COMPONENTS OF *Cichorium glandulosum* SEEDS

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Cichorium glandulosum Boiss et Hout (Compositae, Asteraceae) is usually used as a medicinal plant to treat liver diseases [1]. *C. glandulosum* is widely distributed in Xinjiang. Its chemical composition is little studied with the exception of the essential oil [2] and esculetin [3]. Herein we describe the isolation and structure determination of the isolated compounds.

Seeds (~5 kg) of *C. glandulosum* were extracted with ethanol (70%, 3×). The extracts were combined and evaporated to dryness in vacuo (at reduced pressure). The yield of dry powder was 150 g. The ground powder was dissolved in pure water (800 mL) and separated into three fractions by successive extraction with petroleum ether (PE), ethylacetate (EtOAc), and *n*-butanol (*n*-BuOH). The PE fraction was then separated by petroleum ether:methanol (90%) (1:1, v/v). The fraction soluble in methanol (90%) was separated by column chromatography over Sephadex LH-20 with elution by methanol to produce compound **1**. The EtOAc fraction was purified several times by column chromatography to produce compounds **2-4**. The butanol fraction was first placed on a column packed with polyamide resin and eluted by a gradient of water with a gradually increasing concentration of ethanol. Subfractions were combined according to their TLC behaviors and then chromatographed several times over a silica-gel column and by preparative TLC to produce compounds **5-7**, which were identified using PMR and ^{13}C NMR spectroscopy.

Kaempferol (1), yellowish powder (methanol). PMR spectrum (400 MHz, $CDCl_3$ + DMSO-d_6, ppm, J/Hz): 8.08 (2H, d, J = 8.8, H-2′,6′), 6.96 (2H, d, J = 8.8, H-3′,5′), 6.43 (1H, d, J = 1.6, H-8), 6.29 (1H, d, J = 1.6, H-6). ^{13}C NMR spectrum (100 MHz, $CDCl_3$, δ, ppm): 146.3 (C-2), 136.0 (C-3), 176.0 (C-4), 156.9 (C-5), 98.9 (C-6), 164.2 (C-7), 94.1 (C-8), 159.3 (C-9), 103.4 (C-10), 122.0 (C-1′), 129.6 (C-2′,6′), 115.7 (C-3′,5′), 161.5 (C-4′). Compound **1** corresponded with kaempferol [4].

Esculetin (2). The PMR and ^{13}C NMR spectra corresponded with those published [3].

Dibutylphthalate (3), colorless oil. PMR spectrum (600 MHz, $CDCl_3$, δ, ppm, J/Hz): 7.70 (2H, m, H-2, H-5), 7.50 (2H, m, H-3, H-4); dibutyl protons: 4.30 (4H, t, J = 6.6), 1.71 (4H, m), 1.44 (4H, m), 0.95 (6H, t, J = 7.2, $2CH_3$). ^{13}C NMR spectrum (150 MHz, DMSO-d_6, δ, ppm): 167.5 (C-1), 132.2 (C-2), 130.7 (C-3), 128.7 (C-4), 65.3 (C-5), 30.4 (C-6), 19.1 (C-7), 13.5 (C-8). Compound **3** was identified as dibutylphthalate.

Ethylcaffeate (4), white powder (methanol). PMR spectrum (600 MHz, $CDCl_3$, δ, ppm, J/Hz): 7.59 (1H, d, J = 16.2, H-7), 7.11 (1H, d, J = 1.8, H-2), 7.03 (1H, dd, J = 1.8, 8.4, H-6), 6.88 (1H, d, J = 8.4, H-5), 6.28 (1H, d, J = 16.2, H-8), 4.30 (2H, q, J = 7.2), 1.37 (3H, t, J = 7.2). These data agreed with those published [5].

Astragalin (5), yellow powder (methanol), mp 219-220°C. PMR spectrum (600 MHz, DMSO-d_6, δ, ppm, J/Hz): 12.6 (1H, s, 5-OH), 10.9 (1H, br.s, 7-OH), 10.2 (1H, br.s, 4′-OH), 8.05 (2H, d, J = 7.2, H-2′,6′), 6.89 (2H, d, J = 7.2, H-H-3′,5′), 6.46 (1H, d, J = 1.8, H-8), 6.23 (1H, d, J = 2.4, H-6), 5.47 (1H, d, J = 7.8, H-1″), 3.07-3.58 (glucose protons). ^{13}C NMR spectrum (150 MHz, DMSO-d_6, δ, ppm): 156.6 (C-2), 133.6 (C-3), 177.9 (C-4), 160.4 (C-5), 99.1 (C-6), 164.6 (C-7), 94.0 (C-8), 156.8 (C-9), 104.4 (C-10), 121.3 (C-1′), 131.3 (C-2′,6′), 115.5 (C-3′,5′), 161.6 (C-4′), 101.2 (C-1″), 74.6 (C-2″), 76.8 (C-3″), 70.3 (C-4″), 77.9 (C-5″), 61.2 (C-6″). Compound **5** was identified as astragalin [4].

Chlorogenic acid (6), light yellowish powder. ^{13}C NMR spectrum (150 MHz, DMSO-d_6, δ, ppm): 75.5 (C-1), 39.5 (C-2), 73.7 (C-3), 72.0 (C-4), 72.2 (C-5), 39.7 (C-6), 146.8 (C-α), 115.1 (C-β), 126.0 (C-1′), 116.2 (C-2′), 146.0 (C-3′), 148.7 (C-4′), 115.1 (C-5′), 121.6 (C-6′), 176.3 (C-7), 166.7 (C-8). These data agreed with those in the literature [6].

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Inosine (7), white powder. PMR spectrum (600 MHz, DMSO-d_6, δ, ppm, J/Hz): 12.43 (1H, s, OH-4), 8.36 (1H, s, H-3), 8.09 (1H, s, H-3), 5.80 (1H, d, J = 6.0, H-5), 5.53 (1H, s, OH-7), 5.25 (1H, s, OH-10), 5.11 (1H, s, OH-8), 3.0-5.0 (5H, ribose protons). ^{13}C NMR spectrum (150 MHz, DMSO-d_6, ppm): 139.1 (C-1), 146.3 (C-2), 148.6 (C-3), 157.0 (C-4), 124.8 (C-5), 87.8 (C-1′), 70.7 (C-2′), 74.5 (C-3′), 86.0 (C-4′), 61.7 (C-5′). Compound **7** was identified as inosine.

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