Designation: D4766 – 98 (Reapproved 2014)

Standard Test Method for Vinyl Chloride in Workplace Atmospheres (Charcoal Tube Method)[1]

1. Scope

1.1 This test method describes the determination of vinyl chloride monomer (VCM) in workplace atmospheres using a modified charcoal tube method (see Practice D3686).

1.2 This procedure is compatible with low flow rate personal sampling equipment. It can be used for personal or stationary monitoring. It cannot determine instantaneous fluctuations in concentration to detect maximum values. Alternative on-site procedures, such as gas chromatography or infrared spectrometry, must be used to measure fast-changing concentrations.

1.3 The range of this test method is from the limit of detection of approximately 0.01 to 100 ppm (v).

1.4 The sampling method provides a time-weighted average sample.

1.5 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For more specific precautionary statements, see Section 9 and Warnings in 10.2.3 and 11.1.3.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D3686 Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)

E355 Practice for Gas Chromatography Terms and Relationships

3. Terminology

3.1 *Definitions*—For definitions of terms relating to this test method, refer to Terminology D1356 and Practice E355.

4. Summary of Test Method

4.1 Known volumes of sample air are passed through glass or metal tubes packed with activated charcoal. Vinyl chloride is adsorbed on the charcoal and thus removed from the flowing gas stream (**1-3**).[3]

4.2 A two-section tube containing a front and a backup section of adsorbent is used to collect the sample.

4.3 The vinyl chloride is desorbed with carbon disulfide and analyzed with a gas chromatograph equipped with a flame ionization detector. Two or more different columns are to be used to reveal interferences that might otherwise not be detected.

4.4 Adsorption/desorption efficiencies are determined by the same techniques used for the atmospheric analysis applied to known standards. Instrument calibrations are made with prepared standard solutions of vinyl chloride in carbon disulfide.

5. Significance and Use

5.1 Vinyl chloride monomer (VCM) is the starting material for the manufacture of poly(vinyl chloride) (PVC), which is used extensively in construction, electronics, packaging, and other industries. The vinyl chloride gas can escape during manufacturing, transportation, and polymerization stages. Residual monomer can also escape during subsequent fabrication processes.

5.2 Vinyl chloride is a toxic and explosive hazardous material.

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[1] This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The boldface numbers in parentheses refer to the list of references at the end of this test method.

5.3 Vinyl chloride is suspected to be a carcinogenic agent (**4**), and occupational exposure limits (OELs) have been established for this agent. For instance, the present U.S. Occupational Safety and Health Administration (OSHA) standard for VCM is 1.0 ppm (v) with a 0.5-ppm (v) action level; the maximum length of operator exposure at 5 ppm (v) is 15 min.[4]

5.4 The method is made selective for vinyl chloride by choosing chromatograph columns that will separate vinyl chloride from other organic compounds in the sample.

5.5 The method meets requirements (for example, those of OSHA) for monitoring workplace atmospheres in vinyl chloride and poly(vinyl chloride) production and fabrication processes.

6. Interferences

6.1 Organic components that have the same or nearly the same retention time as vinyl chloride during gas-chromatographic analysis will interfere.

6.2 Other volatile organic compounds in the area samples should be given consideration.

6.3 Such interferences can be minimized by proper selection of gas-chromatographic columns. A mass spectrometric detector can be used to confirm the presence of vinyl chloride.

6.4 Water mists and high humidity affect adsorption efficiencies by reducing the adsorptive capacity of the charcoal for vinyl chloride.

7. Apparatus

7.1 *Charcoal Sampling Tube:*

7.1.1 *Description*—A sampling tube consists of a length of glass tubing containing two sections of activated charcoal that are held in place by nonadsorbent material and sealed at each end.

7.1.2 Sampling tubes are commercially available and consist of a glass tube 110 mm long, 10 mm in outside diameter, and 8 mm in inside diameter. They contain two sections of 20/40 mesh activated charcoal, 800 and 200 mg, each separated by a 2-mm section of urethane foam. The front section is retained by a plug of glass wool and the back section is retained by a second 2-mm portion of urethane foam or other retainer, such as glass wool. The ends of the tube are flame sealed.

7.1.3 The back section of the sample tube (200 mg) adsorbs vapors that penetrate the front section and is used to determine whether the collection capacity of the tube has been exceeded. Instead of a single tube, two tubes in series may be used (see 11.1.11).

