



Standard Test Method for Measurement of Formaldehyde in Indoor Air (Passive Sampler Methodology)¹

This standard is issued under the fixed designation D 5014; 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.

^{e1} NOTE—Editorial corrections were made throughout in March 2000.

1. Scope

1.1 This test method covers personal or area measurements of formaldehyde in indoor air in the range from 0.01 to 17 mg/m³ (0.008 to 14 ppm v/v). Formaldehyde is collected in a passive diffusion sampler, and analyzed by a colorimetric method using 3-methyl-2-benzothiazolinone hydrazone hydrochloride (MBTH). The recommended sampling time is 15 min to 24 h.

1.2 The lower quantification limit of the MBTH test method is 0.03 μ g of formaldehyde per millilitre of absorbing solution used. A formaldehyde concentration of 0.01 mg/m³ (0.008 ppm v/v) can be determined in indoor air, based on using an aliquot of 5 mL of absorbing solution in the prescribed sampler for a period of 24 h and observing a minimum difference of 0.05 absorbance units from the blank when using spectrophotometer cells of path length 1 cm.

1.3 Water soluble aliphatic aldehydes give a significant positive interference (**1**, **2**)² nearly equal to formaldehyde on a molar basis. Further information on estimating potential and actual interferences from aliphatic aldehydes may be found in 6.2 and 10.1.7. Most other compounds which react to produce colored products are not gaseous or water soluble and, consequently, should not interfere.

1.4 The values stated in SI units are to be regarded as the standard.

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.* Specific precautionary statements are given in 8.3 and 8.4.

¹ This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

Current edition approved June 15, 1994. Published August 1994. Originally published as D 5014 – 89. Last previous edition D 5014 – 89.

² The boldface numbers in parentheses refer to the references at the end of this test method.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water³

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres⁴

D 1357 Practice for Planning the Sampling of the Ambient Atmosphere⁴

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): (The Modern Metric System)⁵

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology D 1356. For an explanation of units, symbols, and conversion factors, refer to IEEE/ASTM SI 10.

3.2 Descriptions of Terms:

3.2.1 *Knudsen disk*—a gas-permeable diffusion disk which regulates the transfer of gas by the principles of Knudsen diffusion. Details concerning the construction of a Knudsen disk are described in 7.1.2.

4. Summary of Test Method

4.1 Formaldehyde is absorbed into a 0.05 % aqueous solution of 3-methyl-2-benzothiazolinone hydrazone hydrochloride (MBTH) contained in a sampler consisting of a glass vial with a septum cap that retains a Knudsen disk. During air sampling the vial is inverted to establish contact between the absorbing liquid and the Knudsen disk. Formaldehyde passes from the ambient atmosphere into the MBTH solution through the Knudsen disk at a constant rate. After collection, the resulting azine is oxidized by a ferric chloride-sulfamic acid solution to form a blue cationic dye in acidic medium. The concentration of the blue cation is measured by colorimetry at 628 nm (**1**, **3-5**). The air concentration of formaldehyde is computed from

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 11.03.

⁵ *Annual Book of ASTM Standards*, Vol. 14.03.

the amount of formaldehyde collected divided by the product of the diffusion rate and the time of exposure.

4.2 The mechanism of the present procedure as applied to formaldehyde includes the following steps: reaction of formaldehyde with 3-methyl-2-benzothiazolinone hydrazone hydrochloride, forming the azine which is oxidized to a reactive cation, and formation of the blue cation (1, 3).

5. Significance and Use

5.1 This test method allows field measurement of formaldehyde in indoor air at concentrations from 0.01 to 17 mg/m³ (0.008 to 14 ppm) using sampling times between 15 min and 24 h. A 24-h sampling time is recommended to measure time-weighted average formaldehyde concentrations over the range from 0.01 to 0.2 mg/m³ (0.008 to 0.16 ppm v/v) in offices and residences. An 8-h sampling time allows measurement over the range of 0.03 to 0.6 mg/m³ (0.025 to 0.5 ppm v/v). The test method is suitable for both area or personal monitoring.

5.2 This test method allows sampling and quantification of formaldehyde under field conditions with the aid of a portable field colorimeter, without the need for any laboratory support.

