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A New Ursene Type Triterpenoid from *Crepis napifera*

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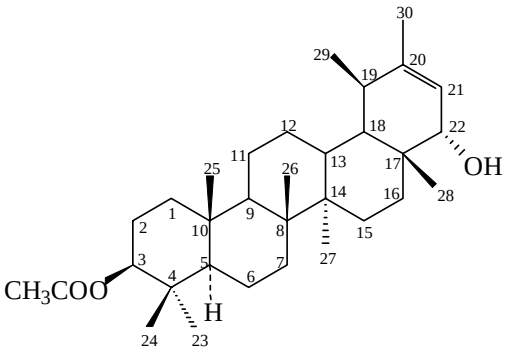
Abstract: A new triterpenoid, urs-20-en-3β-acetoxy-22α-ol, and three known compounds 3β-acetoxy-amyrin, 3β-acetoxy-lupeol, lupeol were isolated from the leaves of *Crepis napifera* (Franch.) Babc. Their structures were determined by means of spectroscopic studies.

Keywords: *Crepis napifera* (Franch.) Babc., Compositae, triterpenoid, urs-20-en-3β-acetoxy-22α-ol, 3β-acetoxy-amyrin, 3β-acetoxy-lupeol, lupeol.

Crepis napifera (Franch.) Babc., which is distributed in Yunnan province (Southwest China), is used for the treatment of inflammation and nourishing the lung to arrest cough in traditional Chinese medicine[1]. Only a triterpene isolated from the plant has been published until now[2]. In our study on chemical constituents of *Crepis napifera*, a new triterpenoid, urs-20-en-3β-acetoxy-22α-ol **1**, together with three known compounds 3β-acetoxy-amyrin[3], 3β-acetoxy-lupeol and lupeol[4-6] were isolated from the leaves of this plant.

Dried and powdered leaves of *Crepis napifera* (2 kg) were extracted with MeOH. The extract was partitioned with petroleum ether. The petroleum ether extract (40 g) was subjected to chromatography repeatedly on silica gel column to give **1**, which was obtained as colorless needles from petroleum ether-acetone, mp 221.5-223.5°C, $[\alpha]^{25}_{D}$ +45.8 (*c* 0.004, $CHCl_3$). Its molecular formula was determined to be $C_{32}H_{52}O_3$ based on EIMS at *m/z* 484 $[M]^+$ and ^{13}C NMR spectrum. The IR spectrum exhibited the presence of a hydroxy group (3522 cm^{-1}) and an estercarbonyl group (1723 cm^{-1}). Comparison of the ^1H and ^{13}C NMR spectra with the reference data[7, 8] indicated that **1** is an ursene derivative with an acetoxyl group at the C-3β position and a hydroxyl group. The ^1H NMR spectrum showed signals of a methyl group at δ 1.66 (3H, *s*) attached to a double bond, an olefinic proton at δ 5.58 (1H, *d*, *J* = 6.4 Hz), and a hydroxymethine proton at δ 3.31 (1H, *d*, *J* = 6.5 Hz). The double bond was located between C-20 and C-21 from the HMBC spectrum, in which long-range correlations were observed from H-29 [δ 1.02 (3H, *d*)] to C-20, and from H-30 [δ 1.66 (3H, *s*)] to C-20 and C-21. The ^1H-^1H COSY spectrum showed the correlation between the hydroxymethine proton at δ 3.31 (1H, *d*, *J* = 6.5 Hz) and the olefinic proton. The α orientation of 22-OH was established on the basis of NOESY spectrum, which showed NOE interactions between H-22 and H_3-28, and between H_3-28 and H_3-29. Therefore, compound **1** was elucidated as

urs-20-en-3β-acetoxy-22α-ol.



1

Compound **1**, ^{1}H NMR δ (400 MHz, $CDCl_3$): 5.58 (1H, *d*, *J* = 6.4 Hz, H-21), 4.46 (1H, *dd*, *J* = 10.5 Hz, *J* = 5.7 Hz, H-3), 3.31 (1H, *d*, *J* = 6.5 Hz, H-22), 2.02 (3H, *s*, CH_3COO), 1.66 (3H, *s*, H-30), 1.04 (3H, *s*, H-26), 1.02 (3H, *d*, H-29), 0.96 (3H, *s*, H-27), 0.85 (3H, *s*, H-25), 0.82 (3H, *s*, H-23), 0.81 (3H, *s*, H-24), 0.63 (3H, *s*, H-28); ^{13}C NMR δ (100 MHz, $CDCl_3$): 38.5 (C-1), 23.7 (C-2), 81.0 (C-3), 37.8 (C-4), 55.5 (C-5), 18.2 (C-6), 34.3 (C-7), 41.2 (C-8), 50.4 (C-9), 37.1 (C-10), 21.6 (C-11), 26.8 (C-12), 38.8 (C-13), 42.3 (C-14), 27.6 (C-15), 29.9 (C-16), 38.2 (C-17), 41.0 (C-18), 36.5 (C-19), 145.6 (C-20), 121.7 (C-21), 74.0 (C-22), 28.0 (C-23), 16.5 (C-24), 16.4 (C-25), 14.7 (C-26), 16.1 (C-27), 18.1 (C-28), 22.9 (C-29), 21.6 (C-30), $OCOCH_3$ (170.9, 21.2).

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