7.1.4 The pressure drop across the charcoal tube should be no greater than 2.5 cm Hg [3.3 kPa] at a flowrate of 1000 mL/min.

7.1.5 Glass tubes must be held in suitable protective holders to prevent breakage during sampling.

7.2 *Syringes:*

7.2.1 *Gas-Tight Syringe,* 1 to 2-mL capacity.

7.2.2 *Microlitre Syringes,* 10, 25, and 100-µL or other convenient sizes for making standards.

7.3 *Polyethylene End Caps,* for capping charcoal tube. Caps must fit snugly to prevent leakage.

7.4 *Vials,* glass, approximately 12 mL (3-dram), for desorbing samples and holding standards, polyethylene or TFE-fluorocarbon-lined screw cap or septum valve. A 1-oz vial with the same types of closure is needed for standard preparation.

7.5 *Plastic Bags*—Poly(vinylidine chloride) or poly(vinyl fluoride), 50 to 100 L, for preparing known concentrations of vinyl chloride. The significant solubility of vinyl chloride in polytetrafluorethylene renders bags made of this type of film unsuitable. Other equivalent containers for preparing known concentrations are also suitable.

7.6 *Sampling Equipment*—See Ref (**5**) for general information on air sampling instrumentation.

7.6.1 Any pump whose flow rate can be accurately determined and set at the desired sampling rate is suitable.

7.6.2 Pumps having stable low flow rates (10 to 200 mL/min) are preferable for long period sampling (up to 8 h) or for short periods when concentration of organic vapors are expected to be high.

7.6.3 All sampling pumps must be carefully calibrated with a charcoal tube in the proper sampling position. (See Practice D3686 and Annex A1.)

7.6.4 *Tubing,* rubber or plastic, 6-mm [¼ in.], bore about 0.9 m [3 ft] long, equipped with a spring clip to hold the tubing and charcoal tube in place on worker's lapel area (Note 1).

NOTE 1—Sampling tubes must not be used with plastic or rubber tubing upstream of the charcoal. Absorption by the tubing may introduce sampling errors.

7.7 *Gas Chromatograph:*

7.7.1 Gas chromatographs that employ either a flame ionization detector or a detector whose specifications are equivalent in sensitivity and selectivity should be used. Detectors must be capable of determining vinyl chloride concentrations of interest with a signal-to-noise ratio of at least 10 to 1. Suitable detectors are capable of detecting approximately 5×10^{-10} g of vinyl chloride per injection.

7.7.2 A gas-chromatographic column capable of separating vinyl chloride from other components is required. A number of suitable columns have been discussed in the literature (**6**). Table 1 lists 14 columns and the oven temperatures used for vinyl chloride determination. Table 2 lists the relative retention times of some potential interferences for four chromatographic columns. Column suitability must be verified by testing with two or more columns of dissimilar packings to ensure the absence of interferences. If the chromatographic peak for vinyl chloride overlaps the peak for other components by no greater than 5 %, the separation is considered to be satisfactory.

7.7.3 Gas chromatographic operating conditions for the instrument being used should be optimized so that the separation required for a successful analysis can be obtained in a reasonable time.

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[4] Federal Register, 39, 194 (1974).

TABLE 1 Common Packings and Oven Temperatures for the Vinyl Chloride Analysis

Column Packing	Oven Temperature, °C	Reference
10 to 20 ft [3 to 6 m] SE-30 on Chromosorb G, Chromosorb W. or Anakrom ABS	ambient to 90	**3**, **7**, **8**
20 ft [6 m] 10 % FFAP on Chromosorb W	65	**3**
6 ft [1.8 m] Poropak Q	100–135	**2**, **9**, **10**
5 to 6 ft [1.5 to 1.8 m] Poropak QS	70–120	**1**, **11**, **12**
6 ft [1.8 m] Chromosorb 101	90–100	**13**, **14**
1.5 to 6 ft [0.5 to 1.8 m] Chromosorb 102[A]	70–145	**15**, **16**, **17**, **18**
6 ft [1.8 m] 10–20 % DC 200 on Chromosorb W or Supelcoport	80	**1**, **16**
20 ft [6 m] Carbowax 4000 on Supelcoport	80	**1**
6 ft [1.8 m] 0.4 % Carbowax 1500 on Carbopak A	ambient	**16**, **19**
6 ft [1.8 m] 5 % OV-101 on Chromosorb W	ambient	**10**
6 ft [1.8 m] 10 % Apiezon M on Chromosorb W	ambient	**10**
6 ft [1.8 m] silica gel	30	**20**
300 ft [90 m] open tubular column, coated with dibutyl maleate	0	**20**
16 ft [4.8 m] 16.7 % triscyano ethoxypropane on Chromosorb W	programmed from 50 to 170	**21**

