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Attendiol, a new pentacyclic triterpenoid isolated from *Caralluma attenuata* root

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Abstract

From the root of *Caralluma attenuata* belonging to the family *Asclepiadaceae*, a novel pentacyclic triterpenoid was isolated and its structure was elucidated on the basis of spectroscopic data. It has two hydroxyl groups and named as *attendiol*. This is the first report of such novel pentacyclic triterpenoid from *Caralluma attenuata* root.

Keywords: *Caralluma attenuata*, *Asclepiadaceae*, *Attendiol*, pentacyclic triterpenoid

Introduction

Isolation and characterization of oxypregnane glycosides [1, 2], pregnane glycosides [3-9], triterpene saponins [10], flavonoids [11], pregnane esters [12], bisdesmosidic glycosides [13, 14], flavone glycosides [15], were earlier reported from the same genus. In the course of our investigation on chemical constituents of *Carallumas* we have earlier isolated and reported three new triterpenoids [16-18]. In this paper we report the structural elucidation of another novel pentacyclic triterpenoid derivative *attendiol* from *Caralluma attenuata*.

Experimental

The plant material of *Caralluma attenuata* was collected in Tirumala forests during Jan -2017. MPs uncorrected IR ν_{\max}^{KBr} , UV I_{\max}^{EtOH} , 1H NMR dppm, 300 MHz $CDCl_3$, ^{13}C NMR, COSY, CC and TLC on silica gel.

Extraction and Isolation

The roots of *Caralluma attenuata* were air dried, powdered and extracted with hexane, benzene, acetone and methanol respectively using soxhlet extractor. n-Hexane extract 50gms was subjected to column chromatography using silica gel 10-40 mesh. It is eluted with mixtures of benzene, benzene and methanol. Benzene fractions (66-72) yielded a white solid which showed single spot on TLC with R_f – 0.3125 (benzene as developing solvent), The solid obtained was recrystallised using benzene and acetone mixture and melting point was determined. MP 268 °C. This was analysed by spectral data.

Spectral Data

1H NMR δ ppm 1.19, 1.8, 1.34, 1.21, 1.3, 1.24, 1.256, 1.94, 1.0, 1.35, 1.45, 5.62, 2.15, 3.45
 ^{13}C NMR δ ppm 18.368, 41.2, 79.2, 40.541, 36.617, 27.70, 29.057, 27.951, 40.765, 34.496, 38.519, 28.890, 21.097, 22.751, 122.045, 141.890, 77.5.

Results and Discussions

The compound was isolated as white crystalline needles with MP 268 ° C and analysed for $C_{29}H_{48}O_2$ $[M^+]$ m/z 427 $[M-H]$, $[M^+]$ m/z 426 $[M-2H]$. The compound showed positive test for Libermann-Burchard reaction, Salkowski test indicating it to be a steroid/terpenoid.

The IR spectrum showed strong absorption at ν_{\max}^{KBr} 3263.15 cm^{-1} as a broad peak indicating the presence of –OH group. A strong absorption at ν_{\max}^{KBr} 1641.52 cm^{-1} indicates the presence of olefinic double bond.

Table 1: The important IR absorption peaks of *attendiol*

S. No.	Absorption cm^{-1}	Group Assignment
1	3263.15	O-H stretching vibration, the peak is broad
2	2955.78, 2847.55	C-H stretching in CH_2
3	1641.52	C = C stretching mode of unconjugated alkenes

The ^1H NMR spectrum showed the characteristic ring protons ranging from δ 0.6-1.8 indicating the presence of pentacyclic triterpenoid [19].

A two proton triplet at δ 3.45 indicates the presence of 1° -OH which is supported by ^{13}C NMR spectra. The ^{13}C NMR signal at δ 77.5 indicates the presence of 1° -OH. This is observed in DEPT as CH_2 carbon. A two proton triplet at δ 2.15 indicates presence of allylic proton supported by ^{13}C NMR δ 34.49 which is also observed in DEPT as CH_2 carbon.

The cross peak in COSY indicates the existence of coupling between allylic protons and CH_2OH supporting that allylic carbon is directly attached to CH_2OH . A singlet signal at δ 1.35 accounting for 3 protons indicates $-\text{CH}_3$ attached to a tertiary carbon with electronegative group accounting for a tertiary hydroxy group at C-3 which is further supported by ^{13}C NMR signal at δ 79.2 which is not seen in DEPT.

