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FORMALDEHYDE IN WORKPLACE ATMOSPHERES
(3M MODEL 3721 MONITOR)



Method Number: ID-205

Matrix: Air

OSHA Permissible Exposure Limit (PEL): 1 ppm Time Weighted Average (TWA)*
3 ppm Short-Term Exposure Limit (STEL)*

Collection Device: Passive badge monitor containing bisulfite-impregnated paper

Recommended Sampling Time: 8 h (4 to 16 h range)

Average Sampling Rate: 0.0614 ± 0.005 L/min (25 °C & 760 mmHg)

Face Velocity: Minimum 4.6 m/min (15 ft/min)

Analytical Procedure: A modified chromotropic acid procedure is used. Sample filters are desorbed using deionized water. Solutions are acidified, and chromotropic acid is added. The color complex formed is analyzed using a UV spectrophotometer at 580 nm.

Detection Limit
Qualitative: 0.039 ppm (4-h sampling time)
Quantitative: 0.11 ppm (4-h sampling time)

Dose Range: 0.8 to 72 ppm-h (as claimed by the manufacturer)

Precision and Accuracy
Validation Range: 0.2 to 4.9 ppm
CV_T: 0.084
Bias^{**}: +0.018
Overall Error^{**}: ±18.6%

Method Classification: Validated Method

Chemist: James C. Ku
Date: December, 1990

* The 3M Model 3721 monitor is recommended for TWA determinations only. It is not recommended for STEL monitoring. Any samples taken for STEL determinations should follow OSHA method No. 52.

** As compared to OSHA method no. ID-102.

Commercial manufacturers and products mentioned in this method are for descriptive use only and do not constitute endorsements by USDOL-OSHA. Although the following sampling procedure uses a specific formaldehyde monitor, other passive monitors can be substituted provided they meet validation requirements.

Branch of Inorganic Methods Development
OSHA Technical Center
Salt Lake City, Utah

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1. Introduction

This method describes the passive monitor collection of airborne formaldehyde in the breathing zone of workplace personnel and the subsequent analysis of those samples using a colorimetric technique. Although this method specifically mentions the 3M Model 3721 monitor, other monitors can be used provided performance requirements have been met. Some examples of validation procedures to determine performance are given in references 5.1 and 5.2.

1.1 History

The simplicity and freedom of the 3M Model 3751 formaldehyde passive monitor showed promise when first offered in 1981 as an industrial hygiene sampling alternative for formaldehyde (5.3); however, subsequent independent studies indicated analyte loss when sampling at low humidities (5.4, 5.5). Consequently, the Model 3751 monitor was removed from the market by 3M in April, 1984. The Model 3721 3M monitor, capable of sample humidification, was introduced in 1985 as a replacement. The changes instituted by 3M and incorporated into the model 3721 are:

