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Thin-layer chromatography of carbazole derivatives

During structural studies of strychnine, PAUSACKER AND ROBINSON[1] observed that the detection of carbazole and 3-methyl carbazole in a mixture offered considerable difficulties. They could only be detected with the help of the IR spectrum of the mixture[2]. Recent investigations on the carbazole alkaloids[3,4] of the family Rutaceae necessitated the separation and isolation of carbazole and its C-methyl derivatives from a mixture of degraded products. The occurrence of these carbazole alkaloids in the taxonomically related genera of plants of the family Rutaceae provides a scope for working out a suitable method for the separation and detection of these groups of compounds.

With a view to separating carbazoles from methyl carbazoles, CHAKRABORTY *et al.*[5] subjected these compounds to paper chromatography. Previous work in this area was carried out with hydroxy carbazoles[6]. A clear-cut separation of the constituents

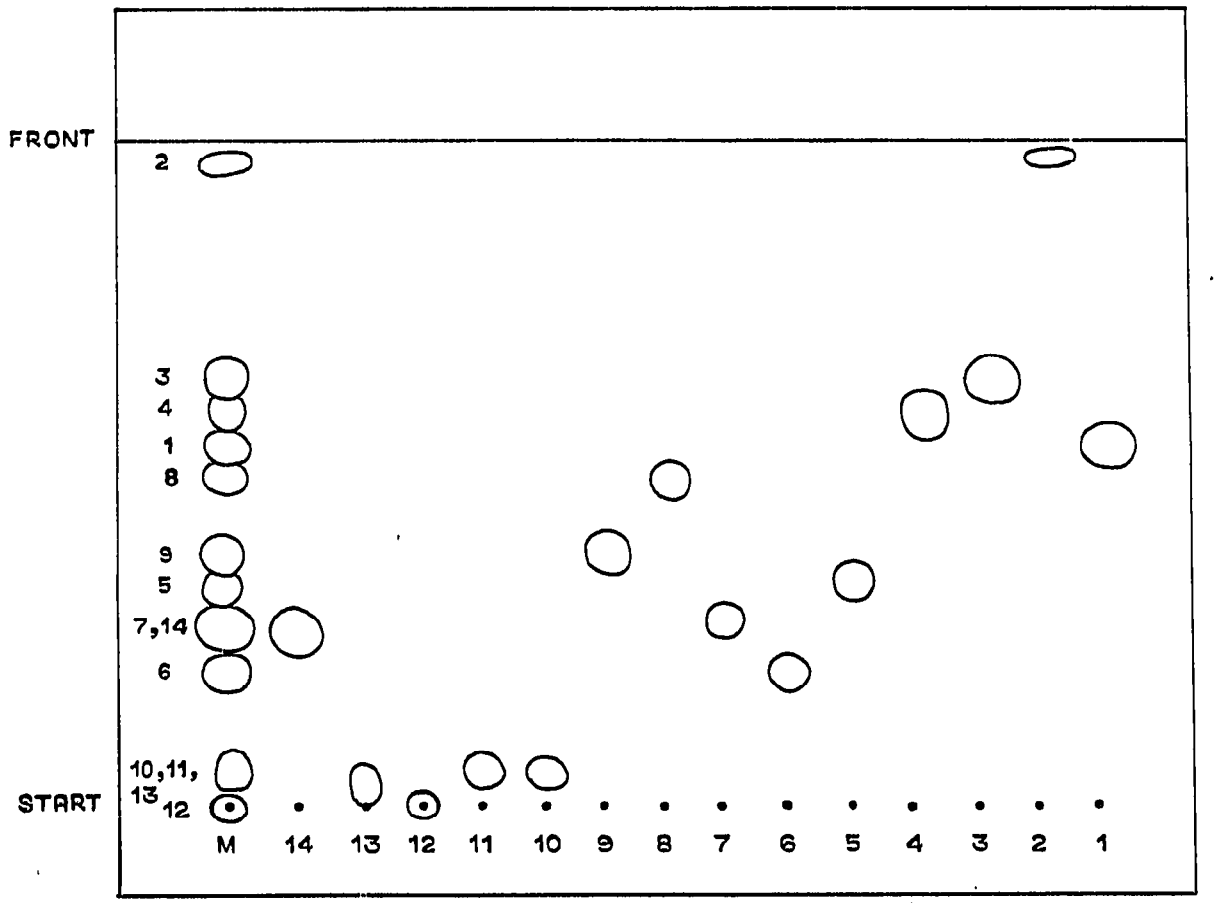

Fig. 1. Thin-layer chromatogram of carbazole compounds. Adsorbent: Aluminium Oxide G; solvent system: petroleum ether (40–60°)–chloroform (10:1). 1 = carbazole; 2 = N-methyl carbazole; 3 = 2-methyl carbazole; 4 = 3-methyl carbazole; 5 = 3-methyl 1,2,3,4-tetrahydrocarbazole; 6 = 2-methoxy carbazole; 7 = 3-methoxy carbazole; 8 = 4-methoxy carbazole; 9 = 3-methoxy-2-methyl carbazole; 10 = 3-formyl-1,4-dimethyl carbazole; 11 = 3-formyl-1,4,6-trimethyl carbazole; 12 = 3,6-diformyl carbazole; 13 = 2-hydroxy carbazole 3-methyl carboxylate; 14 = 3,6-dibromocarbazole; M = mixture of 1–14.

from a mixture could not be accomplished by paper chromatography[5]. We now report a more convenient method for the separation of a group of simple carbazole derivatives using the technique of thin-layer chromatography.

