



Standard Test Method for Determining Formaldehyde Concentrations in Air from Wood Products Using a Small-Scale Chamber¹

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1. Scope

1.1 This test method measures the formaldehyde concentrations in air emitted by wood product test specimens under defined test conditions of temperature and relative humidity. Results obtained from this small-scale chamber test method are intended to be comparable to results obtained from testing larger product samples by the large chamber test method for wood products, ASTM Test Method [E1333](#). The results may be correlated to values obtained from ASTM Test Method [E1333](#). The quantity of formaldehyde in an air sample from the small chamber is determined by a modification of NIOSH 3500 chromotropic acid test procedure. As with ASTM Test Method [E1333](#), other analytical procedures may be used to determine the quantity of formaldehyde in the air sample provided that such methods give results comparable to those obtained by using the chromotropic acid procedure. However, the test results and test report must be properly qualified and the analytical procedure employed must be accurately described.

1.2 The wood-based panel products to be tested by this test method are characteristically used for different applications and are tested at different relative amounts or loading ratios to reflect different applications. This is a test method that specifies testing at various loading ratios for different product types. However, the test results and test report must be properly qualified and must specify the make-up air flow, sample surface area, and chamber volume.

1.3 Ideal candidates for small-scale chamber testing are products relatively homogeneous in their formaldehyde release characteristics. Still, product inhomogeneities must be considered when selecting and preparing samples for small-scale chamber testing.

1.4 The values stated in SI units are the standard values. Any values given in parentheses are for information only.

¹ This test method is under the jurisdiction of ASTM Committee [D07](#) on Wood and is the direct responsibility of Subcommittee [D07.03](#) on Panel Products.

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1.5 *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:²

D3195 Practice for Rotameter Calibration

D5197 Test Method for Determination of Formaldehyde and Other Carbonyl Compounds in Air (Active Sampler Methodology)

D5221 Test Method for Continuous Measurement of Formaldehyde in Air (Withdrawn 1997)³

E77 Test Method for Inspection and Verification of Thermometers

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E741 Test Method for Determining Air Change in a Single Zone by Means of a Tracer Gas Dilution

E1333 Test Method for Determining Formaldehyde Concentrations in Air and Emission Rates from Wood Products Using a Large Chamber

2.2 U.S. Department of Housing and Urban Development Standard:⁴

HUD 24 CFR 3280, Manufactured Home Construction and Safety Standards

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>; request Federal Register, Vol 49, No. 155, Aug. 8, 1984

2.3 *National Institute for Occupational Safety and Health Standard:*

NIOSH 3500 Formaldehyde Method⁵

2.4 *Other Documents:*

Minnesota Statutes Sections 144.495, 325f.18, and 325F.181—Formaldehyde Gases in Building Materials⁶

California Air Resources Board (CARB) California Code of Regulations sections 93120-93120.12, title 17, Airborne Toxic Control Measure to Reduce Formaldehyde Emissions from Composite Wood Products

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *air change rate, (Q/V):* the ratio of the conditioned and filtered air, Q , that enters or is replaced in the small chamber in one hour divided by the interior volume of the small chamber, V , air changes per hour (ACH).

3.1.2 *loading ratio, L: (L = A/V)*, the total exposed surface area (A), excluding panel edges, of the product being tested divided by the test chamber's interior volume, V , in m^2/m^3 .

3.1.3 *make-up air flow, Q:* the quantity of conditioned and filtered air fed into the chamber per unit time, m^3/h . Q can be determined by taking the Q/A value from Table 1 and dividing by A .

3.1.4 *Q/A ratio:* the ratio of air flow through the chamber (Q) to sample surface area (A), m^3/h air per m^2 test area (see Section 8, Table 1)

3.1.5 *sample surface area, A:* the total area of all sample faces exposed in the chamber, m^2 .

3.1.6 *steady state concentration, C_s:* the formaldehyde concentration (expressed in parts of formaldehyde per million parts air (ppm) under the defined environmental test parameters of this method.

3.1.7 *volume of closed system, V:* the interior volume of the test chamber, m^3 .

