

B.F.GOODRICH CHEMICAL COMPANY

Inter-Organization Correspondence

To J. D. Franklin

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Location Calvert City Laboratory

From R. L. Parrish

Subject EFFICIENCY OF CARBON ADSORPTION - CARBON DISULFIDE DESORPTION IN
AMBIENT VINYL CHLORIDE TEST PROCEDURE

Objective

Determine the carbon adsorption-carbon disulfide desorption efficiency as they relate to ambient vinyl chloride testing.

Conclusions

With limitations pointed out in the text of this report, conclusions are:

1. Efficiency of the adsorption of the ambient vinyl chloride onto the activated carbon is 99%.
2. Efficiency of the extraction of vinyl chloride off the activated carbon with carbon disulfide is 100%.

Future Course of Action

Carbon tube testing for ambient vinyl chloride, under the conditions noted herein, will assume 100% efficiency for adsorption and extraction of vinyl chloride in relation to the activated carbon.

Discussion

The vinyl chloride ambient perimeter surveillance program will employ two types of sampling.

Grab samples are to be taken on the wind rosters.

Charcoal tube samples are to be taken at stationary points over a 24 hour sampling period.

Chris Orsborn is looking for a D.C. powered pump that will sample over a 24 hour period. Until one is found, an A.C. powered Millipore pump is available.

The selected sampling rate is 1000 cc per minute of air. Over a 24 hour period, this rate will provide enough sample volume (at the expected VCl concentrations) to assure good analytical accuracy.

It is essential to know the adsorption efficiency, in respect to vinyl chloride, of the activated carbon at this sampling rate.

A laboratory experiment was carried out wherein the carbon adsorption and the carbon disulfide extraction efficiencies were determined.

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Summary of Lab Work

A sampling train is prepared using three 150 mm polyethylene tubes each containing 8.3 grams of Nuchar WV-H activated carbon. Teflon tubing is used to connect the tubes together. The sampling train is connected to a gas cylinder containing a 48 ppm vinyl chloride in air standard gas. A dry gas meter is used to measure the total sample volume. The standard gas is sampled for exactly 8 minutes at a rate of approximately 1000 cc per minute. After sampling, the tubes are extracted with carbon disulfide and analyzed in the manner used for personnel monitoring testing.

Calculations

A. Theoretical Micrograms

The theoretical total micrograms vinyl chloride adsorbed is calculated from the total volume of standard sampled multiplied by the standard concentration:

$$\text{Total } \mu\text{g VC1} = \text{volume} \times \frac{1 \text{ mole air}}{\text{molar volume}} \times \text{std. concentration} \times \text{mole Wt. VC1} =$$

$$7.924 \text{ liters} \times \frac{1 \text{ mole air}}{24.6 \text{ liters}} \times \frac{48 \times 10^{-6} \text{ mole VC1}}{1 \text{ mole air}} \times \frac{62.5 \times 10^{-6} \mu\text{g VC1}}{\text{mole VC1}} = 966 \mu\text{g VC1}$$

Where:

Volume = volume gas sampled as measured by dry gas meter =

$$0.280 \text{ cu. ft.} \times 28.3 \text{ liter/cu. ft.} = 7.924 \text{ liters}$$

$$\text{Molar volume} = 22.4 \text{ liter} \times \frac{760 \text{ mm}}{751.3 \text{ mm}} \times \frac{296^\circ}{273^\circ} = 24.6 \text{ liters}$$

$$\text{Standard concentration} = 48 \text{ mole ppm or } \frac{48 \times 10^{-6} \text{ mole VC1}}{1 \text{ mole air}}$$

$$\text{Mole Wt. VC1} = 62.5 \times 10^{-6} \mu\text{g VC1/mole VC1}$$

B. Sampling Rate

$$\text{Sampling rate} = \frac{\text{Volume sampled}}{\text{Time of sample}} = \frac{7.924 \text{ liters}}{8 \text{ minutes}} = 0.990 \text{ liters/minute or}$$

990 cc per minute

C. Analytically Determined Micrograms

The analytically determined total micrograms vinyl chloride adsorbed is calculated from the analysis of the extract of each tube. A blank (14 μg) is subtracted from the analysis of each tube. The blank is derived from the instrument response for a CS_2 extract of 8.3 grams of fresh activated carbon.

Calculation

1. Calibration of Gas Chromatograph

Method: A 48 mole ppm vinyl chloride in air mixture is the calibration standard gas. With a Precision gas tight syringe, 2.5 cc of the standard gas are injected into the instrument. The area response is recorded and calculated. This response is divided into the micrograms vinyl chloride in the standard injection to obtain the GC calibration factor.

