

March 28, 1974

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RECEIVED

MAR 29 1974

R. N. WHEELER, JR.

VC Exposure

From: Mr. B. L. Murray

Subject: Measuring the Exposure of Personnel to Vinyl Chloride

To confirm our conversation relative to the analysis of Vinyl Chloride collected on carbon tubes, a rough draft of the method of sampling and analysis⁽¹⁾ is attached for your review.

Presently, the Vinyl Chloride analysis is being done by the Technical Center Environmental Health Service Laboratory. As soon as possible, we hope this analytical work could be performed by the Plant Laboratory.

A Model 513 AID Portable Chromatograph for detecting Vinyl Chloride has been recommended for purchase and will require a two (2) months delivery time.

In the meantime, we need the capability of delivering the carbon tube air samples collected to the Plant Laboratory for analysis. We expect at least twelve samples per day, five days per week, during the next 90 days.

We would appreciate your review of the method of analysis and the assurance that the air samples collected can be analyzed at the Plant Laboratory.

(1) Neff, J. E. - Vinyl Chloride - Determination in Air by Adsorption on Activated Charcoal and Analysis by Gas Chromatography.

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Attachments

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Rough Draft

VINYL CHLORIDE

DETERMINATION IN AIR BY ADSORPTION ON ACTIVATED
CHARCOAL AND ANALYSIS BY GAS CHROMATOGRAPHY

- 1 **PURPOSE AND LIMITATIONS** This paper describes a procedure for measuring the exposure of personnel to vinyl chloride in the working environment. The method as written will determine vinyl chloride in the range of 9 to 90 parts per million by volume in air, based on a 4-hour sample at a flow rate of 28 ml per minute. Lower or higher ppm ranges can be determined by preparing additional standards to cover the range desired.
- 2 **PRINCIPLE** The sample is collected by passing air through a glass tube containing activated charcoal which adsorbs any vinyl chloride vapors present. The vinyl chloride is then desorbed from the charcoal by carbon disulfide extraction and analyzed by gas chromatography.
- 3 **INSTRUMENT PARAMETERS**

Chromatograph	AID (Analytical Instrument Development, Inc.) Portable Chromatograph, Model 511, or equivalent chromatograph with flame ionization detector.
Column	Six-foot x 1/8-inch stainless steel packed with 10% di(2-ethyl hexyl) Sebacate on Chromasorb P, 80/100 mesh, NAW.
Column temperature	100°C isothermal
Injection temperature	100°C
Detector	Flame ionization detector
Carrier	Helium or nitrogen
Hydrogen flow rate	23 cc per minute
Air flow rate	133 cc per minute
Sample size	2 µl solvent flush technique
Retention time	0.5 minute (adjust carrier flow until vinyl chloride elutes at this time)

4 REAGENTS AND APPARATUS

- a) Personal sampling pump. MSA Model G or Bendix Micronair.
Insert an in-line orifice made from a cut-off and restricted (pinched) hypodermic needle in the sampling line to the pump in order to obtain a 28 cc per minute flow on these pumps.
- b) Hypo-vials, 15-ml size, Cat. No. 12911; Neoprene septa, Cat. No. 13233; alumina seals, Cat. No. 13214; and hand crimper, Cat. No. 13212, Pierce Chemical Company, Rockford, Illinois.
- c) Carbon disulfide, spectrophotometric grade
- d) Activated charcoal, 12 x 20 mesh size, Cat. No. 4580-26, Barnebey-Cheney, Columbus, Ohio
- e) Soap film flow meter, 10-ml size
- f) Stop watch

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5 PREPARATION OF THE ACTIVATED CARBON ABSORPTION TUBE

- a) Prepare the activated charcoal by placing the charcoal on a 40-mesh sieve and wash with demineralized water until no visible fines appear in the washings.
- b) Dry the carbon in a drying oven at 110°C until dry and free flowing (overnight). Store in a screw-top, glass bottle until needed.
- c) Cut 8 mm O.D. x 6 mm I.D. Pyrex glass tubing into 6-inch lengths and fire polish the ends.
- d) Insert a Pyrex glass wool plug two-thirds of the distance into the glass tubes. The longer section of tubing will be designated as the primary end, while the shorter side of the tubing is the back-up end.
- e) Pack the tubes with the activated carbon, using a length of 60 mm in the primary section and 35 mm in the back-up section. Retain the carbon with glass wool plugs.
- f) Seal the tubes with a red rubber septum or parafilm until ready for use.

6 SAMPLING PROCEDURE

- a) Remove the seals from the charcoal tube and attach the back-up section to a portable pump by means of a length of 1/4-inch Tygon tubing. THE SMALLER SECTION OF CHARCOAL IS USED AS A BACK-UP AND SHOULD BE POSITIONED NEAREST THE SAMPLING PUMP. THE CHARCOAL TUBE SHOULD BE PLACED IN A VERTICAL POSITION.
- b) Set the flow rate at 28 ml/min. through the tube with a calibrated rotameter. (NOTE: The 10-ml soap bubble flow meter can be used to obtain a more precise reading of the air flow through the tube if desired.)

