

VIII. REFERENCES

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IX. APPENDIX I
SAMPLING OF FORMALDEHYDE IN AIR

Sampling

Air samples are collected to represent the breathing zone of employees by drawing air through two all-glass midget impingers in series, each containing 20 ml of distilled water. (If other aldehydes are present, use 20 ml of 1% sodium bisulfite solution.) Under certain conditions, it may be possible to attach the impingers to employees clothing. A personal sampling pump may also be worn by the employee. In other instances, employee movements may make sampling in this manner impractical, but samples should be collected as close to the breathing zone as possible. A prefilter assembly should be used when dusty or smoky conditions prevail and should be connected to the impinger using a minimum amount of tubing. The air being sampled should not pass through any other tubing or equipment before entering the impinger. Sampling is performed for at least 30 minutes at a rate of 1 liter/minute. The flow rate, with the impingers on line, should be checked as a minimum precaution before and after the sample is taken.

Two impingers must be used in series, because under conditions of sampling the collection efficiency of only one impinger is approximately 80% [179]. With two impingers in series, the total collection efficiency is 95% [179]. The contents of each impinger may be analyzed separately if relatively high concentrations are suspected, or may be combined and analyzed as a single sample. If each impinger is analyzed separately and the second impinger is found to contain more than about 30% of the amount

collected in the first impinger, appreciable loss of sample has most likely occurred, and resampling is required to obtain an adequate value.

After sampling, the impinger stems can be removed and cleaned, first tapping the stem gently against the inside wall of the impinger bottle to recover as much of the sampling solution as possible, then washing with a small amount (1-2 ml) of distilled water and adding the wash to the impinger flask. The impinger flask is then sealed tightly with a hard, nonreactive stopper, preferably Teflon, but never with rubber. If shipping the impinger flasks with the stems in, is preferred, the outlets of the stem should be sealed with Parafilm or equivalent nonrubber covers, and the ground glass joints sealed, usually by means of plastic tape. Care should be taken to minimize spillage or loss by evaporation at all times. If analysis cannot be done within a day, samples should be refrigerated to prevent sample loss due to polymerization. Whenever possible, hand delivery of the samples is recommended, or special impinger shipping cases should be used to ship the samples. A blank impinger should be handled in exactly the same manner as the other samples (fill, seal, and transport) except that no air is sampled through this impinger.

Calibration

Since the accuracy of an analysis can be no greater than the accuracy of the volume of air which is measured, the accurate calibration of a sampling device is essential. The frequency of calibration required depends on the use, care, and handling to which the pump is subjected. Pumps should be calibrated if they have been subjected to abuse or if they

have just been repaired or received from a manufacturer. Under certain conditions of heavy usage, more frequent calibration may be necessary.

Ordinarily, pumps should be calibrated in the laboratory both before they are used in the field and after they have been used to collect a large number of field samples. The accuracy of calibration is dependent on the type of instrument used as a reference. The choice of calibration instrument will depend largely upon where the calibration is to be performed. For laboratory testing, a 1-liter buret or wet-test meter is recommended, although other standard calibrating instruments such as spirometer, Marriot bottle, or drygas meter can be used. The actual set-up should be the same for any of the instruments mentioned above. The calibration instrument should be connected in sequence to the sampling train which will be followed by the sampler pump. In this way, the calibration instrument will be at atmospheric pressure. If the personal sampler pump is used, each pump must be calibrated separately. If the buret is used, it should be set up so that the flow is toward the narrow end of the unit.