Fourteen compounds, carbazole and its derivatives, were studied on both Silica Gel G and Alumina G (E. Merck) plates of 0.25 mm thickness activated for 1 h at 110°. A large number of both polar and nonpolar solvent systems were tried. The developments were allowed to continue up to 15 cm over the line of application. For the detection of the spots the developed chromatograms were lightly sprayed with 2 *N* alcoholic sodium hydroxide solution and viewed under UV light. The spots were also detected by spraying the same chromatogram with a saturated solution of picric acid in benzene containing 1% acetic acid. Brick red to chocolate spots appeared against the yellow background within 10–15 min. Of the different solvents tried, the petroleum ether (40–60°)–chloroform (10:1) mixture was used to achieve a complete separation of carbazole, isomeric methyl carbazoles and methoxy carbazoles on an alumina plate. From the typical chromatogram shown in Fig. 1 it can be seen that this solvent system could not separate the formyl carbazole derivatives. These can be separated better, but

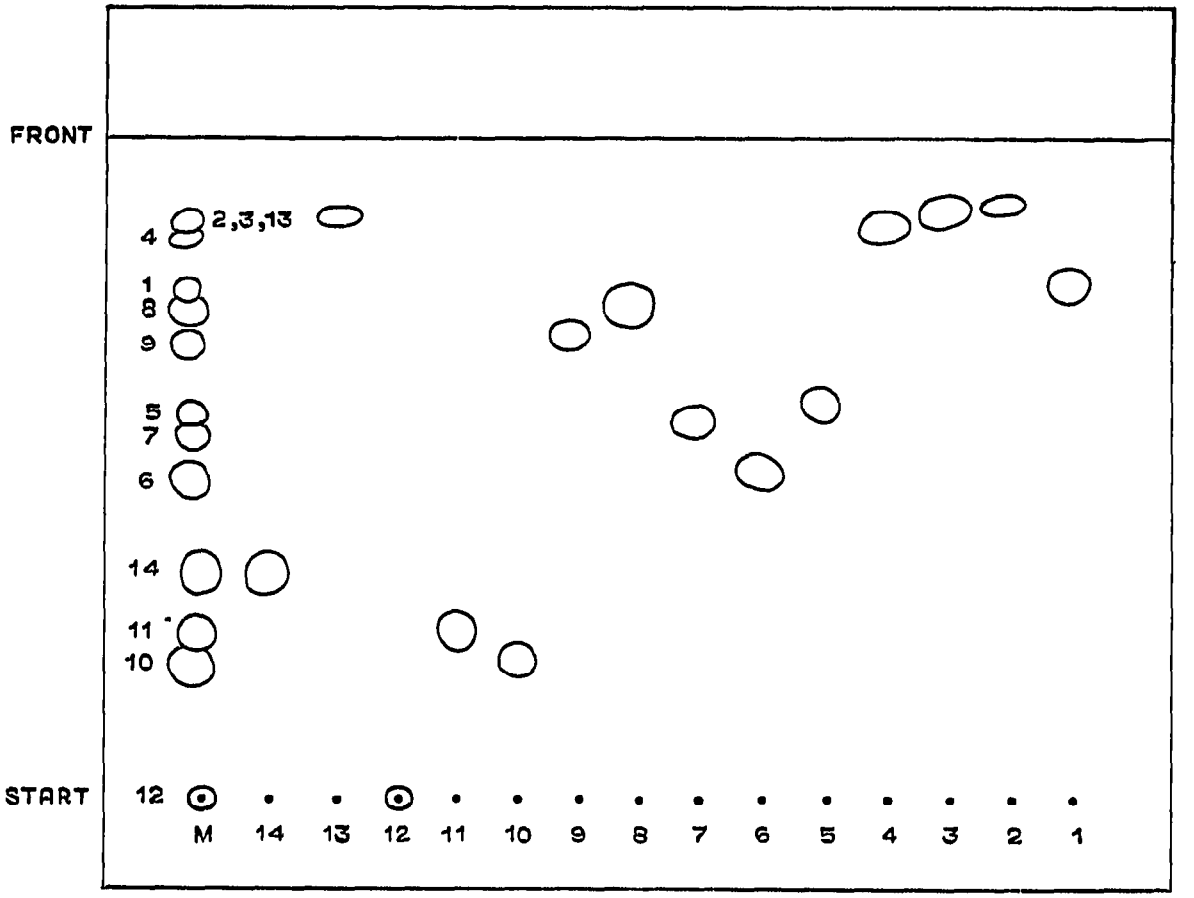

Fig. 2. Thin-layer chromatogram of carbazole compounds. Adsorbent: Aluminium Oxide G; solvent system: petroleum ether (40–60°)–acetic acid. 1 = carbazole; 2 = N-methyl carbazole; 3 = 2-methyl carbazole; 4 = 3-methyl carbazole; 5 = 3-methyl-1,2,3,4-tetrahydrocarbazole; 6 = 2-methoxy carbazole; 7 = 3-methoxy carbazole; 8 = 4-methoxy carbazole; 9 = 3-methoxy-2-methyl carbazole; 10 = 3-formyl-1,4-dimethyl carbazole; 11 = 3-formyl-1,4,6-trimethyl carbazole; 12 = 3,6-diformyl carbazole; 13 = 2-hydroxy carbazole 3-methyl carboxylate; 14 = 3,6-dibromocarbazole; M = mixture of 1–14.

with a corresponding loss in the resolution of methyl carbazoles, on an alumina plate using a mixture of petroleum ether (40–60°)–acetic acid (10:1) (Fig. 2).

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