



Standard Test Method for Measurement of Evolved Formaldehyde from Water Reducible Air-Dry Coatings¹

This standard is issued under the fixed designation D 6191; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method may be used to measure the amount of formaldehyde evolved from air-dry water reducible coatings utilizing latices, resin emulsions, or water reducible alkyds. The results may be used to define the “free” formaldehyde evolved from a sample under controlled laboratory conditions.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 2194 Test Method for Concentration of Formaldehyde Solutions.²

3. Summary of Test Method

3.1 A sample of water reducible coating is placed on the walls of a test tube with a drawdown rod and purged with nitrogen. The evolved formaldehyde is collected in deionized water with tandem impingers and measured colorimetrically using acetylacetone at 412 nm.

4. Significance and Use

4.1 This test method measures the amount of formaldehyde that is released from a coating under laboratory conditions. The amount of formaldehyde available for release from a coating may vary depending on composition, and may decrease as the sample ages.

4.2 This test method may be used for typical air dried paints where water is the major volatile material. The useful range is estimated to be from 10 ppm to 1000 ppm formaldehyde in the sample.

4.3 Significant amounts of other volatile aldehydes, such as acetaldehyde, are reported to cause an interference with the determination of formaldehyde. This limitation is not expected to cause a problem for most common water reducible coatings.

4.4 Samples containing organic solvents as the major volatile component have not been evaluated and are not expected to be compatible with this test method.

5. Apparatus

5.1 *Formaldehyde Collection Apparatus*, assembled using a flowmeter to control the nitrogen flow at ~ 1.0 L/min, a 25 by 200 mm outside diameter long glass test tube, and two 30-mL midjet impingers. Connections are made with glass tubes through a No. 4 two-hole rubber stopper and clear flexible tubing (see Fig. 1).

5.2 *Drawdown Bar*, stainless steel No. 22 wire-wound 6.4-mm rod.

5.3 *Glass Sample Bottles*, (~ 2 oz), capable of holding 30-mL solution.

5.4 *Spectrophotometer*, capable of measuring the absorbance of solutions at 412 nm with a minimum sample light path of 10 mm.

6. Reagents

6.1 *Acetylacetone Solution*—Dissolve 150 ± 0.1 g ammonium acetate in ~ 600 mL deionized water in a 1-L volumetric flask. Add 3.0-mL glacial acetic acid and 2.0-mL acetylacetone (2,4 pentanedione) and dilute to volume.

6.2 *Formaldehyde Solution* —Use 37 % in methanol/water, commercial stock standard solution. If a commercially calibrated 37 % standard solution is not being used, the concentration of formaldehyde may determined in accordance with Test Method D 2194. Then, the actual concentrations of the working calibrations standards are to be calculated using the measured value.

NOTE 1—**Precaution:** Formaldehyde, acetylacetone, and the other reagents used in this test method are toxic or hazardous. Exposure to these materials should be minimized. Consult the supplier’s Material Safety Data Sheet for specific hazard information.

6.3 *Formaldehyde Calibration Standards*—Prepare a 100-ppm stock solution by diluting 25 μ L of the 37 % formaldehyde solution to 100 mL with deionized water. Dilute this intermediate stock solution to prepare at least 5 calibration standards. Pipeting 0.5, 3, 5, 7 and 10 mL aliquots of the stock solution into 100-mL volumetric flasks and diluting with deionized water will prepare calibration standards of 0.5, 3, 5, 7 and 10 ppm formaldehyde.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.21 on Chemical Analysis of Paints and Paint Materials.

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² *Annual Book of ASTM Standards*, Vol 06.04.

