

CERTAIN-TEED PRODUCTS CORPORATION STUDY

CONDUCTED BY EPA, REGION III

AIR QUALITY MONITORING BRANCH

SURVEILLANCE & ANALYSIS DIVISION

APPENDICES A THROUGH I

APPENDIX B
METEOROLOGICAL DATA

5 10 15 20
0 10 20 30 40
20 40 60 80

0-50 mph
RANGE

5 10 20 mph

1
20 mph

10 15 20 25
30 35 40 45
50 55 60 65

5 10 20 mph

360 90

N-0°/360°

SITE B (9)
5/1/78 6:27 PM

DIRECTION CORRECTED
6:27 PM CG

340

350

335

340

STOP SAMPLING 340

5/1/78
12:00 AM
(9)

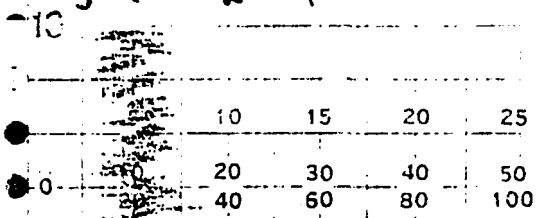
START 9:30 AM

56.172

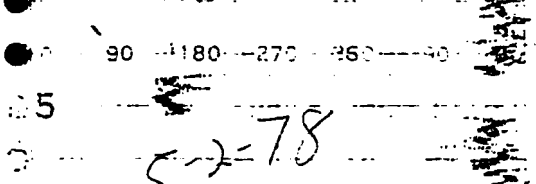
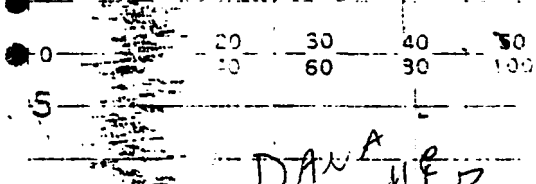
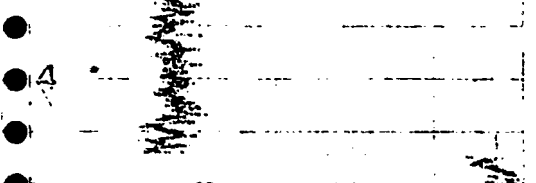
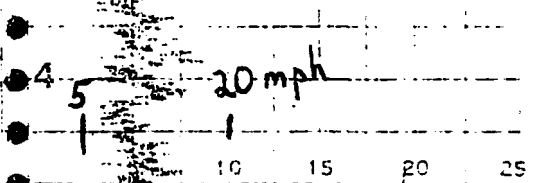
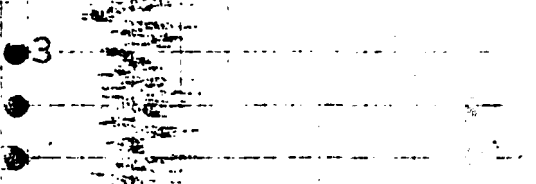
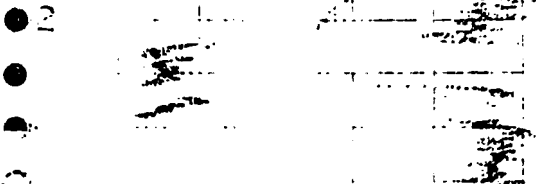
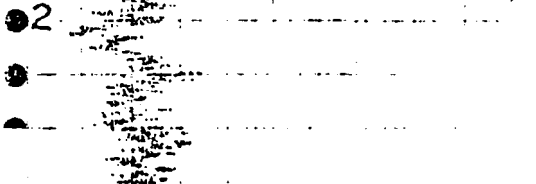
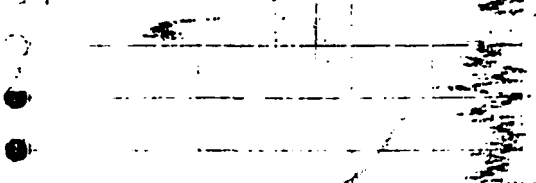
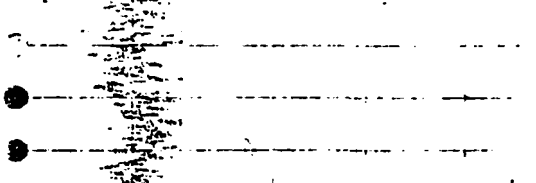
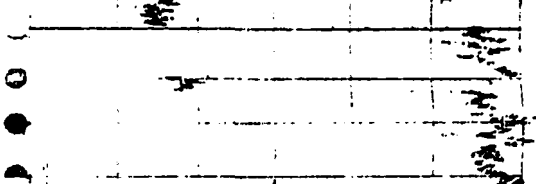
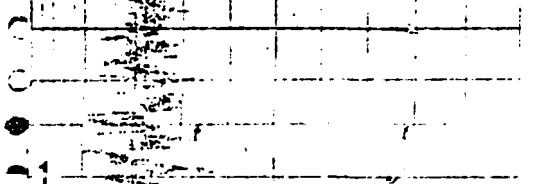
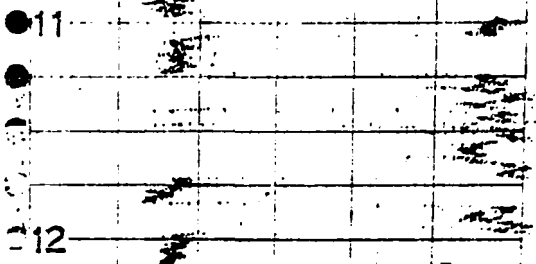
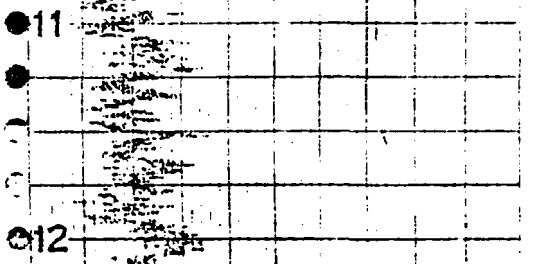
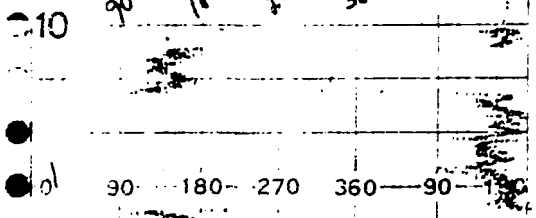
Q

180° = N

5 10 20 mph



90 180 270 360 90 180



DANA
PUMP SITE B

Dana C. B. Ien

6:27 PM

5-2-78

Shot down

6:27 PM

6:27 PM

5-10-20 mph

4 in 20 mph

5	10	15	20	25
10	20	30	40	50
20	30	60	80	100

52

6

5/3/78 (C) stored 12:00AM
Rest w/ 5/3/78 (C) 12:26AM

-12

360 90

● 五

70 180 370

4

55

5:00 AM
5-4-78
N = 148°

of

APPENDIX C
TEST PROCEDURES

METHODS OF AIR SAMPLING AND ANALYSIS

Intersociety Committee

American Conference of Governmental Industrial Hygienists

American Chemical Society

American Industrial Hygiene Association

Association of Official Analytical Chemists

Air Pollution Control Association

American Public Health Association

American Public Works Association

American Society of Civil Engineers

American Society of Mechanical Engineers

American Society for Testing and Materials



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TENTATIVE METHOD OF ANALYSIS FOR FORMALDEHYDE CONTENT OF THE ATMOSPHERE (MBTH— COLORIMETRIC METHOD—APPLICATIONS TO OTHER ALDEHYDES)

43502-02-70T

1. Principle of Method

1.1 The aldehydes in ambient air are collected in a 0.05 per cent aqueous 3-methyl-2-benzothiazolone hydrazone hydrochloride (MBTH) solution. The resulting azine is then oxidized by a ferric chloride-sulfamic acid solution to form a blue cationic dye in acid media, which can be measured at 628 nm (1,2,3).

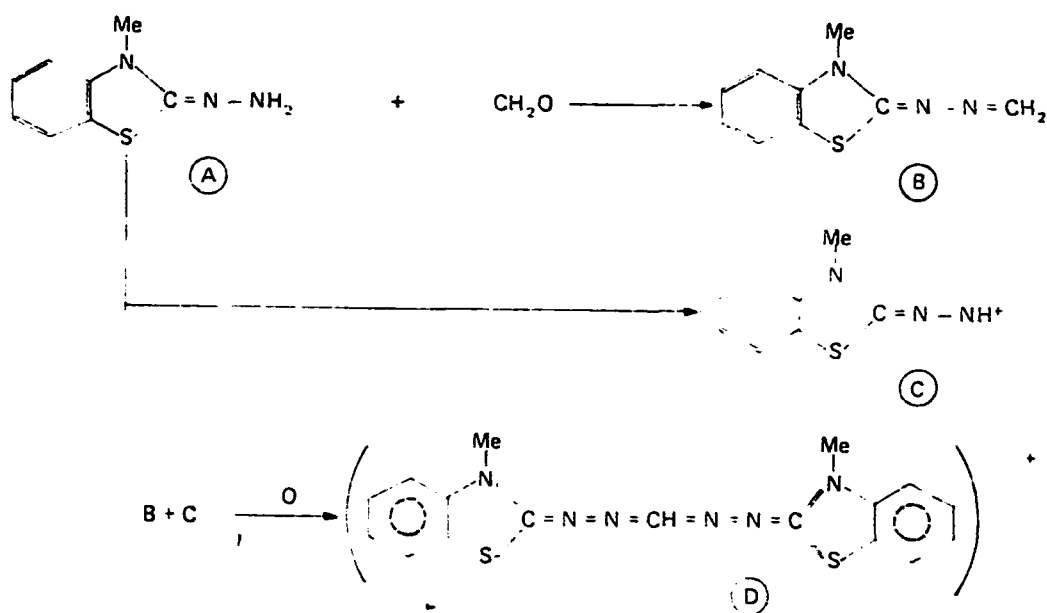
1.2 The mechanism of the present procedure as applied to formaldehyde includes the following steps: reaction of the aldehyde with 3-methyl-2-benzothiazolone hydrazone, A, to form the azine, B; oxidation of A to a reactive cation, C; and formation of the blue cation, D (1).

2. Range and Sensitivity

2.1 From 0.03 $\mu\text{g/ml}$ –0.7 $\mu\text{g/ml}$ of formaldehyde can be measured in the color developed solution (12 ml). A concentration of 0.03 ppm of aldehyde (as formaldehyde) can be determined in a 25 l air sample based on an aliquot of 10 ml from 35 ml of absorbing solution and a difference of 0.05 absorbance unit from the blank.

3. Interferences

3.1 The following classes of compounds react with MBTH to produce colored products. These are aromatic amines, imino heterocyclics, carbazoles, azo dyes, stilbenes, Schiff bases, the ali-



phatic aldehyde 2,4-dinitrophenyl hydrazones, 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 water soluble aliphatic aldehydes in the atmosphere (3).

4. Precision and Accuracy

The method was checked for reproducibility by having three different analysts in three different laboratories analyze standard formaldehyde samples. The results listed in Table 1 agreed within ± 5 per cent.

5. Apparatus

5.1 *Absorbers*—(All glass samplers with coarse fritted tube inlet. Figure 1 shows an acceptable absorber.)

5.2 *Air metering device*—Either a limiting orifice of approximately 0.5 lpm capacity or a wet test meter can be used. If a limiting orifice is used, regular and frequent calibration is required.

5.3 *Air pump*—A pump capable of drawing at least 0.5 l of air/min for 24 hr through the sampling train is required.

5.4 *Spectrophotometer*—An instrument capable of measuring accurately the developed color at the narrow absorption band of 628 nm.

6. Reagents

6.1 *Purity of chemicals*—All reagents must be analytical reagent grade.