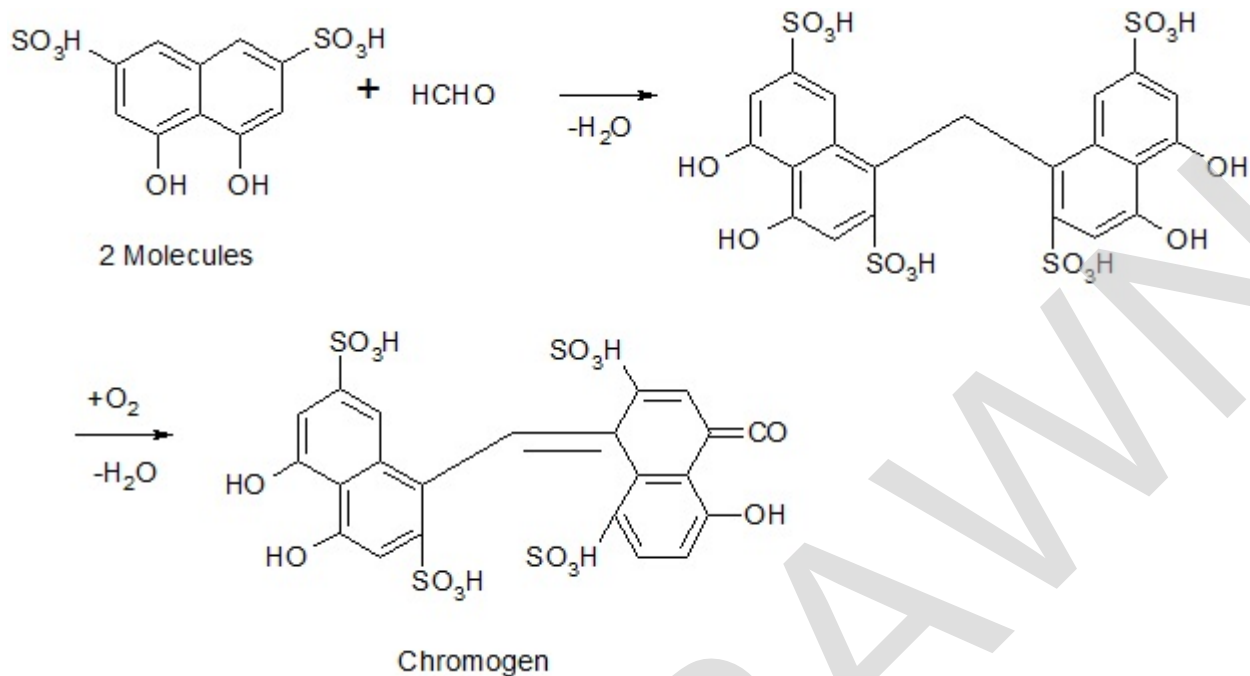
- 1) A water-saturated pad in the bottom section of the monitor has been added for sample humidification.
- 2) Each monitor is now packaged in a sealed metal container. Previously, the Model 3751 monitor was enclosed in a resealable plastic bag.
- 3) The calculated sampling rate has been changed from 0.0659 to 0.0614 L/min.
Note: The sampling rate of 0.0614 L/min is in agreement with a previous OSHA Salt Lake City Analytical Laboratory (SLCAL) study (5.5).

With the exception of the moisturizing pad, the appearance of the Model 3721 is physically identical to the Model 3751 monitor. The Model 3751 monitor has been extensively evaluated by independent laboratories (5.4-5.6). Results from these studies did not indicate serious problems with desorption efficiency, face velocity, reverse diffusion, or post-collection sample storage stability. The recent modifications instituted by 3M suggest sampling performance would not be significantly affected in these areas. As long as the face velocity of the sampled environment is above 4.6 m/min (15 ft/min), the sampling rate of the monitor does not appear to be significantly altered (5.4-5.7). Sampling and analytical procedures are identical for either model monitor; however, result calculations are different since slightly different sampling rates are used.

1.2 Principle

The 3M formaldehyde monitor is a diffusion-type air monitoring assembly worn near the breathing zone of personnel to evaluate potential exposure to formaldehyde (HCHO) vapors. Formaldehyde vapor is adsorbed on bisulfite-impregnated paper located within the assembly. The resulting adduct is desorbed with deionized water. An aliquot of the sample is reacted with chromotropic acid in the presence of sulfuric acid to form a purple mono-cationic chromogen. The absorbance of this colored solution is read in a spectrophotometer at 580 nm and is compared to prepared standards. Although the chemistry of the color formation is not well-established, the following reaction mechanism is proposed in acidic solution (5.8):

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1.3 Advantages and Disadvantages

- 1.3.1 This method has adequate sensitivity for measuring workplace atmosphere concentrations of formaldehyde for TWA determinations.
- 1.3.2 The passive dosimeter used for collection of formaldehyde vapor is small, lightweight, and requires no sampling pumps.
- 1.3.3 The collected formaldehyde sample is stable for at least 30 days.
- 1.3.4 One disadvantage of the method is that the analytical procedure may not be capable of accurately determining STEL exposures at or below 3 ppm.
- 1.3.5 Another disadvantage with the dosimeter is sample rate dependence on face velocity. The dosimeter should not be used in areas where the air velocity is less than 4.6 m/min (15 ft/min). Most industrial work areas have air movement above 7.6 m/min (25 ft/min).
- 1.3.6 A disadvantage concerning the analytical procedure is the use of concentrated H_2SO_4 during sample preparation. Extreme care should be used when handling H_2SO_4 .

