CHEMICAL COMPOSITION OF THE ESSENTIAL OIL OF Artemisia scoparia

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We have previously [1] reported the isolation from the essential oil of oriental wormwood (Artemisia scoparia) gathered in Krasnodar territory and in the Poltava and Dnepropetrovsk regions of the Ukraine of two unidentified components in the form of colorless viscous liquids with the compositions $C_{15}H_{24}O$ and $C_{15}H_{20}O_3$. In the present paper we give information on the identification of these compounds and also on a further study of the component composition of the essential oil of oriental wormwood.

The IR spectrum of the substance with the composition $C_{15}H_{24}O$ had the absorption bands of an OH group (3350 cm^{-1}) and of an isolated double bond (1610 cm^{-1}). With bromine and acetic acid the substance gave a green coloration changing to dark blue, which showed its azulene skeleton. The NMR spectrum (δ, ppm) contained the following signals: 1.0 d (6H, two quaternary methyls); 1.3 s (methyl attached to oxygen); 4.7 s (2H, two vinyl protons). On comparing the results obtained with the literature, we observed agreement with those for spathulenol. A direct comparison with a specimen of spathulenol obtained from chamomile essential oil confirmed their identity. This is the first time that spathulenol has been obtained from the essential oil of oriental wormwood.

The second, unidentified, substance proved to be a mixture of two substances difficult to separate, with M^+ 220 (main substance) and M^+ 248 (minor component). Twofold column chromatography on alumina and silica gel, and also threefold preparative thin-layer chromatography led to the isolation of a substance with the composition $C_{15}H_{24}O$ (M^+ 220), mp 57-59°C (from hexane). The IR spectrum lacked the absorption band of an OH group. The PMR spectrum contained the following signals (δ, ppm): 1.0 d (two quaternary methyls); 1.2 s (methyl attached to oxygen); and 4.85 and 4.95, two singlets (vinyl protons). On comparing the results obtained with the literature characteristics of β-caryophyllene epoxide, isolated previously from the essential oil of this wormwood species [2], we found that they were identical.

In addition to the compounds mentioned above, by column chromatography we isolated methyleugenol, $C_{11}H_{14}O_2$, in the form of a colorless viscous liquid, and capillol, $C_{12}H_{10}O$, mp 85-87°C (from ethanol), and also the hydrocarbons myrcene and aromadendrene.

Thus, the major components of the essential oil of oriental wormwood gathered in the European part of the CIS are α- and β-pinenes, myrcene, ar-curcumene, capillene, capillin, capillol, β-caryophyllene epoxide, spathulenol, methyleugenol, eugenol, and aromadendrene.

The composition of the specimens of oriental wormwood that we investigated differed considerably from that known from the literature. In particular, we did not detect 1-phenylhex-2-ene, 1-phenylhexa-2,5-diyne, and scoparone, which have been described previously as major components of oriental wormwood essential oil [3, 4]. In addition, we have isolated capillin, capillol, spathulenol, and aromadendrene from the essential oil of oriental wormwood for the first time.

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SYNTHESIS OF THE SIDE CHAIN OF A SAPOGENIN FROM THE SOFT CORAL Asterospiculata randalli

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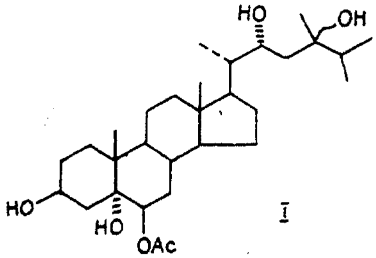
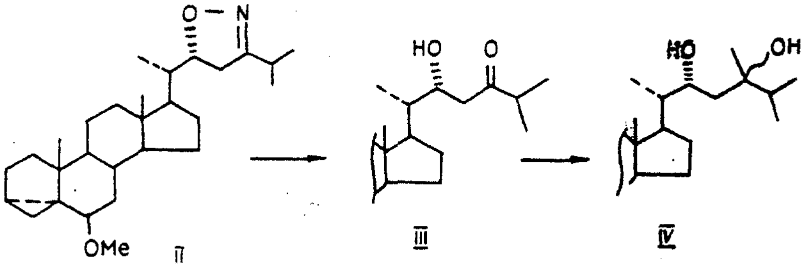
In 1984, the isolation from the soft coral Asterospicularia randalli of the polyhydroxylated steroid (I) with a set of hydroxy functions at C-22 and C-24 unusual for steroids of marine organisms was reported [1].



I

In a preceding paper, a method of constructing the side chain that is characteristic for this genin was proposed for the first time. The method is based on the use of a steroid isoxazoline (II) that we had obtained previously [2]. The reductive cleavage of the heterocyclic nucleus of compound (II) over Raney nickel in the presence of acids (H_3BO_3, AcOH, etc.) led to the formation of the 22R-hydroxy-24-oxo derivative (III). The formation of a β-ketone of this structure was shown by the appearance of the absorption bands of a C=O group (1710 cm^{-1}) and of a hydroxy group (3510 cm^{-1}) in the IR spectrum and the disappearance from the PMR spectrum of the signals of the methylene protons at C-23 characteristic for the isoxazoline ring. The interaction of the resulting compound (III) with methyllithium in ether at 0°C gave the desired product – the 22R,24ξ-diol (IV). It must be mentioned that we obtained both diastereomers of compound (IV) and separated them chromatographically. For the less polar isomer: PMR spectrum (200 MHz, δ, ppm): 0.75 s (3H, 18-Me), 0.85 d (3H, J = 7 Hz, 26-Me), 0.95 d (3H, J = 7 Hz, 21-Me), 0.98 d (3H, J = 7 Hz, 27-Me), 1.02 s (3H, 19-Me), 1.09 s (3H, 28-Me), 2.02 m (1H, C_{25}-H), 3.33 s (3H, OMe), 4.09 m (1H, C_{22}-H). IR spectrum (KBr, cm^{-1}): 3510, 1100. Mass spectrum (m/z): 446 $[M]^+$, 431 $[M - Me]^+$, 414 $[M - MeOH]^+$, 391, 353, 313, 284, 255. For the more polar isomer: PMR spectrum (200 MHz, δ, ppm): 0.75 s (3H, 18-Me), 0.86 d (3H, J = 7 Hz, 26-Me), 0.92 d (3H, J = 7 Hz, 27-Me), 0.95 d (3H, J = 7 Hz, 21-Me), 1.03 s (3H, 19-Me), 1.19 s (3H, 28-Me), 2.78 m (1H, C_6-H), 3.33 s (3H, OMe), 4.10 m (1H, C_{22}-H). IR spectrum (KBr, cm^{-1}): 3510, 1100. Mass spectrum (m/z): 446 $[M]^+$, 431 $[M - Me]^+$, 414 $[MeOH]^+$, 391, 353, 313, 284, 255.



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