6.2 *3-Methyl-2-benzothiazolone hydrazone hydrochloride absorbing solution (0.05 per cent)*—Dissolve 0.5 g of MBTH in distilled water and dilute to 1 liter. This colorless solution is filtered by gravity, if slightly turbid, and is stable for at least 1 week after which it becomes pale yellow. Stability may be increased by storing in a dark bottle in the cold.

6.3 *Oxidizing reagent*—Dissolve 1.6 g of sulfamic acid and 1.0 g of ferric

Table 1. Comparison of Formaldehyde Results from Three Laboratories
(Analysis of Standard Formaldehyde Samples)

Micrograms/ml Formaldehyde	Absorbance		
	Laboratory 1	Laboratory 2	Laboratory 3
0.05	0.078	0.077	0.082
0.10	0.151	0.156	0.146
0.30	0.430	0.457	0.445
0.50	0.720	0.700	0.728
0.70	0.990	1.04	1.02

chloride in distilled water and dilute to 100 ml.

6.4 *Formaldehyde standard solution "A" (1 mg./ml)*—Dilute 2.7 ml of 37 per cent formalin solution to 1 liter with distilled water. This solution must be standardized as described in "Calibration" section. This solution is stable for at least a 3-month period.

6.5 *Formaldehyde standard solution "B" (10 μ g. ml.)*—Dilute 1 ml of standard solution "A" to 100 ml with 0.05 per cent MBTH solution. Make up fresh daily.

6.6 *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.

6.7 *Iodine 0.01 N*—Dilute 100 ml of the 0.1 N iodine solution to 1 liter. Standardize against sodium thiosulfate.

6.8 *Starch solution 1 per cent*—Make a paste of 1 g of soluble starch in 2 ml of water and slowly add the paste to 100 ml of boiling water. Cool, add several mls of chloroform as a preservative, and store in a stoppered bottle. Discard when a mold growth is noticeable.

6.9 *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 and dilute to 1 liter.

6.10 *Sodium bisulfite 1 per cent*—Dissolve 1 g of sodium bisulfite in 100 ml

FORMALDEHYDE

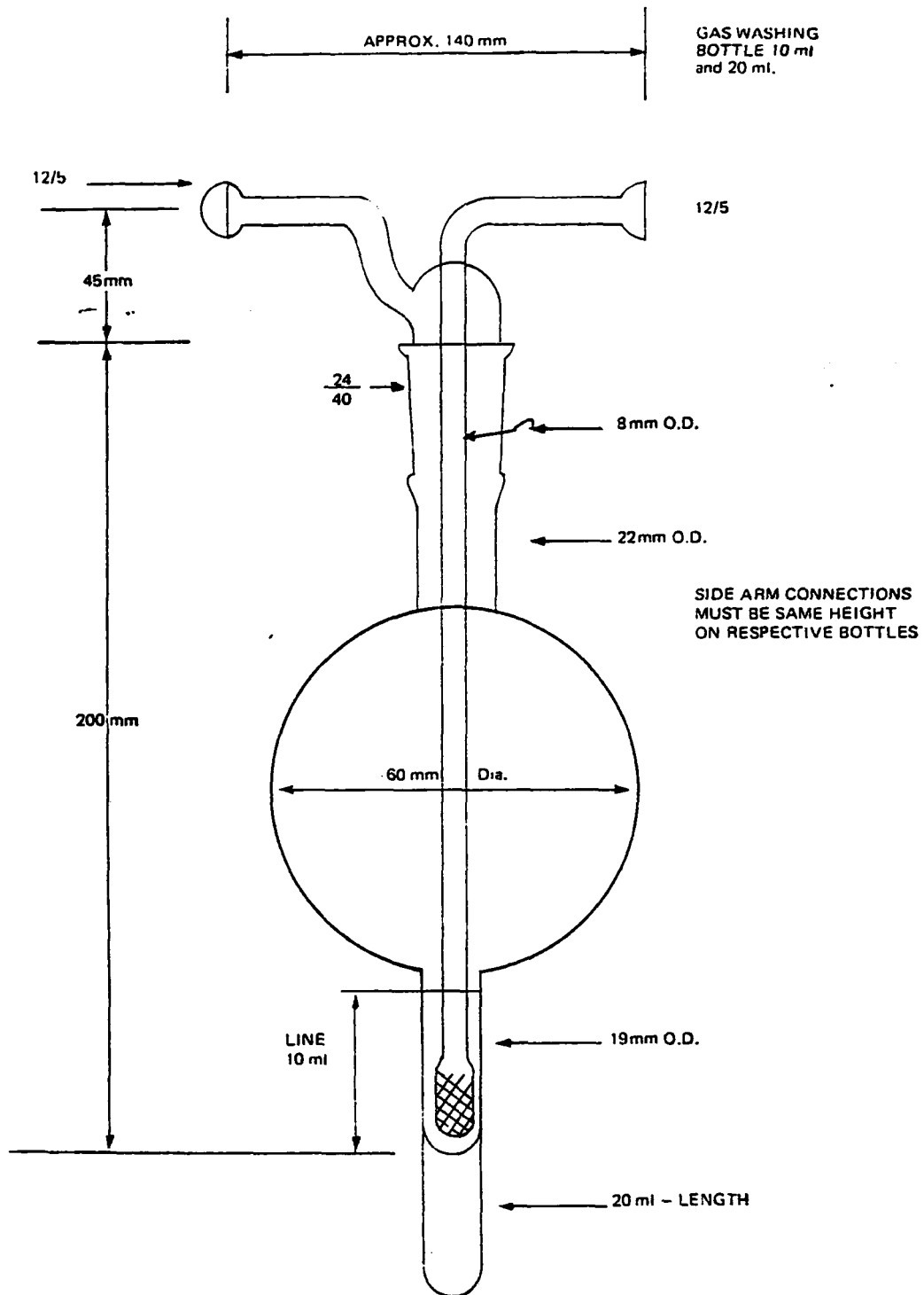


Figure 1—Absorber.

of water. It is best to prepare a fresh solution weekly.

7. Procedure

7.1 *Air sampling*—Draw measured volumes of the vapor laden air at a rate of 0.5 lpm for 24 hr through 35 ml of MBTH absorbing solution contained in the absorber. A shorter sampling time can be used providing enough formaldehyde is collected to be above the lower limit of sensitivity of the method.

The average collection efficiency of formaldehyde in air has been determined to be 84 per cent when air was sampled at a rate of 0.5 lpm over a 24-hr period in 35 ml of collecting reagent (3) in an absorber equipped with an extra coarse (EC) fritted tube inlet. Absorption efficiency may be improved by using a coarse (C) frit although data are lacking on this likelihood.

7.2 Analysis.

7.2.1 Transfer the samples from the sampling bottles to 50 ml graduates, dilute to 35 ml with distilled water and allow to stand for 1 hr.

7.2.2 Pipet a 10 ml aliquot of the sampling solution into a glass stoppered test tube. A blank containing 10 ml of MBTH solution must also be run. If the aldehyde content of the aliquot exceeds the limits of the method, a smaller aliquot diluted to 10 ml with MBTH solution is used.

7.2.3 Add 2 ml of oxidizing solution and mix thoroughly.

7.2.4 After standing for at least 12 min, read at 628 nm on a suitable spectrophotometer using a 1 cm cell. No significant change in absorbance was noted over a 3 hr period after color development. Determine the aldehyde content of the sampling solution from a curve previously prepared from the

standard formaldehyde solution. This will give total aldehyde calculated as formaldehyde.¹

8. Calibration

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

8.2 Add 10 ml of 1 per cent sodium bisulfite and 1 ml of 1 per cent starch solution.

8.3 Titrate with 0.1 *N* iodine to a dark blue color.

8.4 Destroy the excess iodine with 0.05 *N* sodium thiosulfate.

8.5 Add 0.01 *N* iodine until a faint blue end point is reached.

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

8.7 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.

8.8 One ml of 0.0100 *N* iodine is equivalent to 0.15 mg of formaldehyde. Therefore, since 1 ml of formaldehyde standard solution was titrated, the milliliters of 0.01 *N* iodine used in the final titration multiplied by 0.15 mg gives the formaldehyde concentration of the standard solution in mg/ml.

¹ *Note:* The final colored solution tends to form bubbles that cling to the sides of the cuvettes. In order to eliminate this, the solution should be thoroughly shaken periodically during the 12 min standing time waiting for full color development. It has been found that this thorough shaking will eliminate bubble formation.

8.9 Preparation of standard curve.

8.9.1. 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 per cent MBTH solution. These solutions contain 0, 0.05, 0.1, 0.3, 0.5, and 0.7 μg of formaldehyde/ml.

8.9.2. After final dilution let stand for 1-hour.

8.9.3. Transfer 10 ml of each solution to a glass stoppered test tube and add 2 ml of oxidizing reagent and mix.

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

8.9.5. Plot absorbance against micrograms of formaldehyde/ml of solution.

9. Calculation

9.1 The concentration of total aliphatic aldehyde (as formaldehyde) in the sampled atmosphere may be calculated by using the following equation:

$$\text{PPM (Vol.)} = \frac{C \times 35 \times 24.45}{V \times \text{M.W.} \times E}$$

E=correction factor for sampling efficiency (0.84 may be used if absorber contains an EC frit)

V=liters of air sampled.

C= μg /ml of formaldehyde in sampling solution. (Since each sample is diluted to 35 ml, this figure must be multiplied by 35 to give total micrograms in sampling solution.)

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

24.45=ml of formaldehyde gas in one millimole at 760 Torr and 25 C.

10. Effect of Storage

10.1 The time study of the reaction of microgram quantities of formaldehyde with 0.05 per cent MBTH shows that the reaction is complete in approximately 45

min; therefore, a reaction time of 1 hr is selected for this procedure. Formaldehyde is fairly stable in 0.05 per cent MBTH since only approximately 5 per cent of the formaldehyde is lost after standing in the MBTH for 13 days. The samples are, therefore, stable enough for later analysis (3).

11. References

1. Sawicki, E.; T. R. Hauser; T. W. Stanley; and W. Elbert. The 3-Methyl-2-Benzothiazolone Hydrazone Test. *Anal. Chem.* 33:93, 1961.
2. Hauser, T. R., and R. L. Cummins. Increasing the Sensitivity of 3-Methyl-2-Benzothiazolone Hydrazone Test for Analysis of Aliphatic Aldehydes in Air. *Anal. Chem.* 37:679, 1964.
3. Hauser, Thomas R. Determination of Aliphatic Aldehydes: 3-Methyl-2-Benzothiazolone Hydrazone Hydrochloride (MBTH) Method. *Selected Methods for the Measurement of Air Pollutants*. Public Health Service Publication No. 999-AP-11, Page F-1, 1965.

ADDENDUM

Applications to Other Aldehydes

Acetaldehyde and propionic aldehyde both yield a blue dye after reaction with 3-methyl-2-benzothiazolone hydrazone hydrochloride and a ferric chloride-sulfamic acid solution. It has been found that as the length of chain increases, the sensitivity decreases. Therefore when measuring total aldehydes as formaldehyde this method would give low results if any aldehyde other than formaldehyde is present.

From 0.05 μg /ml-1.0 μg /ml of both acetaldehyde and propionic aldehyde can be measured in the color developed solution (12 ml). For the lower concentrations the method has poor reproducibility. However, at higher concentrations (0.30 μg /ml and above) reproducibility was very good. These data are summarized in Tables A and B.

