

| Citation | Requirement | Applies to subpart HHHH | Explanation |
|---------------|--|-------------------------|-------------|
| § 63.15 | Availability of Information/Confidentiality. | Yes. | |

APPENDIX A TO SUBPART HHHH OF
PART 63—METHOD FOR DETERMINING
FREE-FORMALDEHYDE IN UREA-
FORMALDEHYDE RESINS BY SODIUM
SULFITE (ICED & COOLED)

1.0 Scope

This procedure corresponds to the Housing and Urban Development method of determining free-formaldehyde in urea-formaldehyde resins. This method applies to samples that decompose to yield formaldehyde under the conditions of other free-formaldehyde methods. The primary use is for urea-formaldehyde resins.

2.0 Part A—Testing Resins

Formaldehyde will react with sodium sulfite to form the sulfite addition products and liberate sodium hydroxide (NaOH); however, at room temperature, the methanol groups present will also react to liberate NaOH. Titrate at 0 degrees Celsius (°C) to minimize the reaction of the methanol groups.

2.1 Apparatus Required.

- 2.1.1 Ice crusher.
- 2.1.2 One 100-milliliter (mL) graduated cylinder.
- 2.1.3 Three 400-mL beakers.
- 2.1.4 One 50-mL burette.
- 2.1.5 Analytical balance accurate to 0.1 milligrams (mg).
- 2.1.6 Magnetic stirrer.
- 2.1.7 Magnetic stirring bars.
- 2.1.8 Disposable pipettes.
- 2.1.9 Several 5-ounce (oz.) plastic cups.
- 2.1.10 Ice cube trays (small cubes).

2.2 Materials Required.

- 2.2.1 Ice cubes (made with distilled water).
- 2.2.2 A solution of 1 molar (M) sodium sulfite (Na₂SO₃) (63 grams (g) Na₂SO₃/500 mL water (H₂O) neutralized to thymolphthalein endpoint).
- 2.2.3 Standardized 0.1 normal (N) hydrochloric acid (HCl).
- 2.2.4 Thymolphthalein indicator (1.0 g thymolphthalein/199 g methanol).
- 2.2.5 Sodium chloride (NaCl) (reagent grade).
- 2.2.6 Sodium hydroxide (NaOH).

2.3 Procedure.

2.3.1 Prepare sufficient quantity of crushed ice for three determinations (two trays of cubes).

2.3.2 Put 70 cubic centimeters (cc) of 1 M Na₂SO₃ solution into a 400-mL beaker. Begin stirring and add approximately 100 g of crushed ice and 2 g of NaCl. Maintain 0 °C during test, adding ice as necessary.

2.3.3 Add 10–15 drops of thymolphthalein indicator to the chilled solution. If the solution remains clear, add 0.1 N NaOH until the solution turns blue; then add 0.1 N HCl back to the colorless endpoint. If the solution turns blue upon adding the indicator, add 0.1 N HCl to the colorless endpoint.

2.3.4 On the analytical balance, accurately weigh the amount of resin indicated under the “Resin Sample Size” chart (see below) as follows.

RESIN SAMPLE SIZE

| Approximate free HCHO (percent) | Sample weight (gram(s)) |
|------------------------------------|-------------------------------|
| <0.5 | 10 |
| 0.5–1.0 | 5 |
| 1.0–3.0 | 2 |
| 3.0 | 1 |

2.3.4.1 Pour about 1 inch of resin into a 5 oz. plastic cup.

2.3.4.2 Determine the gross weight of the cup, resin, and disposable pipette (with the narrow tip broken off) fitted with a small rubber bulb.

2.3.4.3 Pipette out the desired amount of resin into the stirring, chilled solution (approximately 1.5 to 2 g per pipette-full).

2.3.4.4 Quickly reweigh the cup, resin, and pipette with the bulb.

2.3.4.5 The resultant weight loss equals the grams of resin being tested.

2.3.5 Rapidly titrate the solution with 0.1 N HCl to the colorless endpoint described in Step 3 (2.3.3).

2.3.6 Repeat the test in triplicate.

2.4 Calculation.

2.4.1 The percent free-formaldehyde (%HCHO) is calculated as follows:

$$\% \text{HCHO} = \frac{(\text{mL } 0.1 \text{ N HCl}) (\text{N of Acid}) (3.003)}{\text{Weight of Sample}}$$

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2.4.2 Compute the average percent free-formaldehyde of the three tests.

(NOTE: If the results of the three tests are not within a range of ± 0.5 percent or if the average of the three tests does not meet expected limits, carry out Part B and then repeat Part A.)