1.4 Method Performance (5.5, 5.9)

- 1.4.1 This method was validated over the range of 0.2 to 4.9 ppm.
- 1.4.2 The coefficient of variation (CV_T) for the total analytical and sampling method (50% RH) was 0.084. The overall error (as compared to the reference method OSHA ID-102) was $\pm 18.6\%$.
- 1.4.3 The qualitative detection limit of the analytical method is 0.7 μg of formaldehyde based on a 3.0-mL sample volume. This is equivalent to 0.039 ppm for a 240-min sampling time.
- 1.4.4 The quantitative determination limit for the analytical method is 2 μg of formaldehyde in a 3.0-mL sample volume. This is equivalent to 0.11 ppm for a 240-min sampling time.

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1.4.5 Somewhat variable results were obtained when sampling for a short duration (STEL). Therefore, the 3M Model 3721 monitor is recommended for 4 to 16-h sampling measurements only, and is not recommended for STEL sampling.

1.4.6 The Model 3751 monitor was extensively evaluated in 1982 (5.5) and included storage stability, face velocity, sampling rate, and reverse diffusion experiments. Due to the similarity of the 3751 and 3721 monitors, these experiments were not repeated for the Model 3721. The 3751 experiments indicated (5.5):

- 1) The results of a storage stability test show that the mean recovery of samples stored after 30 days were within $\pm 10\%$ of the mean of monitors analyzed immediately after sampling.
- 2) The results of a face velocity test indicate that the 3M Model 3751 monitor can accurately measure a known concentration as high as 10 ppm at face velocities as low as 15 ft/min.
3. The results of a sampling rate validation test indicate that the average sampling rate was 0.0614 ± 0.005 L/min.
4. The results of a reverse diffusion test indicate that reverse diffusion of collected formaldehyde from the monitor back into the atmosphere should not be a significant factor when sampling over an 8-h sampling period.

1.5 Interferences

1.5.1 When other substances are known or suspected to be present in the air, such information, including their suspected identities, should be transmitted with the sample.

1.5.2 Any compound that has the potential of developing the same color as the formaldehyde/chromotropic acid complex using the conditions described in this method is an interference.

1.5.3 It has been reported by 3M that there is no interference from phenol (5.10). The lack of interference is mainly due to the monitor's inability to collect a significant amount of phenol.

1.6 Uses (5.11)

1.6.1 Formaldehyde (CAS 50-00-0) is used mainly as a raw material for producing synthetic resins. This accounts for over 50% of the total production of formaldehyde.

1.6.2 Potential occupational exposures to formaldehyde are listed:

Anatomists	Glass etchers
Agricultural Workers	Glue and adhesive makers
Bakers	Grease-resistant textile finishers
Beauticians	
Biologists	Greenhouse workers
Bookbinders	Hexamethylenetetramine makers
Botanists	Hide preservers
Deodorant makers	Histology technicians
Disinfectant makers	Ink makers
Disinfectors makers	Lacquerers and lacquer makers
Dress goods store personnel	Mirror workers
Dressmakers	Oil well workers
Drugmakers	Paper makers
Dyemakers	Pentaerythritol makers
Electrical insulation makers	Photographic film makers
Embalmers	Plastic workers
Embalming fluid makers	Resin makers
Ethylene glycol makers	Rubber makers

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Fertilizer makers
Fireproofers
Formaldehyde resin makers
Foundry employees
Fumigators
Fungicide workers
Furniture dippers and sprayers
Fur processors

Soil sterilizers
Surgeons
Tannery workers
Taxidermists
Textile mordanters and printers
Textile waterproofers
Varnish workers
Wood preservers

1.7 Physical Properties (5.11):

Formula	HCHO
Molecular Weight	30.03
Physical state	Gas
Melting point	-92 °C
Boiling point	-21 °C
Specific gravity	0.815
Relative vapor density	1.043 (air = 1)
Solubility	Soluble in water, alcohol, and ether
Color	Colorless
Odor	Pungent and irritating
Explosive limits (Gas)	Gas 7.0-73% by volume in air
Flashpoint (closed cup)	50 °C (122 °F) of aqueous solution

1.8 Toxicology

Note: Information listed within this section is a synopsis of current knowledge of the physiological effects of formaldehyde (HCHO) and is not intended to be used as the basis for OSHA policy.

Formaldehyde is considered a strong irritant and potent sensitizer. Inhalation of large amount of HCHO can cause severe irritation of the upper respiratory tract and death. Data from human exposures indicate that exposure to large concentrations of HCHO gas may lead to pulmonary edema. Even HCHO gas present in the workroom at concentrations of 1 to 11 ppm can cause eye, nose, and throat irritation (5.11). Formaldehyde has the potential to cause cancer in humans (5.12).