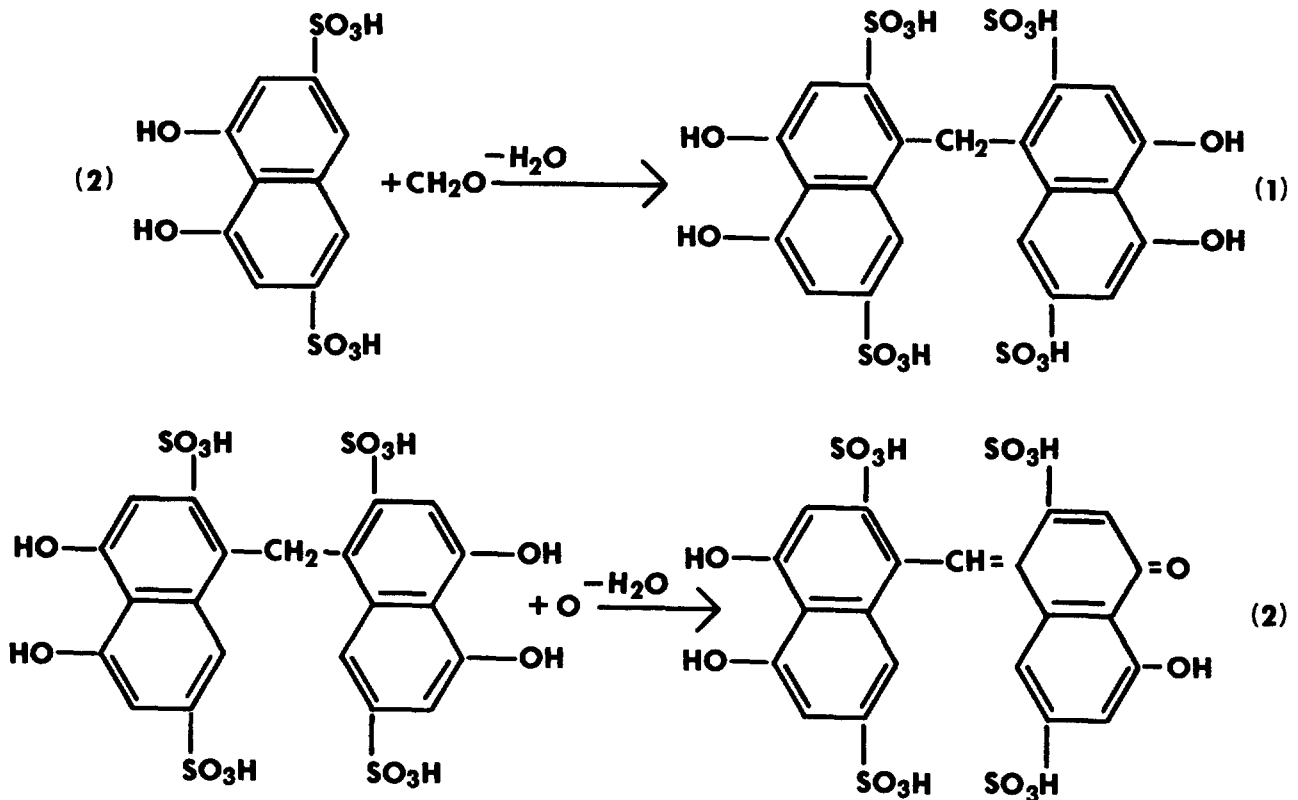
Care must be exercised in the assembly to ensure that seals at the joints are airtight and that the length of connecting tubing is kept at a minimum. Calibration should be performed under essentially the same conditions of pressure and temperature under which it is anticipated the sampling will be performed. The calibrated pump rotameter should be used to set the flow rate in the field.

X. APPENDIX II

ANALYTICAL METHOD FOR FORMALDEHYDE IN AIR

Principle of the Method

Formaldehyde reacts with chromotropic acid-sulfuric acid solution to form a purple monocationic chromogen. The absorbance of the colored solution is read in a spectrophotometer at 580 nanometers (nm) and is proportional to the amount of formaldehyde in the solution. The chemistry of this color reaction is uncertain. Fiegel [212] proposed that the chromogen is formed as follows:



Range and Sensitivity

From 0.1 $\mu\text{g/ml}$ to 2.0 $\mu\text{g/ml}$ of formaldehyde can be measured in the 10-ml final volume of solution.

A concentration as low as 0.16 ppm of formaldehyde can be determined in a 25-liter air sample based on an aliquot of 4 ml from 20 ml of absorbing solution and a difference of 0.05 absorbance unit from the blank.

Interferences

The chromotropic acid procedure has very few interferences [179] from other aldehydes. Saturated aldehydes give less than 0.01% positive interference [179], and the unsaturated aldehyde acrolein results in a few percent positive interference [179]. Ethanol and higher molecular weight alcohols and olefins in mixtures with formaldehyde are negative interferences [179]. However, concentrations of alcohols in air are usually much lower than formaldehyde concentrations and, therefore, do not usually cause a serious interference with the estimation of formaldehyde [179].

Phenols result in a 10-20% negative interference [179] when present at an 8:1 excess over formaldehyde. They are, however, ordinarily present in the atmosphere at lesser concentrations [179] than formaldehyde and, therefore, usually do not cause serious interference with the method.