TABLE 2 Relative Retention Times of Potential Interferences to Vinyl Chloride

(Vinyl Chloride Retention = 1.0)

Compound	Chromosorb[A] 102	Chromosorb[B] 102	Poropak Q[C]	0.4 % Carbowax[D] 1500 on Carbopak A
Methane	0.15	...	0.05	0.20
Ethane	0.21	...	...	0.29
Ethene	0.21	0.33	...	0.26
1,1-Difluoroethylene	...	0.33	...	0.63
Propene	...	0.62	0.46	0.63
Propane	0.54	...	0.52	0.63
Methylacetylene	...	...	0.56	...
Methyl chloride	0.63	...	0.57	0.45
1,1-Difluoroethane	...	0.51	...	...
Chlorodifluoromethane	...	0.53	...	...
Cyclopropane	...	...	0.59	...
Formaldehyde	...	...	0.62	...
1-Chloro-1,1-difluoroethane	...	0.92	...	...
Acetyladehyde	0.93	...	0.95	0.77
Dichlorotetrafluoroethane	...	1.21	...	...
Isobutane	1.22	...	...	...
Isobutylene	1.37	1.25	...	...
Methanol	...	...	...	1.38
1,3-Butadiene	1.57	1.27	...	...
1-Butene	1.43	1.30	...	...
Vinyl methyl ether	...	1.36	...	...
Trans-2-butene	1.57	1.38	...	2.92
Ethyl chloride	1.70	...	...	1.54
Cis-2-butene	1.73	1.43	...	...
Vinyl bromide	...	1.85	...	...
1,1-dichloroethylene	2.00	...	...	...

[A] 6 ft by ⅛ in. Chromosorb 102 (80/100 mesh) at 100°C.

[B] 6 ft by ⅛ in. Chromosorb 102 (80/100 mesh) at 145°C.

[C] 6 ft by ⅛ in. Poropak Q (80/100 mesh) at 100°C.

[D] 6 ft by ⅛ in. 0.4 % Carbowax 1500 on Carbopak A at ambient temperature.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise noted, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other reagents may be used provided it can be demonstrated that they are of sufficiently high purity to permit their use without decreasing the accuracy of determination.

8.2 *Activated Coconut-Shell Charcoal*—Commercially available coconut charcoal (20/40 mesh) has been found to have adequate adsorption capacity.

8.3 *Calibration Standards*—Standard concentrations of vinyl chloride in air may be purchased or prepared in the laboratory. Laboratory standard concentrations are prepared by pressure dilution in stainless steel cylinders, atmospheric pressure dilution in plastic bags, or dynamically with permeation tubes (**22**). These standards are used to determine adsorption/desorption efficiencies of the activated charcoal (Annex A1).

8.4 *Carbon Disulfide* (CS_2), spectroquality.

8.5 *Vinyl Chloride* (VCM), gas cylinder.

9. Safety Precautions

9.1 Carbon disulfide and vinyl chloride vapors are toxic and highly flammable. Usage should be restricted to a well-ventilated hood.

9.2 Small waste quantities of carbon disulfide shall be disposed of only in accordance with local regulations and accepted practices.

9.3 The vapors of vinyl chloride should not be inhaled since they have anesthetic properties and are suspected to be carcinogenic.

9.4 Avoid skin contact with carbon disulfide, solutions of vinyl chloride in CS_2, and vinyl chloride liquid.

10. Calibration and Standardization

10.1 *Sample Pump Calibration*—Calibrate the sample pump flow in accordance with Practice D3686 and Annex A1.

10.2 *Standardization:*

10.2.1 Prepare calibration standards as micrograms of vinyl chloride per 10 mL of carbon disulfide over range of interest.

10.2.2 Pipet 30 mL of carbon disulfide into a 1-oz bottle and seal.

10.2.3 Fill a 1.0-mL gas-tight syringe with pure vinyl chloride vapor from a cylinder of pure vinyl chloride. (**Warning—**Handle the pure vinyl chloride gas in a hood.)

