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Analysis of Halogenated Hydrocarbons in *trans*-1,2-Dichloroethylene by Thick-Film, Wide-Bore, Capillary Gas Chromatography

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Chemical analyses performed in support of toxicological studies must provide a thorough characterization of the impurities in the test chemical. This often includes the need for efficient chromatographic separation of compounds which are closely related to the major component and to each other and are present in the sample at low concentrations relative to the major component. The following work was performed in support of toxicological studies of trans-1,2-dichloroethylene. These studies required that several specific halogenated hydrocarbon impurities be identified and quantitated. Some of these impurities are very similar in volatility to trans-1,2-dichloroethylene. Several gas chromatographic systems have been described for the analysis of halogenated hydrocarbons in complex matrices (Hagenmaier et al. 1982; Eklund et al. 1978; Dahlgran 1981), but these systems were found to be incapable of providing the separation necessary to perform this analysis. These literature methods generally employed a thin-film, narrow-bore (0.25 mm i.d.) capillary column which did not have the capacity necessary to handle the large amount of major component needed for adequate limits of detection. The gas chromatography/mass spectrometry (GC/MS) system described herein may be adaptable to the analysis of low concentration impurities in similar technical grade solvents and especially the separation, identification, and quantitation of rapidly eluting volatile components in these solvents.

MATERIALS AND METHODS

The column used for component separation was a thick-film (3.0 µm) wide-bore (0.53 mm i.d.), fused-silica capillary column chemically bonded with Durabond-1 (J&W Scientific, Inc., Folsom, California). A Perkin-Elmer Sigma 3B gas chromatograph which was linked via a single-stage glass-jet separator to a Finnigan MAT OWA® quadrupole mass spectrometer was used. The sample was introduced into a heated (100°C) Grob injector and split after 30 sec. Helium was used as the carrier gas and was maintained at a flow rate of 10 mL/min. The oven temperature was held at 0°C for 25 min by

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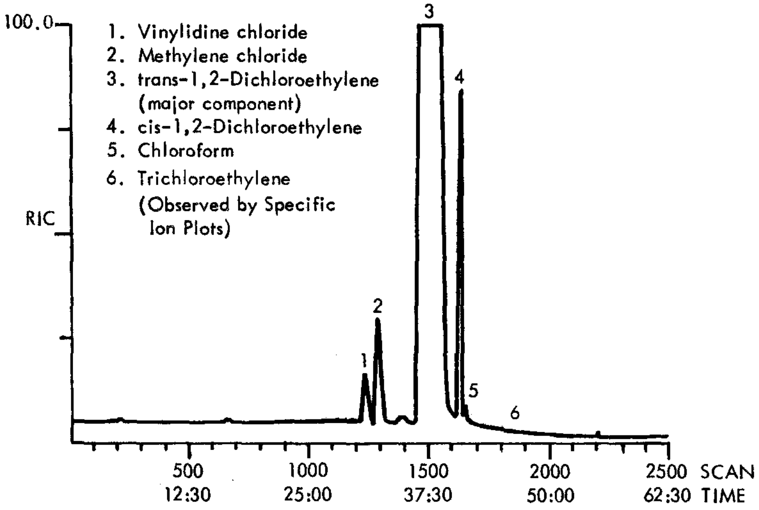

Figure 1. Reconstructed ion-current chromatogram of trans-1,2-dichloroethylene (subambient oven temperature program).

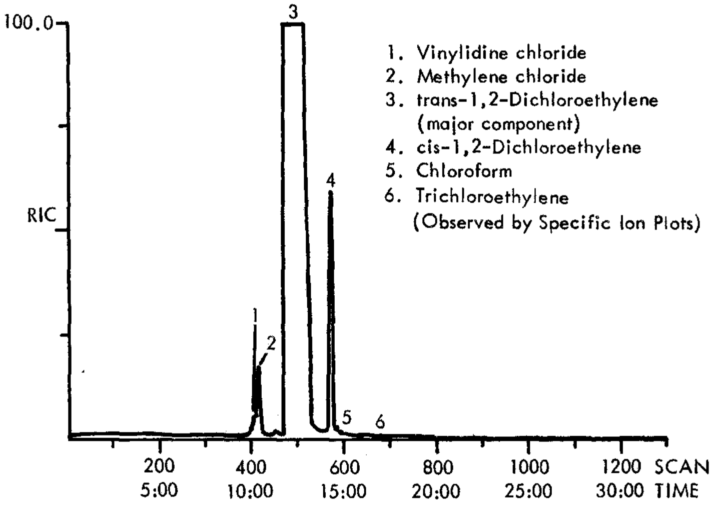

Figure 2. Reconstructed ion-current chromatogram of trans-1,2-dichloroethylene (normal oven temperature program).

using liquid CO_2 and then programmed to 250°C at 5°C/min. The data were processed by using an Incos 2400 Data System.

To determine the importance of the subambient oven temperature to the separation of components, an injection of sample was made on the same GC/MS system by using a more standard oven temperature program. The oven temperature program had an initial temperature of 35°C for 5 min before being programmed to 250°C at 5°C/min.

The sample used for this analysis was a typical industrial grade of trans-1,2-dichloroethylene. Approximately 1 µL of the neat sample was injected.

RESULTS AND DISCUSSION

The use of a thick-film, wide-bore, capillary column held at subambient temperatures was found to provide baseline resolution of all the halogenated hydrocarbons present in the sample (see Figure 1). The chromatographic separation using a normal oven temperature program (see Figure 2) provided adequate separation for identification of all the halogenated impurities, even though some were not baseline resolved, but quantitation could only be achieved by using the subambient temperature program. The halogenated hydrocarbons that were identified and quantitated as impurities in the sample of trans-1,2-dichloroethylene included vinylidine chloride, methylene chloride, cis-1,2-dichloroethylene, chloroform, and trichloroethylene. These impurities were found to be present at 0.024, 0.12, 5.1, 0.002, and < 0.0001% w/w respectively. Several other halogenated hydrocarbons including 1,2-dichloroethane, carbon tetrachloride, 1,1,2-trichloroethane, tetrachloroethylene, chlorobenzene, and 1,1,2,2-tetrachloroethane were easily resolved from the major component by using this system, but these compounds were not found to be present as impurities in the sample. The improved resolution obtained on the system described herein can be attributed to two factors. First, the greater film thickness provided increased column capacity to accommodate the amount of major component that could be placed on the column, enhancing the limits of detection of the impurities. Second, the subambient oven temperature program increased retention times and provided better separation of the volatile impurities eluting before the major component.

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REFERENCES

Hagenmaier H, Werner G, Jäger W (1982) Quantitative determination of volatile halogenated hydrocarbons in water samples by capillary gas chromatography and electron capture detection. Z. Wasser Abwasser Forsch 15:195-198

Eklund G, Josefsson B, Roos C (1978) Determination of volatile halogenated hydrocarbons in tap water, seawater and industrial effluents by glass capillary gas chromatography and electron capture detection. J High Resol Chromatogr Commun 1:34-40

Dahlgran JR (1981) Simultaneous detection of total halogenated hydrocarbons in complex environmental samples. J High Resol Chromatogr Commun 4:393-397

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