HYDROXYCOUMARINS FROM THE LEAVES OF Onobrychis kemularia

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The air-dry comminuted leaves (250 g) of O. kemularia Chinth. were extracted with 80% methanol three times. The extracts were combined and concentrated to 300 ml. The total hydroxycoumarins were obtained and they were separated into their individual component by the new methods for the purification and separation of natural organic substances involving partition chromatography on gel sorbents that we have proposed. In particular, 70 g of Sephadex G-25 (coarse fraction) was added to the concentrated extract and was left to swell. After an hour, it was transferred to a column (d = 7 cm). The column was washed first with 500 ml of petroleum ether and then with 700 ml of petroleum ether–chloroform (3:1) to wash out the chlorophyll and other lipophilic substances, and then with chloroform to wash out the hydroxycoumarins [1]. To separate the combined hydroxycoumarins, the chloroform eluate was concentrated to a dry residue. This residue (0.18 g) was dissolved in 5 ml of the organic phase of the chloroform–butanol–water (1:1:1) system, 2 g of Sephadex LH-20 was added, and it was left to swell for 1 h. In parallel, 30 g of Sephadex LH-20 was left to swell in 250 ml of the organic phase of the same system, was transferred to a funnel, washed with 150 ml of the aqueous phase of this system, transferred to a column (d = 2.1 cm) and washed with 100 ml of the aqueous phase of this system, and on it was deposited the Sephadex swollen in the solution of the material under investigation. The hydroxycoumarins were eluted with the aqueous phase of the same system [2]. The eluates were collected in 50-ml fractions. The fractions containing the same substance were combined and extracted with chloroform, and the extracts were dried with anhydrous sodium sulfate and concentrated under vacuum. The residues obtained were dissolved in 10-12 ml of ethanol and the solution was left at room temperature. This gave colorless crystals of substances provisionally denoted by the letters A (0.97 g) and B (0.011 g).

Substance A ($C_{10}H_8O_4$) has mp 200-202°C, $\lambda_{max}^{C_2H_5OH}$ 327, 255 nm (log ε 3.84, 3.31). The IR spectrum showed absorption bands at 1721 cm^{-1} (α-pyrone), 3410 cm^{-1} (OH group), 1611, 1515, and 1405 cm^{-1} (benzene nucleus), and others. This substance gave no depression of the melting point with an authentic sample of scopoletin. Thus, we identified substance A as scopoletin; substance B [$C_9H_6O_3$, mp 230-231°C, $\lambda_{max}^{C_2H_5OH}$ 321, 265 nm (log ε 3.79, 3.37)] was identified from its physicochemical propeties as umbelliferone [3].

LITERATURE CITED

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