6. Interferences

6.1 The following classes of compounds react with MBTH to produce colored products (1, 5). These are aromatic amines, imino heterocyclics, carbazoles, azo dyes, stilbenes, Schiff bases, the aliphatic aldehyde 2,4-dinitrophenylhydrazones, and compounds containing the p-hydroxy styryl group. Most of these compounds are not gaseous or water soluble and, consequently, should not interfere with the analysis of formaldehyde in the atmosphere.

6.2 Aliphatic aldehydes give a positive response with the MBTH method equal to 75 % or less that of formaldehyde on a molar basis (1). The Knudsen disk discriminates against aldehydes having molecular weights greater than formaldehyde in proportion to the ratio of the square roots of their molecular weights.⁶ See 10.1.7 for discussion of a method for confirming whether or not a positive interference exists in the event that other aldehydes are known or suspected to be present.

6.3 Effect of Storage:

6.3.1 Formaldehyde is fairly stable in 0.05 % MBTH since only approximately 5 % of the formaldehyde is lost after standing in the MBTH for 13 days (1).

6.3.2 Formaldehyde samples stored in the samplers with a solid cap are stable for at least one week at room temperature when stored in the dark. After one week the MBTH solution darkens and a precipitate begins to form. Exposure to elevated temperatures reduces the stability of samples. In one experiment samples stored at 313 K (105°F) darkened noticeably after 24 h (6).

7. Apparatus

7.1 *Passive Air Sampler*—An example of the sampler is shown in Fig. 1. It consists of a cylindrical glass vial 70 mm high by 20.5 mm diameter, with a 20–400 thread finish having



FIG. 1 Passive Sampler Showing Vial, Septum Cap Retaining Knudsen Disk, and Clip for Affixing Sampler for Personal or Area Samples

an opening 13.9 mm (³⁵/₆₄in.) in diameter. Mated to the vial is a septum cap with an opening equal in diameter to that of the vial. The septum cap retains a Knudsen disk of diameter 17.5 mm (¹¹/₁₆ in.). Before and after sampling, the septum cap is replaced with a solid cap lined with polyethylene or polytetrafluoroethylene. A “pencil” style clip is slipped over the vial to allow it to be affixed to clothing or other support for collecting a personal or area sample, respectively.⁷

7.1.1 Vials with other thread finishes or septum caps with a different opening size may be used; however, the sampling rate shall be redetermined. When the alternative septum cap and vial having openings of equal size, the new sampling rate may be computed from the relative ratio of opening sizes as follows:

$$SR_{\text{new}} = SR \times (A_{\text{new}}/A) \quad (1)$$

where:

⁷ The passive sampling device used in the development and performance evaluation of this test method was manufactured by Solutions Environmental Health, 6687 N. Blackstone Ave., Fresno, CA 93710–3524, which is the sole source of supply of the sampler known to the committee at this time. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee¹ which you may attend.

⁶ Supporting data are available from ASTM Headquarters. Request RR:D-22.05-02.

- SR_{new} = sampling rate of new vial-septum cap configuration,
 SR = sampling rate of Knudsen disk in specified vial-septum cap configuration,
 A_{new} = area of new vial-septum cap opening, and
 A = area of specified vial-septum cap configuration (1.52 cm²).

When the septum cap and the vial have different opening sizes, the new sampling rate shall be experimentally determined in accordance with 10.3.

7.1.2 *Knudsen Disk*⁸—The Knudsen disk consists of porous polytetrafluoroethylene membranes of 0.07 mm thickness and 0.02 μm pore sizes thermally bound in such a manner as to preserve their microporous properties to both sides of a 1.6-mm (1/16-in.) thick core of macroporous high-density polyethylene. Using the prescribed sampler, the Knudsen disk allows the ambient atmosphere to be sampled for formaldehyde at a constant rate of approximately 11.6 mL/min independent of drafts over the range from 0.13 to 1.3 m/s (25 to 250 ft/min). The sampling rate normally is provided by the supplier of the Knudsen disks, but may also be determined experimentally in accordance with 10.3.

7.2 *Spectrophotometer*, an instrument capable of measuring accurately the developed color at the narrow absorption band of 628 nm. Portable colorimeters with a sample well that accepts the vial are available (6, 7), and allow the analysis procedure to be carried out in the field. The sensitivity is increased by a factor of two because the measured path length through the color-developed solution is approximately doubled relative to a 1-cm spectrophotometer cell.

7.3 *Standard pH Meter*.

7.4 *Magnetic Stirrer*.

