3M COMPANY

OCCUPATIONAL HEALTH & SAFETY PRODUCTS LABORATORY

Organic Vapor Analytical Method No. 2, Revision A
January, 1982

DETERMINATION OF SELECTED ORGANIC VAPORS IN AIR

SCOPE

This is a procedure which 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 titled "3M Organic Vapor Monitor #3500 Analysis Guide" and "3M Organic Vapor Monitor Sampling Guide".

Calibration curves are generated for each contaminant of interest by injecting known amounts of the compound into a gas chromatograph and recording the response. Interferences will be treated in the Discussion Section.

SUMMARY OF 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.

APPARATUS

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

Features: Sampling rate controlled by molecular diffusion.
Each collecting layer has 160 milligrams of petroleum based charcoal.

Gas Chromatograph - Hewlett Packard, Model 5840A, or equivalent, equipped with a flame ionization detector - available from Hewlett Packard, 2025 West Larpenteur Ave. St. Paul, MN 55113.

Analytical Columns and Parameters - A list of suggested columns is given in Table 1*. Column parameters should always be adjusted to produce sharp symmetrical peaks with as much resolution as possible from interfering components. Carrier gas flow rates are nominally set at 25 cm^3/min. for packed columns 1/8 inch in diameter.

Vials: - Size - 3.2 mm x 1.1 mm, Volume - 1 ml.
Part No. 3-3123
Caps: - Equipped with Teflon liners.

Cap Crimper: - Part No. 3-3195 available from Supelco.

REAGENTS

Carbon Disulfide - Spectroquality or equivalent.

Solvents - Preferrably chromatoquality for all compounds of interest.

* Capillary columns exhibits much better resolution than packed columns, but because of variance in manufacturing and performance, no capillary columns are listed.

Gas Chromoatograph Supplies - Pressurized bottles of:

1. Helium for carrier gas use must meet or exceed laboratory Grade D 99.995% pure.
2. Nitrogen for carrier gas use must meet or exceed laboratory Grade E 99.98% pure.
3. Hydrogen for flame detector must meet or exceed Grade A 99.8% pure.
4. Air for flame detector must meet or exceed industrial Grade Type 1.

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

Calibration Standards - A series of standard solutions are prepared for each solvent to be determined. A microliter syringe is used to inject an appropriate amount of a particular compound into a known volume of carbon disulfide or other suitable solvent. Nominally 10, 25, and 50 ml glass - stoppered volumetric flasks are used. For the more volatile compounds, known amounts are added to measured quantities of solvent contained in vials equipped with Teflon - lined caps. The vials are immediately inverted and left in that position until used. These solutions are used to prepare a reference calibration curve for the particular organic vapor of interest. Normally, 2 uL of each of the standard solutions are injected into a gas chromatograph and the peak area for each concentration is recorded. A best fit line is calculated by the regression technique to correlate peak area with the weight of component(s) present. It is recommended that the calibration and monitor samples be the same size (2 uL) to avoid possible column overloading as well as factorial errors.

An alternate method to the regression line is to average the resultant sensitivity factors of three individually prepared standards. Sensitivity factors are determined by the ratio of weight to area units.

Caution: Carbon disulfide is toxic and should be handled in a hood. Standard solutions should be used as soon as possible. They may be stored in the laboratory refrigerator for no more than five days.

SAMPLING

The monitor is removed from the package and the exposure start time is recorded on the back of the badge. When personal sampling is being performed, the monitor should be attached near the breathing zone. For area sampling, the monitor should NOT be placed in a corner or along a wall where stagnant air may exist. At the conclusion of the sampling period, the badge is removed and the retaining ring along with the membrane are discarded. The elutriation cap is then immediately snapped on terminating monitor exposure and the end time is recorded on the back of the badge. The following information should be recorded for each sample:

1. Monitor number.
2. Date exposed.
3. Employee or Area I.D.
4. Organic vapor of interest along with suspected contaminants which could cause interference.
5. Temperature and relative humidity of the monitored environment if considered extreme.
6. Any comments or unusual circumstances.

