

Ximaosteroids A–D, new steroids from the Hainan soft coral *Scleronephthya* sp.

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ABSTRACT

One novel uncommon steroid, ximaosteroid A (**1**), possessing an unusual tetrahydrofuran moiety and three new pregnane steroids, ximaosteroids B–D (**2–4**), were isolated from the Hainan soft coral *Scleronephthya* sp. Their structures were elucidated on the basis of detailed spectroscopic (IR, MS, and 2D NMR) analysis and by comparison with those reported in the literature.

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1. Introduction

Soft corals are recognized as very prolific sources of novel bioactive substances [1]. Numerous secondary metabolites spanning a wide range of structure class and various biological activities were isolated from different species of soft corals such as genera *Sarcophyton*, *Lobophytum*, *Sinularia*, etc. [2–4]. However, a literature survey revealed that chemical studies on the genus *Scleronephthya* (order Alcyonacea, family Nephtheidae) were relatively rare and only a few metabolites mainly pregnane steroids have been reported to date [5–8]. In the course of our ongoing program towards the isolation of biologically active substances from Chinese marine organisms [9–13], we have recently examined an unknown species of the *Scleronephthya* soft coral, collected off the coast of Ximao Island, Hainan Province, China, resulting in the isolation of a series of steroids [14]. Our continuous studies on the minor constituents of the same collection led to the isolation of four new steroids, named ximaosteroids A–D (**1–4**) (Fig. 1). The present paper deals with the isolation and structural elucidation of these new compounds.

2. Experimental

2.1. General methods

Optical rotations were measured on a PerkinElmer polarimeter 341 at the sodium D-line, cell length 100 mm. UV spectra were recorded on a 756 CRT spectrophotometer. IR spectra were recorded on a Nicolet-Magna FT-IR 750 spectrometer, peaks are reported in cm^{-1} . The NMR spectra were measured on a Bruker Avance-500 spectrometer (500 MHz for ^1H and 125 MHz for ^{13}C), using the residual CHCl_3 signal (δ_{H} 7.26 ppm) as an internal standard for ^1H NMR and CDCl_3 (δ_{C} 77.0 ppm) for ^{13}C NMR. Chemical shifts are expressed in δ (ppm) and coupling constants (J) in Hz. ^1H and ^{13}C NMR assignments were supported by ^1H - ^1H COSY, HSQC, HMBC and ROESY experiments. EIMS and HREIMS data were obtained on a Finnigan-MAT-95 mass spectrometer. ESIMS and HRESIMS spectra were recorded on a Q-TOF Micro-LC-MS-MS mass spectrometer. Reversed-phase HPLC (Agilent 1100 series liquid chromatography using a VWD G1314A detector at 210 nm and a semi-preparative ZORBAX ODS (5 μm , 250 mm \times 9.4 mm (i.d.)) column) was also employed. Commercial Silica gel (Qing Dao Hai Yang Chemical Group Co., 200–300 and 400–600 mesh) was used for column chromatography (CC), and precoated silica gel plates (Yan Tai Zi Fu Chemical Group Co., G60 F-254) were used for analytical TLC.

2.2. Animal material

The soft coral *Scleronephthya* sp. was collected off Ximao Island, Hainan Province, China, in December 2001, at a depth of –20 m and identified by Professor R.-L. Zhou of South China Sea Institute

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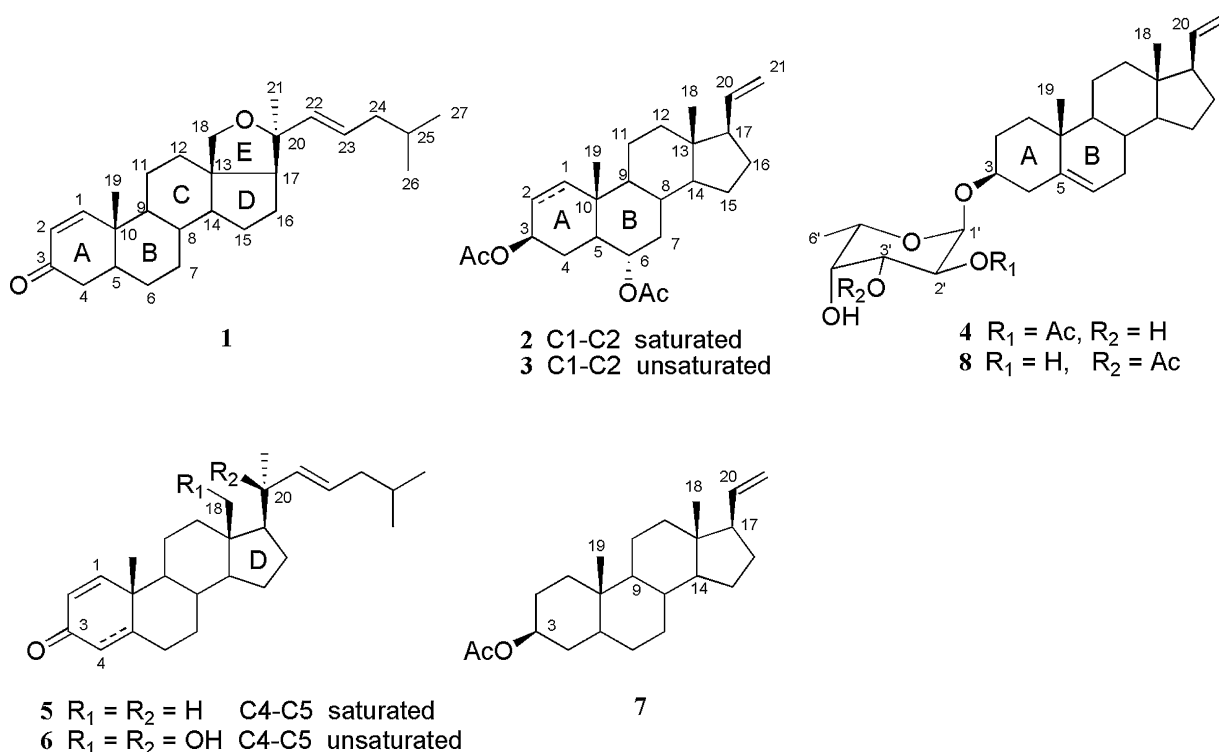