4. Significance and Use

4.1 Upper limits for the formaldehyde emission rates have been established for wood panel building products made with urea-formaldehyde adhesives and permanently installed in homes or used as components in kitchen cabinets and similar industrial products. This test method is intended for use in conjunction with the test method referenced by HUD 24 for manufactured housing and by Minnesota Statutes for housing units and building materials. This method may also be used for monitoring products for compliance to the California Air Resources Board (CARB) regulation for composite wood products. This test method provides a means of testing smaller samples and reduces the time required for testing.

4.2 Formaldehyde concentration levels obtained by this small-scale method may differ from expected in full-scale

indoor environments. Variations in product loading, temperature, relative humidity, and air exchange will affect formaldehyde emission rates and thus likely indoor air formaldehyde concentrations.

4.3 This test method requires the use of a chamber of 0.02 to 1 m^3 in volume to evaluate the formaldehyde concentration in air using the following controlled conditions:

4.3.1 Conditioning of specimens prior to testing,

4.3.2 Exposed surface area of the specimens in the test chamber,

4.3.3 Test chamber temperature and relative humidity,

4.3.4 The Q/A ratio, and

4.3.5 Air circulation within the chamber.

5. Interferences

5.1 The NIOSH 3500 analytical method lists phenols as a negative interference when present at an 8:1 excess over formaldehyde. Modifications in the analytical procedure shall be made when relatively high phenol to formaldehyde concentrations (8:1) are anticipated.^{8,9}

6. Apparatus

6.1 *Test Chamber*—The interior volume of the small chamber shall be from 0.02 to 1 m^3 . The interior of the test chamber shall be free of refrigeration coils that condense water and items such as humidifiers with water reservoirs since water has the potential for collecting formaldehyde and thus influencing test results. The interior surfaces of the small chamber, including any sample support system, shall be a nonadsorbent material. Stainless steel, aluminum, and polytetrafluoroethylene (PTFE) have been found appropriate as chamber lining materials. All joints except for doors used for loading and unloading specimens should be sealed. Doors shall be self-sealing.

6.2 *Make-Up Air:*

6.2.1 The make-up air shall come from a filtered dust-free environment and a formaldehyde concentration in air no more than 0.02 ppm. This can be accomplished by passing make-up air through a filter bed of activated carbon, activated alumina impregnated with potassium permanganate, or other materials capable of absorbing or oxidizing formaldehyde.

6.2.2 Make-up air for the chamber must pass through a calibrated air flow measuring device.

6.2.3 *Air Circulation*—Low speed mixing fans or multi-port inlet and outlet diffusers are two techniques that have been used successfully to ensure mixing of the chamber air over all sample surfaces.

6.2.4 *Air Sampling Port*—The exhaust flow (that is, chamber outlet) is normally used as the sampling point, although separate sampling ports in the chamber can be used. The sampling system shall be constructed of a material to minimize

⁵ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>; request U.S. Dept. of Health and Human Services, 1989.

⁶ Available from Print Communications, Dept. of Administration, 117 University Ave., St. Paul, MN 55155.

⁸ Hakes, D., Johnson, G., and Marhevka, J., *Procedure for Elimination of Phenol Interference in the Chromotropic Acid Method for Formaldehyde*, American Industrial Hygiene Association, April 1984.

⁹ *Technical Bulletin No. 415*, National Council of the Paper Industry for Air and Stream Improvement Inc. (NCASI), 1983.

adsorption (for example, glass, stainless steel), and the system should be maintained at the same temperature as the test chambers.

6.3 Examples of acceptable reagents, materials, and equipment are provided in [Appendix X1](#).

7. Hazards

7.1 *Chromotropic Acid Reagent Treatment*—(See [10.3.4](#) and [10.3.5](#).) During this hazardous operation, the operator must wear rubber gloves, apron, and a full face mask or be protected from splashing by a transparent shield such as a hood window. The solution becomes extremely hot during addition of sulfuric acid. If acid is not added slowly, some loss of sample could occur due to splattering.

7.2 *Cleaning Chemicals for Glassware*—Use appropriate precautions if cleaning chemicals are considered to be hazardous.

8. Test Specimens

8.1 *Specimen Size and Chamber Air Change*—Chambers are operated at a fixed sample size by varying the make-up air (Q), or at fixed Q by varying the product sample size by product type. Either mode is acceptable as long as the appropriate Q/A ratios for the product type are met (see [Table 1](#)).

