# Notes

CHROM. 3326

## Gas chromatographic separation of carbonyl fluoride and carbon dioxide

The simultaneous quantitative evaluation of $CF_2O$–$CO_2$ mixtures is important in numerous kinetic studies including the oxidation of $C_2F_4$ and the pyrolysis of $CF_2O$. The gas chromatographic analysis of $CF_2O$ has been reported by CORDES[1] and BANKS, HASZELDINE AND SUTCLIFFE[2]. Unfortunately neither of the columns used can separate $CF_2O$–$CO_2$ mixtures. HEICKLEN and co-workers[3,4] have measured $CF_2O$ in gas mixtures by quantitatively converting the $CF_2O$ to $CO_2$ on silica gel columns and measuring the $CO_2$ effluent. The purpose of this paper is to describe a gas chromatographic technique for the simultaneous quantitative determination of both $CF_2O$ and $CO_2$.

*Experimental*

*Apparatus.* An Aerograph 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.

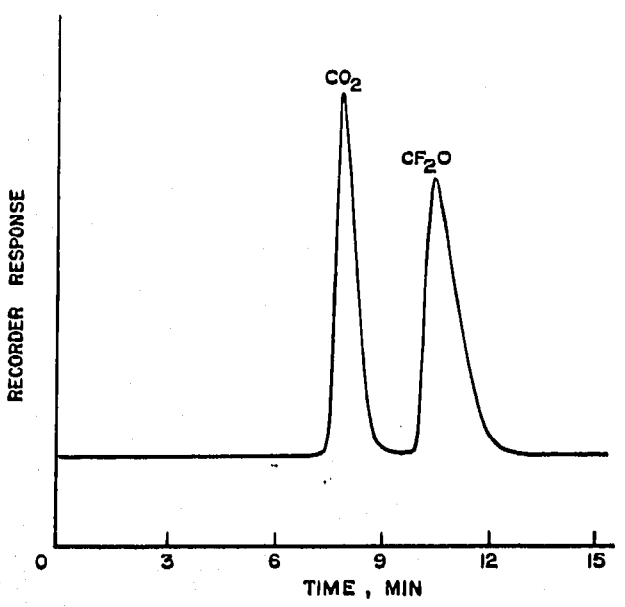


Fig. 1. Typical chromatogram of $CO_2$–$CF_2O$ mixture.

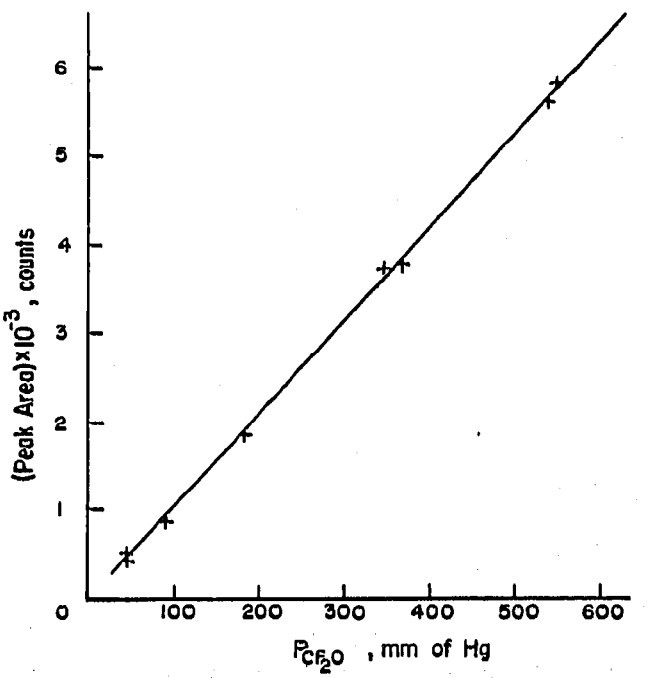


Fig. 2. $CF_2O$ peak area as a function of sample pressure.

*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 $CF_2O$ through it.

*Results and discussion*

A typical chromatogram indicating the separation of $CF_2O$ and $CO_2$ 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 $CF_2O$ peak area as a function of $CF_2O$ 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 $CF_2O$ concentration. The curve given in Fig. 2 approaches the origin as the sample pressure is decreased. This behavior indicated that $CF_2O$ 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 publications[1–5] 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