

3M COMPANY
OCCUPATIONAL HEALTH & ENVIRONMENTAL SAFETY DIVISION
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DETERMINATION OF SELECTED ORGANIC VAPORS IN AIR
USING 3M 3500/3510/3520/3530 ORGANIC VAPOR MONITORS

SCOPE

This procedure covers the method of analyzing samples to determine the amount of a particular organic vapor(s) present in the air. More specifically, this procedure is to be used for those organic vapors which can be collected by 3M Organic Vapor Monitors and desorbed with carbon disulfide or other suitable solvents. A provisional list of organic vapors that can be determined is given in supplementary publications such as "3M Organic Vapor Monitor Sampling and Analysis Guide".

SUMMARY OF THE METHOD

The organic vapors are adsorbed on high activity charcoal, desorbed with carbon disulfide and quantitated using a gas chromatograph equipped with a flame ionization detector (FID). There are certain compounds which have higher recoveries when desorbed with methylene chloride, acetonitrile or other suitable solvents. These are listed in the "3M Organic Vapor Monitor Sampling and Analysis Guide".

EQUIPMENT

The following or equivalent equipment is used:

Gas Chromatograph - Hewlett Packard, Model 5880A or 5890A equipped with a flame ionization detector (FID) and capillary capability.

Analytical Columns - J&W DB5 (nonpolar) (95% dimethyl 5% - diphenyl-polysiloxane), .25 um film thickness, .25 mm I.D., 30 m capillary; J&W DBWAX (polar) (polyethylene glycol), .25 um film thickness, .25 mm I.D., 30 m capillary. Other appropriate columns such as J&W DB225 and DBFFAP may also be used.

REAGENTS/SUPPLIES

The following or equivalent reagents/supplies are used:

Organic Vapor Monitors - 3M Brand No. 3500, 3510, 3520, and 3530.

Features: Sampling rate controlled by molecular diffusion.

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Each collection layer has about 160 milligrams of activated carbon in a Teflon matrix.

Autosampler Vials - 2 ml with Teflon lined caps.

Microliter Syringes - Hamilton (or equivalent) 1uL - 5 mL. These syringes are guaranteed accurate to +/-1% full scale and are used without further calibration.

Volumetric Flasks - Class A (10+/-0.02 mL, 25+/-0.03 mL, 50+/-0.05 mL, 100+/-0.08mL, etc.)

Carbon Disulfide - Aldrich # 27,066-0 (HPLC grade) or #34,227-0 (low benzene)

Solvents - Preferably reagent grade for all compounds of interest.

Compressed Gas Cylinders:

Helium for carrier gas (Linde High Purity).

Hydrogen for FID (Linde Ultra High Purity).

Argon-Methane for ECD (Linde P5).

Caution: High pressure cylinders are hazardous and should be handled with care. Do not store in extreme heat.

Air for FID (house air meeting or exceeding industrial grade Type 1 filtered through Linde Molecular Sieve 5A).

CALIBRATION

Standards containing the compound(s) of interest are prepared (usually three to five) and run with each set of analysis samples. Standards are prepared by diluting an appropriate amount (1 to 100 uL) of the compound of interest to 1.5, 10 or 25 mL with CS₂ or other solvents such as acetonitrile, methylene chloride, isopropanol or 50% dimethylformamide in CS₂. When more dilute working standards are required a 1:10 or 1:100 stock dilution is prepared first. Concentrations of standards are chosen to bracket the amount expected to be found. A sensitivity factor may be calculated by averaging the ug/unit area response for the standards or a calibration curve may be prepared using Slide-Write, Kaleidograph, Excel or other statistical program.

Appropriate quality control samples are run with each set of standards and samples.

Caution: Carbon disulfide is toxic and should be handled in a hood.

The standard solutions should be used as soon as possible. Standards may be stored in the laboratory refrigerator for no more than five days.

For example:

If a monitor is exposed to 50 ppm of toluene for 6 hours, the amount expected is:

$$mg = \frac{(ppm \times SR \times t \times MW \times 10^{-6})}{24.45} = \frac{(50 \times 31.4 \times 360 \times 92.14 \times 10^{-6})}{24.45} = 2.13$$

where:

SR = sampling rate (cm³/min)
MW = molecular weight
t = time (minutes)

If the monitor is eluted with 1.5 mL, the volume of pure toluene required in a 10 mL standard is:

$$uL = \frac{mg \times 10}{d \times 1.5} = \frac{2.13 \times 10}{0.865 \times 1.5} = 16.4$$

where:

d = density

Standards could be prepared by diluting 10, 15 and 20 uL of pure toluene to 10 mL yielding standards of 0.865, 1.298, and 1.73 mg/mL.

SAMPLE ANALYSIS

Using a repipet or a syringe, add 1.5 ml of the desorption reagent to each monitor through the center port (the port is immediately resealed). After standing for 30 minutes with occasional gentle agitation, the eluent is decanted into a marked 2 ml vial, sealed and a 1 to 5 ul sized sample is automatically introduced into the gas chromatograph. The area of the peak of interest is recorded and the amount in mg or ug is determined from the standard curve. If the weight collected for a single contaminant, is greater than the defined capacity listed in "3M Organic Vapor Monitor #3500 Analysis Guide" then the validity of the sample should be questioned. When sampling multiple contaminants, the combined weights of the contaminants collected should not exceed the defined value for the single contaminant with lowest capacity.

CALCULATIONS

Determine the weight of contaminant(s) present in each sample by use of the calibration data (regression equation or sensitivity factor) generated from the prepared standards.

The sample weight should always be corrected by subtracting any interfering contributions made from a control blank.