TABLE 1 Q/A Ratios, $\pm 2\%$

Q/A (m ³ /h air per m ² test area)	Product Type
0.526	hardwood plywood wall paneling
1.172	particleboard flooring panels, industrial particleboard panels, industrial hardwood plywood panels
1.905	medium density fiberboard (MDF)
3.811	particleboard door core

8.2 *Standard Face and Back Configuration Testing*—Loading ratio (L or A/V) is defined as the total exposed specimen surface area, excluding edge area, divided by the chamber volume. Aluminum tape, or coatings with similar performance, shall be used to cover the edges of the specimens if the edge exposure is greater than 5 % of the surface area, thereby retarding formaldehyde emission from the edge.

8.3 *Nonstandard Sample Configuration Testing Products with Single Surface Exposed*—Some products have significantly different formaldehyde release characteristics for each surface. In those cases, panels may be tested back-to-back with edges taped together. The panels shall be identified as tested in the back-to-back mode.

8.4 *Combination Testing*—Different products may be tested in combination. Qualify the test report and note the Q/A ratio used.

9. Sample Material Handling and Specimen Conditioning

9.1 *Handling*—Materials selected for testing shall be wrapped in polyethylene plastic having a minimum thickness of 0.15 mm (6 mil) until sample conditioning is initiated. When

testing wood products that are not newly manufactured such as after original application, installation or use, the method of packaging and shipping the products for testing shall be fully described. Information on the age and history of the product shall be detailed in the test report.

9.2 *Conditioning*—Condition test specimens with a minimum distance of 0.15 m (6 in.) between each specimen for 2 h ± 15 min at conditions of $24 \pm 3^\circ\text{C}$ ($75 \pm 5^\circ\text{F}$) and $50 \pm 5\%$ relative humidity. The formaldehyde concentration in the air within 0.3 m (12 in.) of where panels are conditioned shall be not more than the lesser of 0.10 ppm or the applicable compliance limit when testing for compliance purposes, during the conditioning period. Alternative conditioning intervals may give better correlation, such as seven day conditioning that parallels Test Method [E1333](#).

10. Procedure

10.1 Test Procedure for Materials:

10.1.1 Purge the chamber by running empty or with the use of filters designed to reduce the formaldehyde background concentration in air, or both. The formaldehyde background concentration in air of the empty operating chamber shall not exceed 0.02 ppm. Clean chamber surfaces with water or suitable solvent if formaldehyde background concentrations approach 0.02 ppm.

10.1.2 Locate the specimens in the chamber so that the conditioned air stream circulates over all panel surfaces.

10.1.3 Operate the chamber at $25 \pm 1^\circ\text{C}$ ($77 \pm 2^\circ\text{F}$) and $50 \pm 4\%$ relative humidity. Record the temperature, relative humidity, and barometric pressure during the testing period. Conduct the chamber test at a given Q/A ratio and record this ratio in the report.

10.1.4 After placing samples in chamber, allow time for no less than three full air changes or 15 min, whichever is greater, before beginning air sample collection (see [Note 1](#)).

NOTE 1—For products with very low emissions or to establish equivalence to ASTM Test Method [E1333](#), it may be necessary to allow a longer time period prior to beginning air sampling.

10.2 *Air Sampling*—Purge air sampling lines for 1 min. At the sampling station, bubble air through a single impinger containing 20 mL of a 1 % sodium bisulfite (NaHSO_3) solution. A filter trap may be placed between the impinger and the flowmeter. Set a calibrated flowmeter to maintain an average airflow of 1 ± 0.05 L/min for 30 to 60 min (see [Note 2](#)) with time measured accurately to within 5 s. Following air sampling, analyze the collection solution.

NOTE 2—For products with very low emissions or to establish equivalence to ASTM Test Method [E1333](#), it may be necessary to use the 60 min sampling time.

10.3 Analysis of Air Samples:

10.3.1 Pipet 4 mL of the NaHSO_3 solution from the impinger into each of three 16 by 150-mm screwcap test tubes for triplicate analysis of each impinger sample.

