Journal of Chromatography, 147 (1978) 513–515

CHROM. 10,324

Note

Cactaceae alkaloids

XXVIII. High-performance liquid chromatography of isomeric Cactaceae alkaloids and related tetrahydroisoquinolines

JAN STRÖMBOM and JAN G. BRUHN

Department of Pharmacognosy, Faculty of Pharmacy, Biomedicum, Box 579, S-751 23 Uppsala (Sweden)

(Received June 13th, 1977)

Recent studies of Cactaceae alkaloids have successfully employed thin-layer[1–3] and gas chromatographic[4–6] procedures for the separation and identification of alkaloids. Difficulties occur, however, in attempting complete separation of some isomeric pairs of the major alkaloid groups: tetrahydroisoquinolines and phenethylamines[5]. The main objective of the present study was to develop a high-performance liquid chromatographic (HPLC)[7] method to overcome these difficulties.

EXPERIMENTAL

The analyses were carried out on a liquid chromatograph consisting of the following parts: a Waters 6000 pump, a Waters U6K universal injector, and a Varian Variscan 635 UV detector (8 μm cell; measuring wavelength 254 nm). Two stainless-steel columns (a and b; each 30 cm × 4.5 mm I.D.) equipped with Swagelok connections and Altex stainless-steel frits (2 μm) were interconnected. The columns were packed with LiChrosorb Si 60 (10 μm) (Merck, Darmstadt, G.F.R.) (a) and μPorasil (8 μm) (Waters, Frankfurt/M, G.F.R.) (b). The balanced-density slurry technique was used for filling the columns[8]. The solvents used were *pro analysi* grade (Merck). The analyses were carried out at a flow-rate of 2.0 ml/min and a pressure of 1100 p.s.i. for solvent system I (acetonitrile–conc. ammonia, 96:4) and a flow-rate of 1.0 ml/min at 900 p.s.i. for solvent system II (chloroform–1% conc. ammonia in methanol, 9:1).

RESULTS AND DISCUSSION

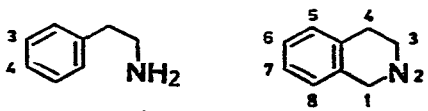
Table I summarizes the liquid chromatographic data for 11 cactus alkaloids and five closely related tetrahydroisoquinolines (general structures, Fig. 1). The latter compounds may be expected to occur in cacti on biogenetic grounds. Each pair of isomers can be clearly separated by HPLC. The separation of a mixture of salsoline, isosalsoline, and a third isomer, arizonine, is shown in Fig. 2. As had been observed in HPLC analysis of other alkaloids[9], addition of alkali to neutral solvent systems resulted in better separations. Tailing was considerably reduced and the alkaloids were eluted much faster. The analytical systems used also permit semi-preparative separations.

TABLE I

LIQUID CHROMATOGRAPHIC DATA FOR CACTACEAE ALKALOIDS AND RELATED COMPOUNDS

*Alkaloid**	*Retention time (min) and k' in solvent system: (t_o = benzene)*			
	I		*II*	
	t_R	k'	t_R	k'
Phenethylamines				
3-Hydroxy-4-methoxyphenethylamine	23.2	6.48	—**	—
4-Hydroxy-3-methoxyphenethylamine	14.4	3.64	—**	—
Tetrahydroisoquinolines				
Salsoline (6-hydroxy-7-methoxy-1-methyl-)	15.0	3.69	—**	—
Isosalsoline*** (7-hydroxy-6-methoxy-1-methyl-)	17.5	4.47	—**	—
Arizonine (8-hydroxy-7-methoxy-1-methyl-)	11.7	2.66	—**	—
O-Methylcorypalline (N-Methyl-6,7-dimethoxy-)	5.2	0.63	11.1	0.79
N-Methyl-7,8-dimethoxy-1,2,3,4-tetrahydroisoquinoline§	3.9	0.22	8.8	0.42
N-Methylsalsoline§	7.7	1.31	33.9	4.38
N-Methylisosalsoline§	7.6	1.48	31.8	4.00
Hydrocotarnine§ (N-methyl-8-methoxy-6,7-methylenedioxy-)	3.9	0.22	9.0	0.45
N-Methyl-6-methoxy-7,8-methylenedioxy-1,2,3,4-tetrahydroisoquinoline§	3.9	0.22	8.0	0.3
Pellotine (1,2-dimethyl-8-hydroxy-6,7-dimethoxy-)	7.4	1.31	29.0	3.60
Gigantine (1,2-dimethyl-5-hydroxy-6,7-dimethoxy-)	7.1	1.22	22.8	2.70
6,7-Dimethoxy-1,2,3,4-tetrahydroisoquinoline***	10.2	2.19	41.5	5.59
Salsolidine (6,7-dimethoxy-1-methyl-)	8.3	1.59	37.4	4.94
Carnegine (1,2-dimethyl-6,7-dimethoxy-)	5.4	0.69	17.8	1.82

* General structures in Fig. 1.
** $t_R > 40$ min.
*** Recently isolated by us from *Pachycereus pecten-aboriginum* (Eng.) Br. & R.
§ Not identified in cacti.



A B

Fig. 1. General structures of phenethylamines (A) and tetrahydrosioquinolines (B).

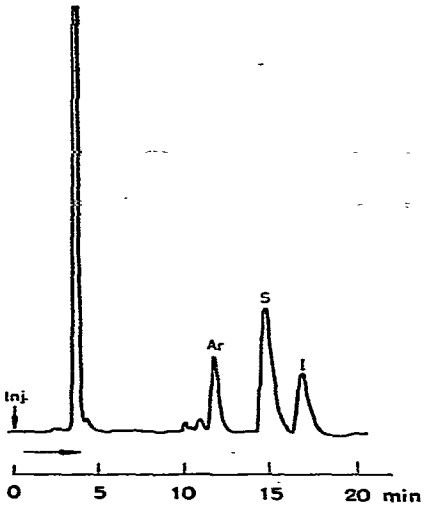


Fig. 2. Chromatogram of salsoline (S), isosalsoline (I), and arizonine (Ar) in solvent system I (see text).

ACKNOWLEDGEMENTS

We are grateful to Professor J. M. Bobbitt and Drs. A. Brossi and H. Dutschewska for samples of reference compounds.

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