The following symptoms have been noted in some individuals (5.12):

<u>Concentration</u>	<u>Symptoms</u>
0.5 to 2 ppm	eyes, nose and throat irritation
3 to 5 ppm	tearing of the eyes
10 to 20 ppm	difficult breathing, nose and throat burning, cough, heavy tearing of the eyes
25 to 30 ppm	severe respiratory tract injury
100 ppm	immediately dangerous to life and health (IDLH)

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2. Sampling

2.1 Precautions:

- 2.1.1 Avoid inhalation of or skin contact with formaldehyde.
- 2.1.2 If the possibility exists that the face velocity of an area being sampled is less than 4.6 m/min (15 ft/min), an active sampling device (i.e., OSHA sampling and analytical method No. 52) should be used instead of the passive monitor.

2.2 Equipment - Passive Monitors (If provided, also follow the 3M Formaldehyde Monitor Model 3721 - Instructions for Use.)

The 3M Model 3721 formaldehyde monitor (3M, St. Paul, MN) contains the following parts:

- 1) Container consisting of two aluminum cans held together by a label. The two cans are labeled can A and can B.
- 2) Can A contains:
Top Section (has a white film and plastic retaining ring),
Sealing Cup (has Date, Start Time, etc. written on it)
- 3) Can B contains:
Bottom Section (has a metal clip attached), Translucent Closure Cap

Note: The original shipping container and aluminum cans can be reused for sample shipment to the lab.

- 2.2.1 Remove the plastic lid from can A. Open each can by grasping the ring tabs and carefully pulling up. Remove the lids from both cans. Examine the contents to make sure all parts are available.
- 2.2.2 Remove the Translucent Closure Cap from the Bottom Section. Save the Closure Cap.
- 2.2.3 Pressing firmly, snap together the Top and Bottom Sections. Make sure the white film and plastic ring are **NOT** removed from the Top Section. The monitor is now ready for sampling.

2.3 Sampling Procedure

- 2.3.1 Immediately begin sampling by attaching the monitor to the employee or by placing it in the sampling area. The white film (Top Section) should face away from the employee.
- 2.3.2 Record the following information:
 - 1) Beginning sampling time
 - 2) Sampling date
 - 3) Monitor serial number
 - 4) Employee or area identification
 - 5) Temperature, pressure, and relative humidity at the sampling site
- 2.3.3 If possible, sample for 8 h. The minimum sampling time recommended is 1 to 2 h. For indoor air quality investigations, sample up to 16 h.
- 2.3.4 Immediately after sampling, remove and discard the white plastic film and purple retaining ring from the monitor. In place of the film/ring, snap on the Translucent Closure Cap by applying some pressure. A "clicking" sound should be heard when the cap is securely fastened.

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- 2.3.5 Be sure both plugs on the Translucent Closure Cap are firmly seated. This will insure a gas-tight seal.
- 2.3.6 Snap the Sealing Cup into place on the Bottom Section of the monitor. Be sure the cup is snapped securely.
- 2.3.7 Record the end sampling time and any drastic change (>10%) in temperature, pressure, or relative humidity that may have occurred during sampling.
- 2.3.8 Assemble a blank sample in the same fashion as mentioned in Sections 2.2.1-2.2.3 and 2.3.4-2.3.6. Do not expose the blank.

2.4 Sample Shipment

- 2.4.1 Place each monitor back into the aluminum container, cover with the plastic cap, and securely wrap each can with an OSHA Form 21 sample seal.
- 2.4.2 Submit at least one blank sample with each set of samples. The blank sample should have been handled in the same manner as the other samples except that it was not exposed. If possible, also submit a "lot blank". This is an unused monitor inside an unopened aluminum container.
- 2.4.3 When other substances are known or suspected to be present in the air, such information should be transmitted with the sample.
- 2.4.4 Send the monitors directly to the laboratory and request formaldehyde analysis. The original shipping carton can be used for shipment.