7.5 *Erlenmeyer Flask*, 125 mL.

7.6 *Capped Analysis Vials*, glass vials able to hold 6 mL conveniently, having closures lined with polyethylene or foil. Avoid closures lined with paper or tetrafluoroethylene.

8. Reagents

8.1 *Purity of Reagents*—Unless otherwise stated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁹ Other grades may be used, provided it is ascertained that use of the reagent does not lessen the accuracy of the test method.

8.2 *Purity of Water*—References to distilled water shall be understood to mean distilled water, which is Type II reagent water conforming to Specification D 1193.

⁸ The Knudsen diffusion disk used in this test method is covered by a patent held by Robert R. Miksch, Ph.D., 548 E. Mallard Circle, Fresno, CA 93720-1227. Interested parties are invited to submit information regarding the identification of acceptable alternatives to this patented item to the Committee on Standards, ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Your comments will receive careful consideration at a meeting of the responsible technical committee which you may attend.

⁹ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.3 *3-Methyl-2-Benzothiazolinone Hydrazone Hydrochloride Absorbing Solution (MTBH) (0.05 %)*—Dissolve 0.5 g of MBTH in distilled water and dilute to 1 L. This colorless solution can be filtered by gravity, if slightly turbid, and is stable for a least one week, after which it becomes pale yellow. Stability may be increased by storing the solution in a dark bottle under refrigerated conditions. Alternatively, MBTH reagent tablets are commercially available, premeasured so that one tablet dissolved in 5 mL of distilled water provides a 0.05 % solution. (6) (**Warning**—Mild irritant. Avoid contact with skin and eyes. Wash with water.)

8.4 *Oxidizing Reagent*—Dissolve 1.6 g of sulfamic acid and 1.0 g of ferric chloride hexahydrate in distilled water and dilute to 100 mL. (**Warning**—Caustic. Avoid contact with skin and eyes. Wash with water.)

8.5 *Formaldehyde Standard Solution “A” (1 mg/mL)*—Dilute 2.7 mL of 37 % formalin solution to 1 L with distilled water. This solution must be standardized as described in Section 9. This solution is stable for one year, after which it should be restandardized.

8.6 *Formaldehyde Standard Solution “B” (10 μg/mL)*—Dilute 1 mL of “A” to 100 mL with 0.05 % MBTH solution. Prepare fresh daily.

8.7 *Sodium Sulfite (2 N)*—Dissolve 31.5 g of anhydrous sodium sulfite in distilled water to 250 mL. Prepare fresh daily.

8.8 *Hydrochloric Acid, Standard Solution (0.05 N)*—Dilute 4.28 mL of concentrated hydrochloric acid (HCl, specific gravity 1.19) to 1 L.

9. Calibration and Standardization

9.1 *Standardization of Formaldehyde Standard Solution “A”*:

9.1.1 Place 50 mL of 2 N sodium sulfite in a 125-mL Erlenmeyer flask. Stir the solution using a magnetic stirrer.

9.1.2 Measure and accurately record the pH. It should be around 10.

9.1.3 Add 25 mL of formaldehyde standard solution “A.” The pH should rise sharply to about 12.

9.1.4 Using the pH meter as a continuous monitor, titrate the solution back to its exact original pH using 0.05 N hydrochloric acid. Approximately 17 mL will be required.

9.1.5 One mL of 0.05 N hydrochloric acid is equivalent to 1.50 mg of formaldehyde. Therefore, since 25 mL of formaldehyde standard solution “A” was titrated, the millilitres of 0.05 N acid used in the titration multiplied by 0.060 equals the formaldehyde concentration of the standard solution in milligrams per millilitre. When stored in a brown glass bottle, formaldehyde standard solution “A” should be recalibrated on a quarterly basis.

9.2 *Preparation of Standard Curve*:

9.2.1 Pipet 0, 0.5, 1.0, 3.0, 5.0, and 7.0 mL of standard formaldehyde solution “B” into 100 mL volumetric flasks. Dilute to volume with 0.05 % MBTH solution. The solutions contain 0, 0.05, 0.1, 0.3, 0.5, and 0.7 micrograms of formaldehyde per millilitre.

