THE ISOCOUMARIN ARTEMIDIN FROM ARTEMISIA DRACUNCULUS

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Khimiya Prirodnykh Soedinenii, Vol. 6, No. 4, pp. 467–468, 1970
UDC 547.588.25

By the successive chromatographic separation of the chloroformic extract of the epigeal part of *Artemisia dracunculus* L. (tarragon) [1] on alumina and silica gel we have obtained a new lactone $C_{13}H_{12}O_2$ with mp 49–50° C, mol wt 200 (mass spectrometry), and we have called it "artemidin" (I).

On a thin layer of silica gel in petroleum ether–ether (9 : 1) and benzene systems, substance I gives a single spot in each case with R_f 0.5 and 0.6, respectively.

The UV spectrum of artemidin has absorption bands at 234, 243, 252, 280, 303, and 340 mμ (log ε 4.58, 4.54, 4.52, 4.60, 4.57, and 4.16, respectively).

The IR spectrum of I (in chloroform) has absorption bands at (cm^{-1}) 1730 (lactone group), 1660 (double bond in a lactone ring), 1625, 1570, and 1490 (aromatic ring), and 970 (trans disubstituted double bond). In the IR spectrum of I taken in paraffin oil, in addition to the bands mentioned above, a band appears clearly at 765 cm^{-1} (1, 2-disubstituted benzene ring).

Artemidin dissolves in caustic alkalis on heating, but is not re-formed when the solutions are acidified.

In the NMR spectrum of I (taken on a JNM-4H-100/100 MHz instrument in $CDCl_3$), in the aromatic region there are signals at τ 1.90 ppm, one-proton quartet with J_{ortho} = 7.5 and J_{meta} = 1.5 Hz, and a three-proton multiplet with a center at τ 2.50 ppm. The paramagnetic shift by 0.6 ppm of the signal of one of the protons (τ 1.90) of the benzene ring relative to the other aromatic protons shows that there must be a carbonyl group in the γ-position with respect to this proton [2]. Since with a value of ν_{CO} of 1730 cm^{-1} a chromone skeleton is impossible [3], the second oxygen atom may be located adjacent to the carbonyl group. In addition, since the IR spectrum of I contains the band of a 1, 2-disubstituted benzene ring, I has an isocoumarin skeleton. The singlet at 3.88 ppm (1H) relates to a proton at C_4 of an α-pyran ring [4]. Consequently, in artemidin the side chain is located at C_3.

In the olefin region, in addition to the C_4—H signal there are a one-proton sextet at τ 3.45 ppm (J_1 = 15.5 and J_2 = J_3 = 6.5 Hz) and a one-proton complex doublet at τ 4.10 ppm (J_1 = 15.5 and J_2 = 1.5 Hz) (long-range spin-spin coupling). These two signals belong to the protons of a —CH=CH— group, and the value J_1 = 15.5 Hz shows their trans arrangement, as is confirmed by a band at 970 cm^{-1} in the IR spectrum. The additional splitting with J_2 = 6.5 Hz shows that there is a CH_2 group adjacent to the double bond, for which the signal of the protons is found at τ 7.84 ppm (2H, quintet), and, together with the triplet at τ 9.03 (3H, J = 6.5 Hz), shows that the side chain has the structure $—CH{=}CH—CH_2—CH_3$.

Thus, the NMR and IR spectra permit the conclusion that the isolated lactone belongs to the isocoumarin series with a butene chain in position 3 of the α-pyrone ring.

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30 April 1970

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