10.2.4 Inject 1.0 mL of vinyl chloride vapor (through the septum) directly into the carbon disulfide in the vial. Mix thoroughly. This standard contains 852 µg of vinyl chloride per 10 mL of carbon disulfide and is equivalent to 13.3 ppm in air based on a 25-L sample. Prepare in duplicate (Note 2).

NOTE 2—The concentration of the standard solution is based on

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[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

injection of 1.0 mL of gas at 25°C and 1 atmosphere (101.3 kPa). For other conditions of temperature and pressure, the concentration should be multiplied by a correction factor, as follows:

$$\text{Correction Factor} = \frac{\text{barometric pressure (kPa)}}{101.3\text{ kPa}} \times \frac{298}{°\text{C} + 273} \quad (1)$$

10.2.5 Prepare a dilute standard by pipetting 1 mL of the above standard solution into a 3-dram vial containing 10 mL of carbon disulfide giving a total volume of 11 mL. Cap immediately with a septum valve. This standard contains 77.5 µg/10 mL of carbon disulfide and is equivalent to 1.18 ppm in air based on a 25-L sample.

10.2.6 Additional standards are prepared by dilution of the above standards with carbon disulfide. Care must be taken in the dilution steps to minimize the chance of vinyl chloride loss.

10.2.7 Analyze each vinyl chloride in carbon disulfide standard in duplicate by gas chromatography.

10.2.8 Draw 3 to 4 µL of the solution to be analyzed into a 10-µL syringe and make sure there are no bubbles in the syringe. Adjust the volume in the syringe to 2 µL. After the needle is removed from the sample and prior to injection, the plunger is pulled back approximately 1 µL to minimize evaporation of the sample from the tip of the needle.

10.2.9 An alternative injection procedure may be used. Before taking a sample from the standard solution, draw about 1 µL of carbon disulfide solvent into a 10-µL syringe. Draw a specified amount, up to 5 µL of the calibration mixture into a barrel of the syringe, leaving a 0.5-µL air space between the CS_2 and the calibration mixture. Draw the entire sample into the barrel of the syringe. Inject the sample plus the CS_2 (flushing plug) into the gas chromatograph.

10.2.10 Prepare a calibration curve by plotting micrograms of vinyl chloride per 10 mL of carbon disulfide versus peak height or peak area.

10.2.11 From the calibration curve, determine the linear range for the analysis. If the plot is linear over the range of the samples, a linear regression equation or an appropriate response factor may be used.

10.2.12 Analyze freshly prepared standards with each batch of samples.

11. Procedure

11.1 *Sampling:*

11.1.1 Break open both ends of the charcoal tube to be used for sampling, ensuring that each opening is at least one half the inside diameter of the tube.

11.1.2 Attach the previously calibrated (10.1) sample pump to the activated charcoal tube with rubber or plastic tubing, placing the backup section nearest the pump.

11.1.3 For a breathing zone sample, fasten the sampling pump to the worker and attach the sampling tube as close to the worker's breathing zone as possible. Position the tube in a vertical position to avoid channeling of air through adsorber sections. (**Warning—**Ensure that the presence of the sampling equipment is not a safety hazard to the worker.)

11.1.4 Turn on the pump, which has previously been adjusted and calibrated to provide a sampling rate of 100 mL/min for a 4-h sample time or 50 mL/min for an 8-h sample time. Note and record the time, flow rate or pump register, temperature, and barometric pressure.

11.1.5 Sample volumes up to 25-L are sufficient for determining vinyl chloride concentrations for compliance purposes. Attempting to collect larger quantities at high concentrations, high flow rates, or long sampling times may result in a "breakthrough" of vinyl chloride and loss of sample. The breakthrough volume is determined in accordance with Annex A2.

11.1.6 At the end of the sampling period, note the flow rate, turn the pump off, and note and record the time, temperature, barometric pressure, and pump register reading if appropriate.

11.1.7 Disconnect the sample tube and seal both ends with polyethylene end caps. Place identifying labels on each tube.

11.1.8 Present at least one charcoal sampling tube for analysis as a blank with every 20 or 30 samples, or for each specific inspection or field study. Break the sealed ends off the tube and cap it with the plastic caps. Do not draw air through the tube, but in all other ways treat it as an air sample.

