

Formaldehyde Release from Fabric, Determination of: Sealed Jar Method

Developed in 1965 by AATCC Committee RR68; reaffirmed 1968, 1972, 1989, 1998, 2003; revised 1975, 1978, 1982, 1984, 1993; technical correction 1983; editorially revised 1985, 1986; editorially revised and reaffirmed (with new title) 1990, 2008.

1. Purpose and Scope

1.1 This test method is applicable to textile fabrics that may release formaldehyde, particularly fabrics finished with chemicals containing formaldehyde. It provides accelerated storage conditions and an analytical means for determining the amount of formaldehyde released under the conditions of accelerated storage (see Section 5 and 10.1).

1.2 An optional accelerated incubation procedure is available (see 13.5).

2. Principle

2.1 A weighed fabric specimen is suspended over water in a sealed jar. The jar is placed in an oven at a controlled temperature for a specified length of time (see 13.5). The amount of formaldehyde absorbed by the water is then determined colorimetrically.

3. Terminology

3.1 **formaldehyde release**, n.—that formaldehyde exuded from textiles under the accelerated storage conditions of this test, including that which is free (unbound or occluded) from unreacted chemicals, or from finish degradation as a result of this test.

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 sensory irritant and potential sensitizer. Its chronic toxicity has not been fully established. 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.

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 of January 1, 1989). 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 13.7).

5. Uses and Limitations

5.1 The procedure is intended for use in the range of releaseable formaldehyde on the fabric up to about 3500 µg/g. The upper limits are 500 µg/g if a 1:1 ratio of Nash reagent to sample solution is used in the analytical portion of the test and 3500 µg/g if a 10:1 ratio is used. The procedure promotes formaldehyde release from odor-free, fully cured durable press fabrics that have been afterwashed (Vail, S. L. and B. A. K. Andrews, *Textile Chemist and Colorist*, Vol. 11, No. 1, January 1979, p. 48). For these reasons, the procedure should not be used to estimate µg/g formaldehyde in air for compliance with any mandated or voluntary standards. The procedure was originally developed to measure the "propensity of a resin-treated fabric to liberate an excessive amount of formaldehyde under hot, humid conditions." (Nuessle, A. C., *American Dyestuff Reporter*, Vol. 55, No. 17, 1966, pp. 48-50; also Reid, J. D., R. L. Arcenaux, R. M. Reinhardt and J. A. Harris, *American Dyestuff Reporter*, Vol. 49, No. 14, 1960, pp. 29-34.)

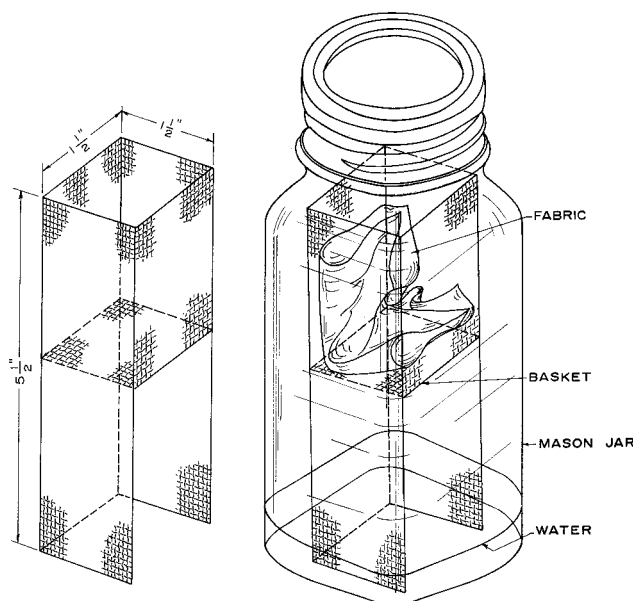


Fig. 1—The wire mesh basket detailed on the left is suspended in a sealed jar with one fabric specimen as shown on the right.

6. Apparatus and Materials

6.1 Mason or equivalent canning jars, 0.95 L (1 qt) and gas sealing caps.

6.2 Small wire mesh baskets (or other suitable means for suspending fabric above the water level inside the jars; see 13.1). As an alternative to the wire mesh baskets, a double strand of sewing thread may be used to make a loop in the fabric that has been folded in half twice, suspended above the water level. The two double-thread ends are draped over the top of the jar and held securely by the jar cap.

6.3 Thermostatically controlled oven, $49 \pm 1^\circ\text{C}$ ($120 \pm 2^\circ\text{F}$) (see 13.5).

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

6.5 Formaldehyde solution, approximately 37%.

6.6 Volumetric flasks, 50, 500 and 1000 mL.

6.7 Mohr pipettes, graduated in tenths of a mL; and 5, 10, 15, 20, 25, 30 and 50 mL volumetric pipettes, all calibrated "to deliver" and meeting Class B volume accuracy and flow time requirements (see 13.2).

6.8 Graduated cylinders, 10 and 50 mL, graduated in mL, calibrated "to deliver" and meeting Class A volume accuracy requirements (see 13.2).

6.9 Photoelectric colorimeter or spectrophotometer (see 10.6).

6.10 Test tubes or colorimeter tubes (see 13.2).

7. Preparation of Nash Reagent

7.1 In a 1000 mL volumetric flask, dissolve 150 g of ammonium acetate in about 800 mL of distilled water; add 3 mL of glacial acetic acid and 2 mL of acetylacetone. Make up to the mark with distilled water and mix. Store in a brown bottle.

7.2 The reagent darkens in color slightly on standing over the first 12 h. For this reason the reagent should be held 12 h before use. Otherwise, the reagent is usable over a considerable period of time, at least 6-8 weeks. However, since the sensitivity may change slightly over a long period of time, it is good practice to run a calibration curve weekly to correct for slight changes in the standard curve.

