Journal of Chromatography, 329 (1985) 178–180
Elsevier Science Publishers B.V., Amsterdam — Printed in The Netherlands

CHROM. 17 769

Note

Separation of carbazole alkaloids by gas–liquid chromatography

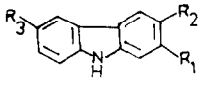
B. K. CHOWDHURY* and A. MUSTAPHA
Department of Pharmaceutical and Medicinal Chemistry, Faculty of Pharmaceutical Sciences, Ahmadu Bello University, Zaria (Nigeria)
and
P. BHATTACHARYYA
Department of Chemistry, Bose Institute, Calcutta 700009 (India)
(First received September 18th, 1984; revised manuscript received April 1st, 1985)

Since the isolation in 1965 of murrayanine[1], the first member of the carbazole alkaloids without any basic nitrogen, there has been considerable progress with this group of alkaloids and various members have been isolated[2] from some plant sources belonging to the genera *Murraya, Glycosmis* and *Clausena*. These carbazole alkaloids contain C_{13}, C_{18} and C_{23} skeletal groups. A single plant species, *Murraya koenigii* Spreng, the richest source of phytocarbazoles so far reported, has been found to give more than twenty carbazole alkaloids[2]. It was therefore of interest to develop an easy and effective method for the separation and detection of such alkaloids. The separation of these alkaloids by thin-layer chromatography has been reported earlier[3]. This paper reports a convenient method for the separation of carbazole alkaloids with C_{13}, C_{18} and C_{23} skeletons using gas–liquid chromatography (GLC).

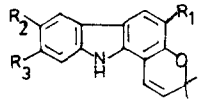
EXPERIMENTAL

Substances

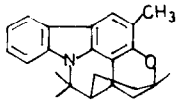
Carbazole (I) and eight carbazole alkaloids (II–IX) were used.



Carbazole (I)	$R_1 = R_2 = R_3 = H$
3-Methylcarbazole (II)	$R_1 = R_3 = H$; $R_2 = CH_3$
Glycozoline (III)	$R_1 = H$; $R_2 = CH_3$; $R_3 = OCH_3$
Mukonal (IV)	$R_1 = OH$; $R_2 = CHO$; $R_3 = H$
Glycozolidine (V)	$R_1 = R_3 = OCH_3$; $R_2 = CH_3$



Heptazolidine (VI)	$R_1 = OCH_3$; $R_2 = CH_3$; $R_3 = H$
Koenimbine (VII)	$R_1 = CH_3$; $R_2 = OCH_3$; $R_3 = H$
Koenidine (VIII)	$R_1 = CH_3$; $R_2 = R_3 = OCH_3$



Murrayazoline (IX)

0021-9673/85/$03.30

Solution

A mixture containing 1 mg of each sample was dissolved in chloroform (0.5 ml) and aliquots (3 μl) were injected into the GLC system.

Apparatus and conditions

A Pye Unicam 204 Series gas chromatograph equipped with a flame ionization detector and a temperature programmer were used. Pre-packed columns, consisting of 3% OV-17 and 3% SE-30 on Gas-Chrom Q packed in 1.5 m × 2.0 mm I.D. glass columns previously conditioned at 300°C for 16 h and silanized with three 5-ml volumes of hexamethyldisilazane, were employed. The carrier gas (nitrogen) flow-rate was 50 ml/min and the hydrogen and air inlet pressures were 27 and 15 p.s.i., respectively, giving flow-rates of 24 and 500 ml/min, respectively.

RESULTS AND DISCUSSION

Fig. 1 shows the separation of the alkaloids on the 3% OV-17 column and Fig. 2 that on the 3% SE-30 column.

The OV-17 column, being slightly more polar than the SE-30 column, requires a higher elution temperature. The order of elution in both columns is identical. With the SE-30 column the peak corresponding to mukonal (IV) was very small. However,