^1H NMR and ^{13}C NMR spectral analysis

Table 2: ^1H NMR and ^{13}C NMR spectral analysis of *attendiol*

Carbon NO	^1H Nmr Data	^{13}C Nmr Data	Carbon NO	^1H Nmr Data	^{13}C Nmr Data
1	δ 1.19 δ 1.8	18.368	2	δ 1.19 δ 1.8	41.2
3	-----	79.2	4	1.9	40.541
5	1.34	36.617	6	δ 1.21 δ 1.8	27.70
7	δ 1.21 δ 1.8	29.057	8	1.3	36.617
9	1.3	36.617	10	1.3	36.617
11	δ 1.19 δ 1.8	29.057	12	δ 1.19 δ 1.8	29.057
13	1.24	36.617	14	1.3	36.617
15	δ 1.21 δ 1.8	29.057	16	δ 1.21 δ 1.8	27.951
17	1.3	36.617	18	1.24	36.617
19	δ 1.19 δ 1.8	40.765	20	-----	34.496
21	δ 1.256 δ 1.94	38.519	22	δ 1.256 δ 1.94	28.890
23	1.0	21.097	24	1.35	18.368
25	1.45	22.751	26	5.62	122.045
27	-----	141.890	28	2.15	34.496
29	3.45	77.5			

A two proton doublet at δ 5.62 indicates the presence of a disubstituted olefinic bond weak C-H absorption in IR at $\nu_{\text{max}}^{\text{KBr}}$ 1397 cm^{-1} indicates the presence of terminal double bonds which is supported by ^{13}C NMR spectra. The ^{13}C NMR signals at δ 141.890 and δ 122.05 indicates the presence of olefinic carbons. ^{13}C NMR signal at δ 141.890 is not observed in DEPT clearly indicates that this olefinic carbon to be a quaternary carbon. The signal at δ 122 is seen in DEPT

as CH_2 carbon indicating a disubstituted terminal olefinic double bond $>\text{C}=\text{CH}_2$. ^1H NMR shows a doublet signal corresponding to 3H at δ 1.0 indicating the presence of methyl attached to methine [$-\text{CH}-$] carbon. It also shows a singlet at δ 1.35 indicates a methyl group attaches to a tertiary carbon. DEPT spectrum showed carbon resonances relating to $-\text{CH}$, $-\text{CH}_2$ and $-\text{CH}_3$ protons present in the compound which are in agreement with ^{13}C NMR resonances.

Table 3: DEPT analysis of *attendiol*

Carbon No	-CH	- CH_2	- CH_3	^{13}C
1	--	18.368	--	18.368
2	--	41.2	--	41.2
3	--	-----	--	79.2
4	40.541	--	--	40.541
5	36.617	--	--	36.617
6	---	27.70	--	27.70
7	--	29.057	--	29.057
8	36.617	---	--	36.617
9	36.617	---	--	36.617
10	36.617	--	--	36.617
11		29.057	--	29.057
12	--	29.057	--	29.057

13	36.617	---	--	36.617
14	36.617	---	--	36.617
15	--	29.057	---	29.057
16	-----	27.951	---	27.951
17	36.671	----	-----	36.617
18	36.617	---	--	36.617
19	----	40.765	--	40.765
20	--	--	---	34.496
21	--	38.519	---	38.519
22	--	28.890	--	28.890
23	--	--	21.097	21.097
24	--	--	18.368	18.368
25	--	--	22.751	22.751
26	--	122.045	-----	122.045
27		----	----	141.890
28	--	34.496	-----	34.496
29	--	77.5	--	77.5

Thus, based on IR, NMR (^1H NMR and ^{13}C) partial structure of the compound *attendiol* can be given as,

