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A New Triterpenoid from the Roots of *Callistemon lanceolatus* DC.

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ABSTRACT

A new triterpenoid of the ursane series, 2α,3β,24-Trihydroxyurs-12-en-28-oic acid 1 was isolated from the roots of *Callistemon lanceolatus*. The structural elucidation of 1 was carried out by extensive studies of both spectral studies and derivatization.

Keywords: 2α,3β,24-Trihydroxyurs-12-en-28-oic acid, *Callistemon lanceolatus* DC, Myrtaceae, Roots.

INTRODUCTION

The plant *Callistemon lanceolatus* DC. syn. *C. citrinus* Skeels belong to family Myrtaceae. *C. lanceolatus* is a small tree, indigenous to Australia and frequently grown in gardens in India. It is commonly called as bottle-brush due to the close resemblance of its inflorescence with it both in shape and size. Aliphatic acids and steroids from flowers[1-4], fruits[1,2], leaves[5-12], stem bark[13] and seeds[13]. Essential oils and various extracts of plant parts exhibited antifungal, antibacterial and antiviral activity as also antitumor activity in bioassay systems[14-18]. The leaf extract showed cholinesterase activity[19]. Seed extract demonstrated haemagglutinating activity against red cells of various animal species tested[20]. The plant extract exhibited mild inhibitory activity against different strains of virus. The essential oils of leaves exhibited pesticidal, growth inhibitory, anthelmintic and fungitoxic activities[21-24].

RESULTS AND DISCUSSION

Compound **1** (Fig. 1), was isolated as white solid, after crystallization with ethyl acetate, m.p. 334-36°C. In the MS spectrum, the cationized cluster ion $[M+Na]^+$ appeared at m/z 511 corresponding to the molecular ion m/z 488. The other important ions in mass spectrum were observed at m/z 470 [M^+–H_2O], 248 [$C_{16}H_{24}O_2$], 235 [$C_{15}H_{23}O_2$], 230 [$C_{16}H_{24}O_2 - H_2O$], 204 [$C_{12}H_{28}O_3$], 203 [$C_{16}H_{24}O_2$ – COOH], 185 [$C_{16}H_{24}O_2$ – (COOH + H_2O)] and 133 [$C_{10}H_{13}$]. The peaks observed in the infrared spectrum, at 3540, 3460 and 3370 for hydroxyl groups with the C-O stretching at 1070, 1050 and 1030 cm^{-1} indicated the presence of three hydroxy functions of which one is primary and the other two of secondary nature. Other important peaks appeared at 1710 (C=O stretching) and 1650 cm^{-1}(C=C stretching). The appearance of peaks at 1395, 1375, 1365, 1315, 1270, 1260 confirmed that of the triterpenoid belonged to the ursane series[25]. The PMR spectrum revealed the presence of six methyls in the region δ 0.74-1.08. The appearance of an upfield methyl at δ 0.74 suggested the presence of carboxylic acid group at C-28 position[26]. A multiplet centered at δ 3.68 and a doublet at δ 3.34 (J = 6Hz) corresponded to the protons geminal to the hydroxy groups placed on C-2 and C-3 respectively. Appearance of an ABq at δ 3.26 and 3.49 (J = 9Hz) indicated the presence of an hydroxymethyl group on C-4. A peak at δ 5.25 could be assigned to an olefinic proton at C-12.

Acetylation of compound **1** furnished a triacetate compound **2** with absorption for acetate groups at 1750, 1740, 1250 and 1240 cm^{-1} in the IR spectrum. In the ^1H NMR of the acetyl derivative (Fig. 1) three singlets at δ 2.17, 2.12 and 2.09 appeared for the CH_3CO groups. The doublet at δ 5.07 (J = 11 Hz) was assigned to the H-3 proton and the double doublet centered at δ 5.14 (J = 10.8, 3.9 Hz) corresponded to the H-2 proton. This downfield shift of the carbinolic protons on acetylation by ≈1.5 confirmed the secondary nature of these –OH groups. The nature and magnitude of the coupling constant (J_{ax-ax} = 10 Hz) suggested the presence of 2β and 3α protons on the carbons bearing the hydroxyl function[27]. The AB quartet now appeared at δ 3.58 and δ 3.84 (J = 12 Hz) where the shift of ≈0.4 was in conformity with the primary nature of this hydroxyl group. The chemical shifts of C-4 and 23-methyl led to the placement of this CH_2OH at C-24 position[28].

