GAS CHROMATOGRAPHIC SEPARATION OF CARBONYL FLUORIDE AND CARBON DIOXIDE

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ABSTRACT

An experimental method for the quantitative analysis of both CO_2 and CF_2O in gas mixtures is described. The analysis is based on thermal conductivity gas chromatography techniques, and it is shown that the detector response is linear over a wide range of CF_2O concentrations.

INTRODUCTION

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 these columns 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/min) which was attached to a Sargent 10 in. recorder.

Column Materials and Preparation. A 6-foot composite column consisting of 2-feet of 50/80 mesh Porapak (Waters Associate, Inc.) type T followed by 4-feet of 50/80 mesh Porapak type N was used for the analysis. The column was packed in 1/4 inch O.D. Type 316 stainless steel tubing. Before final installation in the chromatograph, the column was heated to 200° C and purged with helium (60 ml/min) for 2 hours. Prior to each series of runs the column was conditioned by passing three 250 mm of Hg samples of CF_2O through it.

RESULTS AND DISCUSSION

A typical chromatogram indicating the spearation of CF_2O and CO_2 as obtained with the column described above is given in Figure 1. The operating conditions corresponding to the results given in Figure 1 are; column temperature 23° C; 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 Figure 2. These results indicate that the detector response is linear over an eleven fold increase in CF_2O concentration. The curve given in Figure 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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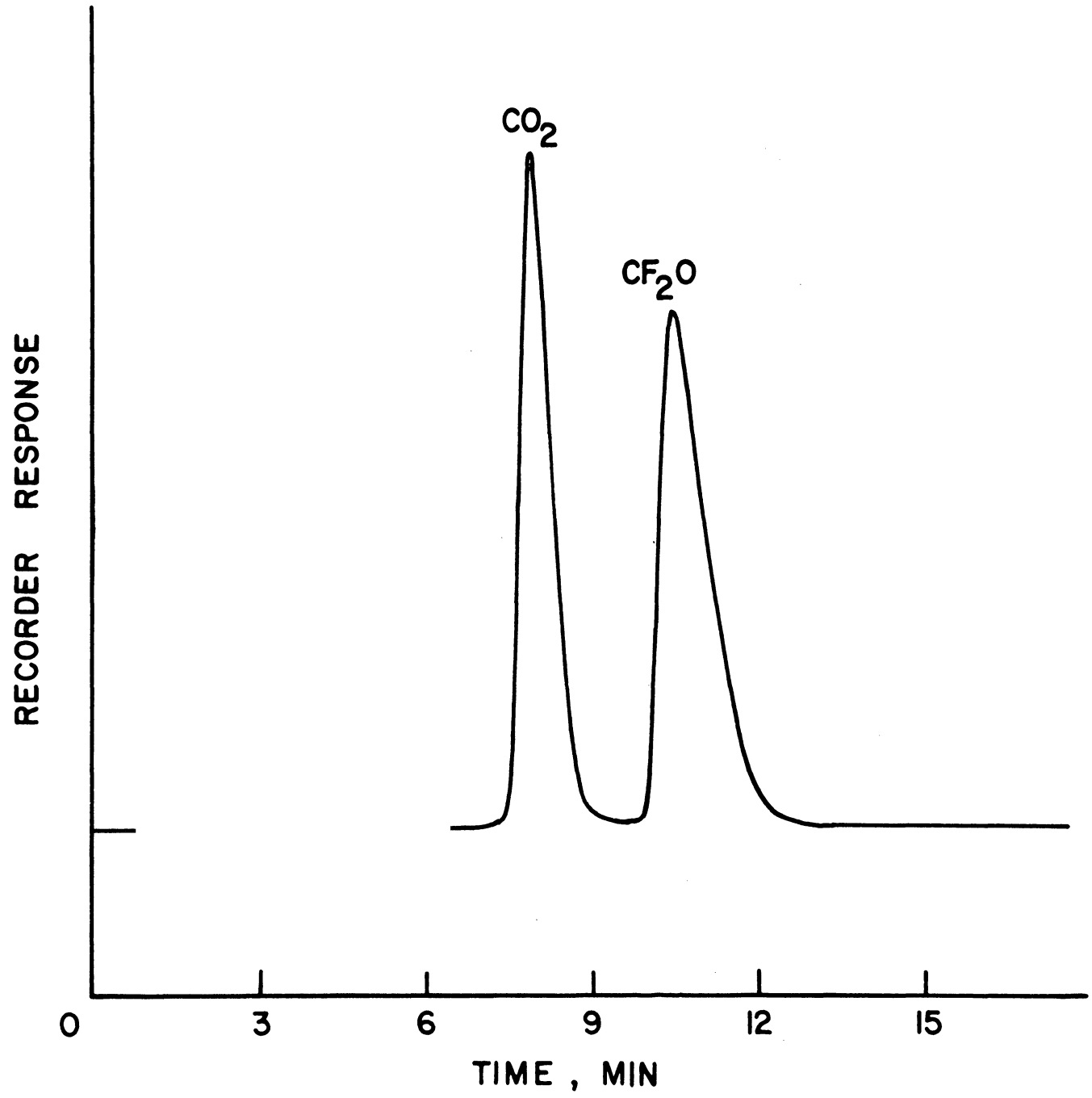