Acetaldehyde (Eastman Kodak Company, Cat. No. 468) and propionic aldehyde (Eastman Kodak Company, Cat. No. 653) were considered to be primary standards when preparing solutions of known concentration. Exactly 1.28 ml of acetaldehyde was diluted to 1 l with

Table A. Acetaldehyde

$\mu\text{g./ml}$	Number of Samples	Average Absorbance	Range	% Variance From Avg.
0.05	29	0.063	0.050-0.074	± 20
0.10	29	0.125	0.106-0.144	± 15
0.30	29	0.339	0.316-0.355	± 7
0.50	29	0.519	0.495-0.538	± 4
0.70	29	0.685	0.660-0.710	± 3
1.00	15	0.900	0.890-0.910	± 1

Table B. Propionic Aldehyde

$\mu\text{g./ml}$	Number of Samples	Average Absorbance	Range	% Variance From Avg.
0.05	29	0.046	0.032-0.057	± 27
0.10	29	0.082	0.063-0.095	± 20
0.30	29	0.243	0.225-0.250	± 5
0.50	29	0.399	0.380-0.422	± 5
0.70	29	0.538	0.515-0.568	± 5
1.00	15	0.732	0.710-0.750	± 2

distilled water and then 1 ml of this solution was diluted to 100 ml with MBTH solution giving a final concentration of 10 $\mu\text{g./ml}$. Exactly 1.24 ml of propionic aldehyde was diluted to 1 l with distilled water and then 1 ml of this solution was diluted to 100 ml with MBTH solution giving a final concentration of 10 $\mu\text{g./ml}$. The strong standard solutions have a 2 month shelf life. The dilute standard solutions must be prepared fresh daily.

A series of 34 ambient air samples were collected in 35 ml of MBTH solu-

tion contained in each of two absorbers in series. The sampling time was 24 hr and the sampling rate was 1 liter/minute. Collection efficiencies varied from 69 per cent to 100 per cent with the average for the 34 samples being 82 per cent.

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and the absorbance measured directly at 510 nm.

8. Standardization of Phenol Solution

8.1 *Stock standard phenol solution*—prepare a 0.1 per cent solution of phenol in distilled water. Into a 500 ml iodine flask transfer 50 ml of stock standard and add 100 ml of distilled water. Add exactly 10 ml of bromide-bromate solution. Add carefully 0.5 ml of concentrated hydrochloric acid. Swirl the flask gently, making certain that the stopper is seated. If, at this point, the color of bromine does not persist, continue to add exactly 10 ml portions of bromide-bromate solution until the reddish-brown bromine color does persist. If the stock solution is made up to contain 1000 mg of phenol/l, 4–10 ml portions of bromide-bromate solution will be required. With the stopper in position let the reaction flask sit for 10 minutes. Add quickly one g of potassium iodide. Prepare a blank in exactly the same manner, using 10 ml of bromide-bromate solution and distilled water. Titrate both blank and sample with 0.025 N sodium thiosulfate, using starch as indicator. Calculate the concentration of phenol solution as follows:

$$\text{Milligrams of phenol per liter} = [(A \times B) - C] \times 7.835$$

A=ml of 0.025 N thiosulfate used for blank.

B=ml of bromide-bromate solution used for sample, divided by 10.

C=ml of 0.025 N thiosulfate used for sample. The factor, 7.835, is based on the use of an exactly 0.025 N thiosulfate solution in the titration.

8.2 *Primary standard phenol solution*—Dilute the stock standard so that 1 ml is equal to 10 µg of phenol.

8.3 *Working standard*—Dilute the primary standard 1:10 with distilled water. This solution contains one µg of phenol in 1 ml.

9. Calculation

9.1 Working standards are used to prepare a concentration vs absorbance curve from which the concentration of phenol in samples is determined. One µg of phenol/l of air is equal to 0.26 ppm and one ppm is equal to 3.84 µg/l at 25 C and standard pressure.

10. Effects of Storage

10.1 The addition of 5 ml of copper sulfate solution to the alkaline solution of phenols will serve to stabilize the sample.

10.2 Cautions.

10.2.1 Equipment which has been lubricated with stopcock grease should not be used.

10.2.2 Temperature variations will affect the blank.

10.2.3 Filtration of the chloroform extract before reading it in the spectrophotometer will remove possible turbidity due to presence of water dispersion.

10.2.4 The chloroform extract of the dye will fade on standing.

10.2.5 It is advisable to work quickly when serial readings are made.

11. References

1. Emerson, E. J. The Condensation of Aminoantipyrene: A New Test for Phenolic Compounds. *J. Organic Chem.*, 8:417, 1943.
2. Ettinger, M. D.; C. C. Ruchhoft; and R. J. Lishka. Sensitive 4-Aminoantipyrene Method for Phenolic Compounds. *Anal. Chem.*, 23:1783-1788, 1951.
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4. Mohler, E. F. and L. N. Jacob. Determination of Phenolic-Type Compounds in Water and Industrial Waste Waters. *Anal. Chem.*, 29:1369-1374, 1957.
5. Smith, R. G., J. D. MacEwen; and R. E. Barrow. Sampling and Analysis of Phenols in Air. *Amer. Indust. Hyg. Assoc. Jour.*, April, 112-118, 1959.

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pare according to classical laboratory method.

7. Procedure

7.1 Air Sampling—Particulates. Draw a 24-hr air sample at a measured flow rate through a flash-fired fiberglass filter using a high volume sampler.¹

7.2 Air Sampling. Vapors and Particulates. Wet method—Draw a 30 min sample (or larger if desired) of air through a 0.1 *N* solution of sodium hydroxide in distilled water, at a standard impinger flow rate of one ft³/minute. If only vapor phenolics are required, use a membrane filter in the sampling train to remove particulates.

7.3 Analysis—(CAUTION—do not use stopcock grease in any apparatus).

7.4 Filter samples—Extract the filter or any desired portion of it in a Soxhlet extractor by refluxing with benzene for 3 hours. Transfer the benzene extract to a separatory funnel, filtering it through a close (Whatman 42 or equivalent) paper. Extract 3 times with 10 ml portions of 1.0 *N* NaOH. Treat according to Section 7.6 *Determination of Phenols*.

7.5 Samples collected in 0.1 *N* NaOH.

7.5.1 Air samples. Proceed to Section 7.6 *Determination of Phenols*.

7.5.2 Exhaust gases or process effluents. Use the whole sample. Add 1 ml of 10 per cent copper sulfate solution. Acidify, using methyl orange as indicator and 10 per cent phosphoric acid solution. Transfer to an all glass distillation apparatus and distill, collecting 90 ml of the distillate. Cool the distillation flask and add 10 ml of distilled water. Continue the distillation until exactly 100 ml of distillate has been col-

lected. Acidify with 0.5 ml of 10 per cent phosphoric acid solution, add 1.0 ml of 10 per cent copper sulfate solution and transfer to a separatory funnel. Add 30 g of reagent grade sodium chloride and extract with 3–10 ml portions of chloroform. Discard the aqueous phase. Shake the chloroform extract with 2–15 ml portions of 0.1 *N* NaOH. Discard the chloroform phase. Heat the alkali extracts until the traces of chloroform have been removed, dilute the alkaline extract to 100 ml volume with distilled water and treat according to Section 7.6.2.

7.6 Determination of Phenols.

7.6.1 Adjust the alkaline extracts to volume of 100 ml, either by aliquoting or diluting to volume with distilled water. Add 1 ml of 10 per cent copper sulfate solution. Acidify with 10 per cent phosphoric acid solution using methyl orange indicator. Distill from an all glass distillation apparatus until 90 ml have been collected. Add 10 ml of distilled water to the cooled distillation flask and continue the distillation until a total volume of 100 ml of distillate have been collected.

7.6.2 Take a 50 ml aliquot of the distillate. Prepare standards containing 0.5, 1.0, 5.0, 10.0 and 20.0 μg of phenol. Adjust the volumes of sample and standards to 100 ml with distilled water. Add 2 ml of ammonium chloride solution. Using a pH meter adjust to pH 10.0 ± 0.2 , with concentrated ammonium hydroxide. Add 1 ml of 4-aminoantipyrine solution and mix. Add 1 ml of potassium ferricyanide solution, transfer to a separatory funnel and wait 3 minutes. Extract with 3–5 ml portions of chloroform and discard the aqueous phase. Make up the chloroform extract to a known volume with chloroform. Using a blank as reference, record the absorbance at 460 nm. For higher concentrations of phenol the chloroform extraction may be omitted

¹ Proper methods for calibrating the high volume sampler or other sampling devices should be provided by the manual supplied by the manufacturer.

2. Range and Sensitivity

2.1 One cubic meter of air containing 1.3 ppb of phenol will produce sufficient sample to give a coupling product absorbance of approximately 0.2 units in a 20 mm cuvet when measured at 460 nm wavelength in a spectrophotometer.

3. Interferences

3.1 Any color, other than that due to the reagents used, interferes with the method. Turbidity, sulfur compounds and certain metallic ions interfere (4). However, the distillation procedure described by Smith eliminates these interferences.

4. Precision and Accuracy

4.1 In the range of 0.49–1.93 ppm the standard deviation is 0.022 and at the 95% confidence level 0.065. At 18–75 ppb the standard deviation is 3.3 and at the 95% confidence level 10.7 ppb. Accuracy is plus or minus two per cent (4).

5. Apparatus

- 5.1 Soxhlet extractors.
- 5.2 Distillation apparatus—all glass.
- 5.3 Iodine bottles—500 ml size.
- 5.4 Impingers—Standard, midget or equipped with fritted absorbers (extra coarse porosity).
- 5.5 Fiber glass filter sheets, flash fired.
- 5.6 Spectrophotometer—any spectrophotometer capable of measuring the absorbance of the solution complex at 460–510 nm, as required.
- 5.7 High volume sampler or other air sampling device for collection of particulate samples, equipped with a calibrated gauge or flow meter to measure air volume flow accurately.

6. Reagents

6.1 Purity of chemicals. All reagents should be ACS analytical grade.

6.2 *4-aminoantipyrine solution*—dissolve 2 g of 4-aminoantipyrine in distilled water and make up to 100 ml. This solution should not be kept longer than 1 week.

6.3 *Potassium ferricyanide solution*—dissolve 8 g of analytical reagent grade potassium ferricyanide in distilled water and make up to 100 ml. Discard when the solution becomes darkened.

6.4 *Ammonium chloride solution*—dissolve 50 g of analytical reagent grade salt in distilled water and make up to one liter.

6.5 *Copper sulfate solution*—prepare a 10 per cent solution of the pentahydrate.

6.6 *Sodium hydroxide sampling solution*—prepare a 1 N solution.

6.7 *Bromide/Bromate solution*—dissolve 2.784 g of analytical reagent grade potassium bromate in distilled water; add 10 g of analytical reagent grade potassium bromide and make up to 1 liter.

6.8 *Ammonium Hydroxide*—Analytical reagent grade.

6.9 *Hydrochloric Acid*—Analytical reagent grade.

6.10 *Phosphoric acid solution*—prepare a 10 per cent solution of orthophosphoric acid.

6.11 *Potassium Iodide*—Analytical reagent grade salt.

6.12 *Sodium thiosulfate solution*—prepare a 0.1 N solution of the salt and standardize according to classical laboratory procedures. Dilute to make an exactly 0.025 N solution.

6.13 *Starch solution*—dissolve one g of soluble starch in 100 ml of distilled water. Prepare a fresh solution daily.

6.14 *Phenol*—reagent grade.

6.15 *Benzene*—reagent grade.

6.16 *Chloroform*—reagent grade.

6.17 *Methyl orange indicator*—pre-

INTERSOCIETY COMMITTEE

116

TENTATIVE METHOD OF ANALYSIS FOR DETERMINATION OF PHENOLIC COMPOUNDS IN THE ATMOSPHERE (4-AMINOANTIPYRINE METHOD)

17320-01-70T

1. Principle of the Method

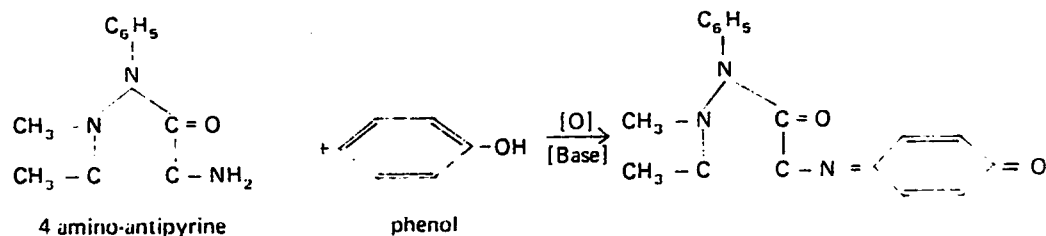
1.1 Air is scrubbed with an alkaline solution in a standard impinger. Particulate phenolic substances are collected by passing air through a fiberglass filter. Phenolic compounds are separated from other compounds by distillation from an acidified system. Phenols are determined by coupling them with 4-aminoantipyrine in an alkaline medium containing an oxidant.