3. Analysis

3.1 Precautions

- 3.1.1 Refer to instrument manuals for proper operation.
- 3.1.2 Observe laboratory safety regulations and practices.
- 3.1.3 **CAUTION:** Sulfuric acid can cause severe burns. Wear protective gloves, laboratory coat, and eyewear when handling concentrated sulfuric acid and the formaldehyde stock solution.
CAUTION: Formaldehyde has the potential to cause cancer in humans (5.12). Extreme care must be observed when handling.
- 3.1.4 Do not store formaldehyde standards or samples in a refrigerator since polymerization will occur. Polymer precipitation may be observed by the appearance of a white milky substance in the formaldehyde solution.
- 3.1.5 Sodium sulfite solutions used for formaldehyde standardization gradually absorb carbon dioxide on exposure to air. Solutions which have stood for more than a week should be discarded.
- 3.1.6 Do not use reagent bottles having caps which contain phenolic resins. Formaldehyde contamination could occur.

3.2 Equipment

- 3.2.1 Spectrophotometer: double beam, 1-cm cell.
- 3.2.2 Meter, pH.

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3.2.3 Miscellaneous volumetric glassware or plasticware: Volumetric burets, graduated cylinders, pipettes, volumetric and Erlenmeyer flasks, other laboratory glassware, syringes. (Note: All glassware or plasticware should be washed and rinsed thoroughly with deionized water and then air dried prior to use.)

3.2.4 Analytical balance (0.01 mg).

3.3 Reagents (All chemicals should be reagent grade or better.)

3.3.1 Deionized water (DI H₂O).

3.3.2 Chromotropic acid sodium salt (C₁₀H₇O₈S₂Na) solution (1%): Dissolve 1 g of chromotropic acid sodium salt (1,8 - dihydroxy-3, 6-naphthalenedisulfonic acid sodium salt) in 100 mL of DI H₂O. Prepare this solution daily. (Note: This reagent is also commonly referred to as 4,5-dihydroxy-2,7-naphthalenedisulfonic acid sodium salt.)

3.3.3 Sulfuric acid (H₂SO₄), concentrated.

3.3.4 Sodium bisulfite (NaHSO₃), 1%: Dissolve 10 g of NaHSO₃ in 1 L of DI H₂O.

3.3.5 Formaldehyde (HCHO) solution, 37%.

3.3.6 Formaldehyde stock solution, ~1,000 µg/mL: Dissolve 2.7 g (about 3 mL) of 37% HCHO solution in 1 L of DI H₂O. Standardize this solution as described in Section 3.4.

The solution is stable for at least 6 months. (Note: After 6 months, the standardization should be repeated).

3.3.7 Reagents for standardization of HCHO stock solution:

- 1) Sodium carbonate (Na₂CO₃), certified, 99.9% minimum purity: Dry the Na₂CO₃ powder at 120 °C for 2 h, then transfer to a desiccator and cool to a constant weight. Use as a primary standard.
- 2) Sulfuric acid, 0.1 N: Dilute 3 mL of concentrated H₂SO₄ slowly to 1 L with DI H₂O.
- 3) Sodium sulfite (Na₂SO₃), 1.13 M: Dissolve 140 g of anhydrous Na₂SO₃ in 980 mL DI H₂O. Store in a refrigerator (approximately 4 °C).
- 4) Bromocresol green/methyl red mixed indicator solution.

3.4 Standard Preparation

3.4.1 Standardization of the HCHO ~1,000 µg/mL stock solution (5.13, 5.14):

1. 1)

Standardize the 0.1 N H₂SO₄ solution using the certified Na₂CO₃ as a primary standard: Weigh 1.00 to 1.20 g of dried Na₂CO₃ into a 250-mL beaker containing 50 mL of DI H₂O, add 3 drops of bromocresol green/methyl red mixed indicator and titrate with the H₂SO₄ to a faint pink color. Heat the titrated solution to a gentle boil for 2 min to expel any dissolved CO₂, then cool the flask contents to room temperature. If the end point has not been overrun, the indicator will reassume its characteristic green color. Complete the titration with H₂SO₄ to a sharp color change. Calculate the normality of the H₂SO₄ solution (N₂) based on the following equation:

$$N_2 = \text{meq of Na}_2\text{CO}_3/V_2$$

where: V₂ = mL of H₂SO₄ solution required to titrate the Na₂CO₃.