FIG.1 TYPICAL CHROMATOGRAM OF CO_2-CF_2O MIXTURE

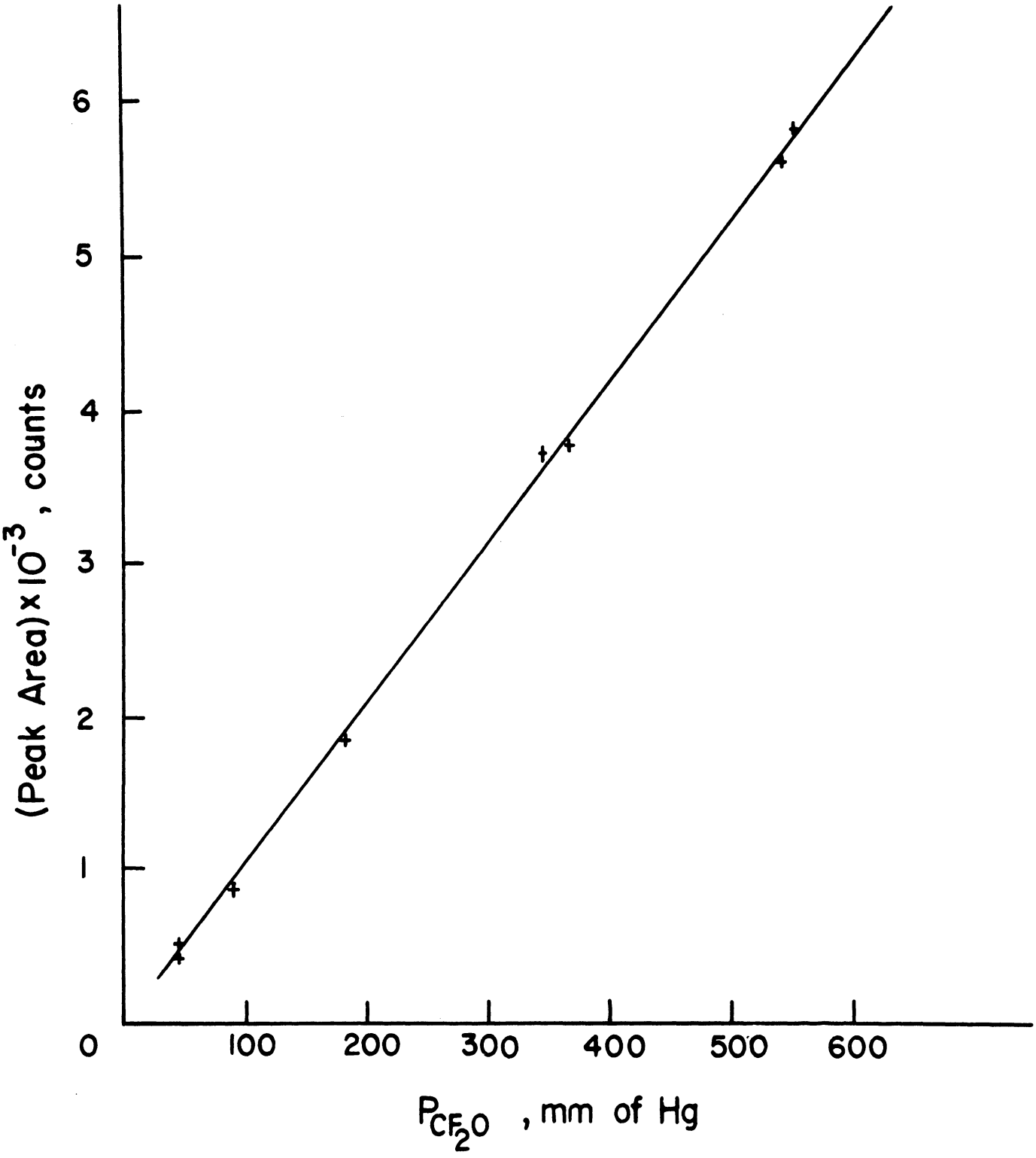

FIG.2 CF_2O PEAK AREA AS A FUNCTION OF SAMPLE PRESSURE

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