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Neochlorogenin from the Fruits of *Solanum nigrum*

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Abstract—A steroidal sapogenin, neochlorogenin, was isolated from *Solanum nigrum*. This is the first report from this plant.

Keywords—*Solanum nigrum* • Solanaceae • steroidal sapogenin • neochlorogenin

Many steroidal saponins and alkaloids have been isolated from *S. nigrum*.[1~2] In our studies on the fruits of *S. nigrum*(Solanaceae), we have now isolated one steroidal sapogenin, neochlorogenin, from the hydrolysate of n-BuOH soluble fraction.

Experimental

Melting point was determined with Yanaco micro-melting point apparatus and is uncorrected. The optical rotation was measured with Jasco DIP 360 automatic polarimeter. The IR spectrum was recorded on Perkin-Elmer 1310 spectrophotometer. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker AM-300 spectrometer using TMS as an internal standard. EI mass spectrum was measured on a Kratos MS 25 RFA spectrometer. TLC chromatography was performed on precoated Kieselgel 60 F_{254} plates (Merck, 5715).

Plant material

The fruits of *S. nigrum*(100g) was collected from Kang-Won Do, Korea in 1990. The specimen has been deposited in College of Pharmacy, Kang Won National University.

Extraction and purification

The dried fruits of *Solanum nigrum*(100 g) were refluxed with MeOH for 3 hr(2 times) and evaporated to dryness. The residue(2.5 g) was partitioned between hexane and H_2O. The aqueous layer was then extracted with n-BuOH and concentrated in vacuo to afford resiue(1.3 g). *n*-BuOH extract(1 g) was hydrolyzed with 5% H_2SO_4 in MeOH for 2 hrs. After cooling, the reaction mixture was diluted with iced water and the precipitate was collected by filteration, and dried. The hydrolysate was subjected to column chromatography over silica gel with hexane-EtOAc(8:5) to yield neochlorogenin(**1**).

Neochlorogenin(1)

Colorless needles from MeOH; mp 252~256°; $[\alpha]_{20}^{D}$=−50.8° (*c* 0.1, pyridine); IR ν_{max}^{KBr} cm^{-1} 3400, 985, 920, 900, 850[intensity 920>900, 25(S)-spiroketal]; EI-MS *m/z* (rel. int.) 432 (M^+, 6.3), 373(4.4), 363(11.7), 360(21.7), 318(22.2), 139(100.0), 115(14.8); ^{1}H-NMR (pyridine-d_5, 300 MHz) δ 0.83(3H, s, 19-CH_3), 0.86(3H, s, 18-CH_3), 1.06(3H, d, *J*=7.1Hz, 27-CH_3), 1.13(3H, d, *J*=7.0 Hz, 21-CH_3), 2.98, 3.35(1H each, br.d, *J*=11 Hz, 26-CH_2), 3.66(2H, m, 3α and 6β-H), 4.51(1H, m, 16-H); ^{13}C-NMR(pyridine-d_5, 75.5 MHz) δ 38.0, 32.4, 71.0, 33.7, 52.8, 68.6, 42.9, 34.4,

54.3, 36.6, 21.4, 40.2, 40.8, 56.5, 32.2, 81.2, 62.9, 16.6, 13.8, 42.5, 14.9, 109.7, 27.5, 26.2, 26.4, 65.1, 16.3 (signals of C-1 to C-27).

Results and Discussion

Neochlorogenin(**1**), mp 252~256°, was positive to the Liebermann-Burchard test and showed characteristic absorption bands due to 25(S)-spiroketal moiety in the IR spectrum.[3] The EI mass spectrum of **1** showed molecular ion peak at *m/z* 432 and the base peak at *m/z* 139. The ^{1}H-NMR spectrum of **1** exhibited two tertiary methyl groups(singlets of δ 0.83 and 0.86) and two secondary methyl group(doublets of δ 1.06 and 1.13) in the strong field, and two broad doublets at δ 2.98 and 3.35 corresponded to the resonances of the two protons of H-26. And a signal at δ 4.51 was due to the resonance of H-16. In addition to the signals described above, there were also a multiplet at δ 3.66, which must be considered as two carbinyl protons. In the ^{13}C-NMR spectral data, the signals due to the A~D rings moiety of **1** were in good agreement with those of chlorogenin,[4] but the signals of E~F rings system were coincident with those of yamogenin[5] or 25(S)-ruscogenin.[6] Consequently, **1** was comfirmed to be 25(S)-spirostane-3β, 6α-diol, neochlorogenin.[7]

Neochlorogenin was already isolated from *S. hispidum*,[7] but the isolation of this sapogenin from *S. nigrum* is first reported.

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