10.3.2 Pipet 4 mL of 1 % NaHSO_3 into a 16 by 150-mm screwcap test tube to act as a reagent blank.

10.3.3 Add 0.1 mL of 1 % chromotropic acid reagent to each test tube. Shake tube after addition.

10.3.4 Slowly and carefully pipet 6.0 mL concentrated sulfuric acid (H₂SO₄) into each test tube (**Warning**—See 7.1.) and allow to flow down the side of test tube. Allow the volumetric pipet to drain. *Do not blow out.* Before placing caps on test tubes, check the condition of the polytetrafluoroethylene (PTFE) cap liners to make sure they are clean and not deteriorated.

10.3.5 Slowly and gently agitate test tubes to affect mixing. Mixing is complete when there is no sign of stratification. Caution needs to be taken due to the exothermic chemical reaction. Rapid mixing will cause heating and a pressure increase which may break the test tube. Vent test tubes to release pressure.

10.3.6 If absorbance readings exceed 1.0 or if spectrophotometric analysis is performed within 2 h, heat capped test tubes to 95°C or place capped test tubes in a boiling water bath for 15 ± 2 min to ensure that the chemical reaction is completed. Remove tubes from water bath and allow to cool to room temperature.

10.4 Absorbance Readings:

10.4.1 Standardize the spectrophotometer using distilled water at 580 nm in accordance with the instrument's operating instructions. The reagent blank shall be read against distilled water. A high absorbance for the reagent blank indicates contamination of reagent blank or improper solution preparation. If absorbance for the reagent blank compared to distilled water is greater than 0.040 (using a 12-mm cell path length) or above 0.030 (using a 10-mm cell path length), repeat the entire standardization procedure.

10.4.2 Zero the instrument using the reagent blank, or the instrument may be left zeroed on distilled water, and the absorbance of the reagent blank subtracted from the absorbance of the standard solutions.

10.4.3 Read and record absorbance at 580 nm for each test tube prepared (see A4.6 – A4.9). If the absorbance of the specimen solution is found to fall outside the preferred absorbance range (>1.0), steps 10.3.1 – 10.3.4 may be repeated using an appropriate dilution of each impinger solution.

11. Calculation

11.1 Convert the volume of air sampled to the volume of air at standard conditions as follows:

$$V_s = \frac{V \times P \times 298}{101 \times (T + 273)} \quad (1)$$

where:

V_s = volume of air at standard conditions (101 kPa and 298 K), L,

V = volume of air sampled, L,

P = barometric pressure, kPa, and

T = temperature of sample air, °C.

11.2 Calculate total micrograms of formaldehyde collected in each impinger sample as follows:

$$C_t = C_a \times F_a \quad (2)$$

where:

C_t = total formaldehyde in the impinger sample, µg,

C_a = total quantity of formaldehyde in the sample aliquots taken from the impinger (as determined from the calibration curve in Annex A4), µg, and

F_a = aliquot factor = $\frac{\text{sampling solution volume, mL}}{\text{aliquot used, mL}}$

11.2.1 Calculate the concentration of formaldehyde in air in the small chamber as follows:

$$C_s = \frac{C_t \times 24.47}{V_s \times 30.03} \quad (3)$$

where:

C_s = parts of formaldehyde per million parts air, ppm,

30.03 = molecular weight of formaldehyde, and

24.47 = µL of formaldehyde gas in 1 µmol at 101 kPa and 298 K.

Round calculated formaldehyde concentrations to the nearest 0.01 ppm. Round up to the nearest 0.01 ppm any value at or in excess of 0.005 ppm. Round down all values below 0.005 to the nearest 0.01 ppm.

11.3 When the chamber temperature differs from 25 by ¼ °C (77 by ½ °F) or more, adjust the formaldehyde concentrations obtained to a standard temperature of 25°C (77°F) using a formula developed by Berge et al.¹⁰ Annex A1 contains a table of conversion factors for use at different observed test temperatures as calculated using this formula. The observed test temperature is the average temperature for the total period of 15 min prior to air sampling plus the time of air sampling.

11.4 The small chamber formaldehyde concentration in air shall be adjusted to a concentration at 50 % relative humidity when the difference in relative humidity from 50 % is greater than or equal to 1 % (see Annex A2).

12. Report

12.1 Report the following information:

12.1.1 Test number.

12.1.2 Title of report shall state if standard face and back configuration testing (see 8.2) or if nonstandard configuration testing (see 8.3) was performed.