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AIR BEING SAMPLED
SHOULD NOT PASS
THROUGH ANY HOSE OR
TUBING BEFORE ENTERING

c) Record the time the air sampling was started and the time when the sampling is completed. A four-hour sample may be taken with this system.

d) ^{Immediately} After sampling, recheck the flow rate and reseal the ends of the charcoal tube. ^{with the supplied plastic caps. Under no circumstances should} Return the tube to the laboratory for analysis.

e) Record the temperature and barometric pressure at the sampling site.

7 ANALYTICAL PROCEDURE

NOTE: High humidity does not present problems in sampling by this method, but the presence of airborne water in liquid form does. Where two or more solvents are known or suspected to be present in the air, and information regarding their suspected identities should be furnished with the sample, with differences in polarity, one may determine the number from the charcoal.

a) Make a small hook at the end of a piece of wire and remove the glass wool plug from the primary end of the charcoal tube. Make sure that no charcoal particles adhere to the glass wool plug.

b) Pour the charcoal into a 10-ml glass-stoppered volumetric flask, pipet 3 ml of carbon disulfide (CS_2) into the flask and stopper securely. (CAUTION: Carbon disulfide is toxic and should be handled under a hood.)

c) Agitate the flask periodically for at least 30 minutes.

d) Solvent Flush Injection Technique. This injection technique is designed to eliminate difficulties arising from blow-back or distillation with the needle of the microliter syringe.

e) Flush a 10 μ l syringe with CS_2 several times to wet the barrel and plunger.

f) Draw 2 μ l of CS_2 into the syringe and remove the tip of the needle from the solvent. Withdraw the plunger an additional 0.5 μ l to separate the CS_2 from the sample with a pocket of air.

g) Dip the needle into the sample solution in the volumetric flask and withdraw the plunger until the air bubble between the solvent and the sample has passed the 2 μ l mark on the syringe.

h) Remove the tip of the needle from the sample solution and adjust the volume in the syringe until the meniscus of the air bubble rests on the 2 μ l mark. Remove the excess sample solution from the tip of the needle.

i) Pull the plunger back an additional 0.5 μ l to prevent the sample solution from evaporating from the tip of the needle.

- j) Inject the entire contents of the syringe into the chromatograph.
- k) Measure the peak height and determine the vinyl chloride content from a previously prepared calibration curve.

8 CALIBRATION CURVE

- a) Pipet 10 ml of carbon disulfide (CS_2) into each of five 15-ml hypo-vials. Cap the vials with the Neoprene septa and aluminum seals, using the hand crimper.
- b) Place the hypo-vials in wet ice to reduce the vapor pressure of CS_2 .
- c) Cap one of the valves on a steel sample cylinder containing vinyl chloride with a 1/8-inch Swagelok tubing nut which has a chromatographic septum installed in the nut.
- d) Insert a hypodermic needle through the septum on the cylinder, open the valve and allow the vinyl chloride to vent through the needle for about 10 seconds to purge the air from the system. Remove the needle.
- e) Using the appropriate gas-tight syringe fitted with a number 27 gauge hypodermic needle, insert the needle through the septum on the cylinder and allow the pressure of the vinyl chloride to displace the volume of the syringe. Flush the syringe two times to remove any air which may have been trapped in the needle.
- f) Into the respective hypo-vials, inject 0.2, 0.3, 0.5, 1.0, and 2.0 cc of vinyl chloride using the appropriate gas-tight syringe.
- g) Shake the hypo-vials for one minute to put the vinyl chloride in solution. These standards will contain 51.0, 76.5, 128, 255, and 510 micrograms vinyl chloride per ml of CS_2 .
- h) Allow the standards to warm up to room temperature, then inject these standards into the chromatograph using the procedure described in Section 7, Paragraphs d through j. Shake the hypo-vials each time just prior to withdrawing a sample.
- i) Plot peak height versus micrograms of vinyl chloride per ml.

9 CALCULATION

$$\frac{A \times 3 \times 24.5}{L \times 62.5} = \text{vinyl chloride, ppm by volume at } 25^\circ\text{C and } 760 \text{ mm}$$

A = gm per ml of vinyl chloride read from calibration curve

L = total liters of air sample (flow rate x time)

This method was developed by the Industrial Hygiene Department at the Texas City Plant.

REFERENCES

- (1) Dees , D. E., "Vinyl Chloride Personnel Monitoring Data - Texas City Plant Summary and Method", Letter, February 25, 1974.
- (2) Reid, F. H. and Halpin, W. R., "Determination of Halogenated and Aromatic Hydrocarbons in Air by Charcoal Tube and Gas Chromatography", Amer. Ind. Hyg. Assoc. J., 29, No. 4, pp. 390-396, July-August, 1968.