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Journal of Asian Natural Products Research

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713454007>

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To cite this Article Lan, W.-J. , Li, H.-J. , Yan, S.-J. , Su, J.-Y. and Zeng, L.-M.(2007) 'New tetraterpenoid from the soft coral *Sarcophyton tortuosum*', Journal of Asian Natural Products Research, 9: 3, 267 – 271

To link to this Article: DOI: 10.1080/10286020600604344

URL: <http://dx.doi.org/10.1080/10286020600604344>

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New tetraterpenoid from the soft coral *Sarcophyton tortuosum*

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(Received 1 August 2005; revised 6 December 2005; in final form 16 December 2005)

A novel tetraterpenoid named methyl tortuoate C (**1**) has been isolated from the soft coral *Sarcophyton tortuosum* Tix.-Dur. The structure of **1** was determined on the basis of spectroscopic methods.

Keywords: Soft coral; *Sarcophyton tortuosum*; Tetraterpenoid; Methyl tortuoate C

1. Introduction

In the previous paper [1] we reported two novel tetraterpenoids, methyl tortuoate A and methyl tortuoate B, isolated from the soft coral *Sarcophyton tortuosum* Tix.-Dur. After further research, we obtained another new tetraterpenoid, methyl tortuoate C (**1**) from the same ethanolic extract of the soft coral. Herein, we report the isolation and structural determination of this compound.

2. Results and discussion

Tetraterpenoid methyl tortuoate C (**1**) was obtained as colourless needles with mp 235–236°C and $[\alpha]_D^{25} + 320$ (c. 0.03, CHCl₃). HRFAB-MS m/z 705.4355 $[M + Na]^+$ revealed a molecular formula of C₄₁H₆₂O₈. The IR spectrum indicated the existence of hydroxy (3354 cm⁻¹), carbonyl (1698 cm⁻¹) and ester groups (1735 cm⁻¹). The ¹³C NMR spectral data revealed 41 carbon atoms for the molecule, which consists of three carbonyl groups (δ_C 214.0 s, 212.9 s, 209.2 s), one ester group (δ_C 174.5 s), eight sp² carbons (δ_C 140.1 s, 138.0 d, 135.3 s, 129.9 s, 127.9 s, 125.2 d, 120.6 d, 120.1 d), nine methyl groups (δ_C 51.1 q, 22.3 q, 21.3 q, 21.4 q, 20.2 q, 19.7 q, 19.1 q, 17.6 q, 17.3 q) and three oxygen-bearing carbons (δ_C 79.8

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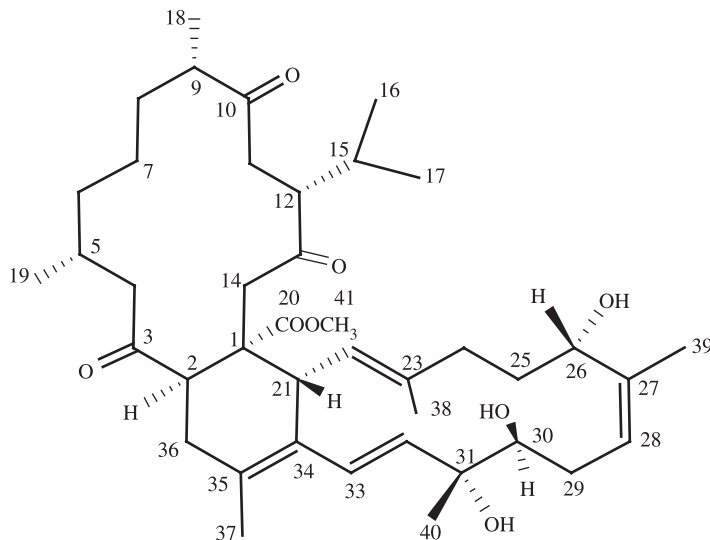


Figure 1. Structure of methyl tortuatoate C (**1**).

d, 75.9 d, 74.0 s). This finding showed that **1** was a tetracyclic tetraterpenoid similar to methyl tortuatoate A and methyl tortuatoate B that we have isolated from this soft coral [1] (figure 1).