12.1.3 The manner in which materials were shipped or stored, or both: wrapped separately in vapor retarder, wrapped collectively in vapor retarder or in original box or container. If materials were shipped unwrapped, or not in the original box or container, it shall be noted in the test report. Information on age and product history, if known, shall be described in the test report.

12.1.4 Name of product manufacturer or name of company submitting material, or both, date of manufacture, and sampling date (if known).

12.1.5 Description of test material or product shall include generic product name, thickness, size, if surface is finished or sealed (both surfaces should be described), and special treatment (if known).

¹⁰ Berge, A., Mellagaard, B., Hanetho, P., and Ormstad, E. B., *Formaldehyde Release from Particleboard-Evaluation of a Mathematical Model*, Holz Als Roh- und Werkstoff 38, 1980, pp. 252–255.

12.1.6 Specimen conditioning details to include average temperature and range nearest ¼ °C (0.5°F), average relative humidity and range (nearest 1 %), and time to the nearest minute.

12.1.7 Formaldehyde background concentration in the air in the area where specimens are conditioned (rounded to the nearest 0.01 ppm).

12.1.8 Chamber volume: nominal length, width, and height.

12.1.9 Chamber Q/A ratio.

12.1.10 Description of specimens as loaded into chamber including number of specimens in charge and number of surfaces exposed.

12.1.11 Average temperature and range nearest ¼ °C (0.5°F), average relative humidity and range (nearest 1 %), and time to the nearest minute during the sampling period.

12.1.12 Chamber formaldehyde concentration in air at test conditions; chamber formaldehyde concentration in air corrected to 25°C (77°F), 50 % relative humidity, rounded to nearest 0.01 ppm.

12.1.13 The analytical method employed if different from the NIOSH 3500 chromatographic acid test procedure.

12.1.14 Formaldehyde background concentration of air in chamber prior to test and formaldehyde concentration of make-up air (rounded to the nearest 0.01 ppm).

12.1.15 Air-sampling rate and length of sample time.

12.1.16 Date of test.

13. Precision and Bias

13.1 A study including seven laboratories and four test materials was conducted in accordance with Practice E691 and resulted in the following statements for precision and bias.

13.1.1 *Repeatability*—Test results indicate a repeatability (within laboratory) precision standard deviation ranging from 0.01 to 0.02 for products emitting 0.06 to 0.24 ppm of formaldehyde.

13.1.2 *Reproducibility*—Test results indicate a reproducibility (between laboratory) precision standard deviation ranging from 0.02 to 0.05 for products emitting 0.06 to 0.24 ppm of formaldehyde, respectively.

13.1.3 *Bias*—No bias statement is available for this test method due to the lack of an acceptable homogeneous formaldehyde off-gassing reference material.

14. Keywords

14.1 airborne; chromatographic acid analysis; formaldehyde concentration in air; small chamber; small-scale test; wood products

ANNEXES

(Mandatory Information)

A1. TEMPERATURE CONVERSION FACTORS FOR FORMALDEHYDE

A1.1 Table A1.1 is based on the Berge et al.¹⁰ formula to correct formaldehyde concentrations in air for temperature:

$$C = C_o \times e^{-R(1/t - 1/t_o)}$$

or

$$C_o = C e^{R(1/t - 1/t_o)}$$

where:

- C = test formaldehyde concentration level,
- C_o = corrected formaldehyde concentration level,
- e = natural log base,
- R = coefficient of temperature (9799),
- t = actual temperature, K, and
- t_o = corrected temperature, K.

TABLE A1.1 Temperature Conversion Table for Formaldehyde

NOTE 1—The Berge et al.¹⁰ equation is an exponential function. The greater the variance between actual and corrected temperature, the greater the potential error. Two horizontal lines within the table delineate the specified test temperature ranges 25 ± 1°C (77 ± 2°F).