8. Preparation of Standard Solution and Calibration (Caution)

8.1 Prepare an approximately 1500 $\mu\text{g}/\text{mL}$ stock solution of formaldehyde by diluting 3.8 mL of reagent grade formaldehyde solution (approximately 37%) to one liter with distilled water. Equilibrate the stock solution for at least 24 h before standardization. Determine the concentration of formaldehyde in the stock solu-

tion by a standard method (see 13.6 or any other suitable procedure such as sodium sulfite titration using 0.1 *N* HCl. Reference: J. Frederick Walker, *Formaldehyde*, 3rd Ed. Reinhold Publ. Co., New York, 1964, p. 486). Record the actual concentration of this standardized stock solution. This stock solution will keep for at least four weeks and is used to prepare standard dilutions. A 1:10 dilution of the standardized formaldehyde stock solution is prepared by pipetting 25 mL of the standardized stock solution into a 250 mL volumetric flask and diluting to the mark with distilled water. If the stock solution is titrated and found to be different than 1500 $\mu\text{g}/\text{mL}$, three methods that can be used for preparing the calibration curves are:

8.1.1 Calculate new volumes of stock solution aliquots to pipette to achieve exactly 1.5, 3.0, 4.5, 6.0 and 9.0 $\mu\text{g}/\text{mL}$, respectively. (For example, if the formaldehyde stock solution were found to be 1470 $\mu\text{g}/\text{mL}$ by titration, not 1500 $\mu\text{g}/\text{mL}$, pipette 5.1 mL, 10.2 mL, 15.3 mL, 20.4 mL and 30.6 mL of the 147 $\mu\text{g}/\text{mL}$ dilution into a 500 mL volumetric flask and dilute to mark with distilled water.) (NOTE: It is easy to make errors using a graduated pipette!)

8.1.2 Pipette 5, 10, 15, 20 and 30 mL of the 1:10 dilution into a 500 mL volumetric flask and dilute to mark with distilled water. (If, for example, the standardized stock solution were found to be 1470 $\mu\text{g}/\text{mL}$ by titration, calculate new values for the calibration curve abscissa; i.e., 1.47, 2.94, 4.41, 5.88, 8.82 $\mu\text{g}/\text{mL}$.) This is the preferred method for those with a microprocessor spectrophotometer or computer. However, it is harder to plot manually.

8.1.3 Calculate a concentration correction factor for each sample. Correct the concentration for the dilution value using this factor. Plot the curve as if each of the dilutions were exactly 1.5, 3.0, 4.5, 6.0 and 9.0 $\mu\text{g}/\text{mL}$. Calculate the correct concentration for each of these values using the factor. For example, if the standardized stock solution were found to be 1470 $\mu\text{g}/\text{mL}$ by titration, then the correction factor (*CF*) is:

$$CF = \frac{\text{Actual}}{\text{Nominal}} = \frac{1470}{1500} = 0.980$$

8.2 When 5, 10, 15, 20 and 30 mL aliquots of the 1:10 dilution of the standardized stock solution from 8.1 are diluted with distilled water in 500 mL volumetric flasks, formaldehyde solutions containing approximately 1.5, 3.0, 4.5, 6.0 and 9.0 $\mu\text{g}/\text{mL}$ formaldehyde respectively will be obtained. Record accurately the concentration of solutions. The equivalent concentrations of the formaldehyde in the test fabric based on the weight of

1 g of the test fabric and 50 mL of water in the test jars, will be 50 times the accurate concentrations of these standard solutions.

8.3 Use 5 mL aliquots of each of the standard solutions and the procedure described in 10.4-10.7 to prepare a calibration chart in which $\mu\text{g}/\text{mL}$ formaldehyde are plotted against absorbance.

9. Test Specimens

9.1 Cut approximately 1 g specimens; weigh each one to ± 0.01 g.

10. Procedures

10.1 Place 50 mL of distilled water in the bottom of each jar. Suspend one fabric specimen above the water in each jar, using a wire mesh basket or other means (see Fig. 1). Seal the jars and place them in the oven at $49 \pm 1^\circ\text{C}$ ($120 \pm 2^\circ\text{F}$) for 20 h (see 13.5).

10.2 Remove and cool the jars for at least 30 min.

10.3 Remove the fabric and baskets, or other support, from the jars. Recap the jars and shake them to mix any condensation formed on the jar sides.

10.4 Pipette 5 mL of Nash reagent into a suitable number of test tubes, small (50 mL) Erlenmeyer flasks, or other suitable flasks (colorimeter or spectrophotometer tubes can be used directly, see 13.2) and pipette 5 mL of the reagent into at least one additional tube for a reagent blank. Add 5 mL aliquots from each of the sample incubation jars to the tubes and 5 mL of distilled water to the tube which is used as a reagent blank.

10.5 Mix and place the tubes in a $58 \pm 1^\circ\text{C}$ water bath for 6 min. Remove and cool.

10.6 Read the absorbance in the colorimeter or spectrophotometer against the reagent blank using a blue filter or a wavelength of 412 nm. **Caution:** Exposure of the developed yellow color to direct sunlight for a period of time will cause some fading. If there is appreciable delay in reading the tubes after color development and strong sunlight is present, care should be exercised to protect the tubes such as by covering them with a cardboard box or by similar means. Otherwise the color is stable for considerable time (at least overnight) and reading may be delayed.

10.7 Determine the $\mu\text{g}/\text{mL}$ formaldehyde (HCHO) in the sample solutions using the prepared calibration curve (see 8.3 and 13.3).

11. Calculation

11.1 Calculate the amount of formaldehyde released for each specimen to the nearest $\mu\text{g}/\text{g}$ using the following equation:

$$F = (C) (50)/W$$