The structure and the assignment of the ^1H and ^{13}C chemical shifts of **1** were further determined by the assistance of 2D NMR (^1H - ^1H COSY, HMQC, HMBC and NOESY). All proton and carbon signals have been assigned from HMQC spectrum. The correlations of ^1H - ^1H COSY revealed seven proton-proton networks, as shown in figure 2. HMBC experiments showed long-range correlations between C-1/H-2, H-14; C-3/H-2, H-4 and H-36; C-10/H-9, H-11 and Me-18; C-13/H-12, H-14; C-20/H-14, H-21; C-23/H-24, Me-38; C-24/Me-38; C-27/H-26, Me-39; C-31/H-30, H-32 and H-33; C-30/Me-40; C-33/H-21;

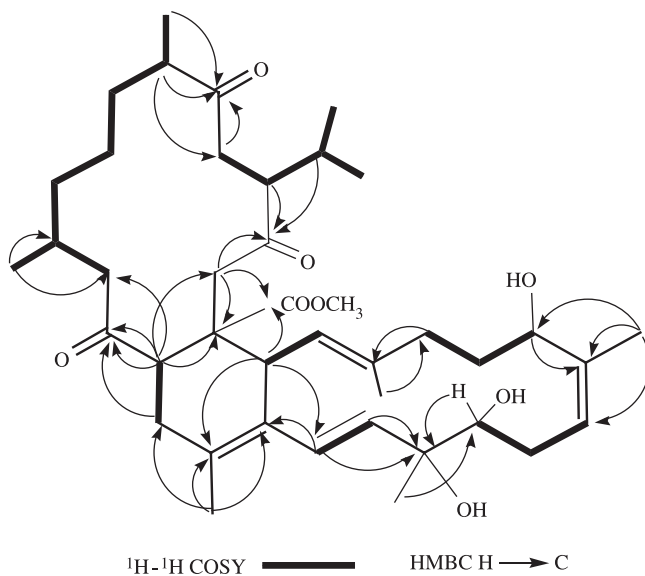


Figure 2. ^1H - ^1H COSY and Key HMBC correlations of **1**.

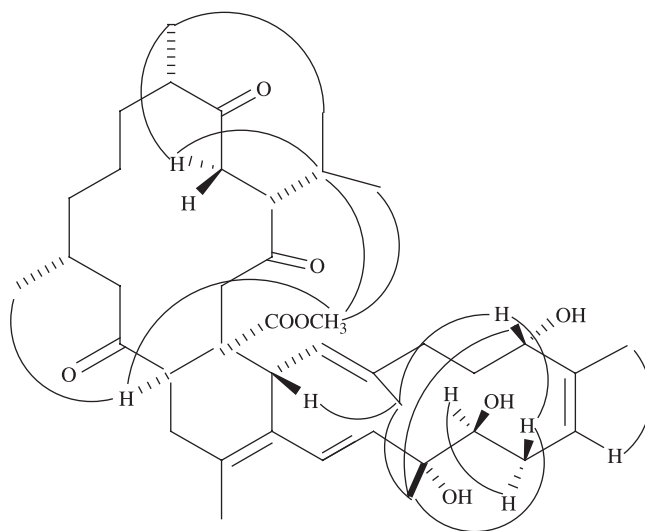


Figure 3. Key NOESY correlations of **1**.

C-34/H-33, H-37; C-35/H-21, Me-37 (figure 2). Thus, the seven moieties of the proton–proton network can be connected and the planar structure of **1** was established.

The relative configurations of the nine chiral carbons of **1**, depicted in figure 3, were determined from NOESY correlation peaks (figure 3). As referred to the lowest chiral carbon (C-1) having *R*-chirality [2], the relative configurations of the chiral centres were determined as 1*R**, 2*R**, 5*R**, 9*S**, 12*R**, 21*R**, 26*R**, 30*S**, 31*R**. The *E*-configuration of the $\Delta^{32,33}$ double bonds was established on the basis of the large couple constant ($J_{32,33} = 16.0$ Hz). Besides, the 22*E*, 27*Z*, and 34*E* configuration of the olefinic bonds were deduced by the ¹³C NMR chemical shifts [3] of the vinyl methyl groups (Me-38, Me-39 and Me-37) of **1**. In the NOE studies, interaction between H-28 and H₃-39 was observed, confirming a *Z* configuration for 27,28-double bond. Alternatively, NOE interactions could not be observed between H-22 and H₃-38, demonstrating an *E* configuration for 22,23-double bond.

3. Experimental

3.1 General experimental procedures

Melting points were determined using an X-6 micromelting point apparatus and are uncorrected. ¹H NMR and ¹³C NMR spectra were recorded at 500 and 125 MHz, respectively, in CDCl₃ using TMS as internal standard. Optical rotations were measured on a Schmidt + Haensch Polaptronic hnqw5 polarimeter. HPLC separation was performed with a Perkin-Elmer series 200 instrument. Reverse-phase HPLC was carried out using a Symmetry prep C₁₈ (7 μm, 7.8 × 300 mm) column at 8 ml/min and monitored at 210 nm.