1.2 The method is based on a reaction discovered by Emerson (1). This procedure is essentially that of Smith

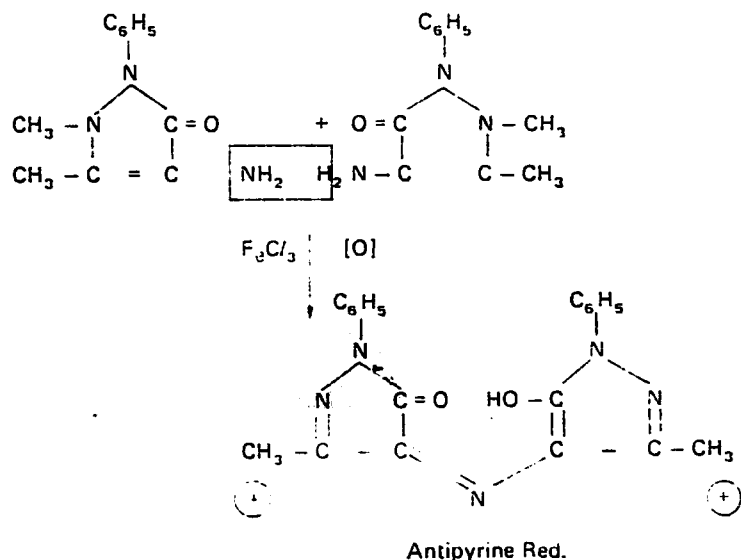
et al (5). Discussions of theory and efficiency are given by Ettinger (2) and Mohler (4).

1.3 In the presence of a strong alkaline oxidizing reagent this coupling reaction will proceed as shown in 1.4 below. If the system is not sufficiently alkaline dimerization of 4-aminoantipyrine to antipyrine red will take place, as in 1.5 below. It is important, therefore, to have a high pH when the coupling reaction is induced.

1.4 Coupling reaction of 4-aminoantipyrine and phenol



1.5 Dimerization of 4-aminoantipyrine



criteria for a recommended standard....

OCCUPATIONAL EXPOSURE TO FIBROUS GLASS



U.S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
Public Health Service
Center for Disease Control
National Institute for Occupational Safety and Health

April 1977

C-12

IX. APPENDIX I

AIR SAMPLING METHOD - MEMBRANE FILTER

General Requirements

The following sampling and analytical methods for fiber counting are adapted from the NIOSH membrane filter method for evaluating airborne asbestos fibers [90].

(a) Air samples representative of the breathing zones of workers must be collected to characterize the exposure from each job or specific operation in each work area.

(b) Samples collected shall be representative of the exposure of individual workers.

(c) Suggested records:

- (1) The date and time of sample collection.
- (2) Sampling duration.
- (3) Total sample volume.
- (4) Location of sampling.
- (6) Other pertinent information.

Sampling

(a) Samples shall be collected so as to be representative of the breathing zones of workers without interfering with their freedom of movement.

(b) Samples shall be collected to permit determination of TWA exposures for every job involving exposure to fibrous glass in sufficient

numbers to determine the variability of exposures in the work situation.

(c) Equipment

The sampling train consists of a membrane filter and a vacuum pump.

(1) Membrane filter: Samples of fibrous glass are collected in the breathing zones of the workers using a personal sampler with cellulose ester membrane filter. The filter is a 0.8- μ m pore size mixed cellulose ester membrane mounted in a open-face sampling cassette which can be attached to the worker near his or her breathing zone.

(2) Pump: A battery-operated pump, complete with clip for attachment to the worker's belt, capable of operation at 2.5 liters/minute or less.

(d) Calibration

The personal sampling pump should be recharged prior to calibration and then calibrated against a bubble meter, wet test meter, spirometer, or similar device at a flowrate of 1.0 to 2.5 liters/minute. The sampling train used in the calibration (pump, hose, filter) shall be equivalent to the one used in the field. The calibration should be performed to an accuracy of $\pm 5\%$.

(e) Sampling Procedure

(1) Sampling is performed using an open-face membrane filter cassette.

(2) The sampler shall be operated at a flowrate between 1.5 and 2 liters/minute.

(3) The temperature and pressure of the atmosphere being sampled are measured and recorded.

(4) One membrane filter is treated in the same manner as the sample filters with the exception that no air is drawn through it. This filter serves as a blank.

(5) Immediately after sampling, personal filter samples should be sealed in individual plastic filter holders for shipment. The filters shall not be loaded to the point where portions of the sample might be dislodged from the collecting filter during handling.

(f) Optimum Sampling Times

A requirement for a minimum count of 100 fibers or 20 fields has been determined to be the optimum choice to achieve low variability of the fiber count (as approximated by a Poisson distribution) and reduced counting times. In other words, the optimum fiber density on the filter should be 1 to 5 fibers/microscope counting field. To estimate optimum sampling times, the approximate field area of the counting scope and the pump flowrate must be known in advance.

The following equation is used to calculate the range of optimum sampling times which can then be plotted on log-log paper:

$$\text{Minutes} = \frac{(\text{FB/FL})(\text{ECA/MFA})}{(\text{FR})(\text{AC})}$$

where: FB/FL = 1 to 5 fibers/field

ECA = Effective collecting area of filter in square millimeters (855 square mm for 37-mm filter)

MFA = Microscope field area in mm (generally 0.003 to 0.006 square mm)

FR = Pump flowrate in cc/minute

AC = Air concentration of fibers in fibers/cc

(NOTE: If air concentrations are expressed
in fibers/cu m they must be changed
to fibers/cc for this equation.)

X. APPENDIX II
ANALYTICAL METHOD - FIBER COUNT

Principle of the Method

- (a) Environmental dust samples are collected by drawing air through a membrane filter by means of a battery-powered personal sampling pump.
- (b) The filter is transformed from an opaque solid membrane to a transparent, optically homogeneous gel.
- (c) The fibers are sized and counted by phase-contrast microscopy at 400-450X magnification.

Range and Sensitivity

- (a) This method has been successfully applied at concentrations of 10,000 to 20,000,000 fibers/cu m (0.01 to 20 fibers/cc) for fibers longer than 5 μ m. Large deviations from the specified conditions of the method may result in filters with either too few or too many fibers. Too few fibers will yield air concentration estimates of low statistical precision.
- (b) A sensitivity of 10,000 fibers/cu m (0.01 fiber/cc) has been reported [JM Dement, written communication, 1975] based on a 4-hour sample at 2 liters/minute air flow.

Interferences

All particulates, such as asbestos or mineral wool, with a length-to-width ratio of 3 to 1 or greater, and length greater than 10 μ m should, in

the absence of other information, be considered as glass fibers and counted as such. Asbestos interference can be eliminated using phase contrast, polarized light microscopy.

Advantages of the Method

(a) The fiber count method allows for repeated counts, and storage for counting at a later time. The method consumes only part of the filter, thereby allowing for at least one replicate sample analysis at a later time.

(b) Fiber counts are assumed to be more toxicologically significant than fiber weight for fibers less than 3.5 μm in diameter.

(c) Fiber size determinations may be performed.

Disadvantages of the Method

(a) The fiber count method is slow and tedious.

(b) Variation in counts may be significant between different observers.

(c) The sensitivity of the method is dependent on the sampling time and flowrate. The sensitivity and useful range of this method has not been determined specifically for fibrous glass but is based on the method recommended for asbestos.

Apparatus

(a) Optical Equipment

(1) Microscope body with binocular head, 10X Huygenian

eyepieces, and Koehler illumination.

(2) Porton reticle.

(3) Mechanical stage, and stage micrometer with 0.01-mm subdivisions.

(4) Abbe or Zernike condenser fitted with phase ring with a numerical aperture equal to or greater than the numerical aperture of the objective.

(5) A phase-ring centering telescope or Bertrand lens and a green filter if recommended by the microscope manufacturer.

(6) Fiber mounting equipment

(A) Microscope slides, and cover slips, usually 0.17 mm thick.

(B) Scalpel, tweezers, lens tissues, and glass rod or spatula for mounting procedures.

(b) Wheaton Balsam Bottle.

Reagents

(a) Dimethyl phthalate.

(b) Diethyl oxalate.

Analysis of Samples

(a) Calibration and Standardization

(1) Porton Reticle and the Counting Field

The fiber count procedure consists of comparing fiber length with calibrated circles, and counting all fibers $> 10 \mu\text{m}$ in length within a

given counting field. A Porton reticle is used for this purpose. The Porton reticle is a glass plate inscribed with a series of circles and rectangles. The square on the left, divided into six rectangles, is defined as the counting field.

(2) Placement in Eyepiece

Place the Porton reticle inside one Huygenian eyepiece, resting it on the field-limiting diaphragm. Keep the reticle clean, since dirt on the reticle will be in focus and will complicate the counting and sizing process.

(3) Stage Micrometer

The Porton reticle cannot be used for counting until it has been properly calibrated with a stage micrometer. Most stage micrometer scales are approximately 2 mm long, divided into units of 10 μm .

(4) Microscope Adjustment

When adjusting the microscope, follow the manufacturer's instructions while observing the following guidelines.

(A) The light source image must be in focus and centered on the condenser iris or annular diaphragm.

(B) The object for examination must be in focus.

(C) The illuminator field iris must be in focus, centered on the sample, and opened only to the point where the field of view is illuminated.

(D) The phase rings (annular diaphragm and phase-shifting elements) must be concentric.

(5) Porton Reticle Calibration Procedure

Each eyepiece-objective-reticle combination on the microscope must be calibrated. Should any of the three be changed (disassembly, replacement, zoom adjustment, etc) the combination must be recalibrated. Calibration may change if the interpupillary distance is changed. For proper calibration, the following procedure should be followed closely.

Using a 10X objective, place the stage micrometer on the mechanical stage and focus and center the image. Change to the 40-45X objective and adjust the first scale division to coincide with the left boundary of the Porton rectangle. Count the number of divisions between the left and right boundaries of the long horizontal dimension of the largest rectangle, estimating any portion of the final division. This measurement represents 200 L units and the measurement is then divided by 200 to find "L." The large rectangle is 100 L units long on the short vertical dimension. The calculated "L" is inserted into the formula $D = L(2N)^{1/2}$ where "N" is the circle number (indicated on the reticle) and "D" is the circle diameter. Since the circle diameters vary logarithmically, every other circle doubles in diameter. For example, number three is twice the diameter of number one; number four is twice the counting field area consisting of the left six smaller rectangles can be calculated from the relation 10,000 L. The reticle calibration is now completed for this specific objective-eyepiece-reticle combination.

(b) Preparation of Mounting Solution

An important part of the sample evaluation is the mounting process which involves a special mounting medium of prescribed viscosity. The proper viscosity is important to expedite filter clearing and to minimize particle migration. Once the sample has been mounted, an elapsed time of

approximately 15 minutes is needed before the sample is ready for evaluation.

Combine the dimethyl phthalate and diethyl oxalate in a 1 to 1 ratio by volume and pour the solution into a Wheaton balsam bottle. Add 0.05 gram of new membrane filter/ml of solution to reach the necessary viscosity. The mixture must be stirred periodically until the filter material is dissolved and a homogeneous mixture is formed. The normal shelf life of the mounting solution is about 6 months. Approximately 300 samples can be prepared from 20 ml of mounting solution.

