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Research Article

# A new pregnane steroid from the roots of Caralluma umbellate

[1]K. Sandhya , [2]Ch.Hemalatha ,[3]K. Suresh babu*

## ABSTRACT

A new pregnane steroid was isolated from the root of *Caralluma umbellata*, together with three known steroid compounds. The structure of **1** was elucidated on the basis of spectral evidence including 2D NMR studies.

***Keywords:*** Caralluma umbellata; Boucerosia; Asclepiadaceae; pregnane steroid.

## 1. Introduction

*Caralluma umbellata* (syn. *Boucerosia*) is an erect, branching, succulent perennial herb growing wild in Tirumala forest and surrounding places of Andhra Pradesh, India. It grows to a height of about 1 foot to 2 feet and the roots are fibrous. The plant, belonging to the family Asclepiadaceae, is medicinally important and rich in pregnane glycosides, which may possess different biological activities [1]. In folkloric medicine, as well as in unani and Ayurvedic systems of medicine, the plants of *Caralluma* are being used for the treatment of diabetic patients and rheumatism [2]. The pregnane steroid isolated from *C. umbellata* was shown to possess anti-inflammatory activity. Carumbelloside-I (3), isolated from *C. umbellata*, exhibited significant analgesic activity and antimicrobial activity [3]. A survey of the literature revealed that species of the genus *Caralluma* have been investigated on alcoholic extracts and found to be a source of steroids, triterpenes, and steroidal glycosides [4– 13]. The isolation and structural elucidation of three pregnane glycosides 2, 3, and 4 and one flavanone glycoside luteolin-4-*O*- neohesperidoside were reported from *C. umbellata* [14]. However, very few investigations were carried out on the extracts of non- polar solvents such as hexane . In order to search for any possible new compounds that could be extracted into non- polar solvents such as hexane, the present investigation was taken up and the results are reported in this paper.

## 2. Results and discussion

Compound 1 was isolated as a white powder. The Molecular formula was deduced from the HREIMS analysis at m/z 285.1 $[M+H]^+$, 13C NMR and DEPT experiments as $C_{21}H_{28}O$. The IR spectrum indicated that a hydroxyl group at 3258.12 $cm^{-1}$. The 1H NMR spectrum of compound 1 showed one proton singlet a δ 5.62 (H-6, *J*=5.5Hz), it suggests a olefinic proton is present. And two sharp singlets integrating each for three protons at δ 0.68 and δ 0.78 indicated the presence of two methyls, suggesting the presence of the pregnane-type nucleus [15]. The aliphatic methylene and methane protons in the region δ1.02 to δ 2.1 could be assigned to the pregnane

[1,3]Department of Sciences and Humanities, Matrusri Engineering college ,Saidabad, Telangana, India.
[2]Department of Freshmen Engineering, Marri Laxman Reddy Institute of technology and management, Hyderabad, Telangana, India

nucleus. The 13 C NMR spectral data (Table. 1) indicated the presence of 20 carbons, which were sorted by DEPT into two methyls, eight methylenes, five methines, and four quaternary carbons. The position of the group was established by HMBC, . The position of the Methyl was established by HMBC, where H-18 is showing correlations with C-12,C-13,C-14.Another Methyl H-19 is showing correlations with C-1,C-5,C-9,C10.Another relation is H-3 is showing correlations with C-2,C-4,C-5, another relation is H-20 is showing correlation with C-20,C-17.On the basis of the above evidence, the structure of 1 was determined as in Figure.1,

These results were supported by HSQC and COSY experiments (Figure 2)

**Table 1. $^{1}$H and $^{13}$C NMR spectral data of 1**

| No | $^{1}$H NMR | $^{13}$C NMR |
|---|---|---|
| **1** | 1.09 (m) | 36.2 |
| **2** | 1.89 (m) | 31.9 |
| **3** | 3.15 | 77.6 |
| **4** | 2.12 (m) | 39.8 |
| **5** | - | 140.1 |
| **6** | 5.62 | 122.1 |
| **7** | 2.08 (m) | 28.7 |
| **8** | 1.81 (m) | 37.8 |
| **9** | 1.45 (m) | 45.9 |
| **10** | - | 37.3 |
| **11** | 1.28 (m) | 26.5 |
| **12** | 2.18 (m) | 38.9 |
| **13** | - | 48.2 |
| **14** | 1.75(m) | 40.6 |
| **15** | 1.91 (m) | 44.5 |
| **16** | 1.97 (m) | 47.5 |
| **17** | 2.02 (m) | 50.1 |
| **18** | 0.68 | 15.1 |
| **19** | 0.78 | 17.5 |
| **20** | 3.45 | 70.8 |
| **21** | 2.13 (m) | 28.0 |
| **22** | 2.12 | 20 |