3.2 Animal material

The soft coral *Sarcophyton tortuosum* Tix.-Dur.(Alcyoniidae) was collected from Sanya Bay, Hainan Island, China, in 2002 and was identified by Dr. Zhican Tang, Institute of

Oceanology, Academia Sinica. A voucher specimen is deposited in the Research Centre of Organic Natural Products, Sun Yat-sen University.

3.3 Extraction and isolation

The soft coral *S. tortuosum* (300 g, dried weight) was extracted with CH₂Cl₂/MeOH (1:1) to give a brown extract, which was extracted with petroleum ether and then with EtOAc. The residue in EtOAc was chromatographed on silica gel eluted with petroleum ether (PE) containing increasing concentrations of EtOAc. Methyl tortuoate A was obtained from the fraction eluted with PE/EtOAc (3:1); methyl tortuoate B was obtained from the fraction eluted with PE/EtOAc (2:1). Fraction C, eluted with PE/EtOAc (1:1), was subjected to flash chromatography on silica gel eluted with PE/EtOAc (1:1) to afford methyl sartortuoate and

Table 1. NMR data of methyl tortuoate C (**1**) (500 MHz for ¹H and 125 MHz for ¹³C in CDCl₃).

Position	¹³ C δ m	¹ H δ (m, J in Hz)	¹ H- ¹ H COSY
1	50.0 s		
2	44.1 d	3.43 (1H,dd, 7.0, 8.5)	H-36
3	209.2 s		
4	53.1 t	2.32 (2H,m)	H-5
5	27.3 d	1.85 (1H, s)	H-4, H-6, H-19
6	37.2 t	1.04 (1H,m); 1.15 (1H, m)	H-5, H-7
7	25.2 t	1.20 (2H, m)	H-6, H-8
8	32.0 t	1.53 (1H,m); 1.67(1H,m)	H-7, H-9
9	47.3 d	2.58 (1H, m)	H-8, H-18
10	213.0 s		
11	33.5 t	1.69 (1H,m); 2.16 (1H, dd, 20.0, 6.5)	H-12
12	52.1 d	3.07 (1H, m)	H-11
13	212.0 s		
14	45.5 t	3.13 (1H,m); 2.82 (1H,d, 18.5)	
15	28.8 d	2.32 (1H, m)	H-16, H-17
16	17.6 q	0.71 (3H, d, 7.0)	H-15
17	21.3 q	1.01(3H, d, 7.0)	H-15
18	17.3 q	1.13 (3H, d, 7.0)	H-9
19	22.3 q	0.85 (3H, d, 7.0)	H-5
20	174.5 s		
21	46.0 d	3.29 (1H, br d, 11.0)	H-22
22	125.2 d	4.74 (1H, d, 11.0)	H-21
23	140.1 s		
24	38.8 t	1.74 (1H,m); 2.35 (1H,m)	H-25
25	33.6 t	1.52 (2H, m)	H-24, H-26
26	79.8 d	3.99 (1H, br d, 9.5)	H-25
27	135.3 s		
28	120.1 d	5.52 (1H, d, 4.0)	H-29
29	24.9 t	1.93 (1H, m); 2.06 (1H, m)	H-28, H-30
30	75.9 d	3.15 (1H, m)	H-29
31	74.0 s		
32	138.0 d	5.78 (1H, d, 16.0)	H-33
33	120.6 d	6.40 (1H, d, 16.0)	H-32
34	127.9 s		
35	129.9 s		
36	32.2 t	2.17 (1H,m); 2.56 (1H,m)	H-2
37	19.7 q	1.86 (3H, s)	
38	19.1 q	1.81 (3H,s)	
39	20.2 q	1.61 (3H,s)	
40	21.2 q	1.28 (3H, s)	
41	51.1 q	3.54 (3H, s)	

a mixture of methyl sartortuoate and methyl tortuoate C. The mixture was then separated by RP-HPLC eluting with MeOH/H₂O (9:1) to give pure methyl sartortuoate (known compound) [4] and pure methyl tortuoate C (**1**) (6.5 mg), respectively.

Methyl tortuoate C (**1**): colourless prisms, mp 235–236°C; $[\alpha]_D^{25} + 320$ (c. 0.030, CHCl₃); IR (KBr) ν_{\max} 3354, 1735, 1698, 1194, 1105 cm⁻¹; ¹H NMR and ¹³C NMR spectra data: see table 1; HRFAB-MS *m/z* 705.4355 [M + Na]⁺ (calcd for C₄₁H₆₂O₈ Na, 705.4350).

Acknowledgements

This work was supported by a grant from the National High Technology Development Project (863 Project, No. 2001AA620403) and a grant from the National Natural Science foundation of China (No. 29932030 and No. 20502036) and the Natural Science Foundation of Guangdong Province (05300667).

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