(c) Sample Mounting

Cleanliness is important. The working area must be kept clean to prevent sample contamination and erroneous counts. The following steps should be followed when mounting a sample.

(1) Clean the slides and cover slips with lens tissue. Lay the slide down on a clean surface with the frosted end up. It is good practice to rest one edge of the cover slip on the slide and the other edge on the working surface. By doing this, you keep from becoming contaminated.

(2) Wipe all the mounting tools clean with lens tissue and place them on a clean surface (such as lens tissue). When mounting a series of filters, wipe the scalpel clean before cutting a sector of each sample [see (5) below].

(3) Apply a small drop of mounting solution onto the center of the slide with a glass rod. It may be necessary to adjust the quantity of solution used or the size of the wedge. The correct amount will result in the solution extending only slightly beyond the filter boundary. If the

quantity is greater than this, adverse particle migration may occur.

(4) With a spatula or a supplemental glass rod, spread the mounting media into a triangular shape. The size of this triangle should coincide with the dimension of the filter wedge.

(5) Separate the middle and bottom sections of the field monitor case to expose the fragile filter. Cut a triangular wedge from the center to the edge of the filter using a scalpel. The size of the wedge should approximate one-eighth of the filter surface. The filter should be handled gently so that no material will be lost.

Grasp the filter wedge with tweezers on the outer area of the filter which was clamped between the monitor case sections. Do not touch the filter with fingers. Place the wedge, fiber-bearing side up, upon the mounting medium.

(7) Lift the cover slip with the tweezers and carefully place it on the filter wedge. Once this contact has been made, do not reposition the cover slip.

(8) Label the slide with the sample number and current date before proceeding to the next filter.

(9) The sample should become transparent after about 15 minutes. If the filter appears cloudy, it may be necessary to press very lightly on the cover slip. This is rarely necessary, however.

(10) Examine the slide within 3 days. The sample mount. should be discarded after 3 days if it has not been counted because crystals which appear similar to glass fibers may begin to grow at the mounting media/air interfaces; they seldom present any problems if the slide is examined within 3 days. In any case, do not perform counting or

sizing around the edges of the filter.

(d) Counting and Sizing--Finding and Inspecting Counting Fields

Place the slide on the mechanical stage and position the center of the wedge under the objective lens and focus upon the sample. Nearly all of the particulates (particles and fibers) will be found in the upper 10-15 μm of the filter surface. When counting and sizing, continued use of the fine focus control is required to insure that nothing is missed. Start counting from one end of the wedge and progress along a straight line to the other end (count in either direction from circumference to wedge tip). Haphazard fields are selected without looking into the eyepieces by slightly advancing the slide in one direction with the mechanical stage control.

(e) Achieving Comparable Results

(1) Size only those fibers with a length-to-width ratio equal to or greater than 3:1.

(2) Count only fibers greater than 10 μm in length. (Be as accurate as possible in accepting or rejecting fibers near this length).

(3) Count up to 100 fields if necessary to yield a total count of at least 100 fibers. Count at least 20 fields even if more than 100 fibers are counted.

(4) Select the field of view without looking through the microscope's eyepieces to minimize unconsciously selecting "heavy" or "light" areas.

(5) The fields are selected along the entire length of a radial line running between the outside perimeter and the tip of the wedge.

(6) When an agglomerate (mass of material) covers a significant portion of the field of view (approximately 1/6 or greater), reject the field and select another. (Do not include this field in the number of fields counted.) Record the agglomerated field even though it is not included in the count.

(7) Bundles of fibers are counted as one fiber unless both ends of a fiber crossing another can be clearly resolved.

(8) For fibers that cross either one or two sides of the counting field, the following procedure is used to obtain a representative count. First, arbitrarily select: a) the left and bottom sides, and b) the upper and lower left corners and vertical direction as "decision aids."

Then count any fiber greater than 10 micrometers in length, but only if the fiber:

- a. lies entirely within the counting area, or
- b. crosses the left or bottom sides, or
- c. crosses the upper or lower left corners, or
- d. crosses both the top and bottom sides.

Reject and do not count all other fibers.

Calculations of Airborne Concentrations

Glass fiber airborne concentration may be calculated from the following formula:

$$C = \frac{(F-B)(W)}{(A)(V)}$$

where:

C = Airborne fiber concentrations in fibers >10 μ m/cu m.

- F = Average fiber count in fibers $>10 \mu\text{m}$ /field.
- B = Average fiber count of blank(s) or control filter(s) in fibers $>10 \mu\text{m}$ /field. (It is subtracted to eliminate the error or background contamination.)
- W = 855 square mm for 37-mm diameter filters (the portion of the membrane filter which is exposed when mounted in the field monitor case, ie, the effective filter area).
- A = The area of the counting field of a calibrated reticle expressed in square mm/field.
- V = Total air volume collected through filter expressed in milliliters.

APPENDIX D

OPERATION OF SAMPLING TRAIN-DATA SUMMARY FROM FIELD LOG BOOK

DATE	SITE	TRAIN CODE	TIME	FOOTMETER	FOOT	SAMPLE INITIAL
5-1-78	A	FG-D	6:01 PM	7.5	1832	DOB
5-1-78	A	F-D	6:01 PM	1.0	1690	
	A	P-D	6:01 PM	3.0	90613	
5-1-78	A	FG-D	7:05 PM	7.5	1832	DOB
	A	F-D	7:05 PM	1.1	1690	
	A	P-D	7:05 PM	3.0	90613	
5-1-78	C	FG-D	9:15 PM	8	1832	DOB
	C	F-D	9:15 PM	.9	1690	
	C	P-D	9:15 PM	2.9	90613	
5-1-78	C	FG-D	9:30 PM	8	1832	DOB
	C	F-D	9:30 PM	.9	1690	
	C	P-D	9:30 PM	2.9	90613	
5-1-78	C	FG-D	10:01 PM	7.9	1832	DOB
	C	F-D	10:01 PM	.9	1690	
	C	P-D	10:01 PM	2.9	90613	
5-1-78	A	FG-D	8:01 PM	7.5	1832	DOB
		F-D		1.0	1690	
		P-D		3.0	90613	
5-1-78	A	FG-D	8:16 PM	7.8	1832	DOB
		F-D		0.9	1690	
		P-D		2.9	90613	
5-1-78	C	FG-D	8:56 PM	7.8	1832	DOB
		F-D		0.9	1690	
		P-D		2.9	90613	
5-1-78	C	FG-D	10:35 PM	7.3	1832	DOB
		F-D		.9	1690	
		P-D		2.9	90613	
5-1-78	C	FG-D	11:05 PM	7.5	1832	DOB
		F-D		.9	1690	
		P-D		3.0	90613	

DATE	SITE	TRAIN CODE	TIME	ROTOMETER	PUMP	SAMPLER INITIAL
5-1-78	C	FG-D F-D P-D	11:25 PM	7.5 .9 2.9	1832 1690 90613	DOB
5-1-78 5-2-78	C	FG-D F-D P-D	MIDNIGHT	7.5 1.0 2.9	1832 1690 90613	DOB
		END SAMPLING		PERIOD AT MIDNIGHT		
5-2-78	A	FG-D F-D P-D	10:00 AM	8 .9 2.4	1832 1690 90613	DOB
5-2-78	A	FG-D F-D P-D	10:30 AM	8 .9 2.5	1832 1690 90613	DOB
5-2-78	D	FG-D F-D P-D	10:55 AM	7.5 .8 2.4	1832 1690 90613	DOB
5-2-78	D	FG-D F-D P-D	11:45 AM	7.6 .8 2.5	1832 1690 90613	DOB
5-2-78	D	FG-D F-D P-D	11:40 AM	7.8 .8 2.6	1832 1690 90613	DOB
5-2-78	D	FG-D F-D P-D	12 NOON	7.8 .8 2.6	1832 1690 90613	DOB
5-2-78	D	FG-D F-D P-D	12:10 PM	7.9 .7 2.6	1832 1690 90613	DOB

DATE	SITE	TRAIN CODE	TIME	PHOTOMETER	PUMP	SAMPLER INITIAL
5-2-78	D	FG-D F-D P-D	12:30 PM	7.8 .6 2.6	1832 1690 90613	DOB
5-2-78	D	FG-D F-D P-D	1:00 PM	7.8 .6 2.6	1832 1690 90613	DOB
5-2-78	D	FG-D F-D P-D	1:30 PM	7.8 .5 2.6	1832 1690 90610	DOB
5-2-78	D	FG-D F-D P-D	2:00 PM	7.4 .7 2.6	1832 1690 90613	DOB
5-2-78	E	FG-D F-D P-D	2:15 PM	7.4 .7 2.6	1832 1690 90613	DOB
5-2-78	E	FG-D F-D P-D	3:00 PM	7.4 .5 2.6	1832 1690 90613	DOB M D
5-2-78	E	FG-D FD - change pump P-D	3:20 PM	7.4 1.4 2.6	1832 145 90613	DOB
5-2-78	E	FG-D FD P-D	3:45 PM	7.4 1.45 3.0	1832 145 90613	DOB
5-2-78	E	FG-D FD P-D	4:00 PM	7.4 1.4 2.6	1832 145 90613	DOB
5-2-78	E	FG-D FD P-D change pump	4:12 PM	2.8 1.4 1.5	167 145 191	DOB
5-2-78	E	FG-D F-D P-D	4:40 PM	2.75 1.4 1.45	167 145 191	DOB

DATE	SITE	TRAIN CODE	TIME	POTIOMETER	PUMP	SAMPLER INITIAL
5-2-78	F	FG-D F-D P-D	5:00 PM	1.75 2.175 1.4 1.4 1.5 1.5	167 145 191	DOB
5-2-78	F	FG-D F-D P-D	5:30 PM	2.70 1.3 1.5	167 145 191	DOB
5-2-78	F	FG-D F-D P-D	6:00 PM	2.7 1.3 1.5	167 145 191	DOB
		END OF SAMPLING PERIOD				
5-4-78	G	FG-D F-D P-D	12:05 AM	3.0 2.7 1.75	167 0580 191	DOB
5-4-78	G	FG-D F-D P-D	12:40 AM	2.75 3.0 1.75	167 0580 191	DOB
5-4-78	G	FG-D F-D P-D	1:00 AM	2.75 3.0 1.75	167 0580 191	DOB
5-4-78	G	FG-D F-D P-D	1:32 AM	2.75 2.9 1.75	167 0580 191	DOB
5-4-78	G	FG-D F-D P-D	2:00 AM	2.75 3.0 1.75	167 0580 191	DOB
5-4-78	G	FG-D F-D P-D	2:30 AM	2.75 3.0 1.75	167 0580 191	DOB
5-4-78	G	FG-D F-D P-D - change pump	2:40 AM	2.8 2.75 1.85	167 0580 145	DOB

DATE	SITE	TRAIN CODE	TIME	ECOMETER	PUMP	SAMPLER INITIAL
5-4-78	G	FG-D F-D P-D change pump	3:00 AM	2.75 2.6 5.3	167 0580 1727	DOB
5-4-78	G	FG-D F-D P-D	3:33 AM	2.75 2.75 5.3	167 0580 1727	DOB
5-4-78	G	FG-D F-D P-D	4:07 AM	2.75 2.8 5.2	167 0580 1727	DOB
5-4-78	G	FG-D F-D P-D	4:30 AM	2.75 2.8 5.1	167 0580 1727	DOB
5-4-78	G	FG-D F-D P-D	5:00 AM	2.75 2.6 5.1	167 0580 1727	DOB
END OF SAMPLING PERIOD						