3.0 Part B—Standard Check

Part B ensures that test reagents used in determining percent free-formaldehyde in urea-formaldehyde resins are of proper concentration and that operator technique is correct. Should any doubts arise in either of these areas, the formaldehyde standard solution test should be carried out.

3.1 Preparation and Standardization of a 1 Percent Formalin Solution.

Prepare a solution containing approximately 1 percent formaldehyde from a stock 37 percent formalin solution. Standardize the prepared solution by titrating the hydroxyl ions resulting from the formation of the formaldehyde bisulfite complex.

3.2 Apparatus Required.

NOTE: All reagents must be American Chemical Society analytical reagent grade or better.

3.2.1 One 1-liter (L) volumetric flask (class A).

3.2.2 One 250-mL volumetric flask (class A).

3.2.3 One 250-mL beaker.

3.2.4 One 100-mL pipette (class A).

3.2.5 One 10-mL pipette (class A).

3.2.6 One 50-mL graduated cylinder (class A).

3.2.7 A pH meter, standardized using pH 7 and pH 10 buffers.

3.2.8 Magnetic stirrer.

3.2.9 Magnetic stirring bars.

3.2.10 Several 5-oz. plastic cups.

3.2.11 Disposal pipettes.

3.2.12 Ice cube trays (small cubes).

3.3 Materials Required.

3.3.1 A solution of 37 percent formalin.

3.3.2 Anhydrous Na_2SO_3 .

3.3.3 Distilled water.

3.3.4 Standardized 0.100 N HCl.

3.3.5 Thymolphthalein indicator (1.0 g thymolphthalein/199 g methanol).

3.4 Preparation of Solutions and Reagents.

3.4.1 Formaldehyde Standard Solution (approximately 1 percent). Measure, using a graduated cylinder, 27.0 mL of analytical reagent 37 percent formalin solution into a 1-L volumetric flask. Fill the flask to volume with distilled water.

(NOTE: You must standardize this solution as described in section 3.5. This solution is stable for 3 months.)

3.4.2 Sodium Sulfite Solution 1.0 M (used for standardization of Formaldehyde Standard Solution). Quantitatively transfer, using distilled water as the transfer solvent, 31.50 g of anhydrous Na_2SO_3 into a 250-mL volu-

metric flask. Dissolve in approximately 100 mL of distilled water and fill to volume.

(NOTE: You must prepare this solution daily, but the calibration of the Formaldehyde Standard Solution needs to be done only once.)

3.4.3 Hydrochloric Acid Standard Solution 0.100 M. This reagent should be readily available as a primary standard that only needs to be diluted.

3.5 Standardization.

3.5.1 Standardization of Formaldehyde Standard Solution.

3.5.1.1 Pipette 100.0 mL of 1 M sodium sulfite into a stirred 250-mL beaker.

3.5.1.2 Using a standardized pH meter, measure and record the pH. The pH should be around 10. It is not essential the pH be 10; however, it is essential that the value be accurately recorded.

3.5.1.3 To the stirring Na_2SO_3 solution, pipette in 10.0 mL of Formaldehyde Standard Solution. The pH should rise sharply to about 12.

3.5.1.4 Using the pH meter as a continuous monitor, titrate the solution back to the original exact pH using 0.100 N HCl. Record the milliliters of HCl used as titrant. (NOTE: Approximately 30 to 35 mL of HCl will be required.)

3.5.1.5 Calculate the concentration of the Formaldehyde Standard Solution using the equation as follows:

$$\% \text{HCHO} = \frac{(\text{mL HCl}) (\text{N HCl}) (3.003)}{\text{mL sample}}$$

3.6 Procedure.

3.6.1 Prepare a sufficient quantity of crushed ice for three determinations (two trays of cubes).

3.6.2 Put 70 cc of 1 M Na_2SO_3 solution into a 400-mL beaker. Begin stirring and add approximately 100 g of crushed ice and 2 g NaCl. Maintain 0 °C during the test, adding ice as necessary.

3.6.3 Add 10–15 drops of thymolphthalein indicator to the chilled solution. If the solution remains clear, add 0.1 N NaOH until the solution turns blue; then add 0.1 N HCl back to the colorless endpoint. If the solution turns blue upon adding the indicator, add 0.1 N HCl to the colorless endpoint.

3.6.4 On the analytical balance, accurately weigh a sample of Formaldehyde Standard Solution as follows.

3.6.4.1 Pour about 0.5 inches of Formaldehyde Standard Solution into a 5-oz. plastic cup.

3.6.4.2 Determine the gross weight of the cup, Formaldehyde Standard Solution, and a disposable pipette fitted with a small rubber bulb.