Calculation of GC factor

$$\text{GC factor} = \frac{\text{Std. injection} \times 1 \text{ liter} \times 1 \text{ mole air} \times \text{mole conc. std.} \times \text{mole Wt. VCl}}{1000 \text{ cc} \times \text{mole volume} \times \text{Instrument response}}$$

$$= \frac{2.5 \text{ cc} \times 1 \text{ liter} \times 1 \text{ mole air} \times 48 \times 10^{-6} \text{ mole VCl} \times 62.5 \times 10^6 \text{ ug VCl}}{1000 \text{ cc} \times 24.6 \text{ liter} \times 1 \text{ mole air} \times 1 \text{ mole VCl}} \\ = 1.23 \times 10^{-5} \text{ ugs VCl/mm}^2 \quad 24837 \text{ mm}^2$$

Where:

$$\text{mole volume} = 22.4 \text{ liter} \times \frac{760 \text{ mm}}{751.3 \text{ mm}} \times \frac{296^\circ}{273^\circ} = 24.6 \text{ liter}$$

$$\text{Instrument response} = 138.6 \text{ mm} \times 1.4 \text{ mm} \times 128$$

2. Calculation of Dilution Factor

The dilution factor is the ratio of the microfilters extract injected into the instrument to the total volume of the extract.

Therefore:

$$D = \frac{1}{2 \text{ ul}} \times \frac{1000 \text{ ul}}{1 \text{ ml}} \times 50 \text{ ml} = 2.5 \times 10^4$$

3. Micrograms VCl per Tube

Calculation

$$\text{Micrograms VCl per tube} = (\text{GC factor} \times \text{response} \times \text{dilution factor}) - \text{blank}$$

Tube #1 (9-26-74)

$$\text{ugs VCl} = (1.23 \times 10^{-5} \text{ ugs/mm}^2 \times 166.4 \text{ mm} \times 0.6 \text{ mm} \times 32 \times 2.5 \times 10^4) - 14 \text{ ugs} \\ = 968 \text{ ugs}$$

Tube #2 (9-26-74)

$$\text{ugs VCl} = (1.23 \times 10^{-5} \text{ ugs/mm}^2 \times 61 \text{ mm} \times 1.3 \text{ mm} \times 1 \times 2.5 \times 10^4) - 14 \text{ ugs} \\ = 10 \text{ ugs}$$

Tube #3 (9-26-74)

$$\text{ugs VCl} = (1.23 \times 10^{-5} \text{ ugs/mm}^2 \times 48.6 \text{ mm} \times 0.9 \text{ mm} \times 1 \times 2.5 \times 10^4) - 14 \text{ ugs}$$

$$= 0 \text{ ugs}$$

4. Total Micrograms on the 3 Tubes

$$\text{Total ugs 3 tubes} = \text{ugs Tube \#1} + \text{ugs Tubes \#2} + \text{ugs Tube \#3} =$$

$$968 \text{ ugs} + 10 \text{ ugs} + 0 \text{ ugs} = 978 \text{ ugs.}$$

D. Adsorption Efficiency

In routine sampling, a single carbon tube is in the sampling train. Therefore we are interested in the vinyl chloride adsorption on the 1st tube (in the experiment). So the adsorption efficiency is here defined as the percent vinyl chloride adsorbed on the 1st tube.

Below this efficiency is calculated by two different methods.

In the first method, the percent adsorption efficiency is the ratio of the analytically determined VCl on tube #1 versus total analytically determined VCl on the 3 tubes.

In the second method, the percent adsorption efficiency is the ratio of analytically determined VCl on tube #1 versus the theoretical VCl purged through the tubes.

The two methods are in good agreement ascertaining that essentially 100% of the VCl in the air being sampled is adsorbed on the 1st tube.

Calculations

Method 1

$$\% \text{ Adsorption efficiency tube \#1} = \frac{\text{ugs VCl tube \#1}}{\text{ugs VCl on 3 tubes}} \times 100\% =$$

$$\frac{968 \text{ ugs}}{978 \text{ ugs}} \times 100\% = 99\%$$

Method 2

$$\% \text{ Adsorption efficiency tube \#1} = \frac{\text{ugs VCl tube \#1}}{\text{theoretical ugs VCl}} \times 100\% =$$

$$\frac{968 \text{ ugs}}{966 \text{ ugs}} \times 100\% = 100\%$$

E. Extraction Efficiency

The extraction efficiency is a measurement of the ability of the CS₂ solvent to "wash" the VCl off the carbon into the CS₂. It is defined herein by the following calculation:

% extraction efficiency = $\frac{\text{ugs VCI tube \#1}}{(\text{theoretical ugs VCI}) (\% \text{ adsorption eff. Method 1})} \times 100$

$$= \frac{968 \text{ ugs}}{(966 \text{ ugs}) (99\%)} \times 100\% = 101\%$$

Comments in Conclusion of Report

This experiment is intended to give a relatively precise estimate of the adsorption and extraction efficiencies. This is essential to analytical accuracy.

It is recognized that some parameters that could affect adsorption are not evaluated in this work. A high concentration of contaminants (humidity, other hydrocarbons, etc.) can decrease the efficiency.

The lab work was done on September 26, 1974.



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