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FASTIGENIN, HARPAGIDE, AND 8-O-ACETYLHARPAGIDE FROM *Teucrium orientale*

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We have investigated the epigeal part of *Teucrium orientale* L., family *Lamiaceae* (oriental germander) collected in the budding-flowering phase in the region of Lake Sevan, Armenian SSR.

An aqueous methanolic (80% MeOH) extract was dried in vacuum, diluted with water (1:1), and filtered, and the filtrate was extracted successively with benzene, chloroform, and ethyl acetate.

By the chromatographic elimination of pigments [1] of the benzene extract on a silica gel column with chloroform, a light yellow crystalline substance (I) was isolated with the composition $C_{18}H_{16}O_7$, M^+ 344, mp 194-196°C (methanol). On the basis of its UV and IR spectra (3400 (OH), 1670 (C=O of a γ-pyrone), 1620, 1570 (C=C of a γ-pyrone), 1595, 1500 cm^{-1} (benzene ring) and the cyanidin reaction [2], substance (I) was assigned to the flavones. According to the results of UV spectroscopy with various additives [3], there were free hydroxy groups in positions 4' and 5: $\lambda_{max}^{CH_2OH}$, 275, 340 nm (log ε 4.07; 4.18); CH_3COONa; 274, 338 nm; $CH_3COONa + H_3BO_3$; 275, 343 nm; $AlCl_3$: 263, 286, 370 nm; $AlCl_3$ + HCl: 263, 293, 368 nm; CH_3ONa 272, 300 sh, 328 sh., 378 nm; $ZrOCl_2$ 294, 371 nm; $ZrOCl_2$ + citric acid; 273 sh., 344 nm.

When the flavone was acetylated with acetic anhydride in pyridine, a diacetate was formed $C_{22}H_{20}O_9$, M^+ 428 (28%), mp 164-166°C (methanol).

According to its IR spectrum (2850, 1740 cm^{-1}) and PMR spectrum, the flavone contained three methoxy groups in positions 3',6, and 7, which was confirmed on the basis of SSCCs ($CDCl_3$, HMDS, Varian T-60): 7.47 d, J = 2 Hz (H-2'); 7.40 dd, J_1 = 2 Hz, J_2 = 9 Hz (H-6'); 6.92 d, J = 9 Hz (H-5'); 6.37 s, (H-8), 6.35 s, (H-3); 3.98 s, 6H (2 OCH_3); 3.90 s, 3H (OCH_3), and also from the results of mass spectrometric fragmentation of the substances: formation of ions with m/z 181 (24%) and m/z 149 (27%) [4].

On the basis of the result obtained, the structure of 4',5-dihydroxy-3',,6,7-trimethoxyflavone, known in the literature as fastigenin [5], was established for substance (I). This is the first time that fastigenin has been detected in plants of the genus *Teucrium*.

By column chromatogrrphy on silica gel in the chloroform–methanol, (7%) solvent system, from the resins obtained from both the chloroformic and the ethyl acetate extracts we isolated a colorless crystalline substance (II) with the composition $C_{17}H_{26}O_{11}$, mp 154-155°C (methanol), $[\alpha]_D^{17.4}$ −104.1° (s 1.58; methanol), R_f 0.38 (ethyl acetate–methanol–water (7:2:1), spots detected with benzidine, the vanillin reagent and Ehrlich's reagent) and substance (III), composition $[\alpha]_D^{20.8}$ $-163.7 \pm 0.28°$ (s 3.54; methanol), R_f 0.27. No substances gave a positive color reaction for iridoids [6].

The IR, PMR, and mass spectra of compounds (II) and (III) agreed well with those for the natural glycosides 8-O-acetylharpagide [7] and harpagide [8], respectively.

The identity of substances (II) and (III) as 8-O-acetylharpagide and harpagide was confirmed by the preparation from each substance on acetylation of a separable mixture of harpagide hexa- and heptaacetates, and also by the formation of harpagide on the alkaline saponification of substance (II) [8].

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In the aqueous fraction remaining after the ethyl acetate extraction of the aqueous methanolic extract, 8-O-acetylharpagide, harpagide, and sucrose were detected on a column of polyamide identified by TLC and melting points in comparison with an authentic sample.

This is the first time that harpagide and 8-O-acetylharpagide have been isolated from oriental germander although harpagide has been detected in this plant previously by paper chromatography [9].

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MOLECULAR STRUCTURE OF THE SESQUITERPENE LACTONE ARTEANNUIN B

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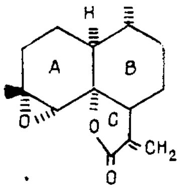
On the basis of the results of NMR, IR, and CD spectroscopy, the following structure has been proposed for the sesquiterpene lactone arteannuin B isolated from *Artemisia annua* L.:



The results of an x-ray structural analysis of this compound that we have performed have confirmed the proposed structure and permitted the conformations of the three linked rings to be determined unambiguously. The spatial structure of the lactone arteannuin B (I) is shown in Fig. 1 in projection on the (010) plane, which shows the cis-linkage of rings A/B and the trans-linkage of B/C. The methyl groups at C(4) and C(10) have the β- and α-orientations, respectively. Ring A has the half-chair conformation: the C(3)...C(6) atoms are located in one plane, and C(1) and C(2) depart from this plane in opposite directions by 0.29 and 0.59 Å, respectively. The second six-membered ring, B, has the chair conformation. The lactone ring has the envelope conformation; the departure of the C(6) atom from the plane of the other four is determined with an accuracy of 0.08 Å and amounts to −0.60 Å. The values of the valence angles and of the bond lengths (see Table 1 and Fig. 1) agree, within the limits of error, with those given in [3] and with standards [4]. The errors in the determination of the bond lengths are not greater than 0.01 Å.

Crystals were obtained from a 1:4 mixture of hexane and benzene. The space group and the parameters of the elementary cell were determined by a photographic method and were refined on a Syntex $P2_1$ diffractometer: a = 9.045; b = 12.263; c = 12.275 Å; space group $P2_12_12_1$, z = 4. The calculations made use of 731 nonzero reflections. The structure was interpreted by

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