9.2.2 After final dilution, let standards stand for 1 h.

9.2.3 Transfer 5 mL of each solution to an analysis vial, add 1 mL of oxidizing reagent, and mix.

9.2.4 After 12 min read the absorbance at 628 nm in a suitable spectrophotometer using 1 cm cells.

9.2.5 Prepare a calibration line by plotting absorbance versus micrograms of formaldehyde per millilitre of solution. Determine by a least squares analysis the best fit line slope and intercept. A calibration line should be prepared concurrently with each set of samples analyzed. The absorbance of the solution containing 0.0 µg of formaldehyde/mL is used as the laboratory blank in the calculations performed in 11.1.

9.3 *Calibration of Knudsen Disks*—The sampling rate of Knudsen disks must be determined experimentally; however lots in excess of several tens of thousands of Knudsen disks may be prepared that have very precise sampling rates. The disks are stable and the sampling rate will remain unchanged provided that the disks are not subjected to physical abuse. The sampling rate normally is provided by the supplier, but may be determined by the two following procedures.

9.3.1 Knudsen disks may be calibrated using a laboratory test atmosphere of formaldehyde generated by the gas phase depolymerization of trioxane (8). Samplers are tested using a manifold consisting of septum caps affixed to perforations in a poly(vinyl chloride) tube with a diameter 4.75 cm (1.875 in.), taking care that the velocity of the test atmosphere passing by the sampling devices is within the desired range.⁶ Exposure to varying amounts of formaldehyde generates a calibration line whose slope yields the sampling rate.

9.3.2 Knudsen disks may be calibrated in the field by comparison with a reference method, such as NIOSH P&CAM 125 (9). Samples shall be collected simultaneously by the Knudsen disk sampler and the reference method. A plot yields the relative response of the former to the latter, normally described by a line whose slope and intercept may be used to interpret subsequent measurements.

9.4 From Knudsen theory,⁶ the rate at which gas passes through a porous structure in which Knudsen diffusion applies varies as the square root of temperature. If Knudsen disks are calibrated at a temperature other than 298 K (25°C), the sampling rate shall be corrected using the following equation:

$$SR_{298} = (SR_T \times 17.26)/T^{1/2} \quad (2)$$

where:

- SR_{298} = sampling rate of Knudsen disk at 298 K,
- SR_T = sampling rate of Knudsen disk determined at temperature T,
- 17.26 = square root of 298 K, and
- $T^{1/2}$ = square root of temperature, K, at which sampling rate of Knudsen disk was determined.

10. Procedure

10.1 *Air Sampling:*

10.1.1 Select a sampling location and sampling period for determining indoor air concentration or exposure level in accordance with Practice D 1357. To obtain valid results, avoid placing the sampler at a site where the air is stagnant (air movement is less than 0.13 m/s (25 ft/min)). The temperature during sampling should be within typical indoor conditions of 288 to 308 K (15 to 35°C, or 60 to 95°F). Record indoor and outdoor temperature, humidity, air pressure, ventilation rate, air

mixing rate, and any other parameters that are relevant for appropriate data interpretation.

10.1.2 Prepare sampler by assembling the following:

- 10.1.2.1 A glass vial,
- 10.1.2.2 A metal clip,
- 10.1.2.3 A cap with a hole in it, and
- 10.1.2.4 A fresh Knudsen disk.

10.1.3 Place 5.0 mL of 0.05 % MBTH absorbing solution in the sampler. Place a fresh diffusion disk and holed cap on the sampler. Note starting time and any identification information. The sampler starts to collect formaldehyde as soon as the MBTH solution is added, though at a reduced sampling rate while the sampler is upright.

10.1.4 Place the sampler in inverted position so that the Knudsen disk is on the bottom, and the MBTH solution is in contact with the Knudsen disk. The bubbler may be clipped to a person's clothing for a personal breathing zone sample, or it may be placed on a stand for an area measurement. The sampler shall be disk-side-down at all times during sampling to maintain the contact between the absorbing solution and the disk.

10.1.5 To stop sampling turn the vial upright, and replace the septum cap with the solid cap. Note the stop time. Allow the sample to stand for 1 h after the completion of sampling before analysis. A study of the reaction time variation of the reaction of microgram quantities of formaldehyde with 0.05 % MBTH shows that the reaction is complete in approximately 45 min (1).

10.1.6 A field blank shall be collected concurrently with field measurements, and analyzed in the same manner as a field sample. The only difference between a field blank and a field measurement is that for the field blank the Knudsen disk and holed cap are not placed on the sampler. Instead, the solid closure is kept on at all times. The field blank shall be analyzed at the same time and in the same manner as field measurements.