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- 2) Use a pH meter and adjust the pH of 25.0 mL of the 1.13 M Na₂SO₃ solution to 9.6 with the standardized 0.1 N H₂SO₄.
- 3) Place 50.0 mL of the HCHO ~1,000 µg/mL stock solution into a 250-mL beaker.
- 4) Add the previously adjusted Na₂SO₃ solution to the 250-mL beaker and titrate to a pH of 9.6 with the standardized 0.1 N H₂SO₄. Calculate the concentration of HCHO as follows:

$$\text{HCHO, } \mu\text{g/mL} = \frac{(A - B)(C)(D)}{E}$$

where:

A = mL of H₂SO₄ solution required to titrate the sample

B = mL of H₂SO₄ solution required to titrate the blank

C = normality of the H₂SO₄ solution (meq/mL)

D = (30 mg/meq of HCHO)(1,000 µg/mg) = 30 X 10³ µg/meq of HCHO

E = mL of formaldehyde used

3.4.2 Preparation of standards

To a series of 25-mL Erlenmeyer flasks already containing 2 mL of 1% NaHSO₃, carefully add 1.0, 3.0, 5.0, 10.0, 15.0, and 20.0 µL of the ~1,000 µg/mL HCHO stock solution. If the stock solution is prepared as exactly 1,000 µg/mL HCHO after standardization, these aliquots are equivalent to 1.0, 3.0, 5.0, 10.0, 15.0, and 20.0 µg of HCHO. As an alternative, standards can be prepared in 1% NaHSO₃ using serial dilution of the ~1,000 µg/mL stock solution.

3.5 Sample Preparation

- 3.5.1 Assemble and prepare a "lot blank" for analysis, if available (also see Section 2.4.2).
- 3.5.2 Open both ports of the Translucent Closure Cap of each monitor.
- 3.5.3 Using the center port of the Translucent Closure Cap and a small pipette or syringe, add 3 mL of DI H₂O to each monitor. Reseal the ports.
- 3.5.4 After 30 min, with occasional gentle agitation, transfer a 2-mL aliquot of the solution into a 20-mL screw-cap glass vial and reserve for color development.

3.6 Analysis

- 3.6.1 Develop the color of samples, standards, and blank solutions by adding 1 mL of 1% chromotropic acid solution, and after thorough mixing, 5 mL of concentrated H₂SO₄.

(Note: Add the sulfuric acid **slowly** and **carefully**. Add H₂SO₄ to the samples and standards in the same fashion since heat catalyzes the color formation.)

- 3.6.2 Allow the solutions to cool to room temperature, then measure the absorbance of each solution at 580 nm using a 1-cm cell.
- 3.6.3 If the sample absorbance is larger than the absorbance of the highest standard, take a smaller aliquot from the monitor, dilute to 2 mL, and repeat Sections 3.6.1-3.6.2. Use the appropriate dilution factor in calculations if an aliquot other than 2 mL is taken.

3.7 Calculations

- 3.7.1 Use a least squares regression program to plot a concentration-response curve of peak absorbance versus the amount (µg) of formaldehyde in each standard.

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- 3.7.2 Determine the amount (μg) of formaldehyde, A, corresponding to the absorbance in each analyzed sample aliquot from this curve.
- 3.7.3 Calculate the total amount (μg) of formaldehyde, W, in each sample:

$$W = \frac{(A)(\text{Sample vol, mL})(DF)}{(\text{Aliquot, mL})}$$

where: DF = Dilution Factor (if none, DF = 1)

- 3.7.4 Blank correct each sample and calculate the concentration of formaldehyde in each sample:

$$\text{ppm formaldehyde} = \frac{(W - W_b) \times MV}{MW \times AV}$$

where:

AV = $ST \times 0.0614 \times (T_1/T_2)^{1.5} \times (P_2/P_1)$

W_b = Total μg of formaldehyde in the blank sample

MV = Molar volume at 25 °C and 760 mmHg (24.45 L/mole)

MW = Molecular weight of formaldehyde (30 g/mole)

where:

ST = Sampling time (min)

0.0614 = Sampling rate (L/min) at 25 °C and 760 mmHg

T_1 = Sampling site temperature (K)

T_2 = 298 K

P_1 = Sampling site pressure (mmHg)

P_2 = 760 mmHg

3.8 Reporting Results

Report results to the industrial hygienist as ppm formaldehyde.

4. Backup Report

See Reference 5.9 for complete information.

5. References

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