Actual		To Convert to 25°C (77°F)	Actual		To Convert to 25°C (77°F)
°C	(°F)	Multiple by	°C	(°F)	Multiple by
22.2	(72)	1.36	25.3	(77.5)	0.97
22.5	(72.5)	1.32	25.6	(78)	0.94
22.8	(73)	1.28	25.8	(78.5)	0.91
23.1	(73.5)	1.24	26.1	(79)	0.89
23.3	(74)	1.20	26.4	(79.5)	0.86
23.6	(74.5)	1.17	26.7	(80)	0.83
23.9	(75)	1.13	26.9	(80.5)	0.81
24.2	(75.5)	1.10	27.2	(81)	0.78
24.4	(76)	1.06	27.5	(81.5)	0.76
24.7	(76.5)	1.03	27.8	(82)	0.74
25.0	(77)	1.00			

A2. RELATIVE HUMIDITY CONVERSION FACTORS FOR FORMALDEHYDE

A2.1 **Table A2.1** is based on the Berge et al.¹⁰ formula to correct formaldehyde concentrations in air for relative humidity:

$$C = C_o [1 + A(H - H_o)]$$

or

$$C_o = \frac{C}{1 + A(H - H_o)}$$

TABLE A2.1 Relative Humidity Conversion Table for Formaldehyde

Actual RH %	To Convert to 50 % RH Multiply by	Actual RH %	To Convert to 50 % RH Multiply by
46	1.08	51	0.98
47	1.06	52	0.97
48	1.04	53	0.95
49	1.02	54	0.93
50	1.00		

where:

- C = test formaldehyde concentration level,
- C_o = corrected formaldehyde concentration level,
- A = coefficient of humidity (0.0175),
- H = actual relative humidity, and
- H_o = relative humidity, %.

A3. STANDARD SOLUTIONS A AND B

A3.1 Standardization of Formaldehyde Standard Solution A (1.0 mg/mL)

A3.1.1 Pipet 2.70 mL of 37.0 % formaldehyde solution into a 1 L volumetric flask. Dilute to mark with freshly distilled water and mix well. This solution is stable for at least one month in a closed container at laboratory conditions.

A3.1.2 Calibrate the pH meter with standard buffer solution of pH 9.0.

A3.1.3 Pipet two 50 mL aliquots of formaldehyde standard Solution A into two 150-mL beakers for duplicate analysis and add 20 mL of 1 *M* sodium sulfite (Na_2SO_3) to each beaker. Sodium sulfite solution can age, thus the 1 *M* sodium sulfite solution should be adjusted to a 9.5 pH before adding to standard Solution A aliquots.

A3.1.4 Place solution on magnetic stirrer. Immerse pH electrodes into the solution and carefully titrate with 0.100 N hydrochloric acid (HCl) to the original pH of the solution. Record volume of HCl and corresponding pH intermittently. Make a graph of pH versus volume of HCl.

A3.1.5 Calculate the concentration, C_A , of formaldehyde standard Solution A in milligrams per millilitre as follows:

$$C_A = \frac{V \times N \times 30.03 \text{ (mg per milliequivalent)}}{50 \text{ (mL)}}$$

where:

- V = 0.100 N HCl required at pH of 9.5 from the graph prepared in **A3.1.4**, mL, and
- N = normality of HCl. The concentration of standard Solution A will be the average of the two analyses conducted.

A3.1.6 Record the concentration value (mg/mL) of Standard Solution A (C_A) which is the average of the two analyses conducted.

A3.2 Standard Solution B

A3.2.1 Prepare a 1 % sodium bisulfite (NaHSO_3) solution by dissolving 10 g of NaHSO_3 in a 1000 mL volumetric flask and diluting to the mark with distilled water

A3.2.2 Prepare formaldehyde standard Solution B in a 1000 mL volumetric flask by adding 5 mL of standard Solution A and diluting to the mark with the 1 % sodium bisulfite solution. The target concentration of Solution B is 5 µg/mL.

$$C_B = (C_A \times 1000 \times 5 \text{ mL})/1000\text{mL} \quad (\text{A3.1})$$

where:

C_B = concentration of Standard Solution B, µg/mL,
 C_A = concentration of Standard Solution A, mg/mL,
 1000 = constant conversion factor,
 5 mL = amount of Standard Solution A added, and
 1000 mL = size of flask used to prepare Standard Solution B.

A3.2.3 Record the value.

A4. CALIBRATION CURVE

A4.1 Prepare a 1 % sodium bisulfite (NaHSO₃) solution by dissolving 10 g of NaHSO₃ in a 1000 mL volumetric flask and diluting to the mark with distilled water. This solution is stable at room temperature and should be prepared on a weekly basis.