11.1.9 Ship samples as soon as possible, store in a freezer (−20°C) until they are analyzed, and analyze within 3 weeks. If the sample tubes are shipped in ice, place them in leak proof vials.

11.1.10 Do not store samples at room temperature longer than 6 days. Migration or equilibration of the sampled material within the sampling tube during prolonged or adverse storage or handling could be confused with breakthrough.

11.1.11 If samples cannot be analyzed within 6 days or stored in a freezer, collect samples using a 1-g charcoal tube for the front section and a 150-mg charcoal tube connected in series for the back section. The tubes are separated immediately after sampling.

11.2 *Analysis:*

11.2.1 Prepare a set of vials by placing appropriate labels on them, indicating the identification number and designating whether they are front (F) section of the sample tube or the backup (B) section.

11.2.2 Pipet exactly 10.0 mL of carbon disulfide into a vial, seal, and chill in dry ice for 10 min.

11.2.3 Remove the plastic cap from the front end of the sampling tubes or score and break the tubes just above the front retainer plug.

11.2.4 Remove the plug of glass wool that holds the front portion of charcoal and add it to the vial. Slowly add the charcoal to the chilled carbon disulfide in the vial. Reseal the vial.

11.2.5 Discard the foam spacer and pour the charcoal from the back section into a second vial containing chilled carbon disulfide.

11.2.6 Desorb a previously unopened sample tube in the same manner to serve as a reagent blank.

11.2.7 Agitate at room temperature for 30 min for complete desorption.

11.2.8 Inject the sample mixture into the gas chromatograph as described in 10.2.8 or 10.2.9.

11.2.9 Determine the peak height or peak area for the vinyl chloride.

11.2.10 Calculate the concentration of vinyl chloride as directed in Section 12.

11.2.11 Analyze samples, standard solutions used for calibration, and the samples used to determine adsorption/desorption efficiencies in the same manner. Use the same injection technique and injection volume for samples and standards.

12. Calculations

12.1 Subtract the quantity of vinyl chloride found in the reagent blank (11.2.6) from the amount found in the sample.

12.2 Determine the micrograms of vinyl chloride found in the front and back-up portions of the sample tube using the calibration curve prepared in 10.2.9 or calculate directly from the standard as follows:

$$\text{VCM, µg} = \frac{A \times C}{B} \tag{2}$$

where:

A = micrograms of VCM in standard/10 mL solution,
B = peak height or area of standard, and
C = peak height or area of sample.

12.3 Sum the values of the front and back sections. If the backup section contains more than 10 % of the total amount of vinyl chloride, discard the sample as unreliable. In cases where the calculated airborne concentration exceeds the health standard, despite breakthrough, it is meaningful to report the results as greater than the calculated value.

12.4 Correct the total micrograms of VCM found for desorption efficiency as follows:

$$\text{VCM, µg/mL (corrected)} = \frac{\text{VCM found}}{E} \tag{3}$$

where E equals desorption efficiency written as a decimal (see Annex A1).

12.5 Calculate the volume of air collected for sample pumps with flow rate meters as follows:

$$\text{Sample volume, L} = \text{flow rate (L/min)} \times \text{sample time (min)} \tag{4}$$

$$\times \frac{\text{barometric pressure (kPa)}}{101.3\ \text{kPa}} \times \frac{298}{°\text{C} + 273}$$

12.6 Calculate concentration of vinyl chloride in air:

$$\text{ppm} = \frac{\text{corrected µg VCM} \times 24.47}{\text{litres of air samples} \times 62.5} \tag{5}$$

where 62.5 is the molecular weight of vinyl chloride.

12.6.1 Or milligram per cubic metre

$$\text{mg/m}^3 = \text{µg/L} = \frac{\text{corrected µg VCM}}{\text{litres of air sampled}} \tag{6}$$

12.7 Results from the field blanks (11.1.8) shall not be used to correct sample results. If a field blank shows contamination, the samples taken during the test must be assumed to be contaminated.

13. Precision [6]

13.1 *Reproducibility:*

13.1.1 Differences in the determination of vinyl chloride for duplicate samples, in the range from 0.2 to 4.0 mg per tube, should be considered suspect if they exceed $0.26 \times \bar{X}$ mg, where $\bar{X}$ is the average of the two values (95 % confidence level).

