

**Pt. 63, Subpt. NNN, App. B**

**40 CFR Ch. I (7-1-13 Edition)**

NOTE: On air duct products, remove shiplaps and overspray.

3.3 Place specimen in furnace at 540 °C (1,000 °F), ±10 °C (50 °F) for 15 to 20 minutes to insure complete oxidation. After ignition, fibers should be white and should not be fused together.

3.4 Remove specimen from the furnace and cool to room temperature.

3.5 Weigh cooled specimen and wire tray to the nearest 0.1 gram. Deduct the weight of the wire tray and then calculate the loss in weight as a percent of the original specimen weight.

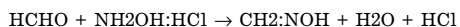
**APPENDIX B TO SUBPART NNN OF PART 63—FREE FORMALDEHYDE ANALYSIS OF INSULATION RESINS BY HYDROXYLAMINE HYDROCHLORIDE**

*1. Scope*

This method was specifically developed for water-soluble phenolic resins that have a relatively high free-formaldehyde (FF) content such as insulation resins. It may also be suitable for other phenolic resins, especially those with a high FF content.

*2. Principle*

2.1 a. The basis for this method is the titration of the hydrochloric acid that is liberated when hydroxylamine hydrochloride reacts with formaldehyde to form formaldoxime:



b. Free formaldehyde in phenolic resins is present as monomeric formaldehyde, hemiformals, polyoxymethylene hemiformals, and polyoxymethylene glycols. Monomeric formaldehyde and hemiformals react rapidly with hydroxylamine hydrochloride, but the polymeric forms of formaldehyde must hydrolyze to the monomeric state before they can react. The greater the concentration of free formaldehyde in a resin, the more of that formaldehyde will be in the polymeric form. The hydrolysis of these polymers is catalyzed by hydrogen ions.

2.2 The resin sample being analyzed must contain enough free formaldehyde so that the initial reaction with hydroxylamine hydrochloride will produce sufficient hydrogen ions to catalyze the depolymerization of the polymeric formaldehyde within the time limits of the test method. The sample should contain approximately 0.3 grams free formaldehyde to ensure complete reaction within 5 minutes.

*3. Apparatus*

- 3.1 Balance, readable to 0.01 g or better.
- 3.2 pH meter, standardized to pH 4.0 with pH 4.0 buffer and pH 7 with pH 7.0 buffer.

- 3.3 50-mL burette for 1.0 N sodium hydroxide.
- 3.4 Magnetic stirrer and stir bars.
- 3.5 250-mL beaker.
- 3.6 50-mL graduated cylinder.
- 3.7 100-mL graduated cylinder.
- 3.8 Timer.

*4. Reagents*

- 4.1 Standardized 1.0 N sodium hydroxide solution.
- 4.2 Hydroxylamine hydrochloride solution, 100 grams per liter, pH adjusted to 4.00.
- 4.3 Hydrochloric acid solution, 1.0 N and 0.1 N.
- 4.4 Sodium hydroxide solution, 0.1 N.
- 4.5 50/50 v/v mixture of distilled water and methyl alcohol.

*5. Procedure*

- 5.1 Determine the sample size as follows:
  - a. If the expected FF is greater than 2 percent, go to Part A to determine sample size.
  - b. If the expected FF is less than 2 percent, go to Part B to determine sample size.
  - c. Part A: Expected FF ≥ 2 percent.

Grams resin = 60/expected percent FF

- i. The following table shows example levels:

Expected % free formaldehyde	Sample size, grams
2 .....	30.0
5 .....	12.0
8 .....	7.5
10 .....	6.0
12 .....	5.0
15 .....	4.0

- ii. It is very important to the accuracy of the results that the sample size be chosen correctly. If the milliliters of titrant are less than 15 mL or greater than 30 mL, reestimate the needed sample size and repeat the tests.

d. Part B: Expected FF < 2 percent

Grams resin = 30/expected percent FF

- i. The following table shows example levels:

Expected % free formaldehyde	Sample size, grams
2 .....	15
1 .....	30
0.5 .....	60

- ii. If the milliliters of titrant are less than 5 mL or greater than 30 mL, reestimate the needed sample size and repeat the tests.

5.2 Weigh the resin sample to the nearest 0.01 grams into a 250-mL beaker. Record sample weight.

5.3 Add 100 mL of the methanol/water mixture and stir on a magnetic stirrer. Confirm that the resin has dissolved.

## Environmental Protection Agency

## § 63.1400

5.4 Adjust the resin/solvent solution to pH 4.0, using the prestandardized pH meter, 1.0 N hydrochloric acid, 0.1 N hydrochloric acid, and 0.1 N sodium hydroxide.

5.5 Add 50 mL of the hydroxylamine hydrochloride solution, measured with a graduated cylinder. Start the timer.

5.6 Stir for 5 minutes. Titrate to pH 4.0 with standardized 1.0 N sodium hydroxide. Record the milliliters of titrant and the normality.

### 6. Calculations

$$\% \text{ FF} = \frac{\text{mL sodium hydroxide} \times \text{normality} \times 3.003}{\text{grams of sample}}$$

### 7. Method Precision and Accuracy

Test values should conform to the following statistical precision:

Variance = 0.005

Standard deviation = 0.07

95% Confidence Interval, for a single determination = 0.2

### 8. Author

This method was prepared by K. K. Tutin and M. L. Foster, Tacoma R&D Laboratory, Georgia-Pacific Resins, Inc. (Principle written by R. R. Conner.)

### 9. References

9.1 GPAM 2221.2.

9.2 PR&C TM 2.035.

9.3 Project Report, Comparison of Free Formaldehyde Procedures, January 1990, K. K. Tutin.

## APPENDIX C TO SUBPART NNN OF PART 63—METHOD FOR THE DETERMINATION OF PRODUCT DENSITY

### 1. Purpose

The purpose of this test is to determine the product density of cured blanket insulation. The method is applicable to all cured board and blanket products.

### 2. Equipment

One square foot (12 in. by 12 in.) template, or templates that are multiples of one square foot, for use in cutting insulation samples.

### 3. Procedure

3.1 Obtain a sample at least 30 in. long across the machine width. Sample should be free of dirt or foreign matter.

3.2 Lay out the cutting pattern according to the plant's written procedure for the designated product.

3.2 Cut samples using one square foot (or multiples of one square foot) template.

3.3 Weigh product and obtain area weight (lb/ft<sup>2</sup>).

3.4 Measure sample thickness.

3.5 Calculate the product density:

Density (lb/ft<sup>3</sup>) = area weight (lb/ft<sup>2</sup>)/thickness (ft)

## Subpart OOO—National Emission Standards for Hazardous Air Pollutant Emissions: Manufacture of Amino/Phenolic Resins

SOURCE: 65 FR 3290, Jan. 20, 2000, unless otherwise noted.

### § 63.1400 Applicability and designation of affected sources.

(a) *Applicability.* The provisions of this subpart apply to the owner or operator of processes that produce amino/phenolic resins and that are located at a plant site that is a major source as defined in § 63.2.

(b) *Affected source.* The affected source is:

(1) The total of all amino/phenolic resin process units (APPU);

(2) The associated heat exchange systems;

(3) Equipment required by, or utilized as a method of compliance with, this subpart which may include control devices and recovery devices;

(4) Equipment that does not contain organic hazardous air pollutants (HAPs) and is located within an APPU that is part of an affected source;

(5) Vessels and equipment storing and/or handling material that contain no organic HAP and/or organic HAP as impurities only;

(6) Equipment that is intended to operate in organic HAP service for less than 300 hours during the calendar year;

(7) Each waste management unit; and

(8) Maintenance wastewater.