A4.2 Prepare eight standard solutions in 200 mL volumetric flasks by pipetting the following amounts of solution “B” followed by dilution to the mark with 1% sodium bisulfite (NaHSO₃) solution which was prepared in A4.1.

Flask Number	Solution B, mL	Target Flask HCHO Concentration, µg/mL
1	0.00	0.000
2	5.00	0.125
3	7.00	0.175
4	10.00	0.250
5	12.00	0.300
6	16.00	0.400
7	20.00	0.500
8	30.00	0.750

Concentration of each flask is calculated as follows:

$$\text{flask HCHO concentration (µg/mL)} = C_B(\text{µg/mL}) \times \text{Solution B added (mL)} \div 200\text{mL} \quad (\text{A4.1})$$

A4.2.1 Record the concentration value for each flask.

A4.3 Pipet a 4 mL aliquot from each flask specified in A4.2 into three test tubes for triplicate analyses.

A4.3.1 Note that no Solution B was added to Flask No. 1 and therefore it will act as the reagent blank.

Test Tube (TT) Set	Target Test Tube HCHO Content (µg)
1	0.000
2	0.500
3	0.700
4	1.000
5	1.200
6	1.600
7	2.000
8	3.000

$$\text{test tube HCHO content (µg)} = \text{flask concentration (µg/mL)} \times 4\text{mL} \quad (\text{A4.2})$$

Record the content value for each test tube. That value will be used in A4.9.2.

A4.4 Add 0.1 mL of 1 % chromotropic acid reagent to each test tube. Shake tube after addition.

A4.5 Slowly and carefully pipet 6.0 mL concentrated sulfuric acid (H₂ SO₄) into each test tube (**Warning**—See 7.1.) and allow to flow down the side of the test tube. Allow the volumetric pipet to drain. *Do not blow out.* Before placing caps on test tubes, check the condition of the polytetrafluoroethylene (PTFE) cap liners to make sure they are clean and not deteriorated.

A4.5.1 Slowly and gently agitate test tubes to affect mixing. Mixing is complete when there is no sign of stratification. Carefully vent test tubes to release pressure. Rapid mixing will cause heating and a pressure increase with the potential for breaking the test tube. If absorbance readings exceed 1.0 or if spectrophotometric analysis is performed within 2 h, heat capped test tubes to 95°C or place in a boiling water bath for 15 ± 2 min to ensure that the chemical reaction is complete. After removal, allow the test tubes to cool to room temperature.

A4.6 Standardize the spectrophotometer using distilled water at 580 nm in accordance with the instrument’s operating instructions. The reagent blank (Flask #1, TT 1) shall be read against distilled water. A high absorbance for the reagent indicates contamination of reagent blank or improper solution preparation. If absorbance for the reagent blank compared to distilled water is greater than 0.040 (using a 12 mm cell path length), or above 0.030 (using a 10-mm cell path length), repeat the entire standardization procedure.

A4.7 Zero the instrument using the reagent blank (Flask #1 TT 1), or the instrument may be left zeroed on distilled water, and the absorbance of the reagent blank subtracted from the absorbance of the standard solutions. Recovery shall be within ±5 % of reagent blank.

A4.8 Read and record absorbance at 580 nm for each standard prepared (solutions from Flask Nos. 2 – 8).

A4.9 Plot absorbance against micrograms of formaldehyde in the color developed solution. Note the amount of formaldehyde in micrograms is based upon the concentration of formaldehyde in standard Solution Flasks, which is dependent upon the standardization carried out on standard Solution A in Annex A3.

A4.9.1 The absorbance of each tube would be plotted against the total micrograms of formaldehyde in each tube.

A4.9.2 The average absorbance would be plotted against the average total micrograms of formaldehyde from each test tube set.

A4.9.3 The absorbance of each chamber impinger aliquot specimen determined in 10.4.3 is compared to this calibration curve, and the total micrograms of formaldehyde in the aliquot is represented as C_a in 11.2.

NOTE A4.1—The calibration curve as described in this annex is provided as an example. If absorbance readings are outside of this range, dilute the solution with distilled water to a concentration that is within the calibration curve. If absorbance readings exceed 1.0, place capped test tubes in a boiling water bath for 15 ± 2 min to ensure that the chemical reaction is completed. Vent test tubes to release pressure. Remove tubes

from water bath and allow to cool to room temperature.