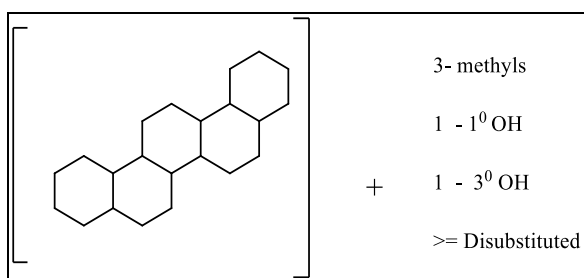


Fig 1: partial structure of the compound *attendiol*

Based on the triterpenoids isolated from the same plant the position of tertiary -OH in *attendiol* can be given as

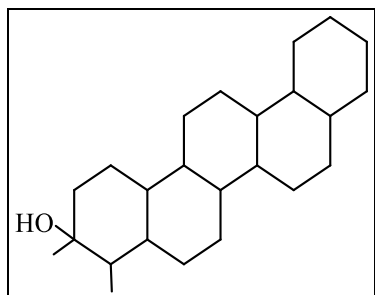


Fig 2: partial structure of *attendiol* with tertiary -OH group

Based on ^{13}C NMR, DEPT and COSY supported by mass spectral fragmentation the structure of the compound *attendiol* can be given as,

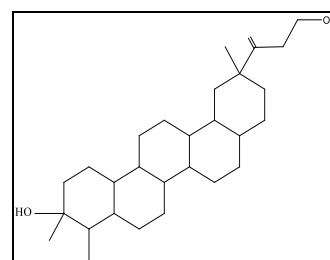
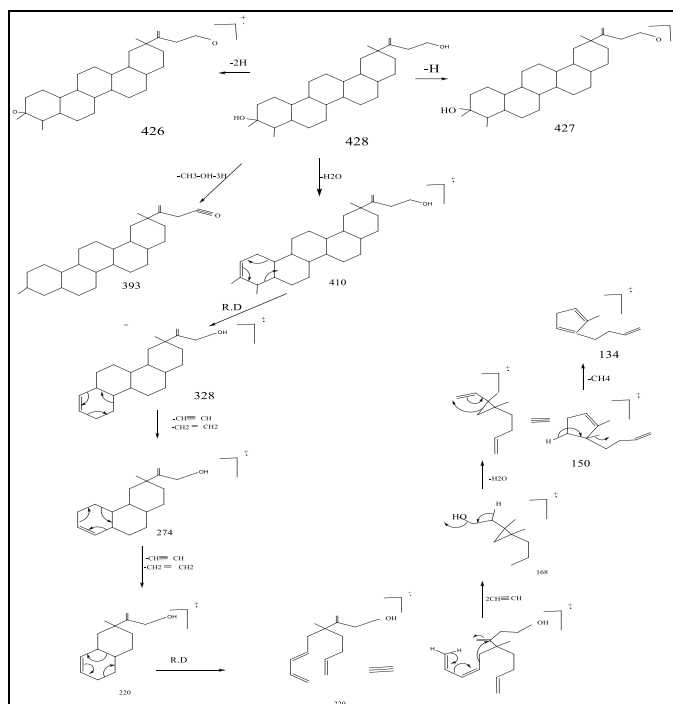


Fig 3: Structure of *attendiol*

This structure is further supported by the mass spectral fragmentation as given in scheme-I which explains most of the mass spectral peaks. This terpenoid is isolated and reported for the first time from this plant as well as from nature.

Table 4: The mass spectral data of *attendiol*

S. No.	m/z	Ion formed
1	427	[M - H]
2	426	[M - 2H]
3	393	[M - CH ₃ -OH-3H]
4	274	[M - H ₂ O - C ₆ H ₁₀ - C ₂ H ₂ - C ₂ H ₄]
5	150	[M - H ₂ O - C ₆ H ₁₀ - C ₂ H ₂ - C ₂ H ₄ - C ₄ H ₆ - C ₄ H ₄ - H ₂ O]
6	134	[M - H ₂ O - C ₆ H ₁₀ - C ₂ H ₂ - C ₂ H ₄ - C ₄ H ₆ - C ₄ H ₄ - H ₂ O - CH ₄]