The time-weighted-average concentration of the environment sampled can be calculated by knowing the length of the sampling period, the contaminant weight determined by gas chromatography, the recovery coefficient and the molecular weight.

$$\text{ppm} = \frac{\left(\frac{\text{mg}}{\text{mL}}\right)(\text{mL})(24.45)}{(\text{SR})(t)(r)(\text{MW})}$$

where:

mg/mL = amount found from calibration curve

mL = elution volume (usually 1.5 mL)

SR = sampling rate (cm³/min)

t = sampling time (min)

MW = molecular weight

r = recovery

Alternatively, the following simplified equations can be used when the amount found on the monitor is determined in micrograms.

The calculation constant A is used to calculate the concentration when expressed in units of milligrams per cubic meter (mg/m³) and constant B when expressed in units of parts per million (ppm). The calculation constants A and B have been determined for all the compounds listed in "3M Organic Vapor Monitor Sampling and Analysis Guide".

3500 and 3510 Organic Vapor Monitor

The time-weighted-average concentration in milligrams per cubic meter (mg/m³) in the environment sampled can be calculated from the following expression:

$$C(\text{mg/m}^3) = \frac{W \times A}{r \times t}$$

The time-weighted-average concentration in parts per million (ppm) of the contaminant can be calculated from the following expression:

$$C(\text{ppm}) = \frac{W \times B}{r \times t}$$

where W = weight found (micrograms) corrected for blank and sample elution volume (usually 1.5 mL)
 r = recovery coefficient
 t = length of sampling period (minutes)
 A&B=calculation constants found in the Sampling and Analysis Guide or calculated as follows:

$$A = \frac{1000}{SR}$$

$$B = \frac{(1000)(24.45)}{(SR)(MW)}$$

Note: Each laboratory should determine their own recovery coefficients for greatest accuracy as described in the 3M bulletin "Recommended Procedure For Determination of Recovery Coefficients". Typical recoveries determined in our laboratories are shown in the "3M Organic Vapor Monitor Sampling and Analysis Guide". Additional information on the determination of recoveries can be found in the 3M Monitor Validation Procedure.

The above expressions calculate the time-weighted-average concentrations at a sampling temperature of 25 °C and pressure of 760 mm Hg. When sampling at other environmental conditions, the previous expressions need to be corrected only for variations in temperature. The previous expressions can be multiplied by the following temperature correction factors (CFt) for samples collected at temperatures other than 25 °C (77 °F).

Sampling Temperature (°C)	Sampling Temperature (°F)	Correction Factor (CFt)
44	111	.97
37	99	.98
31	88	.99
25	77	1.00
19	66	1.01
13	55	1.02
7	45	1.03
2	36	1.04
- 3	27	1.05
- 8	18	1.06

As shown in the above table, every 10-11 degrees above or below 77 °F requires a one percent correction to the calculated time- weighted-average concentration.

If the temperature correction is desired, the time-weighted-average concentration can be calculated by multiplying the concentration calculated above by the CFT.

3520 and 3530 Organic Vapor Monitor with back-up section.

Upon analysis of each section, the validity of the sample can be determined. The ratio of the contaminant weight (W_s) on the secondary section to the contaminant weight (W_p) on the primary section must meet the following criteria.

$$W_s/W_p < .50$$

The calculations for the 3520 and 3530 are as follows:

$$C \text{ (mg/m}^3\text{)} = \frac{(W_p + 2.2 \times W_s) \times A}{r \times t}$$

$$C \text{ (ppm)} = \frac{(W_p + 2.2 \times W_s) \times B}{r \times t}$$

where:

W_p = weight collected on the primary section

W_s = weight collected on the secondary section

A&B=calculation constants found in the Sampling and Analysis Guide

Example Calculation (Trichloroethylene):

Length of sampling period (t) 465 minutes

Temperature (T) 19 °C

Calculation constant A = 32.2 or B = 5.98

Weight (W) 768 micrograms

Recovery coefficient 1.01

Using calculation constant (A):

$$C(\text{mg/m}^3) = (768 \times 32.2 \times 1.01)/(1.00 \times 465)$$

$$C = 53.7 \text{ mg/m}^3$$

Using calculation constant (B):

$$C(\text{ppm}) = (768 \times 6.00 \times 1.01)/(1.00 \times 465)$$

OVM Analytical Method

$$C = 10.0 \text{ ppm}$$

LIMIT OF QUANTITATION (LOQ)

The routine limit of quantitation (LOQ) for benzene and ethylene dichloride is 4 ug. The routine LOQ for all other analyzed compounds is 5-10 ug. The LOQ may be lowered if necessary by increasing the injection volume, reducing the split, or using a splitless injection.

APPENDIX A

Typical Parameters for HP5880 and HP5890 Gas Chromatographs:

Oven Temp	40 °C
Initial Time	3 min
Equib Time	0.5 to 2 min
Prgm Rate	10 °C/min
Final Value	180-225 °C
Final Time	3 min
Det Temp	250 °C
Inj Temp	225 °C

Chart Speed	.50 cm/min
Attn	2 ³
%Offset	10
Threshold	0
Peak Width	.04
Split Ratio	30:1

APPENDIX B

SUGGESTIONS FOR PACKED COLUMN GAS CHROMATOGRAPHS

10% SP1000 (or FFAP), 80/100 Supelcoport, 20 ft. X 1/8 in. SS

15% Carbowax 20M, Chromosorb W, 7.5 ft. X 1/8 in. SS

Poropak Q, 6 ft. X 1/8 in. SS

10% SP2100, Supelcoport, 10 ft. X 1/8 in. SS

20% SP2100/0.1% Carbowax 1500, 100/120 mesh Supelcoport, 10 ft. X 1/8 in SS