		TIME	PERCENTER	TEMP	STIRLER INITIAL	
5/1/78	B	PHENOL - UPWIND	6:00 PM	3.8	1756	CG
5/1/78	B	FORMALDEHYDE - UPWIND	6:01 PM	3.35	1727	CG
5/1/78	B	FIBERGLASS - UPWIND	6:01 PM	8.8	580	CG
5/1/78	B	PHENOL - UPWIND	7:00 PM	3.4	17566	TE
5/1/78	B	FIBERGLASS - UPWIND	7:00 PM	8.8	580	TE
5/1/78	B	FORMALDEHYDE - UPWIND	7:00 PM	2.8	1727	TE
5/1/78	B	PHENOL - UPWIND	7:30 PM	3.0	1756	TE
5/1/78	B	FIBERGLASS - UPWIND	7:30 PM	8.6	580	TE
5/1/78	B	FORMALDEHYDE - UPWIND	7:30 PM	2.8	1727	TE
5/1/78	B	PHENOL - UPWIND	8:01 PM	3.1	1756	TE
5/1/78	B	FORMALDEHYDE - UPWIND	8:01 PM	2.7	1727	TE
5/1/78	B	FIBERGLASS - UPWIND	8:01 PM	8.6	580	TE
END OF TEST						
5/1/78	B	PHENOL - UPWIND	8:15	3.4	1756	CG
5/1/78	B	FORMALDEHYDE - UPWIND	8:15	3.4	1727	CG
5/1/78	B	FIBERGLASS - UPWIND	8:15	7.9	580	CG

0-2

				SAMPLE INITIAL	
5/1/78	B	PHENOL-UPWIND	8:45	3.0	1756 T.E.
5/1/78	B	FIBERGLASS-UPWIND	8:45	7.7.	580 T.E.
5/1/78	B	FORMALDEHYDE-UPWIND	8:45	2.7	1727 T.E.
5/1/78	B	PHENOL-UPWIND	9:00	3.0	1756 T.E.
5/1/78	B	FIBERGLASS-UPWIND	9:00	7.7.	580 CG
5/1/78	B	FORMALDEHYDE-UPWIND	9:00	2.7	1727 CG
5/1/78	B	PHENOL-UPWIND	9:30	3.0	1756 CG
5/1/78	B	FIBERGLASS-UPWIND	9:30	7.7.	580 CG
5/1/78	B	FORMALDEHYDE-UPWIND	9:30	2.2	1727 CG
5/1/78	B	PHENOL-UPWIND	10:03	3.1	1756 CG
5/1/78	B	FIBERGLASS-UPWIND	10:03	8.1	580 CG
5/1/78	B	FORMALDEHYDE-UPWIND	10:03	3.0	1727 CG
			END OF TEST		
5/1/78	B	PHENOL-UPWIND	10:32	3.1	1756 T.E.
5/1/78	B	FORMALDEHYDE-UPWIND	10:32	2.7	1727 T.E.
5/1/78	B	FIBERGLASS-UPWIND	10:32	7.7.	580 T.E.

7-10

DATE	TIME	TESTS	TIME	POTOMETER	TEMP	WIND	WIND
5/1/78	B	PHENOL-UPWIND	11:05	3.1	1756	TE	
5/1/78	B	FORMALDEHYDE-UPWIND	11:05	2.7	1727	TE	
5/1/78	B	FIBERGLASS-UPWIND	11:05	7.7	580	TE	
5/1/78	B	PHENOL-UPWIND	11:30	3.1	1756	CG	
5/1/78	B	FORMALDEHYDE-UPWIND	11:30	2.7	1727	CG	
5/1/78	B	FIBERGLASS-UPWIND	11:30	7.7	580	CG	
5/1/78	B	PHENOL-UPWIND	12:02	3.0	1756	TE	
5/1/78	B	FORMALDEHYDE-UPWIND	12:02	2.7	1727	TE	
5/1/78	B	FIBERGLASS-UPWIND	12:02	7.7	580	TE	
		END OF TEST					
5/2/78	B	PHENOL-UPWIND	10:00 AM	3.7	1756	CG	
5/2/78	B	FORMALDEHYDE-UPWIND	10:00 AM	3.4	1727	CG	
5/2/78	B	FIBERGLASS-UPWIND	10:00	8.4	580	CG	
5/2/78	B	PHENOL-UPWIND	10:30	3.7	1756	CG	
5/2/78	B	FORMALDEHYDE	10:30	3.4	1727	CG	
5/2/78	B	FIBERGLASS	10:30	8.2	580	CG	
5/2/78	B	FIBERGLASS	11:00	8.2	580	CG	

8-0

DATE	TIME	TEST	TIME	POTOMETER	TUMP	REMARKS
5/2/78	B	PHENOL-UPWIND	11:00	3.0	1756	CG
5/2/78	B	FORMALDEHYDE-UPWIND	11:00	3.1	1727	CG
5/2/78	B	PHENOL-UPWIND	11:30	2.9	1756	DL
5/2/78	B	FORMALDEHYDE-UPWIND	11:30	3.1	1727	DL
5/2/78	B	FIBERGLASS-UPWIND	11:30	8.1	0580	DL
5/2/78	B	PHENOL-UPWIND	12:00	2.9	1756	CG
5/2/78	B	FORMALDEHYDE-UPWIND	12:00	3.1	1727	CG
5/2/78	B	FIBERGLASS-UPWIND	12:00	8.1	580	CG
END OF TEST						
5/2/78	B	PHENOL-UPWIND	12:05	3.4	1756	CG
5/2/78	B	FORMALDEHYDE-UPWIND	12:05	3.4	1727	CG
5/2/78	B	FIBERGLASS-UPWIND	12:05	8.4	0580	CG

DATE	TIME	ROTAMETER	PUMP	SAMPLER INITIAL		
5/2/78	B	PHENOL - UPWIND	12:30	2.8	1756	CG
5/2/78	B	FORMALDEHYDE - UPWIND	12:30	3.0	1727	CG
5/2/78	B	FIBERGLASS - UPWIND	12:30	8.0	0580	CG
5/2/78	B	PHENOL - UPWIND	1:00	3.3	1756	CG
5/2/78	B	FORMALDEHYDE - UPWIND	1:00	3.0	1727	CG
5/2/78	B	FIBERGLASS - UPWIND	1:00	8.0	0580	CG
5/2/78	B	PHENOL - UPWIND	1:30	2.6	1756	CG
5/2/78	B	FORMALDEHYDE - UPWIND	1:30	3.0	1727	CG
5/2/78	B	FIBERGLASS - UPWIND	1:30	8.0	0580	CG
5/2/78	B	PHENOL - UPWIND	2:05	3.2	1756	CG
5/2/78	B	FORMALDEHYDE - UPWIND	2:05	3.1	1727	CG
5/2/78	B	FIBERGLASS - UPWIND	2:05	8.1	0580	CG
END OF TEST						
5/2/78	B	PHENOL - UPWIND	2:10	2.8	1756	CG
5/2/78	B	FORMALDEHYDE - UPWIND	2:10	3.0	1727	CG
5/2/78	B	FIBERGLASS - UPWIND	2:10	8.6	580	CG

D.T.O.

DATE	TYPE	TESTS	TIME	ROTOMETER	PUMP	SAMPLER INITIAL	
5/2/78	B	PHENOL-UPWIND	2:30	2.7	1756	CG	
5/2/78	B	FORMALDEHYDE-UPWIND	2:30	3.0	1727	CG	
5/2/78	B	FIBERGLASS-UPWIND	2:30	8.0	0580	CG	"
5/2/78	B	PHENOL-UPWIND	3:00	2.7	1756	CG	
5/2/78	B	FORMALDEHYDE-UPWIND	3:00	3.0	1727	CG	
5/2/78	B	FIBERGLASS-UPWIND	3:00	8.0	0580	CG	
5/2/78	B	PHENOL-UPWIND	3:30	2.7	1756	TE	
5/2/78	B	FORMALDEHYDE-UPWIND	3:30	3.1	1727	TE	
5/2/78	B	FIBERGLASS-UPWIND	3:30	8.0	0580	TE	
5/2/78	B	PHENOL-UPWIND	4:11	2.7	1756	TE	
5/2/78	B	FORMALDEHYDE-UPWIND	4:11	3.0	1727	TE	
5/2/78	B	FIBERGLASS-UPWIND	4:11	8.0	0580	TE	
END OF TEST							
5/2/78	B	PHENOL-UPWIND	4:47	144 1.5	144	TE	
5/2/78	B	FORMALDEHYDE-UPWIND	4:47	1727 2.7	1727	TE	
5/2/78	B	FIBERGLASS-UPWIND	4:47	368 2.8	368	TE	

30

			TIME	ROTAMETER	PUMP	SAMPLER INITIAL
5/1/78	B	PHENOL-UPWIND	5:35	1.45	144	TE
5/1/78	B	FORMALDEHYDE-UPWIND	5:35	2.70	1727	TE
5/1/78	B	FIBERGLASS-UPWIND	5:35	2.50	368	TE
5/2/78	B	PHENOL-UPWIND	6:01	1.40	144	TE
5/2/78	B	FORMALDEHYDE-UPWIND	6:01	2.50	1727	TE
5/2/78	B	FIBERGLASS-UPWIND	6:01	2.70	368	TE

END OF TEST

5/1/78	D H	PHENOL-UPWIND	12:26AM	1.85	144	TE
5/1/78	D H	FORMALDEHYDE-UPWIND	12:26AM	2.65	368	TE
5/1/78	D H	FIBERGLASS-UPWIND	12:26AM	1.4	613	TE
5/1/78	H	PHENOL-UPWIND	1:05	1.85	144	TE
5/1/78	H	FORMALDEHYDE-UPWIND	1:05	2.65	368	TE
5/1/78	H	FIBERGLASS-UPWIND	1:05	1.4	613	TE
5/1/78	H	PHENOL-UPWIND	2:00	1.79	144	TE
5/1/78	H	FORMALDEHYDE-UPWIND	2:00	2.60	368	TE
5/1/78	H	FIBERGLASS-UPWIND	2:00	1.4	613	TE

0-12

			TIME	ROTAMETER	COMP	SAMPLER INITIAL
5/4/78	H	PHENOL-UPWIND	2:20	1.79	144	CG
5/4/78	H	FORMALDEHYDE-UPWIND	2:20	2.40	368	CG
5/4/78	H	FIBERGLASS-UPWIND	2:20	1.4	613	CG
END OF TEST						
5/4/78	H	PHENOL-UPWIND	3:00	1.79	144	CG
5/4/78	H	FORMALDEHYDE-UPWIND	3:00	2.55	368	CG
5/4/78	H	FIBERGLASS-UPWIND	3:00	1.6	613	CG
5/4/78	H	PHENOL-UPWIND	3:30	1.79	144	CG
5/4/78	H	FORMALDEHYDE-UPWIND	3:30	2.55	368	CG
5/4/78	H	FIBERGLASS-UPWIND	3:30	1.6	613	CG
5/4/78	H	PHENOL-UPWIND	4:00	1.70	144	TE
5/4/78	H	FORMALDEHYDE-UPWIND	4:00	2.50	368	TE
5/4/78	H	FIBERGLASS-UPWIND	4:00	1.6	613	TE
5/4/78	H	PHENOL-UPWIND	4:30	1.70	144	CG
5/4/78	H	FORMALDEHYDE-UPWIND	4:30	2.45	368	CG
5/4/78	H	FIBERGLASS-UPWIND	4:30	1.2	613	CG
5/4/78	H	PHENOL-UPWIND	5:00	1.80	144	TE
5/4/78	H	FORMALDEHYDE-UPWIND	5:00	1.4	368	TE
5/4/78	H	FIBERGLASS-UPWIND	5:00	2.5	613	TE