13.1.2 This statement is based on the pooled data from three laboratories and includes effects of transportation, storage, and multi-operator analysis. The 143 samples analyzed were prepared in the laboratory and may not reflect the precision of actual field samples.

14. Keywords

14.1 air monitoring; charcoal tube method; sampling and analysis; vinyl chloride; workplace atmosphere

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D22-1007. Contact ASTM Customer Service at service@astm.org.

ANNEXES

(Mandatory Information)

A1. ADSORPTION/DESORPTION EFFICIENCY

A1.1 The efficiency of the adsorption and desorption process is determined for each lot or batch of activated charcoal used.

A1.2 Sample a known concentration of vinyl chloride in air, measuring the total volume with a calibrated dry test meter or with a rotameter and a stop watch. The sample may be passed through the flowmeter and into the charcoal tube from a pressurized source, or drawn from a plastic bag through the tube and flowmeter by a sample pump. The pressure drop across the charcoal section shall not be greater than 2.5 cm Hg [3.3 kPa] at a flowrate of 1000 mL/min.

A1.3 The charcoal tubes must be from the same lot of tubes to be used for sampling.

A1.4 The flowrate of the vinyl chloride standard shall be in the range from 50 to 200 mL/min. The total volume must be less than the breakthrough volume.

A1.5 Desorption efficiency for the commercial carbon tubes is usually in the range from 90 to 100 %.

A1.6 If the field samples are to be collected under high temperature (>90°F or >35°C) and humidity (>85 % relative humidity) conditions, warm the charcoal tube to the same temperature or above, and saturate the gas stream containing the vinyl chloride with water. Breakthrough would show the need to reduce the sample volume.

A1.7 At the end of the sampling period, disconnect the charcoal tube. Seal the tube with polyethylene caps and label them. Prepare five tubes at the same sample volume that will be used to collect field samples. Three samples for each level are sufficient if the adsorption/desorption is to be tested at different levels.

A1.8 Desorb vinyl chloride from the tubes using the procedure for samples and analyze in exactly the same manner that the samples and standards are analyzed.

A1.9 Calculate the adsorption/desorption efficiency, E, as follows:

$$E = A/B \tag{A1.1}$$

where:

A = milligrams of vinyl chloride found in CS_2, and
B = milligrams of vinyl chloride in the air that passed into the tube.

A2. BREAKTHROUGH VOLUME

A2.1 The breakthrough volume is the amount of air sample that can be passed through or drawn through the charcoal adsorbent before vinyl chloride begins to be emitted from the exhaust end of the tube. The breakthrough volume is effected by high concentrations of vinyl chloride, high concentrations of other impurities in the air, and extreme conditions of humidity and temperature.

A2.2 The breakthrough volume can be determined for a specific size tube and type of charcoal. Assemble a gas train consisting of a pressurized cylinder containing a known concentration of vinyl chloride, a dry-test meter or calibrated rotameter, and a hydrogen flame ionization or equivalent, detector. Adjust the attenuation of the detector so that a concentration of 5 % of the assault concentration can easily be detected. The assault concentration of vinyl chloride should be at least as high as the maximum concentration expected.

A2.3 Pass the gas through the flowmeter at a known rate of 100 mL/min. Note the time the flow was initiated.

A2.4 When the vinyl chloride begins to emerge, the detector will show a response. Record the time when a concentration of 5 % of the assault concentration is detected in the effluent.

A2.5 Calculate the breakthrough volume by multiplying the flow rate expressed as litres per minute by the elapsed time in minutes.

A2.6 Breakthrough volumes for activated coconut charcoal are typically in excess of 25 L sample volume per gram (**1**).

A2.7 Determine the effect of moisture on the breakthrough volume by humidifying the gas stream to 100 % relative humidity. A gas-scrubbing flask or tube containing water may be used. The breakthrough volume is then determined as described above.

REFERENCES

(1) Severs, L. W., and Skory, L. K., Monitoring Personnel Exposure to Vinyl Chloride, Vinylidene Chloride and Methyl Chloride in an Industrial Work Environment, *American Industrial Hygiene Association Journal*, Vol 36, 1975, p. 669.

(2) Cuddeback, J. E., Burg, W. R., and Birch, S. R., Performance of Charcoal Tubes in Determination of Vinyl Chloride, *Environmental Science and Technology*, Vol 9, 1975, p. 1168.