10.1.6.1 The absorbance of the field blank should be within 0.005 absorbance units of the laboratory blank prepared in 9.2. If a larger difference is observed then the field samples must be regarded as suspect.

10.1.7 If interference by other aldehydes is suspected, the sample may be partitioned after collection and analyzed by both the MBTH method and a method using chromotropic acid (10, 11). The latter method is specific for formaldehyde, and will yield lower results than the MBTH method if other aliphatic aldehydes are present.

NOTE 1—This approach takes advantage of the fact that, under the strong acid conditions of the chromotropic acid method, the MBTH-formaldehyde adduct formed during sampling reverts to free starting materials, and the MBTH does not interfere (10, 11). The sensitivity of the chromotropic acid method is approximately sevenfold less than that of the MBTH method, and appropriate adjustments to the sampling time and sample dilution should be made.

10.2 *Analysis:*

10.2.1 Add distilled water to the sampler to bring it back to 5 mL mark if necessary.

10.2.2 Add 1 mL of the oxidizing agent (8.4) to the sampler, and mix thoroughly.

10.2.3 After standing for a minimum of 12 min, read at 628 nm on a suitable spectrophotometer. No significant change in the absorbance occurs over a period of 3 h after color development.

11. Calculation

11.1 Determine the formaldehyde content of the sample solution from the slope and intercept of the standard curve prepared previously:

$$C = (A_s - A_b)/S \quad (3)$$

where:

C = concentration of formaldehyde in sampler solution, $\mu\text{g/mL}$,

A_s = absorbance of sample solution,

A_b = measured absorbance of laboratory blank for solution containing 0.0 $\mu\text{g/mL}$ of formaldehyde, and

S = slope of calibration line, (absorbance units \times mL)/ μg .

11.2 Determine the total mass of formaldehyde, F (μg), collected by multiplying the concentration by the total sample volume, V (mL):

$$F = C \times V \quad (4)$$

11.3 The concentration of formaldehyde in the sampled atmosphere may be calculated from the total mass collected as follows:

$$\text{mg/m}^3 \text{ of formaldehyde at } T, P = \frac{16.6 \times F \times 17.26}{t \times SR_{298} \times T^{1/2}} \quad (5)$$

where:

T = temperature at the time sample was collected, K,
 P = pressure at the time sample was collected, kPa (see 12.3.1),

16.6 = combination of unit conversions [(1 mg/1000 μg) \times (1 h/60 min) \times (10^6 mL/m³)],

F = total formaldehyde captured by sampler in micrograms (μg)

17.26 = square root of 298 K,

t = sampling time, h, and

SR_{298} = sampling rate of Knudsen disk, approximately 11.6 mL/min as provided by supplier or obtained experimentally.

This equation corrects the sampling rate for the effects of temperature based on the Knudsen diffusion theory, which states that Knudsen diffusive transfer varies as the square root of temperature. See 9.4.

11.3.1 By convention, concentrations of contaminants in air under varying conditions of T and P sometimes are compared after concentrations are corrected to 298 K (25°C) and 101.3 kPa (1 atm), referred to as normal temperature and pressure (NTP) conditions. From kinetic gas theory, the sampling volume varies as P/T , and therefore the milligram per cubic metre concentration of formaldehyde is corrected as follows:

$$\text{mg/m}^3 \text{ at NTP} = \frac{(\text{mg/m}^3 \text{ at } T, P) \times T \times 101.3}{298 \times P} \quad (6)$$

In order to apply this correction, the pressure at the time of sampling must be known. Unless conditions are extreme, correcting concentrations of NTP conditions usually will change their value by less than 5 %.

11.3.2 Permissible or recommended exposure levels set by regulatory agencies are often expressed in concentration units of ppm (the number of parts of formaldehyde per million parts of ambient air). The ppm concentration of formaldehyde may be computed from the following:

$$\text{formaldehyde, ppm} = \frac{(\text{mg/m}^3 \text{ at } T, P) \times 24.45}{30.03} \quad (7)$$

where:

24.45 = molar volume under NTP conditions, L/mole, and

30.03 = gram molecular weight of formaldehyde.