D-13

APPENDIX E

HASTINGS MASS FLOW METER DATA



HASTINGS-RAYDIST

A TELEDYNE COMPANY

HAMPTON, VIRGINIA 23361

(703) 721-6311

Specification Sheet **505c**

HASTINGS MASS FLOWMETER LF SERIES - NON LINEAR

FOR LOW FLOW RATE MEASUREMENTS OF AIR AND GASES

RANGES: 0-20 to 0-20,000 STD CC/MINUTE

MAJOR FEATURES

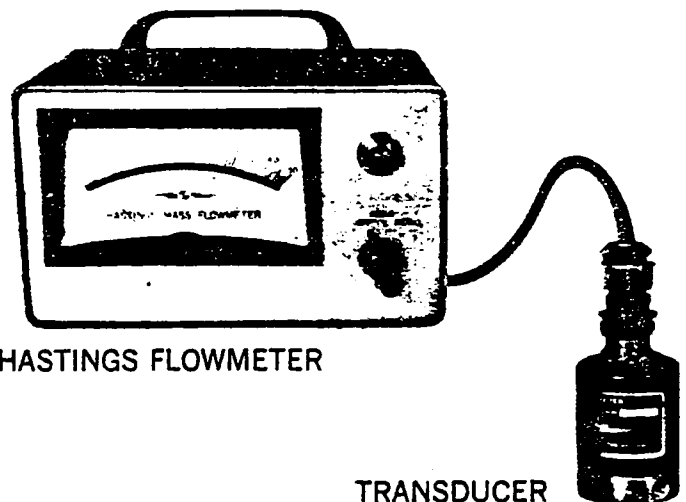
- MEASURES EXTREMELY LOW FLOW RATES
- READABILITY TO 1/2 % OF RANGE.
- ACCURATE AND STABLE WITHIN 2% FROM 0.1 psia TO 250 psia
- NO CORRECTION NECESSARY OVER WIDE RANGES OF TEMPERATURE AND PRESSURE
- RUGGED, VERSATILE, TROUBLE-FREE TRANSDUCER

LONG LIFE

The Hastings LF Mass Flowmeter features a thermal technique wherein the flow transducer sensing element is completely external to the flow stream and has no moving parts to wear out. It is safe for toxic and hazardous gases. Gas flow contacts only monel family alloys, solder and brass. Models with all monel family alloys are available for measuring flow rates of highly corrosive gases.

RUGGED - EASY TO INSTALL - RELIABLE

The LF meter circuitry is 100% solid state for maximum reliability and stability. Transducer can withstand extreme vacuum, pressure and flow rates without damage. No special tools or techniques needed. **E-1**



HASTINGS FLOWMETER

TRANSDUCER

HIGH STABILITY AND ACCURACY

With the Hastings LF Mass Flowmeter pressure and temperature need not be measured to determine mass as with volume flowmeters. No ambient temperature correction is required from 32°F to 110°F. No gas pressure correction is required from 0.1 psia to 250 psia or gas temperatures up to 200°F. Accuracy is within 2% over this range.

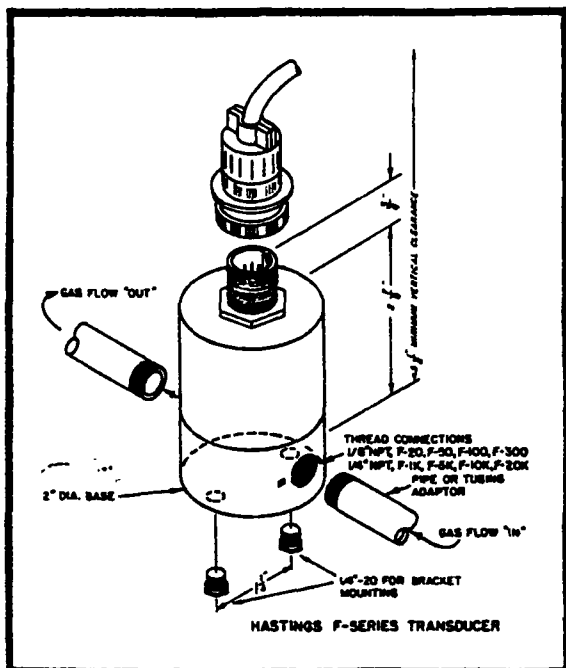
Measurement repeatability is within 1%. Standard factory calibration is for air. Curves for other usual gases are available. Once installed and in use the instrument needs no recalibration. Pressure drop through the flow tube is nominal for most ranges.

MODELS FOR TUNGSTEN HEXAFLUORIDE

Three of the models are especially constructed and calibrated for directly reading the mass flow of tungsten hexafluoride gas. They are also useful for rhenium hexafluoride and similar corrosive gases. Transducer construction of all monel alloys and "teflon" seals. Slightly larger transducer (3" diameter").

Model: ALF-100W-0-100 sccm

ALF-300W-0-300 sccm

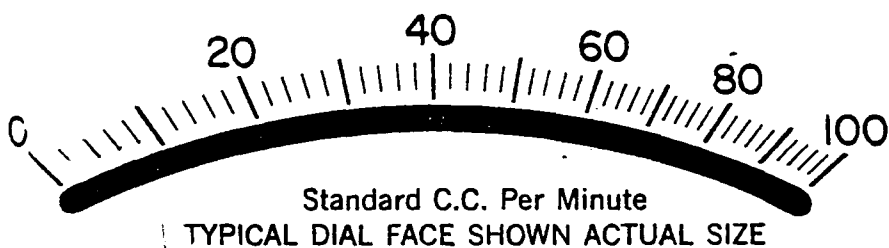


OPERATING PRINCIPLES

The Hastings LF Mass Flowmeter consists of an electrically heated tube and an arrangement of thermocouples to measure the differential cooling caused by a gas passing through the tube. Thermoelectric elements generate d-c voltage proportional to the rate of mass flow of gas through the tube. (No fragile sensing elements project into the stream. This design depends only on the mass flow and specific heat of the particular gas and is, therefore, practically insensitive to pressure changes in temperature, thermal conductivity and viscosity.

APPLICATIONS

Hastings LF Mass Flowmeters have wide applications in the measurement of leak rates and flow rates of gases in the manufacture of tubes, lamps, neon signs, semiconductors, fuel cells, valves and capillaries. They are also used for leak testing flanges and valves in cryogenic gas lines, missile fueling lines and for gas flow metering or for mixing gases in atomic research, magnetohydrodynamics (MHD) research, and in mass spectrometer type leak detectors. Write details of your particular application and requirements for recommendations by our Engineering Department.



CHARACTERISTICS

POWER:	115 volt a-c (105 to 125 V a-c) 50-400 cycles, 15 watts
INDICATOR:	Dimensions: 7 $\frac{3}{4}$ " X 5 $\frac{3}{4}$ " X 5 $\frac{3}{4}$ " Weight: 6 lbs.
TRANSDUCER:	Operating pressures: .1 psia to 250 psia. Gas flow temperatures: Up to 200°F. Ambient temperatures: From 32°F to 110°F. Sensitivity: 0.5% Full Scale. Response Time: Approximately 5 seconds for most models. Weight: 20 oz.
CABLES:	8-foot power and transducer cables included with instrument.

SELECTION CHART

Range Std. CC/Min	Model	Transducers (see Notes)	Pressure Dr @ Full Scale Inches H ₂ O
0-20	LF-20	F-20	70
0-50	LF-50	F-50	12
0-100	LF-100	F-100	1
0-300	LF-300	F-300	3
0-1000	LF-1K	F-1K	1
0-5000	LF-5K	F-5K	1
0-10,000	LF-10K	F-10K	1
0-20,000	LF-20K	F-20K	1

NOTES:

Transducers are available in standard brass "monel" construction. Monel type denotes all materials in contact with the gas are monel family alloy. Transducers are rated to 250 psi.

All models include switch and binding posts for connection to high impedance potentiometer type recorder. Output signal is approximately 0-2.4 mil volts dc.

Also available as a complete flow recorder utilizing G. E. #520 recorder, direct reading scale and 0-100 linear chart paper.

For linear type flowmeters and higher flow rates to 200 scfm, see Hastings Linear Mass Flowmeter Specification Sheet 508-A.

F

APPENDIX F

ROTOMETER CALIBRATION DATA

Flow Calibration Sheet

1

Hastings Mass Flow Meter versus Bubble Meter

4-30-78 Verification

Bubble	Hastings
638	625

658	650
-----	-----

550	535
-----	-----

418	400
-----	-----

446	450
-----	-----

least squares $H = .990 B - 4.9$

Pearson r correlation coefficient .997

5-2-78 Verification

Bubble	Hastings
--------	----------

2410	2420
------	------

1195	1150
------	------

450	440
-----	-----

1760	1690
------	------

3245	3140
------	------

least squares $H = .979 B - 5.2$

Pearson r correlation coefficient .999

Calibration of Rotometer

Pump # EOZ-1756

5-1	Flow	Rot.	5-3	Flow	Rot.
x	1220	43	x	1090	39
x	680	30	x	510	25
x	920	3.8	x	460	35
			x	1350	52

0.5 1.0 1.5 2.0 2.5 3.0 3.5 4.0 4.5 5.0 5.5 6.0 6.5 7.0 7.5 8.0 8.5 9.0 9.5 10.0 10.5 11.0 11.5 12.0 12.5 13.0 13.5 14.0 14.5 15.0 15.5 16.0 16.5 17.0 17.5 18.0 18.5 19.0 19.5 20.0 20.5 21.0 21.5 22.0 22.5 23.0 23.5 24.0 24.5 25.0 25.5 26.0 26.5 27.0 27.5 28.0 28.5 29.0 29.5 30.0 30.5 31.0 31.5 32.0 32.5 33.0 33.5 34.0 34.5 35.0 35.5 36.0 36.5 37.0 37.5 38.0 38.5 39.0 39.5 40.0 40.5 41.0 41.5 42.0 42.5 43.0 43.5 44.0 44.5 45.0 45.5 46.0 46.5 47.0 47.5 48.0 48.5 49.0 49.5 50.0 50.5 51.0 51.5 52.0 52.5 53.0 53.5 54.0 54.5 55.0 55.5 56.0 56.5 57.0 57.5 58.0 58.5 59.0 59.5 60.0 60.5 61.0 61.5 62.0 62.5 63.0 63.5 64.0 64.5 65.0 65.5 66.0 66.5 67.0 67.5 68.0 68.5 69.0 69.5 70.0 70.5 71.0 71.5 72.0 72.5 73.0 73.5 74.0 74.5 75.0 75.5 76.0 76.5 77.0 77.5 78.0 78.5 79.0 79.5 80.0 80.5 81.0 81.5 82.0 82.5 83.0 83.5 84.0 84.5 85.0 85.5 86.0 86.5 87.0 87.5 88.0 88.5 89.0 89.5 90.0 90.5 91.0 91.5 92.0 92.5 93.0 93.5 94.0 94.5 95.0 95.5 96.0 96.5 97.0 97.5 98.0 98.5 99.0 99.5 100.0

Calibration of Rotometers

2

Pump # DO 90613 (PU)

5-1	Flow	Rot	5-3	Flow	Rot
	x 1300	4.0		1990	55
	x 1050	34		x 1170	35
	x 820	25		x 570	19
				400	13
				380	9
				250	5
				190	00

Least square FR = $34.1 \text{ Rot} - 62.4$
CC .993

Pump # E02 1727 (PO)

5-1	Flow	Rot	5-3	Flow	Rot
	x 650	36		x 260	20
	x 700	38		x 800	39
				1340	53

Least square FR = $26.25 \text{ Rot} - 270$
CC .989

Pump # E02-1690 (FB)

5-1	Flow	Rot	5-3	Flow	Rot
	820	28		1220	35
	x 490	1.4		720	20
				x 420	12

Least square FR = $21.7 \text{ Rot} + 161$
CC .973

290	8
180	00

Pump # 004-0580 (FGU) 3

5-1	Flow	Roto	5-3	Flow	Roto
x	2530	84	x	3280	97
x	2400	80	x	2480	80
x	2000	68		1270	50
				810	37
least square FR = 43.9 Roto - 1060			560		30
CC	.985		300		23

Pump # E02-1822 (FGD)

5-1	Flow	Roto	5-3	Flow	Roto
x	2620	78	x	3210	88
x	2230	65	x	2400	70
			x	2100	77

least square FR = 41.9 Roto - 537
CC .984

Pump # 02-73-144 (PU)

