LACTONES FROM THE RESIN OF THE ROOTS OF Ferula pseudooreoselinum

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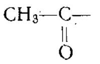
UDC 547.314

Some species of Ferula contain substances with a peculiar structure which it is of interest to study. After the elimination of the glycoside reoselin [1] from a concentrated ethereal solution of the neutral fraction of an extract of Ferula pseudooreoselinum (Regel et Schmalh, K.-Pol.), on prolonged storage a precipitate deposited which consisted of a mixture of two isomeric (according to the IR, UV, NMR, and mass spectra) substances $C_{19}H_{24}O_6$ with mp 107 and 138° C (from aqueous ethanol), which we have called α- and β-reolones.

α-Reolone is inactive and has mol. wt. 348 (mass spectrometry) with the following spectroscopic characteristics: UV spectrum, λ_{max} (ethanol), nm: 322, 283, 232 (log ε 3.756, 4.064, 3.798); IR spectrum, ν_{max} ($CHCl_3$), cm^{-1}: 1765 and 1715 ($A \cdot 10^{-4}\ mol^{-1} \cdot liter \cdot cm^{-2} = 4.9$ and 2.0), 1630 v.s, 1515 m, 1275 s, 1220 s, 1180 s; NMR spectrum, δ ($CDCl_3$), ppm: 1.07 (doublet, 3H, J = 7 Hz), 1.38, 2.17, and 3.92 (singlets, 3H each), 1.77 and 2.55 (broad bands, 4H and 2H), 3.14 (quartet, 1H, J = 7 Hz), 4.3 (doublet, 1H, J = 12 Hz), 6.45 (singlet, 1H), 6.50 and 7.70 (doublets, 1H each, J = 9 Hz), and 12.3 (singlet, 1H).

The spectral characteristics permit the assumption that the molecule of α-reolone contains a lactone ring, a ketone group in an open chain, and an aromatic system with hydroxy and methoxy groups conjugated with a double bond.

The saponification of α-reolone with 5% alkali at a ratio of 1 mole of substance to 2 moles of alkali led to the opening of the lactone ring and the formation of a hydroxy acid with mp 142° C (from aqueous ethanol) not containing a peak at 1765 cm^{-1} in the IR spectrum. The acidification of this acid re-formed the α-reolone, but the saponification of α-reolone with an excess of 5% alkali (15 moles of alkali to 1 mole of substance) yielded an acid $C_8H_8O_4$ (20%) with mp 157° C (from aqueous ethanol) which was identified as 2-hydroxy-4-methoxybenzoic acid. Its methyl ester had mp 48° C [2].

2-Hydroxy-4-methoxybenzoic acid was not obtained when hydrogenated α-reolone was saponified with an excess of alkali or when it was subjected to acid hydrolysis in glacial acetic acid with a few drops of sulfuric acid. Consequently, we assumed that α-reolone does not contain an ester group and the peak with ν_{max} 1715 cm^{-1} is possibly due to a ketone group in an open chain (A = 2.0) [3]. Some confirmation of this is given by the signal at 2.17 ppm in the NMR spectra and by a fragment with m/e 43 (22%) in the mass spectrum, the appearance of these features also being possibly connected with the presence of a  $CH_3{-}\underset{\underset{O}{\|}}{C}{-}$ group in the α-reolone molecule. The study of the structure of α-reolone is continuing.

LITERATURE CITED

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V. L. Komarov Botanical Institute, Academy of Sciences of the USSR. Translated from Khimiya Prirodnykh Soedinenii, No. 2, pp. 204-205, March, 1971. Original article submitted November 23, 1970.

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