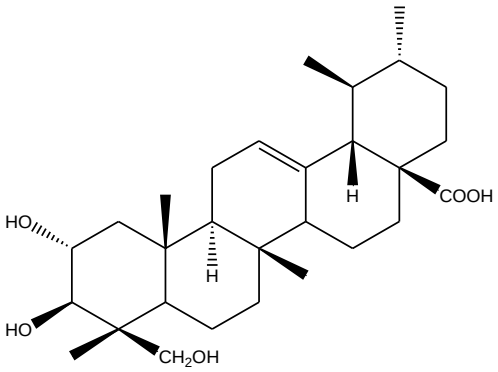

Figure 1.

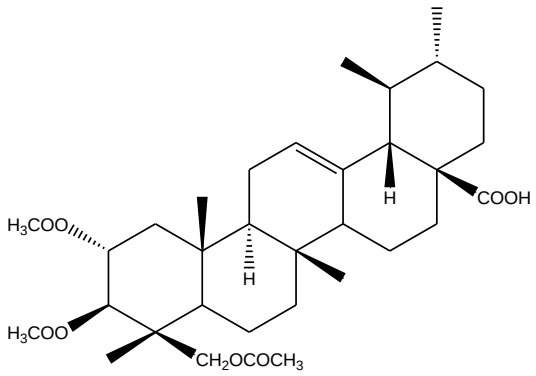

Figure 2.

EXPERIMENTAL

General details

Melting points were determined in glass capillary tubes in an electrothermal melting point apparatus. All solvents used were of analytical grade. The column and thin layer chromatography were conducted on silica gel (60-120 mesh). Spots on TLC plates were visualized in UV light, by spraying with 2% ceric ammonium sulphate in 2N H_2SO_4. The infrared spectra were recorded as KBr pellets on Perkin-Elmer 557 model spectrometer and A400S, Shimadzu, FT-IR spectrometer. ^{1}H NMR spectra were recorded on and Bruker DRX 200 FT NMR and Jeol Al 500 MHz instruments using $CDCl_3$ as solvents and TMS as an internal reference. EIMS spectra was recorded on a Hitachi model RMU 6E and Jeol D-300 mass spectrometer.

Plant material

The roots of *C. lanceolatus* DC. were collected from the Rajasthan University Campus, Jaipur and identified for authenticity in the Department of Botany, University of Rajasthan, Jaipur (Herbarium Sheet No. RUBL 20125).

Extraction and isolation

The roots were air-dried, powdered and exhaustively extracted with ethanol (95%) on a steam bath for 8 hrs thrice. The extract was concentrated under reduced pressure when a dark brown semi-solid was obtained. The ethanolic extract was re-extracted with pet. ether and ethyl acetate successively whereby on concentration under reduced pressure brown pet. ether, and reddish-brown ethyl acetate fractions were obtained. Since both the fractions exhibited a similar TLC profile (benzene : ethyl acetate, 1:1), they were mixed together and The combined fractions were chromatographed over a column of silica gel. Elution was carried out with solvents of increasing polarity, viz., pet.ether, benzene, ethyl acetate and methanol. The fractions were collected with benzene-ethylacetate (2:3) was repeatedly crystallized with ethyl acetate to afford compound **1** as white solid, m.p. 334-36^{o}C.

^{1}H-NMR (300 MHz, $CDCl_3$): 5.25 (1H, t), 3.68 (1H, m), 3.49, 3.26 (2H, ABq), 3.34 (1H, d), 1.92-113 (15H, m), and 1.08-0.74 (18H, d,s).

IR (ν_{max}) cm^{-1} (KBr): 3540, 3460, 3370, 3050, 2880, 1710, 1650, 1395, 1375, 1365, 1315, 1290, 1260, 1070, 1050, 1030, 1020, 860 and 800.

Mass (m/z): 488 [M^+], 470, 248, 235, 230, 204, 203, 185 and 133.

3.4 *Acetate of 2α,3β,24-Trihydroxyurs-12-en-28-oic acid*

Compound (50 mg), acetic anhydride (5 ml) and pyridine (2-3 drops) were refluxed for 4 hrs and the resulting mixture was poured in ice-cold water. The solid was filtered washed well with cold water and crystallized with ethyl acetate as white solid, m.p. 300-02 °C.

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