Indian Journal of Chemistry
Vol. 40B, September 2001, pp. 867-868

Note

New sesquiterpenoid from the soft coral *Sinularia dissecta*†

P Ramesh, V Ravikanth & Y Venkateswarlu*

Natural Products Laboratory, Organic Chemistry Division-I
Indian Institute of Chemical Technology, Hyderabad 500 007, India

Fax: +91-40-7173757/7173387. E-mail: root@csiict.ren.nic.in

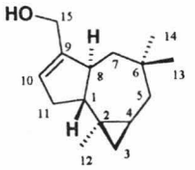
New sesquiterpenoid 15-hydroxy-Δ^9-africanene **1** has been isolated from the soft coral *Sinularia dissecta*. Its structure has been elucidated based on spectral analysis.

In continuation of our interest[1-3] in the bioactive secondary metabolites from the marine organisms, we examined the soft coral *Sinularia dissecta* Tixier Durivault (*Alcyoniidae*) collected from the Mandapam Coast, Tamilnadu during June 1996. From this soft coral *Sinularia dissecta*, we reported five new polyoxygenated steroids[4,5], two new diterpenes[6,7], four sesquiterpenoids[8] and a β-alanine methyl ester derivative[9] for the first time. We report herein the isolation and structural elucidation of 15-hydroxy-Δ^9-africanene **1**.

The dichloromethane - methanol (1:1) extract of the soft coral *S. dissecta* was partitioned between water and ethyl acetate. The concentrated ethyl acetate extract was subjected to gel filtration chromatography followed by silica gel chromatography to afford the new compound 15-hydroxy-Δ^9-africanene **1**.

Compound **1** was obtained as colourless oil, analyzed for $C_{15}H_{24}O$ by EIMS (M^+ 220) and requires four degrees of unsaturation. Its IR spectrum showed strong absorptions at 3500 and 1650 cm^{-1} for the presence of hydroxyl group and double bond, respectively.

The ^1H NMR spectrum of compound **1** showed signals for the presence of three tertiary methyls at δ 1.05 (3H, s), 0.98 (3H, s) and 0.90 (3H, s) and a trisubstituted cyclopropane ring at δ 0.21 (1H, m) and 0.50 (2H, m). Further, the downfield ^1H NMR spectrum showed signals for an olefinic proton at δ 5.56 (1H, br s) and methylene protons bearing oxygen atom at δ 4.05 (2H, br s). The foregoing spectral data were similar to those of $\Delta^{9(15)}$-africanene (**2**)[10,11], except for the signals at δ 4.05 and 5.56 and devoid of exocyclic methylene protons at δ 4.68 (1H, br s) and 4.84 (1H, br s). This suggests that the presence of hydroxy group at C-15 and the migration of double bond between C-9 and C-10 positions.



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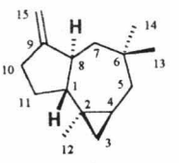

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These assignments were supported by its ^{13}C NMR spectral signals at δ 146.8 (s), 131.2 (d) and 60.95 (t). The foregoing spectral data and a literature survey established the structure of compound **1** as 15-hydroxy-Δ^9-africanene.

Experimental Section

^1H and ^{13}C NMR spectra were recorded on Varian Unity 400 MHz and Varian Gemini 200 MHz spectrometers using TMS as internal standard. Chemical shifts are reported in parts per million, coupling constants (*J*) are expressed in Hertz and ^{13}C NMR spectra are fully ^1H decoupled. IR spectra were recorded on Perkin-Elmer 240-C instrument and mass spectra on a VG Auto spec-M instrument.

Collection, Extraction and Isolation

Fresh specimens of the soft coal *S. dissecta* (Tixier Durivault) were collected at 20 feet depth on the Mandapam Coast, Tamilnadu during June 1996, and a voucher specimen (IIC-233) deposited at the National Institute of Oceanography, Goa, India. The soft coral *S. dissecta* (2 kg dry weight) was extracted with CH_2Cl_2-MeOH (1:1, 3 × 3 L) at room temperature and the combined extracts were filtered and the solvent evaporated under reduced pressure to yield a greenish gum (90 g). The crude extract was partitioned between water and ethyl acetate. The organic layer was concentrated under vacuum and subjected to gel filtration chromatography (Sephadex LH-20; 1:1 dichloromethane-methanol) followed by silica gel chromatography eluting with hexane, hexane/ethyl acetate mixtures, and finally with ethyl acetate. The new compound 15-hydroxy-Δ^9-africanene **1** was obtained in hexane-ethyl acetate (90:10) eluent.

†IICT Communication No. 4464

15-Hydroxy-Δ^9-africanene 1: Colourless oil, yield 8 mg; $[\alpha]_D$ =6.43 (*c* 0.45 $CHCl_3$); IR (neat): 3500, 1650 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.21 (1H, m), 0.50 (2H, m), 0.65-0.82 (1H, m), 0.90 (3H, s), 0.98 (3H, s), 1.05 (3H, s), 1.21 (2H, m), 1.40 (1H, m), 2.05-2.32 (3H, m), 2.78 (1H, m), 4.05 (2H, br s), 5.56 (1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.63 (s), 20.47 (d), 21.75 (q), 22.67 (t), 23.45 (q), 32.36 (t), 33.92 (s), 33.92 (q), 43.31 (t), 44.41 (d), 48.54 (d), 52.83 (t), 60.95 (t), 131.20 (d), 146.8 (s); EIMS: m/z 220 [M^+], 202, 149, 139, 95, 69, 41.

Acknowledgement

We are thankful to Dr (Mrs) Jayasree for identifying the soft coral. We thank Dr A C Kunwar for providing NMR data. We also thank the Director, IICT and Dr J S Yadav for their encouragement. This research work was supported by the Department of Ocean Development, New Delhi, India, and the authors (P R and V R) are thankful to the UGC, New Delhi and CSIR, New Delhi for providing fellowships.

References

1 Venkateswarlu Y, Ramesh P, Sridevi K V & Prabhakara Rao T, *Indian J Chem*, 37, **1998**, 832.
2 Venkateswarlu Y, Srinivasa Reddy N, Ramesh P, Rama Rao M & Siva Ram T, *Indian J Chem*, 37, **1998**, 1264.
3 Ramesh P, Srinivasa Reddy N & Venkateswarlu Y, *J Nat Prod*, 62, **1999**, 780.
4 Venkateswarlu Y, Ramesh P, Srinivasa Reddy N & Prabhakara Rao T, *Nat Prod Lett*, 13, **1999**, 229.
5 Ramesh P & Venkateswarlu Y, *Steroids*, 64, **1999**, 785.
6 Ramesh P, Srinivasa Reddy N, Venkateswarlu Y, Venkata Rami Reddy M & Faulkner D J, *Tetrahedron Lett*, 39, **1998**, 8217.
7 Ramesh P & Venkateswarlu Y, *J Chem Res (S)*, **2000**, 48.
8 Ramesh P, Srinivasa Reddy N, Prabhakar Rao T & Venkateswarlu Y, *J Nat Prod*, 2, **1999**, 1019.
9 Ramesh P, Srinivasa Reddy N, Prabhakar Rao T, Venkateswara Rao J & Venkateswarlu Y, *Bio Syst Ecol*, 27, **1999**, 661.
10 Kashman Y, Bodner M, Finer-Moore J S & Clardy J, *Experientia*, 36, **1980**, 891.
11 Braekman J C, Daloze D, Tursch B, Hall S E, Declereq J P, Germail G & Meerssche M V, *Experientia*, 36, **1980**, 893.