12. Report

12.1 Reports of formaldehyde measurements are made in accordance with Practice D 1357 to include the following:

12.1.1 Date of test.

12.1.2 Name and address of laboratory or individual performing the test.

12.1.3 Statement of test objective.

12.1.4 Sample site, sampling period and other data deemed necessary for proper interpretation of the results as selected in 10.1.1.

12.1.5 Description of apparatus used.

12.1.6 Description of any deviation from standard test procedure.

12.1.7 Values for standard curve prepared in 9.2.

12.1.8 Results of calculations performed in Section 11.

12.1.9 Final results of test.

12.1.10 Signature of responsible individual.

13. Precision and Bias ⁴

13.1 *Statistical Methods:*

13.1.1 For each independent test point examined during laboratory and field experiments a mean, bias (percentage difference between mean and known concentration), standard deviation, and coefficient of variation (standard deviation divided by the mean expressed as a percentage) were computed. An overall mean coefficient of variation (MCV) for the independent test points included in each experiment was determined according to the following:

$$MCV = \sqrt{\frac{[(n_1 - 1)(CV_1)^2 + \dots + (n_i - 1)(CV_i)^2]}{\sum_{i=1}^p (n_i - 1)}} \quad (8)$$

where:

$(n_i - 1)$ = degrees of freedom, equal to number of observations minus one at the i^{th} level,

CV_i = coefficient of variation of the observations at the i^{th} level, and

p = number of independent test points

13.1.2 An overall mean (pooled) bias also was computed for the independent test points included in each experiment as follows:

$$\text{mean bias} = [b_1 n_1 + \dots + b_p n_p] / \sum_{i=1}^p n_i \quad (9)$$

where:

n_i = number of measurements made at the i^{th} level, and

p = number of independent test points.

13.1.3 The overall system accuracy (OSA) of the method then was estimated by applying a statistical evaluation procedure which combined the mean bias and the *MCV* according to the following:

$$\text{OSA} = (\text{absolute mean bias}) + 2 \times \text{MCV} \quad (10)$$

13.1.3.1 As has been discussed by others (11, 12) the OSA estimates the “width” required by an interval to include both the “true” value and 95 % of the measurements made by a measurement method. As a point of reference, in the workplace, NIOSH recommends that 95 % of the measurements made by a sampling method should be accurate within ± 25 % in the range from 0.5 to 2.0 times the environmental standard (13).

13.2 Laboratory Results:

13.2.1 The MBTH method of analysis was checked for reproducibility by having three different laboratories analyze standard formaldehyde samples. The results agreed within ± 5 % (1).

13.2.2 During development work, five independent sets of ten samplers each were exposed to laboratory test atmospheres of formaldehyde between 0.1 and 1 mg/m³ for 4-h periods. A linear relationship between formaldehyde concentration and the amount collected by the sampler was observed. The mean coefficient of variation and bias determined for the five sets of data were 5.0 and 1.2 %, respectively.

13.2.2.1 Additional experiments examined the effect of the velocity of the test atmosphere impinging upon the sampler. Four independent sets of twenty devices each exposed to face velocities between 0.13 and 1.3 m/s (25 and 250 ft/min). The *MCV* and mean bias for these data were 4.1 % and 2.1 % respectively.

13.2.2.2 When all nine sets of data were considered together the *MCV* was 4.1 % and the mean bias was 1.8 %. The OSA for the nine independent sets of development data was computed to be ± 10.0 %.

13.2.3 An independent laboratory evaluation study exposed fourteen groups of five samplers each to formaldehyde con-

centrations between 0.15 and 1.5 mg/m³ over 8-h periods under conditions of low and high, 20 and 85 % humidity, respectively. The mean bias and *MCV*, computed according to Eq 8 and 9, were -7 and 4.9 %, respectively, leading to an OSA of ± 16.8 %.

13.2.3.1 A second set of experiments examined twelve groups of five samplers each exposed to formaldehyde concentrations between 1.8 and 4.6 mg/m³ over 15-min periods. The mean bias and *MCV* were -10 and 3.4 %, respectively. The OSA for these data was also ± 16.8 %.

13.3 Field Results:

13.3.1 An independent field evaluation study exposed eight groups of ten samplers each to formaldehyde concentrations between 0.05 and 0.5 mg/m³ for 5-h periods in a carpeted room with gypsum board walls. Reference samples were collected over 30-min periods at approximately 45 min intervals following the procedure recommended in NIOSH P&CAM 125. The mean bias and *MCV* were -4.8 and 7.3 %, respectively, leading to an OSA of ± 19.8 %.