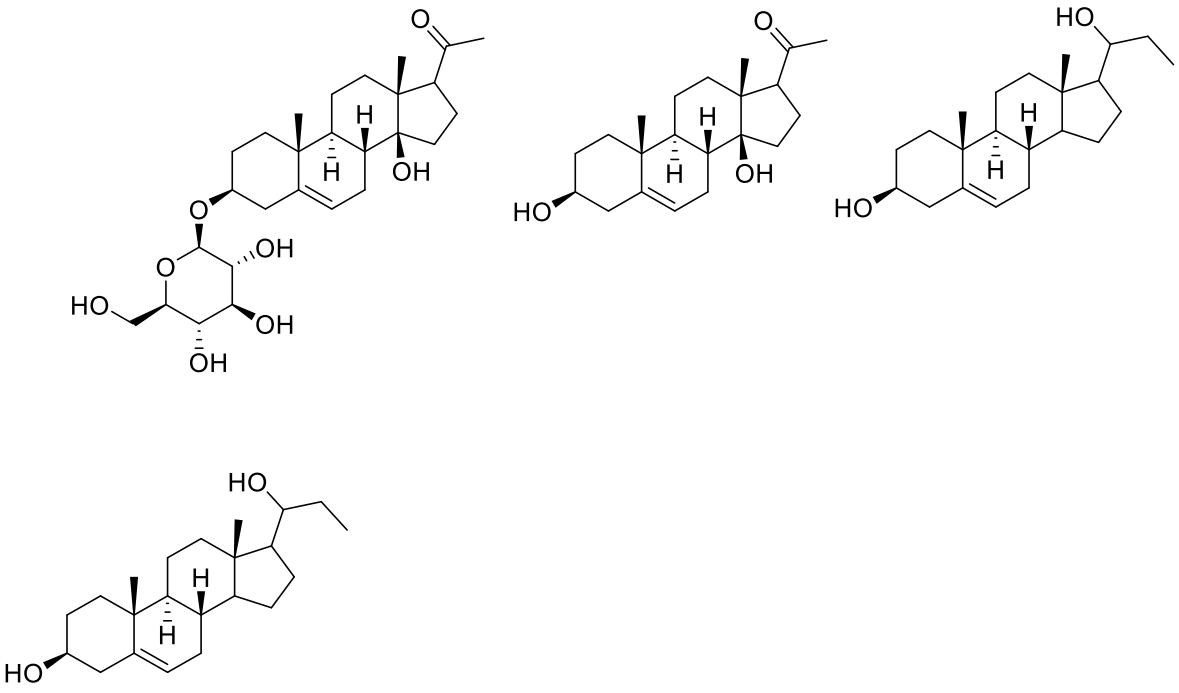

**Figure 1**

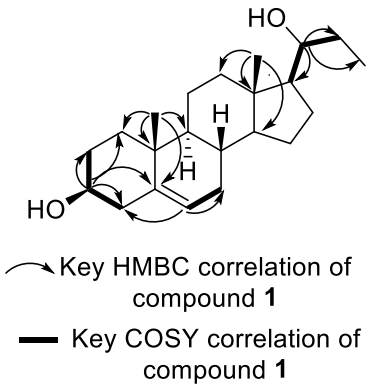


**Figure 2**

## 3. Experimental

*3.1 General experimental procedure*

Melting point was recorded on a Fisher–John apparatus. The specific rotation was recorded on a Perkin–Elmer precision Model-343. The IR spectra were recorded on an IFS- 120H spectrometer. The $^{1}H$ NMR and $^{13}C$ NMR spectra were obtained on a Bruker 300 MHz, 75 MHz spectrometer, using TMS as an internal standard. ESIMS was recorded on a ZAB-HS mass spectrometer and HREIMS was recorded on the Agilent Technologies 6510 Q-TOF LC/MS. Column chromatography was performed on silica gel (100–200 mesh). TLC was carried out on coated silica gel G glass plates with a thickness of 1 mm (PF 254, art 7747, Merck). Solvents and reagents were purified according to standard procedures.

### *3.2 Plant material*

The stems of *C. umbellata* (15 kg) were collected from the forests of Tirumala, Andhra Pradesh, India, in January 2007. It was identified by Dr K. Madhava Chetty, Department of Botany, Sri Venkateswara University, Tirupati. A voucher specimen of the plant is deposited in Herbarium, Department of Botany, with the accession number 1553.

### *3.3 Extraction and isolation*

The air-dried stems of *C. umbellata* (50 g) was powdered and extracted with petroleum ether, benzene, chloroform, and methanol successively. The petroleum ether extract was evaporated under reduced pressure to obtain a residue (7.0 g). The residue was adsorbed on silica gel and subjected to column chromatography eluted with benzene and by mixtures containing increasing amounts of EtOAc. The fractions eluted at 2% EtOAc in benzene were collected separately, concentrated, and repeated column chromatography using silica gel led to obtain compound **1** (20 mg) in pure form.

#### *3.3.1* Compound (1)

White powder , m.p.> $200^0$C, $[\alpha]_D^{20}$ = -180 (c 0.005, $CHCl_3$); IR $V_{max}$ (KBr) ($cm^{-1}$): 1733.69 (C=O); 1H NMR and 13 C NMR spectral data : see Table 1; HREIMS m/z 285.1 $[M + H]^+$ (calcd for $C_{21}H_{28}O$ $H^+$ 285.1025); ESIMS m/z 285. 1 [M + H] +.

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