Ethylene and propylene in a 10:1 excess over formaldehyde result in a 5-10% negative interference, and 2-methyl-1,3-butadiene in a 15:1 excess over formaldehyde showed a 15% negative interference [179]. Aromatic

hydrocarbons may produce a negative interference [188]. It has recently been found that cyclohexanone causes a bleaching of the final color [179].

Precision and Accuracy

The method was checked for reproducibility by having three different analysts in three different laboratories analyze standard formaldehyde samples. [179,180] The results listed in Table X-1 agreed within $\pm 5\%$.

TABLE X-1
COMPARISON OF FORMALDEHYDE RESULTS FROM THREE LABORATORIES

<u>Formaldehyde</u>	<u>Absorbance</u>		
<u>Micrograms</u>	<u>Lab. 1</u>	<u>Lab. 2</u>	<u>Lab. 3</u>
1	0.057	0.063	0.061
3	0.183	0.175	0.189
5	0.269	0.279	0.262
7	0.398	0.381	0.392
10	0.566	0.547	0.537
20	1.02	0.980	1.07

Apparatus

(a) Sampling Equipment

The sampling unit for the impinger collection method consists of the following components:

(1) Two graduated midget impingers containing distilled water.

(2) A pump capable of delivering a flow rate of 1 liter/minute. The sampling pump is protected from splashover or water condensation by an absorption tube loosely packed with a plug of glass wool and inserted between the exit arm of the impinger and the pump.

(3) An integrating volume meter such as a dry-gas or wet-test meter, or a calibrated rotameter.

(4) Thermometer.

(5) Manometer.

(6) Stopwatch.

(b) Spectrophotometer

An instrument capable of measuring the absorbance of a colored solution at 580 nm.

(c) Associated laboratory glassware for use with a spectrophotometer.

Reagents

(a) Chromotropic acid reagent

Dissolve 0.10 g of 4,5-dihydroxy-2,7-naphthalenedisulfonic acid disodium salt in water and dilute to 10 ml. Filter if necessary and store

in a brown bottle. Make up solution weekly, but discard if solution turns yellow or brown.

(b) Concentrated sulfuric acid

(c) Formaldehyde standard solution "A" (1 mg/ml)

Dilute 3.0 ml of 37% formalin solution to 1 liter with distilled water. This solution must be standardized as described below. The solution is stable for at least a 3-month period. Alternatively, sodium formaldehyde bisulfite can be used as a primary standard. Dissolve 4.4703 g in distilled water and dilute to 1 liter.

(d) Formaldehyde Standard Solution "B" (10 μ g/ml)

Dilute 1 ml of standard solution "A" to 100 ml with distilled water. Make up solution daily.

(e) Iodine, 0.1 N (approximate)

Dissolve 25 g of potassium iodide in about 25 ml of water, add 12.7 g of iodine and dilute to 1 liter.

(f) Iodine, 0.01 N

Dilute 100 ml of the 0.1 N iodine solution to 1 liter. Standardize using either sodium thiosulfate or arsenic trioxide.

(g) Starch solution, 1%

Make a paste of 1 g of soluble starch and 2 ml of water and slowly add the paste to 100 milliliters of boiling water. Cool, add several milliliters of chloroform as a preservative, and store in a stoppered bottle. Discard if a mold growth is noticeable.

(h) Sodium carbonate buffer solution

Dissolve 80 g of anhydrous sodium carbonate in about 500 ml of water. Slowly add 20 ml of glacial acetic acid to give a final pH of 9.6, and dilute to 1 liter.

- (i) Sodium bisulfite, 1%

Dissolve 1 g of sodium bisulfite in 100 ml of water. It is best to prepare a fresh solution weekly.

Procedure

- (a) Cleaning of equipment

Care must be exercised to ensure the absence of probable contaminants like organic materials that can be charred by concentrated sulfuric acid. After normal cleaning with detergent solution, glassware should be soaked for 1 hour in a 1:1 mixture of nitric and sulfuric acids, followed by thorough rinsing with doubly deionized water to remove all possible organic contaminants.

- (b) Collection and shipping of samples

Pour 20 ml of the absorbing solution (distilled water) into each graduated midget impinger and collect formaldehyde from air and prepare samples as described in Appendix I.

- (c) Analysis of samples

(1) Transfer the sample from each impinger to either a 25-ml or 50-ml graduate. Note the volume of each impinger solution.