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 30 minutes, with occasional gentle agitation, the eluent is decanted into a marked auto-sampler vial where a 2 uL sized sample is automatically introduced into the gas chromatograph. Alternatively, a 2 uL aliquot is removed from the center port for manual injection. The resultant analysis is recorded on a chromatogram where area units of the desired component(s) are correlated with the amount (weight) present during the sampling period. The weight found is recorded and converted to mg/m^3 and/or ppm. 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 singl contaminant with the lowest capacity.

Calculation of Contaminant Concentrations

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 interferring 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 calculation constant either A or B. The calculation constant A is used to calculate the concentration when expressed in units of milligrams per cubic meter (mg/m^3) and constant B when expressed in units of parts per million (ppm). The calculation constant A and B have been determined for all the compounds listed in "3M Organic Vapor Monitor #3500 Analysis Guide".

The time-weighted-average concentration in milligrams per cubic meter of any (listed) contaminant present in the environment sampled can be calculated from the following expression:

$$C(mg/m^3) = \frac{W \text{ (micrograms)}}{rxt \text{ (minutes)}} \times A$$

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

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

Where W = corrected contaminants weight recovered.

r = recovery coefficient

t = length of sampling period

$$A = \frac{1000}{\text{Sampling Rate}}$$

$$B = \frac{(1000)}{\text{Sampling Rate}} \times \frac{(24.45)}{\text{Molecular Weight}}$$

The above expressions calculate the time-weighted-average concentrations at a sampling temperature of 25°C (298°K) and pressure of 760 mm. When sampling at other environmental conditions, the above expressions need to be corrected only for variations in temperature. The above expressions can be multipled by the following temperature correction factors (CF_T) for samples collected at temperatures other than 25°C (77°F).

Sampling °C	Temperature °F	Temperature Correction Factor CF_T
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

From the previous table, every 10-11° 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 the following expression:

$$C(mg/m^3) = \frac{W \text{ (micrograms)}}{rxt \text{ (minutes)}} \times A \times CF_T$$

$$C(ppm) = \frac{W \text{ (micrograms)}}{rxt \text{ (minutes)}} \times B \times CF_T$$

Example Calculation (Trichloroethylene)

Length of sampling period (t)	465 minutes
Temperature (T)	19°C
Calculation constant (A)	32.2
or constant (B)	6.00
Corrected Toluene Weight (W)	768 micrograms
Recovery coefficient (r)	1.00

Using calculation constant (A):

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

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

Using calculation constant (B):

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

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

DISCUSSION

Experience teaches that a single contaminant is rarely found by itself in a given industrial atmosphere. Depending on the number and nature of the contaminating organic vapors, it is possible that interferences may occur. To help insure the accuracy of an analytical result, a sample may be analyzed on two chemically different natured columns (Carbowax vs SE-30). If the results are essentially the same for both columns, there is a good chance no interferences are present. However, no absolute identification can be made for any contaminant(s) by retention time only, regardless of the number of analytical columns used.

H. E. Mullins
January 20, 1982

TABLE 1

Analytical Columns	Use
10% FFAP, Chromosorb W AW, 10 ft. x 1/8 in. SS	General, Free Fatty
15% carbowax 20m, Chromosorb W, 7.5 ft. x 1/8 in. SS	General, Chlorinate
Porapak Q, 6 ft. x 1/8 in. SS	Low MW Halogens
10% FFAP, Chromosorb W AW, 20 ft. x 1/8 in. SS	General, Solvent
25% CEF, Chromosorb P AW, 10 ft. x 1/8 in. SS	Benzene, Aromatics
5% OV-17, Supelcoport, 6 ft. x 4 mm glass	General, Drugs, Pes
10% OV-101, Supelcoport, 10 ft. x 1/8 in. SS	General
10% SP-2100, Supelcoport, 10 ft. x 1/8 in. SS	General, Hydrocarbo
1.75% Bentone 34/5% SP1200, 10 ft. x 1/8 in. SS	Xylene, Aromatics