General provisions citation	Requirement	Applies to subpart DDD?	Explanation
63.10(d)(2)	Performance Test Results	Yes.	
63.10(d)(3)	Opacity or VE Observations	No	Subpart DDD does not include VE/opacity standards.
63.10(d)(4)–(d)(5)	Progress Reports/ Startup, Shutdown, and Malfunction Reports.	Yes.	
63.10(e)(1)–(e)(2)	Additional CMS Reports	No	Subpart DDD does not require CEMS or CMS performance evaluations.
63.10(e)(3)	Excess Emissions/CMS Performance Reports.	Yes.	
63.10(e)(4)	COMS Data Reports	No	Subpart DDD does not require COMS.
63.10(f)	Recordkeeping/Reporting Waiver	Yes.	
63.11(a)	Control Device Requirements Applicability.	Yes.	
63.11(b)	Flares	No	Flares not applicable.
63.12	State Authority and Delegations	Yes.	
63.13	Addresses	Yes.	
63.14	Incorporation by Reference	Yes.	
63.15	Information Availability/Confidentiality.	Yes.	

Appendix A to Subpart DDD of Part 63—Free Formaldehyde Analysis of Insulation Resins by the Hydroxylamine Hydrochloride Method

1. Scope

The method in this appendix 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 formaldoxine:

HCHO + NH2OH:HCl → CH2:NOH + H2O + HCl

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 (g) 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 in 5.1.c to determine sample size.
b. If the expected FF is less than 2 percent, go to Part B in 5.1.d 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 percent 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 percent 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.

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

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

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. Tutin.