5-2	Flow	Roto	5-3	Flow	Roto
x	1240	190		2420	265
x	770	165		3140	320
				1690	220

least square FR = 13.3 Roto - 1328
CC .984

Pump # 02-73-191 (PD)

4

5-2 Flow Rotor

5-3 Flow Rotor

x 760 155

1700 235

x 1550 210

least squares FR = 14.2 Rotor - 1436

x 1000 170

cc .999

520 140

250 120

150 10

Pump # 02-78-181 (FU)

5-2 Flow Rotor

5-3

Flow Rotor

x 560 125

3390 320

x 950 150

2450 260

1880 220

least squares FR = 11.2 Rotor - 822

1595 200

cc .997

1210 180

x 750 140

x 540 120

x 400 110

Pump # 02-78-145 FD

5-2 Flow Rotor

5-3

Flow Rotor

x 640 145

3140 320

x 430 125

2420 265

1690 220

least squares FR = 11.2 Rotor - 983

1150 185

cc .980

x 440 130

Pump# 03-78-368 (FGU)

5

5-2	Flow	Rotor	5-3	Flow	Rotor
X	2930	295	X	2800	290
X	2440	260	X	2710	245
				1480	200

least squares FR = 15.3 Rotor - 1600 950 160

C.C. .991 700 140
210 100

Pump# 02-78-167 (FGD)

5-2	Flow	Rotor	5-3	Flow	Rotor
X	3050	305	X	3300	320
X	2500	270	X	2710	260
				1410	200
				1740	160

least squares FR = 16.3 Rotor - 1914 500 140

C.C. 1.000 110 100
280 120

APPENDIX G

LIST OF SAMPLES AND TEST RESULTS

APPENDIX G

LIST OF SAMPLES AND TEST RESULTS

CODE	DESCRIPTION	SAMPLE TIME (5-2)	FIBERS FOR FG FOR P&F LAB RESULT	25°C 760mm TOTAL VOLUME SAMPLED M ³	ug/M ³ Conc.	ppb
FG-D-A	Fibrous Glass sites A & D - downwind	10:00-10:49 a.m.				
FG-D-D	Monitors moved during sampling	10:00-12:00 p.m.		.350		
P-D-A	Phenol sites A & D - downwind	10:00-10:49 a.m.				
P-D-D	Monitors moved during sampling	10:50-12:00 p.m.	0.8	.092	9	2
F-D-A	Formaldehyde sites A & D - downwind	10:00-10:49 a.m.				
F-D-D	Monitor moved during sampling	10:50-12:00 p.m.	7.6	.041	181	151
FG-U-B	Fibrous Glass site B - upwind	10:00-12:00 p.m.		.325		
P-U-B	Phenol site B - upwind	10:00-12:00 p.m.	9.2	.084	110	29
F-U-B	Formaldehyde site B - upwind	10:00-12:00 p.m.	LOST	.076,		
FG-D-D	Fibrous Glass site D - downwind	12:10- 2:00 p.m.		.312		
P-D-D	Phenol site D - downwind	12:10- 2:00 p.m.	< 0.5	.090	< 6	< 1
F-D-D	Formaldehyde site D - downwind	12:10- 2:00 p.m.	6.8	.032	223	173
F-U-G	Fibrous Glass site B - upwind	12:05- 2:05 p.m.		.325		
P-U-B	Phenol site B - upwind	12:05- 2:05 p.m.	0.6	.071	9	2
F-U-G	Formaldehyde site G - upwind	12:05- 2:05 p.m.	4.2	.076	55	45
FG-D-E	Fibrous Glass site E - downwind	2:15- 4:00 p.m.		.296		
P-D-E	Phenol site E - downwind	2:15- 4:00 p.m.	0.6	.086		
F-D-E	Formaldehyde site E - downwind	2:15- 3:05 p.m. }	8.4	.014 }	308	251
F-D-E(prime)	Changed pump and new solution	3:20- 4:00 p.m. }	3.6	.024 }		
FG-U-B	Fibrous Glass site B - upwind	2:10- 4:11 p.m.		.315		
P-U-B	Phenol site B - upwind	2:10- 4:11 p.m.	4.2	.074	57	14
F-U-B	Formaldehyde site B - upwind	2:10- 4:11 p.m.	6.8	.069	98	80

APPENDIX G

LIST OF SAMPLES AND TEST RESULTS

CODE	DESCRIPTION	(5-2) SAMPLE TIME	FIBERS FOR FG FOR P&F LAB RESULTS	25°C 76° mm TOTAL VOLUME M ³	ug/M ³ Conc	ppb
FG-D-E	Fibrous Glass site E&F downwind	4:12-5:00 p.m.				
FG-D-F	monitor moved during sampling	5:00-6:00 p.m.		.301		
P-D-E	Phenol site E&F downwind	4:12-5:00 p.m.				
P-D-F	Monitor moved during sampling	5:00-6:00 p.m.	0.6	.079	8	2
F-D-E	Formaldehyde moved during sampling	4:12-5:00 p.m.				
F-D-F	Monitor moved during sampling	5:00-6:00 p.m.	3.4	.065	52	43
FG-U-B	Fibrous Glass site B - upwind	4:47-6:01 p.m.		.197		
P-U-B	Phenol site B - upwind	4:47-6:01 p.m.	0.9	.043	21	5
F-U-B	Formaldehyde site B - upwind	4:47-6:00 p.m.	4.2	.035	120	98
FG-D-G	Fibrous Glass site G downwind	(5-4) 12:15-5:00 p.m.		.844		
P-D-G	Phenol site G downwind	12:15-2:30 a.m.	9.8	.155	63	17
F-D-G	Formaldehyde site G downwind	12:15-2:30 a.m.	2.7	.076	36	29
FG-U-H	Fibrous Glass site H upwind	12:26-5:00 a.m.		.693		
P-U-H	Phenol site H upwind	12:26-2:26 a.m.	< 0.5	.136	< 4	< 1
F-U-H	Formaldehyde site H upwind	12:26-2:26 a.m.	2.7	.054	50	41
P-D-G	Phenol site G downwind	2:40-3:05 a.m.				
	changed pump	3:05-5:00 a.m.	ND	.187	0	0
F-D-G	Formaldehyde site G downwind	2:45-5:00 a.m.	0.4	.057	7	6
P-U-H	Phenol site H upwind	2:31-5:00 a.m.	< 0.5	.157	< 3	< 1
F-U-H	Formaldehyde site H upwind	2:31-5:00 a.m.	ND	.114	0	0

APPENDIX G

LIST OF SAMPLES AND TEST RESULTS

<u>CODE</u>	<u>DESCRIPTION</u>	<u>SAMPLE TIME</u>	<u>FIBERS mg</u> <u>ug FOR P&F LAB</u> <u>RESULTS</u>	<u>25° 76°mm</u> <u>Total of</u> <u>sampled M³</u>	<u>ug/M³</u> <u>Conc.</u>	<u>ppb</u>
	(5-1)					
FG-D-A	Fibrous Glass-site A-downwind	6:01-8:01 p.m.		.330		
P-D-A	Phenol - site A- downwind	6:01-8:01 p.m.	< 0.5	.124	< 4	< 1
F-D-A	Formaldehyde - site A - downwind	6:01-8:01 p.m.	2.7	.045	60	49
FG-U-B	Fibrous Glass - site B - upwind	6:01-8:01 p.m.	--	.357		
P-U-B	Phenol - site B - upwind	6:01-8:01 p.m.	< 0.5	.088	< 6	< 2
F-U-B	Formaldehyde - site B - upwind	6:01-8:01 p.m.	*ND	.061	0	0
FG-D-A	Fibrous Glass - sites A&C - downwind	8:16-8:56 p.m.	--			
FG-D-C	Monitors moved during sampling	8:56-10:01 p.m.		.302		
F-D-A	Phenol - sites A&C downwind	8:16-8:56 p.m.				
F-D-C	Monitors moved during sampling	8:56-10:01 p.m.	< 0.5	.104	< 4	< 1
F-D-A	Formaldehyde- sites A&C downwind	8:16-8:56 p.m.				
F-D-C	monitors moved during sampling	8:56-10:01 p.m.	1.1	.036	31	25
FG-U-B	Fibrous Glass - site B - upwind	8:15-10:03 p.m.		.281		
P-U-B	Phenol - site B - upwind	8:15-10:03 p.m.	1.8	.068	265	69
F-U-B	Formaldehyde - site B - upwind	8:15-10:03 p.m.	*ND	.055	0	0
FG-D-C	Fibrous Glass - site C - downwind	10:15-12 midnight		.289		
P-D-C	Phenol - site C - downwind	10:15-12 midnight	8.5	.104	82	21
F-D-C	Formaldehyde - site C - downwind	10:15-12 midnight	*ND	.039	0	0
FG-U-B	Fibrous Glass - site B - downwind	10:32-12:02 (5-2) a.m.		.221		
P-U-B	Phenol - site B - upwind	10:32-12:02 (5-2) a.m.	< 0.5	.056	< 10	< 2
F-U-B	Formaldehyde - site B - upwind	10:32-12:02 (5-2) a.m.	0.4	.042	10	8

G-3

*ND = Non-detectable

APPENDIX H
HEALTH EFFECTS DATA

TABLE 15. HUMAN RESPONSES FROM EXPOSURE TO PHENOL VAPORS³⁰

Concentration (ppm)	Duration of exposure	Response	Reference
0.047	minutes	Odor threshold	4
0.0 - 3.3	8 hrs/day	No ill effect. Rise in urinary phenol	33
1.5 - 5.2	8 hrs with two 30 min breaks	No ill effect; 60 to 88 percent of phenol absorbed by lungs. Rise in urinary excretion of phenol during exposure with a return to pre-exposure levels within 24 hours	34
48.0 (plus 8 ppm HCHO)	5 to 10 min/hr, 8 hr/day	Marked irritation of the nose, throat and eyes. Formaldehyde may be primary cause.	35

TABLE 14. HUMAN RESPONSES FROM EXPOSURE TO FORMALDEHYDE VAPORS

Concentration (ppm)	Duration of expose	Response	Reference
0.01	5 min	Eye irritation threshold	13
0.05	-	Odor threshold	3
0.4 - 0.8	Occupational exposure	Acute exposures caused eye, nose, throat irritation, and lower respiratory tract symptoms	1
0.13 - 0.45	Occupational exposure	Burning, stinging eyes, headaches, intolerable irritation of eyes, nose and throat; one illness	21
0.25 - 1.39	Occupational exposure	Upper respiratory tract irritations, burning of eyes and nose, sneezing-coughing, headaches	20
0.9 - 1.6	Occupational exposure	Intense irritation, itching of eyes dry and sore throat, increased thirst disturbed sleep	22
1.0	-	Detectable by nearly all people	23
4.0	5 min	Severe eye irritation	24
4.0 - 5.0	10 to 30 min	Intolerable to most people; lachrymation, discomfort, throat irritation	23,25
10.0	few minutes	Profuse lachrymation	23
16 - 30	Occupational exposure (8 hr/day)	Skin reaction	26
50 - 100	5 - 10 min	May cause serious injury; serious bronchial inflammation	17

APPENDIX H

HEALTH EFFECTS DATA

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4. Petrov, V. I. Causes of Phenol Vapor Poisoning During Coke Slaking With Phenol Water, in Levine BS (trans): USSR Literature on Air Pollution and Related Occupational Diseases - A Survey. Springfield, VA., U.S. Dept. Comm. (NTIS 63-11570) 8:219-21. 1963.

APPENDIX I

Participants in Survey

All participants are employees of the Environmental Protection Agency, Surveillance and Analysis Division, Region III.

Robert Kramer	-	Task Manager
Theodore Erdman	-	Test Manager and Test Site Leader
David O'Brien	-	Test Site Leader
David Lorentz	-	Test Technician
Carmella Gualtieri	-	Test Technician