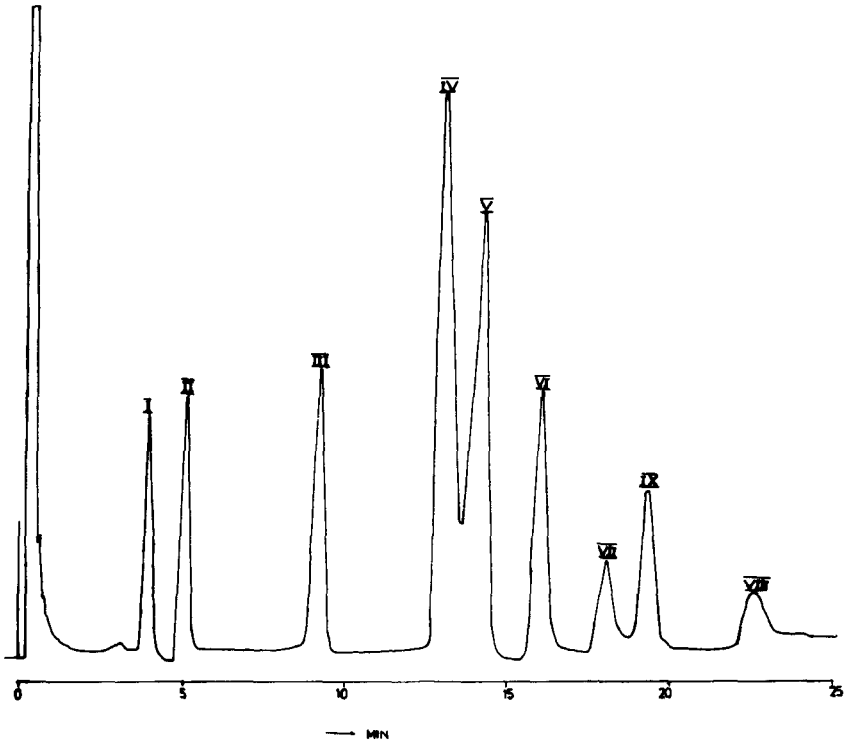


Fig. 1. Gas–liquid chromatogram of a mixture of carbazole (I) and eight carbazole alkaloids (II–IX) on 3% OV-17. Column temperature, programmed from 220 to 295°C at 4°C/min after a 3-min initial delay; detector and injector temperature, 300°C.

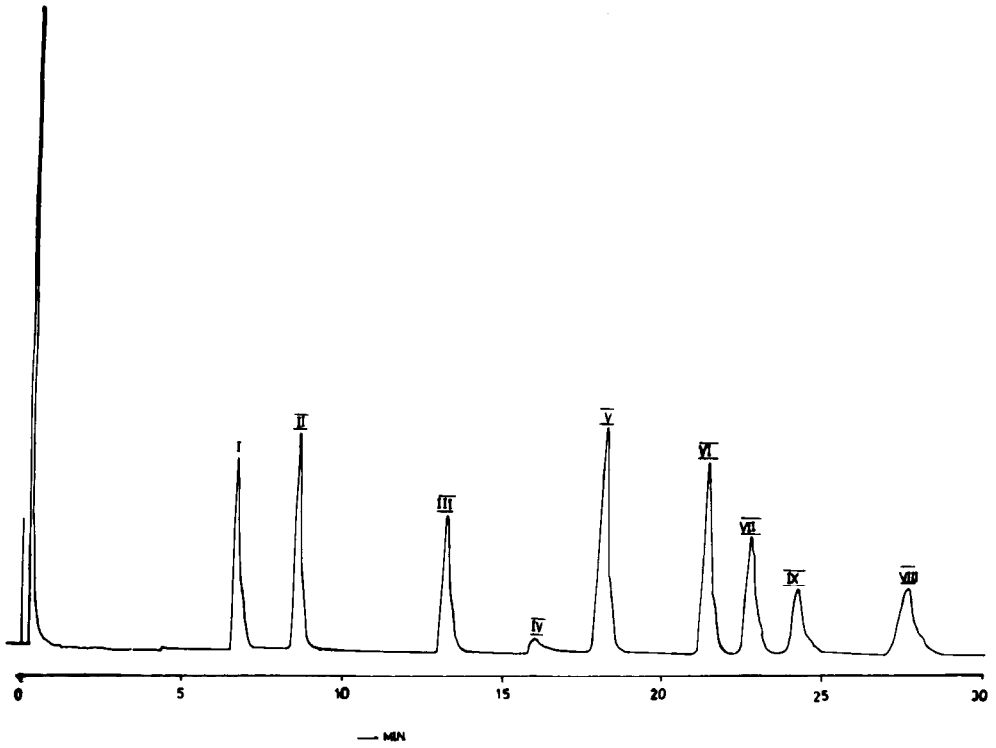


Fig. 2. Gas–liquid chromatogram of a mixture of carbazole (I) and eight carbazole alkaloids (II–IX) on 3% SE-30. Column temperature, programmed from 150 to 245°C at 4°C/min after a 3-min initial delay; detector and injector temperature, 300°C.

no decomposition of mukonal was found to have occurred in the SE-30 column at temperatures up to 230°C.

An examination of the neutral fraction of the light petroleum extract of the root bark of *Glycosmis pentaphylla* (Retz) DC on both OV-17 and SE-30 columns using the present technique showed that glycozoline (III) and glycozolidine (V), the two alkaloids present in the plant, could be detected in the chromatograms. Further work on the analysis of plant extracts and on quantitative aspects of the separation is in progress.

GLC provides a method for the resolution and identification of complex mixtures of carbazole alkaloids. It may be mentioned here that degradation of carbazole alkaloids by zinc dust distillation is a common technique for establishing the presence of the carbazole or 3-methylcarbazole skeleton. This degradation furnishes carbazole or 3-methylcarbazole or both, which can be easily detected using the present technique.

REFERENCES

1 D. P. Chakraborty, B. K. Barman and P. K. Bose, *Tetrahedron*, 21 (1965) 681.
2 D. P. Chakraborty, *Fortschr. Chem. Org. Naturst.*, 34 (1977) 299.
3 S. Roy and D. P. Chakraborty, *J. Chromatogr.*, 96 (1974) 266.