Journal of Chromatography, 104 (1975) 47–49 © Elsevier Scientific Publishing Company, Amsterdam — Printed in The Netherlands

CHROM. 7869

DUAL FLAME THERMOCOUPLE DETECTOR FOR GAS CHROMATO-GRAPHY

B. K. DESAI and P. N. SAHGAL

Chemical Engineering Department, Indian Institute of Technology, New Delhi 110029 (India) (First received June 20th, 1974; revised manuscript received August 19th, 1974)

SUMMARY

A simple, low-cost, robust and easily fabricated dual flame thermocouple detector for gas chromatography has been developed. This detector is responsive to all combustible vapours, with a sensitivity of ca. $4 \cdot 10^{-8}$ and a noise level of $2 \cdot 10^{-8}$ measured in terms of g-mole/sec of propane.

INTRODUCTION

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The object of this work was to develop a simple low-cost detector which could be fabricated in an ordinary workshop. The need for such a detector is greatest in laboratories where analyses of combustible materials by gas chromatography are frequently carried out, and where high accuracy and sophistication involving high cost are unnecessary.

As a starting point, we considered the flame thermocouple detector described by Scott¹. No further development of this simple detector has been reported, perhaps because of the progressive development and refinement of the dual flame ionization detector, which is the current detector of choice for volatile organic compounds.

Scott's detector suffers from two disadvantages: it is highy sensitive to changes in hydrogen flow-rates and a potentiometer is used to back off the standing e.m.f. generated. The dual flame principle in the differential mode (as in flame ionization detectors) was used as an improvement over Scott's detector in the present study.

EXPERIMENTAL

A schematic diagram of the detector is shown in Fig. 1. The hydrogen flame jets were made from nipples used in a domestic kerosene stove. The thermocouples (connected in opposition) were made from 24 s.w.g. chromel-constantan wires insulated with asbestos paper. The distance between the flame jets and the thermo-couple tips was adjustable so as to permit zero adjustment of the standing e.m.f. During the passage of combustible material through the active flame, an error signal was generated which was recorded on a Kent Mk. III potentiometric strip-chart recorder.



Fig. 1. Dual flame thermocouple detector.



Fig. 2. Chromatogram obtained using dual flame thermocouple detector. Sample, 0.1 ml indane; column, squalane; recorder sensitivity, 1 mV/25 cm; chart speed, 10 mm/min; carrier gas (hydrogen) flow-rate, 30 ml/min; air, 7 p.s.i. Peaks: 1, ethane; 2, propane; 3, isobutane; 4, *n*-butane.

RESULTS AND DISCUSSION

As constructed, the detector takes about 30 min to become stabilized. After this period, both the drift and noise fall to very low levels. The chromel-constantan thermocouple used has a e.m.f. response of 61 μ V/°C. At a recorder setting of 1 mV per 25 cm of chart width, the noise level observed is of the order of \pm 0.3 cm or 0.5°. This noise is due almost completely to flame noise, as observed by extinguishing both flames. A sample increasing the flame temperature by 1° can easily be observed.

The inherent linearity of this detector is limited by changes in flame geometry when larger samples of combustible vapours enter the flame. In the linear ranges, the response depends directly on the calorific value of the column effluent, which in turn depends upon the amount of combustible material present and its heat of combustion. Hence, the detector is particularly suitable for the analysis of combustible mixtures, the area of any peak being directly proportional to the mass present, after correction for the heats of combustion. A typical chromatogram recorded for a sample injection of 0.1 ml of L.P.G. (indane) is shown in Fig. 2.

The noise level for the detector was found to be of the order of $2 \cdot 10^{-8}$ g-mole/ sec of propane, and the sensitivity was $4 \cdot 10^{-8}$ g-mole/sec of propane.

Further improvements can be anticipated by thermostating the detector, using filtered air for combustion and changing the flame geometry.

ACKNOWLEDGEMENT

The authors are grateful for the critical and stimulating advice given by Dr. M. K. Sarkar during the course of this work.

REFERENCE

1 R. P. W. Scott, Nature (London), 176 (1955) 793.