13.3.2 A second independent field evaluation study examined TWA and STEL measurements in the wood products industry. Measurements were made in the area of a paper machine using a urea-formaldehyde treatment, and particle-board manufacturing facility. The study examined workplace air drawn through an exposure chamber, with and without addition of generated spikes, and side-by-side area samples. Reference samples were collected using the procedure recommended in NIOSH P&CAM 125.

13.3.2.1 A total of six sets of either four or five samplers were used to make time-weighted average measurements of formaldehyde levels. The mean bias and *MCV* were -13.2% and 4.0%, respectively, leading to an OSA of $\pm 21.2\%$.

13.3.2.2 One set of four and one set of five samplers were used to make 15 min STEL measurements of formaldehyde levels. The mean bias and *MCV* were $+13.1\%$ and 5.9%, respectively, leading to an OSA of $\pm 24.9\%$.

14. Keywords

14.1 3-methyl-2-benzothiazolinone hydrazone hydrochloride; air sampling; aliphatic aldehydes; colorimetric method; formaldehyde; indoor air; Knudsen diffusion; MBTH; passive monitor; passive sampler

REFERENCES

- (1) Intersociety Committee APCA ACS AICHe APWA ASCE ASME AOAC HPS ISA, "Method 117: Tentative method of analysis for formaldehyde content of the atmosphere (MBTH—Colorimetric Method—Application to Other Aldehydes)," *Methods of Air Sampling and Analysis*, Third Ed., James P. Lodge, Jr., Ed., 1989, Intersociety Committee, Chelsea, MI, pp. 279–284.
- (2) Ahonen, R., Priha, T., and Aijäkä, M., "Specificity of Analytical Methods Used to Determine Formaldehyde in Workroom Air," *Chemosphere*, Vol 13, No. 4, 1984, pp. 521–525.
- (3) Sawicki, E., Hauser, T. R., Stanley, T. W., and Elbert, W., "The 3-Methyl-2-Benzothiazolinone Hydrazone Test," *Analytical Chemistry*, Vol 33, 1961, p. 93.
- (4) Hauser, T. R., and Cummins, R. L., "Increasing the Sensitivity of 3-Methyl-2-Benzothiazolinone Hydrazone Test for Analysis of Aliphatic Aldehydes in Air," *Analytical Chemistry*, Vol 37, 1964, p. 679.
- (5) Hauser, T. R., "Determination of Aliphatic Aldehydes: 3-Methyl-2-Benzothiazolinone Hydrazone Hydrochloride (MBTH) Method", *Selected Methods for the Measurement of Air Pollutants. Public Health Service Publication No. 999-AP-11*, 1965, p. F-1.
- (6) Air Technology Labs, Inc., 548 East Mallard Circle, Fresno, CA 93710.
- (7) LaMotte Chemical Products Co., P.O. Box 329, Chestertown, MD 21620.
- (8) Geisling, K., Miksch, R. R., and Rappaport, S. M., "Generation of Dry Formaldehyde at Trace Levels by the Vapor-Phase Depolymerization of Trioxane," *Analytical Chemistry*, Vol 54, 1981, pp. 140–142.
- (9) National Institute for Occupational Safety and Health, *NIOSH Manual of Analytical Methods*, Second and Third Eds. with supplements, Vols 1–7, Washington, DC, 1973 and 1984.
- (10) Miksch, R. R., and Anthon, D. W., "A Recommendation for Combining the Standard Analytical Methods for the Determinations of Formaldehyde and Total Aldehydes in Air," *American Industrial Hygiene Association Journal*, Vol 43, 1982, pp. 362–365.
- (11) National Council of the Paper Industry for Air and Stream Improvement, "Evaluation of Four Recently Commercially Available Passive Diffusion Badge Monitors for Determination of Workplace Formaldehyde," *NCASI Technical Bulletin No. 553*, New York, NY, 1988.
- (12) Lautenberger, W. J., Kring, E. V., and Morello, J. A., "A New Personal Badge Monitor for Organic Vapors," *American Industrial Hygiene Association Journal*, Vol 41, 1980, pp. 737–747.
- (13) National Institute for Occupational Safety and Health, *Documentation of the NIOSH Validation Tests*, Publication No. 77-185, Washington, DC, 1977.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).