

VINYL CHLORIDE SUPPLEMENT TO P&CAM #127

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This document is designed to aid the analyst in adapting the procedures of P&CAM #127, "Organic Solvents in Air" to the determination of vinyl chloride in workplace air. Since in-house research on vinyl chloride analysis is still in progress, the following information is as yet incomplete.

Sampling

In-house experiments on the capacity of the 150-mg two-section activated charcoal sampling tube suggest the following: (1) if a concentration of vinyl chloride in air of around 2 ppm (v/v) is anticipated, no more than 3.5 l of the atmosphere should be sampled; (2) if a concentration of around 50 ppm (v/v) is anticipated, sample no more than 1.2 l. Sampling flow rates of 200 ml/min or less are recommended.

Sample Storage

Samples of vinyl chloride on charcoal appear to be stable for at least 3 weeks if stored at -20°C or at least 2 days if stored at 25°C . During storage at room temperature, the vinyl chloride slowly equilibrates throughout the tube, giving the appearance of migrating to the backup section. At -20°C this equilibration is very slow.

Analytical Procedure

Vinyl chloride is desorbed from the charcoal with ~~0.5 ml~~ 1.0 ml of CS_2 . Vials having a volume of 2 ml are used (thus keeping the headspace

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above the solution small) and are capped with septa after addition of the CS₂ to the charcoal. The columns used in the analysis of vinyl chloride by GC include:

Column	Temperature(°C)	Reference
20-ft 10% SE-30 on 80-100 mesh Chromosorb W AW DMCS	60	in-house
20-ft 10% FFAP on 80-100 mesh Chromosorb W AW DMCS	65 (initial)	in-house
5-ft 5% SP-1000 on 80-100 mesh Chromosorb W AW DMCS	65 (initial)	in-house
6-ft 20% Carbowax 600 on C-22 Firebrick	?	1
6-ft Porapak Q, 80-100 mesh	30-50 (initial)	2
6-ft Carbowax 20M alkaline on 60-80 mesh Chromosorb W AW	?	3
100-ft Squalane coated capillary	30	4
100-ft 25% GE-96 silicone/75% oxybis- (2-ethyl benzonte) coated capillary	25	4
200-ft hexadecane coated capillary	25	4
10-ft 7% OV-101 on 60-80 mesh Chromosorb G AW	50	5

Using the 20-ft GC column of 10% SE-30 on Chromosorb W at 60°C, our standard curve extends as low as 0.2 ng/injection. Impurities in the CS₂ interfered with the analyses for low levels of vinyl chloride using the FFAP or SP-1000 columns.

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Standards are prepared in either of two ways. (A) Pure vinyl chloride is slowly bubbled into a tared amount of toluene (~ 5 ml) contained in a 10-ml volumetric flask. About 100-400 mg of vinyl chloride are collected in 3 min. The solution is reweighed and diluted to the mark with CS₂. Standards are prepared from this stock solution using CS₂ for the dilution. (B) A series of standard solutions are prepared by injecting known volumes of pure vinyl chloride (gas) into CS₂ in 5- or 10-ml volumetric flasks and diluting to the marks. The relative accuracies of these and other methods has not been determined.

The desorption efficiency is determined using a synthetic atmosphere of vinyl chloride in clean air contained in a Tedlar (in house) or a Seran (reference 5) bag. Known volumes of this atmosphere are sampled using the charcoal tubes and the vinyl chloride desorbed and determined by GC analysis. Concentrations thus obtained are compared with those from GC analyses of 1-ml aliquots of the synthetic atmosphere to give the desorption efficiency (DE):

$$DE = \frac{\text{weight vinyl chloride on charcoal/volume atmosphere sampled}}{\text{concentration of vinyl chloride in atmosphere}}$$

In house values of the desorption efficiency for about 300 µg of vinyl chloride on the charcoal tubes have been around 0.8. Since the desorption efficiency may differ with loading on the charcoal, desorption procedure, and charcoal batch, each laboratory should determine its own values, preferably for the loading expected if an atmosphere containing vinyl chloride at the OSHA standard were sampled.

REFERENCES

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