Fig. 1. Structures of 1–8.

of Oceanology, Chinese Academy of Sciences. A voucher specimen (HN-54) is available for inspection at Shanghai Institute of Materia Medica, CAS.

2.3. Extraction and isolation

The frozen animals (150 g dried weight) were cut into pieces and exhaustively extracted with acetone at room temperature (1.5 L 3×). The organic extract was evaporated to give a residue, which was partitioned between Et₂O and H₂O. The Et₂O solution was concentrated under reduced pressure to give a dark green residue (2.8 g), which was fractionated by gradient silica gel CC (0–100% acetone in petroleum ether), yielding 10 fractions. Fraction 2 was chromatographed on a silica gel column (400–600 mesh, petroleum ether/Et₂O, 95:5) to afford 7 (2.1 mg). Fraction 3 was firstly subjected to a silica gel CC (400–600 mesh, petroleum ether/Et₂O, 90:10), and then RP-HPLC [MeOH/H₂O (3:1), 2.0 mL/min] to give compounds 2 (1.6 mg) and 3 (1.3 mg). Fraction 5 gave compound 1 (1.3 mg) after CC on silica gel (400–600 mesh, petroleum ether/Et₂O, 70:30). Fraction 6 was purified by silica gel CC (400–600 mesh, petroleum ether/Et₂O, 65:35), followed by CC on Sephadex LH-20 (petroleum ether/CHCl₃/MeOH, 2:1:1) to yield compound 4 (1.4 mg).

2.3.1. Ximaosteroid A (1)

Colorless oil; $[\alpha]_D^{20} +12.0$ (c 0.13, CHCl₃); UV (MeOH) λ_{\max} (log ϵ) 223 (3.30) nm; IR (KBr) ν_{\max} 2926, 2868, 1734, 1682, 1464, 1240, 1047, 976, 779 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) and ¹³C NMR (CDCl₃, 125 MHz) data, see Table 1; EIMS 70 eV m/z (rel. int.%) 396 [M]⁺ (28), 381 (100), 339 (51), 270 (23); HREIMS m/z 396.3012 [M]⁺ (calcd for C₂₇H₄₀O₂, 396.2995).

2.3.2. Ximaosteroid B (2)

Colorless oil; $[\alpha]_D^{20} +33.0$ (c 0.16, CHCl₃); IR (KBr) ν_{\max} 3448, 2949, 2854, 1736, 1650, 1450, 1375, 1246, 1030, 990, 910, 608 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) and ¹³C NMR (CDCl₃, 125 MHz) data, see Tables 2 and 3; EIMS 70 eV m/z (rel. int.%)

Table 1

¹H and ¹³C NMR data of ximaosteroid A (1)^a and ¹³C NMR data of 5 and 6.

Position	1	5	6
	δ_H mult. (J in Hz)	δ_C mult.	δ_C mult.
1	7.11 d (10.3)	158.2 d	158.7 d
2	5.86 d (10.3)	127.5 d	127.4 d
3	–	200.0 s	200.4 s
4 α	2.23 dd (17.6, 3.3)	41.0 t	41.1 t
4 β	2.37 dd (17.6, 14.3)	–	–
5	1.91 m	44.3 d	44.4 d
6	1.42 m	27.5 t	28.7 t
7	1.82 m	31.6 t	31.4 t
8	1.29 m	38.0 d	35.7 d
9	1.01 m	49.3 d	50.1 d
10	–	38.9 s	39.1 s
11 α	1.84 m	23.2 t	21.3 t
11 β	1.05 m	–	–
12 α	2.14 m	37.4 t	39.7 t
12 β	1.39 m	–	–
13	–	42.5 s	42.7 s
14	1.21 m	55.7 d	56.5 d
15	1.88 m	26.1 t	24.2 t
16	1.56 m	26.3 t	27.7 t
17	2.01 m	55.8 d	56.0 d
18 α	3.55 d (9.0)	72.3 t	12.4 q
18 β	3.63 d (9.0)	–	–
19	0.92 s	12.9 q	13.0 q
20	–	84.7 s	40.2 d
21	1.25 s	25.0 q	20.9 q
22	5.45 d (15.6)	138.1 d	138.0 d
23	5.57 dt (15.6, 7.2)	126.5 d	126.5 d
24	1.93 m	41.6 t	42.0 t
25	1.65 m	28.6 d	28.6 d
26	0.90 d (6.6)	22.5 q	22.4 q
27	0.91 d (6.6)	22.3 q	22.3 q

^a Spectra recorded at 500 MHz in CDCl₃. Assignments were based on DEPT, ¹H–¹H COSY, HMQC, and HMBC experiment.

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