(3) Hill, R. H., Jr., McCammon, C. S., Saalwaechter, A. T., Teass, A. W., and Woodfin, W. J., Gas Chromatographic Determination of Vinyl Chloride in Air Samples Collected on Charcoal, *Analytical Chemistry*, Vol 48, 1976, p. 1395.

(4) Maltoni, C., Lefemine, G., Lincei-Rendiconte Dello Classe Di Sience, Tesiche, *Mathmatische*, Vol 56, 1974, p. 1.

(5) American Conference of Governmental Industrial Hygienists, "Air Sampling Instruments for Evaluation of Atmospheric Contaminants," Fifth Edition, Cincinnati, Ohio, 1978.

(6) Lande, S. S., Measurement of Atmospheric Vinyl Chloride, *American Industrial Hygiene Association Journal*, Vol 40, 1979, p. 96.

(7) Rosen, J. D., Morano, J. R., Pareles, S. R., Giacin, J. R., and Gilbert, S. G., Analysis of Vinyl Chloride by Mass Fragmentography, *Journal of the Association of Official Analytical Chemists*, Vol 58, 1975, p. 700.

(8) Ernst, G. F., and Van Lierop, J. B. H., A Simple, Sensitive Determination and Identification of Vinyl Chloride by Gas Chromatography with a Hall Detector, *Journal of Chromatography*, Vol 109, 1975, p. 439.

(9) Krishen, A., and Tucker, R. G., Elimination of Interference of Acetaldehyde in Gas Chromatographic Quantitation of Low Levels of Vinylchloride, *Analytical Chemistry*, Vol 48, 1976, p. 455.

(10) Williams, D. T., and Miles, W. R., Gas-Liquid Chromatographic Quantitation of Low Levels of Vinylchloride, *Analytical Chemistry*, Vol 48, 1976, p. 455.

(11) Ives, N. F., Sensitive Trapping and Gas Chromatographic Method for Vinyl Chloride in Air Samples, *Journal of the Association of Official Analytical Chemists*, Vol 58, 1975, p. 272.

(12) Ahlstrom, D. H., Kilgour, R. J., and Liebman, S. A., Trace Determination of Vinyl Chloride by a Concentrator/Gas Chromatography System, *Analytical Chemistry*, Vol 47, 1975, p. 1411.

(13) Bellar, T. A., Lichtenberg, J. J., and Eichelberger, J. W., Determination of Vinyl Chloride at mg/L Level in Water by Gas Chromatography, *Environmental Science and Technology*, Vol 19, 1976, p. 926.

(14) Myers, S. A., Quinn, H. J., and Zook, W. C., Determination of Vinyl Chloride Monomer at the Sub-ppm Level Using a Personal Monitor, *American Industrial Hygiene Association Journal*, Vol 36, 1975, p. 332.

(15) Purcell J. E., Gas Chromatographic Analysis of Vinyl Chloride, *American Laboratory*, Vol 7(7), 1975, p. 99.

(16) Going, J. E., Sampling and Analysis of Selected Toxic Substances—Task III—Vinyl Chloride, Secondary Sources, PB-25-966, *National Technical Information Service*, Springfield, VA, 1976.

(17) Lao, R. C., Thomas, R. S., and Monkman, J. L., Improved Methods for Sampling and Analysis of Vinyl Chloride, *American Industrial Hygiene Association Journal*, Vol 37, 1976, p. 1.

(18) Gabany, A. G., and Senman, H., A Sealed System for the Analysis of Vinyl Chloride, *American Laboratory*, Vol 7, 1976, p. 49.

(19) McClenny, W. A., Martin, B. E., Baumgardner, R. E., Jr., Stevens, R. K., and O'Keeffe, A. E., Detection of Vinyl Chloride and Related Compounds by a Gas Chromatographic, Chemiluminescence Technique, *Environmental Science and Technology*, Vol 10, 1976, p. 810.

(20) Gay, B. W., Lonneman, W. A., Bridbord, K., and Moran, J. B., Measurements of Vinyl Chloride from Aerosol Sprays, *Ann. New York Academy of Science*, Vol 246, 1975, p. 288.

(21) Pu, H. H., and Hicks, S. Z., Determination of Vinyl Chloride Monomer by Gas Chromatography, *Journal of Chromatography*, Vol 132, 1977, p. 495.

(22) *Calibration in Air Monitoring, ASTM STP 598*, 1976.