(2) Pipet a 4-ml aliquot from each of the sampling solutions into glass stoppered test tubes. A blank containing 4 ml of distilled water must also be run. If the formaldehyde content of the aliquot exceeds the limit of the method, use a smaller aliquot diluted to 4 ml with distilled water. Alternatively, aliquots from each impinger can be combined for a single analysis

(3) Add 0.1 ml of 1% chromotropic acid reagent to the solution and mix.

(4) Into the solution from step 3, pipet slowly and cautiously 6 ml of concentrated sulfuric acid. The heat produced by the addition of the sulfuric acid is required to promote the reaction, but the acid should be added sufficiently slowly to prevent loss of sample because of boiling and spattering.

(5) Allow to cool for 20 minutes. Read absorbance at 580 nm in a suitable spectrophotometer using a 1-cm cell. Determine the formaldehyde content of the sampling solution from a curve previously prepared from standard formaldehyde solutions.

(6) During the analysis, it is good practice to group together the two impingers from each sampling series and label them as "A" and "B". The formaldehyde content calculated in "A" is added to that calculated in "B" to give the total amount of formaldehyde collected by the impingers in series.

Calibration and Standards

(a) Standardization of formaldehyde solution

(1) Pipet 1 ml of formaldehyde standard solution "A" into an iodine flask. Into another flask, pipet 1 ml of distilled water. This second flask serves as the blank.

(2) To each flask, add 10 ml of 1% sodium bisulfite and 1 ml of 1% starch solution.

(3) Titrate with 0.1 N iodine to a dark blue color.

(4) Destroy the excess iodine with 0.05 N sodium thiosulfate.

(5) Add 0.01 N iodine until a faint blue end point is reached.

(6) The excess inorganic bisulfite is now completely oxidized to sulfate, and the solution is ready for the assay of the formaldehyde bisulfite addition product.

(7) Chill the flask in an ice bath and add 25 ml of chilled sodium carbonate buffer. Titrate the liberated sulfite with 0.01 N iodine, using a microburet, to a faint blue end point. The amount of iodine added in this step must be accurately measured and recorded.

(8) One milliliter of 0.00100 N iodine is equivalent to 0.15 mg of formaldehyde. Therefore, since 1 milliliter of formaldehyde standard solution was titrated, the milliliter of 0.01 N iodine used in the final titration multiplied by the factor, 0.15, gives the formaldehyde concentration of the standard solution in mg/ml.

(9) The factor 0.15 must be adjusted or determined in accord with the exact normality of the iodine solution.

(b) Preparation of Standard Curve

(1) Pipet 0, 0.1, 0.3, 0.5, 0.7, 1.0, and 2.0 ml of standard solution "B" into glass stoppered test tubes.

(2) Dilute each standard to 4 ml with distilled water.

(3) Develop the color as described in the analysis procedure under Section (C).

(4) Plot absorbance against micrograms of formaldehyde in the color developed solution. Note that the microgram concentration of the

formaldehyde is determined based on the standardization value obtained for solution A.

Calculations

(a) Convert the volume of air sampled (V) to the volume of air at standard conditions (Vs) of 760 mm of mercury and 25 degrees C, using the correction formula:

$$V_s = \frac{V \times P \times 298}{760(T + 273)}$$

where:

Vs = volume of air in liters at standard conditions

V = volume of air sampled in liters

P = barometric pressure in mm of mercury

T = temperature of sample air, C

(b) Determine the total concentration (Ct) of formaldehyde present in the two sample impingers in series, A and B.

$$C_t = C_a \times F_a + C_b \times F_b$$

where:

Ct = total µg of formaldehyde in the sample

Ca and Cb = respective formaldehyde concentration in µg of the sample aliquots taken from impingers A and B as determined from the calibration curve

Fa and Fb = respective aliquot factor; sampling soln. vol. in ml
ml aliquot used

(c) The concentration of formaldehyde in the sampled atmosphere may be calculated by using the following equation, assuming standard conditions are taken as 760 mm of mercury and 25 degrees C:

$$\text{ppm (volume)} = \frac{Ct \times 24.47}{V_s \times \text{M.W.}}$$

where:

V_s = liters of air sampled at standard conditions

M.W. = molecular weight of formaldehyde (30.03)

24.47 = μl of formaldehyde gas in one micromole at 760 mm Hg
and 25 degrees C.