25, 30 and 100-mL, all calibrated "to deliver" and meeting Class A volume accuracy and flow time requirements, or a digital, variable-volume pipette capable of meeting Class A requirements.

6.8 A 1-L clean brown glass bottle.

6.9 Spectrophotometer, capable of reading absorbance to a minimum of three decimal places, at a wavelength of 412 nm.

6.10 Test tubes (see 14.4).

6.11 Distilled or deionized water. Hereafter, referred to as deionized water.

6.12 pH meter, calibrated and accurate to 0.01

7. Preparation of Nash Reagent

7.1 Nash Reagent: In a 1000-mL volumetric flask, dissolve 150 g of ammonium acetate in 800-mL of deionized water; add 3.0 ± 0.1 mL of glacial acetic acid and 2.0 ± 0.1 mL of acetylacetone. Add deionized water up to the 1000-mL level and mix. Store in a capped brown bottle.

7.1.1 Newly made Nash Reagent requires a 12 h wait period before being used as the reagent darkens slightly in color during that time period. The reagent may be used up to 8 weeks after it is made. However, since the sensitivity may change slightly over a long period of time, it is good practice to run a calibration curve weekly or when used to correct for the slight changes in the standard curve.

8. Preparation of Standard Solution and Calibration

8.1 Prepare a 1500 µg/mL stock solution of formaldehyde by diluting 3.8-mL of reagent grade formaldehyde solution (approximately 37%, see 4.3) to 1.0-L with deionized water in a volumetric flask. Equilibrate the stock solution for a minimum of 24 h in a capped/stoppered flask before standardization. Determine the concentration of formaldehyde in the