Gas chromatographic separation of carbonyl fluoride and carbon dioxide

The simultaneous quantitative evaluation of CF₂O–CO₂ mixtures is important in numerous kinetic studies including the oxidation of C₂F₄ and the pyrolysis of CF₂O. The gas chromatographic analysis of CF₂O has been reported by CORDES¹ and BANKS, HASZELDINE AND SUTCLIFFE². Unfortunately neither of the columns used can separate CF₂O–CO₂ mixtures. HEICKLEN and co-workers³,⁴ have measured CF₂O in gas mixtures by quantitatively converting the CF₂O to CO₂ on silica gel columns and measuring the CO₂ effluent. The purpose of this paper is to describe a gas chromatographic technique for the simultaneous quantitative determination of both CF₂O and CO₂.

**Experimental**

*Apparatus.* An Acrograph model No. 202-B gas chromatograph equipped with a thermal conductivity cell was used for this study. Mixtures were introduced into the gas chromatograph through a gas sampling valve used in conjunction with a 2 ml sample volume. Peak areas were measured with a Disc Integrator (5000 counts per min) which was attached to a Sargent 10 in. recorder.

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Fig. 1. Typical chromatogram of CO₂–CF₂O mixture.

Fig. 2. CF₂O peak area as a function of sample pressure.

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Column materials and preparation. A 6 ft. composite column consisting of 2 ft. of 50/80 mesh Porapak (Waters Associate, Inc.) type T followed by 4 ft. of 50/80 mesh Porapak type N was used for the analysis. The column was packed in 1/4 in. O.D. Type 316 stainless steel tubing. Before final installation in the chromatograph, the column was heated to 200° and purged with helium (60 ml/min) for 2 h. Prior to each series of runs the column was conditioned by passing three 250 torr samples of CF₂O through it.

Results and discussion

A typical chromatogram indicating the separation of CF₂O and CO₂ as obtained with the column described above is given in Fig. 1. The operating conditions corresponding to the results given in Fig. 1 are: column temperature 23°; helium flow rate 60 ml/min. A plot of CF₂O peak area as a function of CF₂O pressure in the 2 ml sample volume is shown in Fig. 2. These results indicate that the detector response is linear over an eleven fold increase in CF₂O concentration. The curve given in Fig. 2 approaches the origin as the sample pressure is decreased. This behavior indicated that CF₂O absorption on this column is essentially nonexistent.

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Synthetic diamond—A solid adsorbent for corrosive gases

A satisfactory chromatographic system for the analysis of corrosive halogen gases has proven to be an illusive objective. Recent publications¹-⁶ in this area are indicative of continuing difficulties encountered in the separation of these reactive materials. Primarily chromatographic separations have been concerned with inorganic penta- and hexa-fluorides, chlorine trifluoride and anhydrous hydrofluoric acid. No substrate or support has been recommended for gaseous mixtures containing