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FLAVONOIDS AND PHENOLIC COMPOUNDS FROM SEEDS OF THE CHINESE PLANT *Nigella glandulifera*

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Seeds of *Nigella glandulifera* Freyn (Ranunculaceae) have been used since antiquity in Uyghur folk medicine for kidney failure, stimulation of menstrual flow and diuresis, improvement of mental capacity, and lactose clearance [1]. A total of 16 compounds have been isolated from the plant [2-5]. We studied flavonoids and phenolic compounds from this plant.

Defatted seeds of *N. glandulifera* (10 kg) were extracted with ethanol (50%). Ethanol was evaporated in vacuo to afford a syrupy residue that was suspended in distilled water and fractionated successively with petroleum ether, $CHCl_3$, ethylacetate, and *n*-butanol. Rechromatography of the butanol fraction over columns of polyamide, silica gel, and Sephadex LH-20 produced compounds **1**-**8**. The isolated compounds were identified using spectral analysis (UV, IR, NMR). Compounds **1**, **3**, **4**, **5**, **7**, and **8** were isolated for the first time from *N. glandulifera*. Comparison of the results with the literature identified **1** as kaempferol [6]; **3**, quercetin [6-8]; and **4**, rutin [9].

Kaempferol-3-*O*-β-D-glucopyranosyl-(1$\rightarrow$2)-β-D-galactopyranosyl-(1$\rightarrow$2)-β-D-glucopyranoside (2), brown powder. IR spectrum (KBr, ν_{max}, cm^{-1}): 3380 (OH), 1653 (C=O), 1606, 1570, 1496, 1072, 1041.

PMR spectrum (CD_3OD, 600 MHz, δ, ppm, J/Hz): 8.09 (2H, dd, J = 8.4, 8.4, 2′,6′-H), 6.97 (2H, dd, 3′,5′-H), 6.39 (1H, s, 8-H), 6.19 (1H, 6-H), 5.44 (1H, d, J = 7.4, Glu-1H), 4.77 (1H, d, J = 7.7, Gal-1H), 4.71 (1H, d, J = 7.5, Glc-1H). ^{13}C NMR spectrum (CD_3OD, 150 MHz, δ, ppm): 158.45 (C-2), 134.81 (C-3), 179.7 (C-4), 161.51 (C-5), 99.81 (C-6), 166.69 (C-7), 94.68 (C-8), 158.55 (C-9), 105.79 (C-10), 122.79 (C-1′), 132.43 (C-2′), 116.3 (C-3′), 161.51 (C-4′), 116.3 (C-5′), 132.43 (C-6′), 100.80 (GluC′-1), 85.22 (GluC′-2), 78.81 (GluC′-3), 71.05 (GluC′-4), 78.45 (GluC′-5), 62.38 (GluC′-6), 104.91 (GalC-1), 83.56 (GalC-2), 75.01 (GalC-3), 69.61 (GalC-4), 76.32 (GalC-5), 61.81 (GalC-6), 106.37 (GluC″-1), 76.28 (GluC″-2), 77.59 (GluC″-3), 70.38 (GluC″-4), 77.37 (GluC″-5), 62.38 (GluC″-6) [5, 9].

Salicylic acid (5), white powder, mp 157-158°C (acetone). IR spectrum (KBr, ν_{max}, cm^{-1}): 3237 (OH), 2857, 2600, 1660, 1480. UV spectrum (λ_{max}, MeOH, nm): 238, 302.

The PMR spectrum agreed with that for salicylic acid.

^{13}C NMR spectrum (CD_3OD, 150 MHz, δ, ppm): 113.86 (C-1), 163.2 (C-2), 118.13 (C-3), 136.59 (C-4), 120.03 (C-5), 131.52 (C-6), 173.52 (C-7) [10].

4-Hydroxybenzoic acid (6), white flakes, mp 213-214°C (MeOH).

PMR spectrum (CD_3OD, 600 MHz, δ, ppm, J/Hz): 7.91 (2H, d, J = 7.2, H-2,6), 6.85 (2H, d, J = 6.6, H-3,5).

^{13}C NMR spectrum (CD_3OD, 150 MHz, δ, ppm): 122.66 (C-1), 133.00 (C-2,6), 116.02 (C-3,5), 163.36 (C-4), 170.07 (–COOH) [11].

Methyl-4-hydroxybenzoate (7), white needles, mp 128-130°C ($CHCl_3$:CH_3OH). IR spectrum (KBr, ν_{max}, cm^{-1}): 3450, 2950, 2870, 1750, 1700, 1680, 1480, 1320. UV spectrum (λ_{max}, MeOH, nm): 210, 264.

PMR spectrum (CD_3OD, 600 MHz, δ, ppm, J/Hz): 7.90 (2H, d, J = 7.2, H-2,6), 6.85 (2H, d, J = 6.6, H-3,5), 3.87 (3H, s, OMe).

^{13}C NMR spectrum (CD_3OD, 150 MHz, δ, ppm): 168.64 (–COO–), 122.18 (C-1), 132.74 (C-2,6), 163.50 (C-4), 116.13 (C-3,5), 52.22 ($-OCH_3$) [12, 13].

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Pyrogallol (8), white needles, mp 131-133°C ($CHCl_3$). IR spectrum (KBr, ν_{max}, cm^{-1}): 3340, 3249, 1662, 1484, 1330, 1245, 1197, 1065, 1004, 868, 829, 767, 702. UV spectrum (λ_{max}, MeOH, nm): 267.

PMR spectrum (CD_3OD, 600 MHz, δ, ppm, J/Hz): 6.53 (1H, d, J = 7.8, H-5), 6.35 (2H, d, J = 8.4, H-4,6). ^{13}C NMR spectrum (CD_3OD, 150 MHz, δ, ppm): 147.15 (C-1,3), 134.30 (C-2), 120.06 (C-5), 108.26 (C-3,6) [14, 15].

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