URSOLIC AND 3-EPIURSOLIC ACIDS FROM WASTES FROM THE PRODUCTION OF LAVENDER OIL

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Khimiya Prirodnykh Soedinenii, Vol. 4, No. 1, pp. 53–54, 1968

As reported previously [1] we have isolated from lavender (population N 13 of the Nikita Botanical Garden) a mixture of ursolic and 3-epiursolic acids. Recently, the 3-epi isomer has been obtained by German workers [2] semisynthetically from ursolic acid.

We have now developed a method for the separation of the mixture isolated into its epimers. The raw material for the work was the calyxes of lavender taken after the essential oil had been distilled from the plant. They were dried in the air and extracted with petroleum ether and then with ethanol. The ethanolic extract (yield 3.1% of the weight of the calyxes) formed a green amorphous powder consisting mainly of impure ursolic and 3-epiursolic acids. The mixture of acids was freed from impurities by passing an ethereal solution of the extract through a column of Norite (100 g of extract to 400 g of Norite). This gave 68 g of a white powder which, after four recrystallizations from methanol–toluene (3:2) was chromatographed on silica gel. For this purpose, 10 g of the substance was dissolved in benzene and transferred to a column containing 500 g of adsorbent. Benzene–ether (9:1, and then 8:2) eluted 3-epiursolic acid (1.09 g). After three recrystallizations from methanol, the substance had mp 238–239° C, $[\alpha]_D^{20}$ + 65° (in chloroform). These figures agree with those given in the literature [2]. Ether eluted the ursolic acid.

The two hydroxy acids were identified by means of their IR spectra [3] and their derivatives, methyl esters, acetates, and acetates of the methyl esters. To confirm the difference in the configurations of the molecules at the third asymmetric center, the ursolic and 3-epiursolic acids were interconverted. For this purpose, each of the acids was oxidized with chromic anhydride in pyridine. In both cases, ursonic acid was obtained. The reduction of this with sodium borohydride gave ursolic acid. However, when the ursonic acid was reduced by the Meerwein-Ponndorf method with subsequent chromatographic separation of the reduction products on silica gel, 3-epiursolic acid was obtained. This confirms literature data according to which the hydroxy group in 3-epiursolic acid occupies the axial position and that in ursolic acid the equatorial position.

3-Epiursolic acid has not previously been found in nature; we have described the first case of its isolation from a plant.

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18 July 1967

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UDC 615.43+615.177/779

THE CONTENT OF ARBUTIN IN SOME SPECIES OF THE GENUS SERRATULA

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Khimiya Prirodnykh Soedinenii, Vol. 4, No. 1, p. 54, 1968

From the leaves of Serratula isophylla Claus (family Compositae) by chromatography on Kapron we have isolated a phenolic glycoside $C_{12}H_{16}O_7$ with mp 146–147° C, $[\alpha]_D^{20}$ −60° C. Its acetyl derivative $C_{22}H_{26}O_{12}$ contains five acetyl groups and has mp 146° C, $[\alpha]_D^{20}$ −28.2° (in acetone).

The dry residue of an ethyl acetate extract of a concentrated aqueous extract of the raw material that had previously been purified with chloroform was subjected to chromatography. The column was washed with chloroform until the eluate was colorless, after which elution was carried out with a mixture of chloroform–ethanol–acetic acid (900:100:1). The combined eluates were evaporated and the dry residue was recrystallized several times from water. The glycoside crystallized in the form of thin white needles.