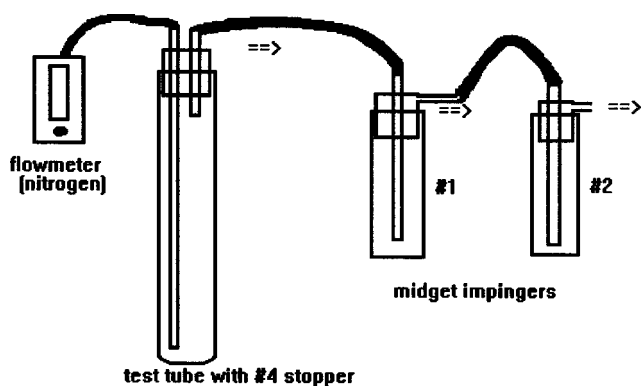


FIG. 1 Formaldehyde Collection Apparatus

7. Calibration

7.1 Prepare a series of at least 5 standards (6.3) plus a blank by adding 5 mL of the formaldehyde calibration standards or 5 mL deionized water into test tubes. Process the standard along with the samples as described in 8.5 to 8.6.

7.2 Construct a calibration curve by plotting the absorbance of the calibration standards versus concentration of formaldehyde (ppm). Prepare a new calibration curve with every batch of samples being analyzed.

8. Procedure

8.1 Assemble the collection apparatus as shown in Fig. 1. The ~1.0 L/min. flow rate should be determined by timing the flow with a bubble tube. If the impingers are not accurately graduated, pipet 15-mL deionized water into the impinger tubes and mark the level for future reference.

8.2 Place about 1 to 1.5 g of the liquid sample into a tared test tube and evenly spread the sample on the walls with a No. 22 drawdown rod. The bottom couple of inches of the test tube will not be coated and the top ~1/2 in. of the tube should be wiped free of coating with a paper towel to avoid having paint on the stopper. Quickly weigh the coated test tube and record the weight of the liquid sample.

8.3 Without delay, connect the test tube to the apparatus containing 15-mL deionized water in each impinger and purge with nitrogen at ~1.0 L/min for 2 h.

8.4 With the nitrogen flowing, remove the impinger tube from the No. 2 impinger. If the sampling liquid is less than the original 15 mL volume, restore the volume to 15 mL while rinsing the impinger tube with deionized water. Disconnect the No. 1 impinger in the series and rinse the impinger tube with

deionized water in order to restore the volume to 15 mL. Pour the No. 1 impinger solution, which should contain most of the formaldehyde, into a ~2-oz. bottle. Use the No. 2 impinger solution to wash the No. 1 impinger tube and mix the combined solutions in the sealed bottle.

8.5 Pipet 5 mL of the impinger solution (or a diluted solution) into a ~20-mL glass test tube and add 5 mL of the acetylacetone solution. Mix the prepared test solutions and heat the test tubes in a 40 ± 0.5°C water bath for 30 min.

8.6 Remove the test tubes from the water bath and let stand for 30 min to cool. Measure the absorbance at 412 nm with a spectrophotometer using a 1-cm cell and deionized water as the reference.

8.7 From the calibration curve, determine the concentration of the combined impinger solution. If the concentration of the test solution is greater than the highest standard, make appropriate dilutions of the impinger solution and repeat 8.5 to 8.6 with freshly prepared standards. Some analysts may prefer to prepare dilutions initially rather than repeat the standard preparation process.

9. Calculation

9.1 Calculate the amount of formaldehyde that is evolved from the sample as follows:

$$\text{ppm Formaldehyde} = \frac{C \times 30 \times D}{W} \quad (1)$$

where:

C = concentration from calibration curve (8.7),

30 = volume (mL) of water in the impingers,

D = dilution factor if diluted, and

W = sample weight, g.

10. Report

10.1 Report the concentration of evolved formaldehyde from the sample as parts per million rounded to two significant digits.

11. Precision and Bias

11.1 *Repeatability*—To be determined.

11.2 *Reproducibility*—To be determined.

11.3 *Bias*—The bias can not be determined since no valid stable reference material is available.

12. Keywords

12.1 air-dry water reducible coatings; evolved formaldehyde

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