A4.10 Preparation of the calibration curve (A4.3 – A4.9) shall be repeated at least once more and the final calibration line shall reflect the composite of the determinations (or the curve shall be calculated using a linear least squares fitting technique). The calibration curve may not be linear at high formaldehyde concentrations (high absorbance readings). If the plot in A4.9 shows the last few points deviating from linearity, omit the points from calculations or repeat entire procedure. Further, the curve should be frequently checked based on changes in reagent lot numbers, past experience, data scattering, or instrument instability.

APPENDIX

(Nonmandatory Information)

X1. REAGENTS, MATERIALS, AND EQUIPMENT FOUND SUITABLE FOR USE

X1.1 Air-Sampling Apparatus

NOTE X1.1—Other apparatus and instruments may be used if equivalent results are anticipated.

X1.1.1 *Midget Impingers.*

X1.1.2 *Rotameters, 1 L/min.*

X1.1.3 *Line Filter, with desiccant (to dry the air before entering rotameters).*

X1.1.4 *Polytetrafluoroethylene (PTFE) Tubing.*

X1.1.5 *Buret, 250 or 500 mL (to calibrate rotameters).*

X1.1.6 *Impinger Pumps.*

X1.1.7 *Film-Type Laboratory Calibrators or Bubble Tube, for calibrating pumps and rotameters.*

X1.2 Analytical Apparatus

X1.2.1 *Spectrophotometer.*

X1.2.2 *Spectrochek, for calibration of the spectrophotometer.*

X1.2.3 *Beaker, 150 mL, low form.*

X1.2.4 *Volumetric Flask, 1000 mL.*

X1.2.5 *Volumetric Flask, 100 mL.*

X1.2.6 *Volumetric Flasks, two, 10 mL.*

X1.2.7 *Buret, 25 mL, Class A.*

X1.2.8 *pH meter.*

X1.2.9 *Magnetic Stirrer.*

X1.2.10 *Pipet, volumetric, 4 mL.*

X1.2.11 *Pipet, volumetric, 50 mL, Class A.*

X1.2.12 *Pipet, volumetric, 6 mL, Class A.*

X1.2.13 *Pipet, long-tip Mohr type, 2 by 0.01 mL.*

X1.2.14 *Pipet, Mohr, 10 by 0.1 mL.*

X1.2.15 *Safety Bulb, for pipeting.*

X1.2.16 *Test Tubes, 16 by 150 mm, with polytetrafluoroethylene (PTFE) lined screw caps.*

X1.2.17 For repetitive analyses of sample solutions and for added safety, use of automatic pipeting equipment may be desirable. Use of the following have been found suitable.

X1.2.17.1 *Brinkman Dispensers, volume 0.1 to 0.5 mL (for chromotropic acid), volume 1 to 10 mL (for sulfuric acid), and volume to 25 mL (for distilled water).*

X1.2.17.2 *Oxford Macro-Set Pipet.*

X1.2.17.3 *Tips, 250, for transferring 4 mL aliquots.*

X1.2.18 *Volumetric Flask, 200 mL.*

X1.3 Reagents

X1.3.1 *Chromotropic Acid Reagent*—Dissolve 0.10 g of chromotropic acid (4,5-dihydroxy-2,7-naphthalene-disulfonic acid disodium salt) in freshly distilled water and dilute to 10 mL. This solution is to be made up daily.

X1.3.2 *Sulfuric Acid (H₂SO₄), concentrated, reagent grade. Nitrate concentration shall be less than 10 ppm.*

X1.3.3 *Buffer Solution, pH 9.0.*

X1.3.4 *Hydrochloric Acid, (HCl) 0.100 N, standard.*

X1.3.5 *Sodium Sulfite Solution, 1.0 M*—Dissolve 12.67 g anhydrous sodium sulfite (Na₂SO₃) (ACS assay 99.5 %) in a 100-mL volumetric flask and dilute to the mark with freshly distilled water. The correct amount to be dissolved should be 12.6/ACS assay of the anhydrous sodium sulfite actually being used (read assay from bottle label).

X1.3.6 *Formaldehyde Solution, weight 37 %.*

X1.3.7 *Sodium Bisulfite, (NaHSO₃), reagent grade.*

X1.3.8 *Mild Liquid Soap.*

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