

ISOLATION OF DEHYDROMOSKACHAN C FROM  
*RUTA CHALEPENSIS* VAR. *LATIFOLIA*

AYHAN ULUBELEN\*

Faculty of Pharmacy, University of Istanbul, Istanbul, Turkey

and HÜSEYİN GÜNER

Faculty of Sciences, University of Ege, Izmir, Turkey

In a previous study with the roots of *Ruta chalepensis* var. *latifolia* (Salisb.) Fiori (Rutaceae) we have obtained graveoline, graveolinine, kokusaginine, dictamine, 1-hydroxy-*N*-methylacridone, and the new alkaloid 5-methoxydictamine, as well as the coumarins chalepentin, rutacultin, and isopimpinellin (1). In addition to the above named compounds, 1-hydroxy-*N*-methylfuroacridone, chalepin, marmasin, bergapten, xanthatoxin, angustifolin 7-methyl ether, as well as two shikimic acid derivatives, moskachan D and the new compound, dehydromoskachan C [1] have now been isolated from the CHCl<sub>2</sub> extract of the aerial parts of the same plant. Similar aromatic compounds, moskachan A, B, C, and D, have been obtained previously from *Ruta angustifolia* (2).

The new compound 1 showed characteristic aromatic peaks in its uv spectrum at 287 nm and 234 nm. <sup>1</sup>H-nmr spectra clearly indicated its structure. The aromatic proton signals at δ 6.71 (1H, d, *J* = 7.8 Hz, H-6), 6.64 (1H, dd, *J* = 7.8 Hz and 1.6 Hz, H-5), 6.58 (1H, d, *J* = 1.6 Hz, H-3), and the dioxymethylene group signal at δ 5.95 (2H, s) were similar to those of the moskachans (2). The lack of a methyl signal in the spectrum and the presence of vinylic

protons at δ 5.81 (1H, dd, *J* = 10 Hz and 17 Hz, H-5'), 5.02 (1H, dd, *J* = 1.6 Hz and 17 Hz, H-6'), and 4.95 (1H, dd, *J* = 1.6 Hz, H-6') indicated the presence of an end vinylic group; therefore, the loss of water from moskachan C [2] had occurred between C<sub>5</sub>' and C<sub>6</sub>'. Methylene peaks were at δ 2.5 (2H, t, *J* = 7.4 Hz, C<sub>4</sub>'), 2.0 (2H, q, *J* = 7.3 Hz, C<sub>4</sub>'), and 1.5 (4H, br t, C<sub>2</sub>' and C<sub>3</sub>'). The mass spectrum indicated a molecular formula of C<sub>13</sub>H<sub>16</sub>O<sub>2</sub> ([M]<sup>+</sup> 204, 70%), and the base peak at *m/z* 135 (C<sub>8</sub>H<sub>7</sub>O<sub>2</sub>) was characteristic for the moskachan-type compounds (2).

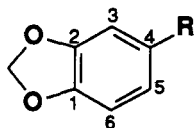
The known alkaloids and coumarins were identified by spectral data (uv, ir, <sup>1</sup>H nmr, and ms) as well as by direct comparison with authentic samples, except for moskachan D and angustifolin 7-methyl ether; spectral data of these were compared to those in the literature (2,3).

## EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—Uv spectra were recorded on a Varian Techtron model 635 instrument in MeOH; ir on a Perkin-Elmer 577 using CHCl<sub>3</sub>; <sup>1</sup>H nmr on a Bruker FT 200 MHz using CDCl<sub>3</sub>; ms on a Varian MAT 711. Kieselgel 60F 254 (E. Merck) tlc plates were used for preparative separation.

PLANT MATERIAL.—Seeds of *R. chalepensis* var. *latifolia* were cultivated as described in a previous paper (1). A voucher specimen (No. 2025) was deposited in the Herbarium of the Faculty of Sciences, University of Ege, Izmir, Turkey.

EXTRACTION AND FRACTIONATION.—Dried, powdered aerial parts (2.4 kg) were extracted with CHCl<sub>3</sub> in a Soxhlet apparatus. After filtration and drying in vacuo, 40 g of residue was obtained; 20 g of this residue was used for isolating the compounds.



- 1 R = (CH<sub>2</sub>)<sub>4</sub>CH=CH<sub>2</sub>  
2 R = (CH<sub>2</sub>)<sub>4</sub>CHOH-CH<sub>3</sub>

SEPARATION AND ISOLATION OF THE COMPOUNDS.—The  $\text{CHCl}_3$  concentrate (20 g) was fractionated in a Si gel column ( $4 \times 50$  cm), eluting with  $\text{C}_6\text{H}_6$ . A gradient of  $\text{CHCl}_3$  was added up to 100%, and then EtOH to 100%. Fractions were further cleaned and/or separated on preparative tlc plates. The yields of the compounds, calculated from the crude extract, were as follows: arborinine (250 mg, 1.25%), graveoline (5 mg, 0.025%), chalepentin (1 g, 5%), chalepin (10 mg, 0.05%), marmasin (10 mg, 0.05%), bergapten (15 mg, 0.075%), 1-hydroxy-N-methylacridone (10 mg, 0.05%), skimmianine (50 mg, 0.25%), 1-hydroxy-N-methylfuroacridone (5 mg, 0.025%), moskachan D (10 mg, 0.05%), dehydromoskachan C (10 mg, 0.05%), angustifolin 7-methyl ether (10 mg, 0.05%), xanthatoxin (5 mg, 0.025%).

DEHYDROMOSKACHAN C [1].—Uv  $\lambda$  max 287 nm ( $\log \epsilon$  3.60), 234 nm ( $\log \epsilon$  3.70); ir  $\nu$  max 3050, 2900, 2840, 2770, 1635, 1605, 1520, 1480, 1430, 1355, 1240, 1180, 1040,

990, 930, 900, 850, 800, 720  $\text{cm}^{-1}$ .  $^1\text{H}$  nmr given in the text; eims  $m/z$  (rel. int.)  $[\text{M}]^+$  204 ( $\text{C}_{13}\text{H}_{16}\text{O}_2$ ) (70),  $[\text{M} - \text{C}_3\text{H}_5]^+$  163 (22),  $[\text{M} - \text{C}_4\text{H}_7]^+$  149 (25),  $[\text{C}_8\text{H}_7\text{O}_2]^+$  135 (100), 91 (15), 77 (22), 51 (11).

#### ACKNOWLEDGMENTS

The authors wish to thank Prof. Dr. F. Bohlmann, Berlin, West Germany, for mass spectra and TUBITAK, Gebze, Turkey, for  $^1\text{H}$ -nmr spectra.

#### LITERATURE CITED

1. A. Ulubelen, H. Güner, and M. Çetindağ, *Planta Med.*, (in press).
2. J.B. del Castillo, M. Secundino, and F. Rodriguez Luis, *Phytochemistry*, **25**, 2209 (1986).
3. J.B. del Castillo, F. Rodriguez Luis, and M. Secundino, *Phytochemistry*, **23**, 2095 (1984).

Received 7 March 1988