Scheme 1: MASS spectral fragmentation of compound

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References

- Abdel-Sattar E, Al-Yahya MMA, Nakamur N, Hattori M. Penicillosides A-C oxypregnane glycosides from *Caralluma penicillata*. *Phytochemistry*. 2001; 57:1213-1217.
- Abdel-Sattar E, Meselhy RM, Al-Yahya MAA. New oxypregnane glycosides from *Caralluma penicillata*. *Planta Med*. 2002; 68:430-434.
- Ahmad VU, Khan U, Rizwani GH. New pregnane glycosides from *Caralluma russeliana*. *J Nat. Prod*. 1988; 51(6):1092-1097.
- Al-Yalhya MAA, Abdel-Sattar E, Guittet E. Pregnane glycosides from *Caralluma russeliana* *J Nat. Pro*. 2000; 63:1451-1453.
- Braca A, Badar A, Morelli I, Scarpato R, Turchi G, Pizza C, Tammasi D *et al*. New pregnane glycosides from *Caralluma negevensis*. *Tetrahedron*. 2002; 58:5837-5842.
- Halim AF, Khalil AT. Pregnane glycosides from *Caralluma retropiciens*. *Phytochemistry*. 1996; 42(4):1135-1139.
- Hayash K, Lida L, Nakao Y, Kaneko K. Four pregnane glycosides *Boucerosides* AI AII, BI and BII from *Boucerosia aucheriana*. *Phytochemistry*. 1998; 27(12):3919-3924.
- Lin LJ, Lin GLLR, Cordell LGA, Ramesh M, Srilatha B, Reddy BM, Appa Rao AVN *et al*. Pregnane glycosides from *Caralluma umbellate*. *Phytochemistry*. 1994; 35(6):1549-1553.
- Tanaka T, Tsukamoto S, Hayashi K. Pregnane glycosides from *Boucerosia aucheriana*. *Phytochemistry*. 1990; 29(1):229-237.
- Jen-Woo Jhoo, Kantle Shengmin Sang, Xiaofong Cheng, Nargurzhuth Ruth, Stark E, Qun Yizheng *et al*. *J Agri. Food Chem*. 2001; 40:5969-5974.
- Kamil M, Jayaraj AF, Ahmad F, Gunasekhar C, Samuel S, Chan K *et al*. Identification and quantification of flavanoids from *Caralluma Arabica* and their quality control studies. *J Pharm. Pharmacol*. 1999; 51:225.
- Khalil AT. *Fitoterapia*. Pregnane esters from *Caralluma retropiciens*. 1995; 66(3):261-264.
- Qiu SX, Lin L, Cordell GA, Ramesh M, Ravi kumar B, Radhakrishna M, Krishnamohan G *et al*. Acylated C-21 steroidal from *Caralluma lasiantha*. *Phytochemistry*. 1997;46(2):333-340.
- Qiu SX, Cordell, Ramesh M, Ravikuma B, Radhakrishna M, Mohan GK *et al*. Bisdesmosidic pregnane glycosides from *Caralluma lasiantha*. *Phytochemistry*. 1999; 50:485- 491.
- Ramesh M, Nageswara Rao Y, Ramakumar Kumar M, Krishna Mohan G, Ravikumar B, Madhakumar AVN *et al*. Flavone glycosides from three *Caralluma lasiantha*. *Biochemical systematics and Ecology*. 1999; 27:85-86.
- Jayalakshmi G, Anuradha V, Kalyani K, Sivaram Babu S. A Novel pentacyclic triterpenoid isolated from *Caralluma attenuata* root. *European journal of pharmaceutical and medical research*. 2016; 3(6):342-344.
- Jayalakshmi G, Kavitha K, Ratnakumari S, Anuradha V, Sivaram Babu S. A Novel pentacyclic triterpenoid isolated from *Caralluma attenuata* root. *European journal of pharmaceutical and medical research*. 2017; 4(6):710-712.
- Jayalakshmi G, Anuradha V, Ratna Kumari S, Sivaram babu S, Rabbani Basha SK. Structural elucidation of a New Novel pentacyclic triterpenoid from *Caralluma attenuata* Root. *Derpharma chemical*. 2019; 11(3):13-18.
- Jin-Woo Jhoo, Kan He Shengmin Sang, Xiafang Chenm, Nanqun Zhu, Ruth E, Stark *et al*. Characterisation of the terpene saponins of the Roots and Rhizomes of Blue Cohosh (*Caulophyllum thalictroides*) *J Agri. Food Chem*. 2001; 49:12.