Journal of Chromatography, 103 (1975) 180–181

CHROM. 7774

Note

Rapid determination of response factors for the gas chromatographic thermal conductivity detector

EUGENE F. BARRY
Department of Chemistry, Lowell Technological Institute, Lowell, Mass. 01854 (U.S.A.)
and
DOUGLAS M. ROSIE
Department of Chemistry, University of Rhode Island, Kingston, R.I. 02881 (U.S.A.)
(Received July 4th, 1974)

Previous investigations of the response of the gas chromatographic thermal conductivity detector (TCD) have shown that the use of peak area correction factors, such as relative molar response data, is essential for accurate quantitative analysis[1,2]. Guillemin *et al.*[3,4] have reported on the utilization of the gas density balance (GDB) as a calibrating detector in the determination of relative response factors for other types of detectors. Since the GDB exhibits adequate sensitivity, predictable response, and may be used with any carrier gas, it is an ideal detector for the calibration of thermal conductivity and flame ionization detectors. Vermont and Guillemin[4] employed a parallel flow system in the calibration of a flame detector in order to avoid any influence of the GDB on the detector. In the present study we have constructed a TCD–GDB detector system connected in series and maintained at 100° in the same chromatographic oven. This arrangement has greatly facilitated the determination of response factors since the GDB provides reliable weight per cent compositions of mixtures.

EXPERIMENTAL

The Gow-Mac Model 9677 TCD equipped with thermistor beads each having a resistance of 8000 ohms at 25° was connected to a Gow-Mac Model 091 gas density balance having W-2X rhenium tungsten filaments. Currents of 6 and 150 mA were delivered to the TCD and GDB, respectively, by means of constant current power supplies. Nitrogen flow-rates of 37 ml/min and 110 ml/min delivered by separate supplies were held constant by rotameters through the sensing channels of the detectors and the reference compartment of the GDB, respectively. Any desired flow through the sensing path can be selected by adjustment of the calibrated rotameter and re-establishment of the reference flow-rate; Walsh and Rosie[5] have found a reference/sensing flow ratio of 3 to be optimum with nitrogen. A schematic diagram of the entire apparatus is presented in Fig. 1. Preferably two recorders and two integrators should also be employed for more rapid data collection.

The TCD–GDB combination in series offers several attractive features. Since

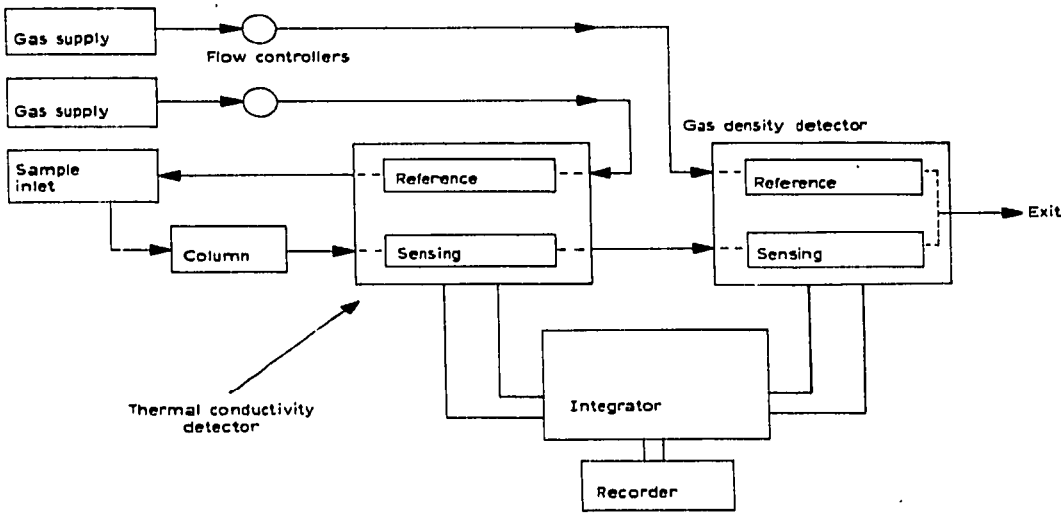

Fig. 1. Schematic diagram of TCD–GDB arrangement in series.

the GDB responds to the effluent of the TCD and not to what is injected onto the column, problems associated with purity of standards, preparation of standard mixtures and irreversible adsorption are eliminated. In addition, a series arrangement with a TCD does not require a flow divider, thermally stable micro-needle valves and/or capillary tubes.

REFERENCES

1 E. F. Barry, R. S. Fischer and D. M. Rosie, *Anal. Chem.*, 44 (1972) 1559.
2 D. M. Rosie and R. L. Grob, *Anal. Chem.*, 29 (1957) 1263.
3 C. L. Guillemin, M. F. Auricourt, J. DuCrest and J. Vermont, *J. Chromatogr. Sci.*, 7 (1969) 493.
4 J. Vermont and C. L. Guillemin, *Anal. Chem.*, 45 (1973) 775.
5 J. T. Walsh and D. M. Rosie, *J. Gas Chromatogr.*, 5 (1967) 232.