3.6.4.3 Pipette approximately 5 g of the Formaldehyde Standard Solution into the stirring, chilled Na_2SO_3 solution.

3.6.4.4 Quickly reweigh the cup, Formaldehyde Standard Solution, and pipette with the bulb.

3.6.4.5 The resultant weight loss equals the grams of Formaldehyde Standard Solution being tested.

3.6.5 Rapidly titrate the solution with 0.1 N HCl to the colorless endpoint in Step 3 (3.6.3).

3.6.6 Repeat the test in triplicate.

3.7 *Calculation for Formaldehyde Standard Solution.*

3.7.1 The percent free-formaldehyde (% HCHO) is calculated as follows:

$$\% \text{HCHO} = \frac{(\text{mL } 0.1 \text{ N HCl})(\text{N Acid})(3.003)}{\text{Weight of Formaldehyde Standard Solution}}$$

3.7.2 The range of the results of three tests should be no more than ± 5 percent of the actual Formaldehyde Standard Solution concentration. Report results to two decimal places.

3.8 Reference.

West Coast Adhesive Manufacturers Trade Association Test 10.1.

APPENDIX B TO SUBPART HHHH OF PART 63—METHOD FOR THE DETERMINATION OF LOSS-ON-IGNITION

1.0 Purpose

The purpose of this test is to determine the loss-on-ignition (LOI) of wet-formed fiberglass mat.

2.0 Equipment

2.1 Scale sensitive to 0.001 gram (g).

2.2 Drying oven equipped with a means of constant temperature regulation and mechanical air convection.

2.3 Furnace designed to heat to at least 625°C ($1,157^{\circ}\text{F}$) and controllable to $\pm 25^{\circ}\text{C}$ ($\pm 45^{\circ}\text{F}$).

2.4 Crucible, high form, 250 milliliter (mL).

2.5 Desiccator.

2.6 Pan balance (see Note 2 in 4.9)

3.0 Sample Collection Procedure

3.1 Obtain a sample of mat in accordance with Technical Association of the Pulp and Paper Industry (TAPPI) method 1007 "Sample Location."

3.2 Use a 5- to 10-g sample cut into pieces small enough to fit into the crucible.

3.3 Place the sample in the crucible. (NOTE 1: To test without the use of a crucible, see Note 2 after Section 4.8.)

3.4 Condition the sample in the furnace set at $105 \pm 3^{\circ}\text{C}$ ($221 \pm 9^{\circ}\text{F}$) for 5 minutes ± 30 seconds.

4.0 Procedure

4.1 Condition each sample by drying for 5 minutes ± 30 seconds at $105 \pm 3^{\circ}\text{C}$ ($22 \pm 5^{\circ}\text{F}$).

4.2 Remove the test sample from the furnace and cool in the desiccator for 30 minutes in the standard atmosphere for testing glass textiles.

4.3 Place the empty crucible in the furnace at $625 \pm 25^{\circ}\text{C}$ ($1,157 \pm 45^{\circ}\text{F}$). After 30 minutes, remove and cool the crucible in the standard atmosphere (TAPPI method 1008) for 30 minutes.

4.4 Identify each crucible with respect to each test sample of mat.

4.5 Weigh the empty crucible to the nearest 0.001 g. Record this weight as the tare mass, T.

4.6 Place the test sample in the crucible and weigh to the nearest 0.001 g. Record this weight as the initial mass, A.

4.7 Place the test sample and crucible in the furnace and ignite at $625 \pm 25^{\circ}\text{C}$ ($1,157 \pm 45^{\circ}\text{F}$).

4.8 After ignition for at least 30 minutes, remove the test sample and crucible from the furnace and cool in the desiccator for 30 minutes in the standard atmosphere (TAPPI method 1008).

4.9 Remove each crucible, and test each sample separately from the desiccator, and immediately weigh each sample to the nearest 0.001 g. Record this weight as the ignited mass, B. (NOTE 2: When it is known that no ash residue separates from the test sample during the weighing and igniting processes, you may weigh the sample separately without the crucible. When this occurs, the tare mass (T) equals zero. With appropriate care, you can dry and weigh a single piece of mat and place with tongs into the ignition oven on appropriate refractory supports. When the ignition time is over, remove the sample as an intact fragile web and weigh it directly on a pan balance.)

5.0 Calculation

5.1 Calculate the LOI for each sample as follows:

$$\% \text{ LOI} = 100 \times (A - B) / (A - T)$$

Where: