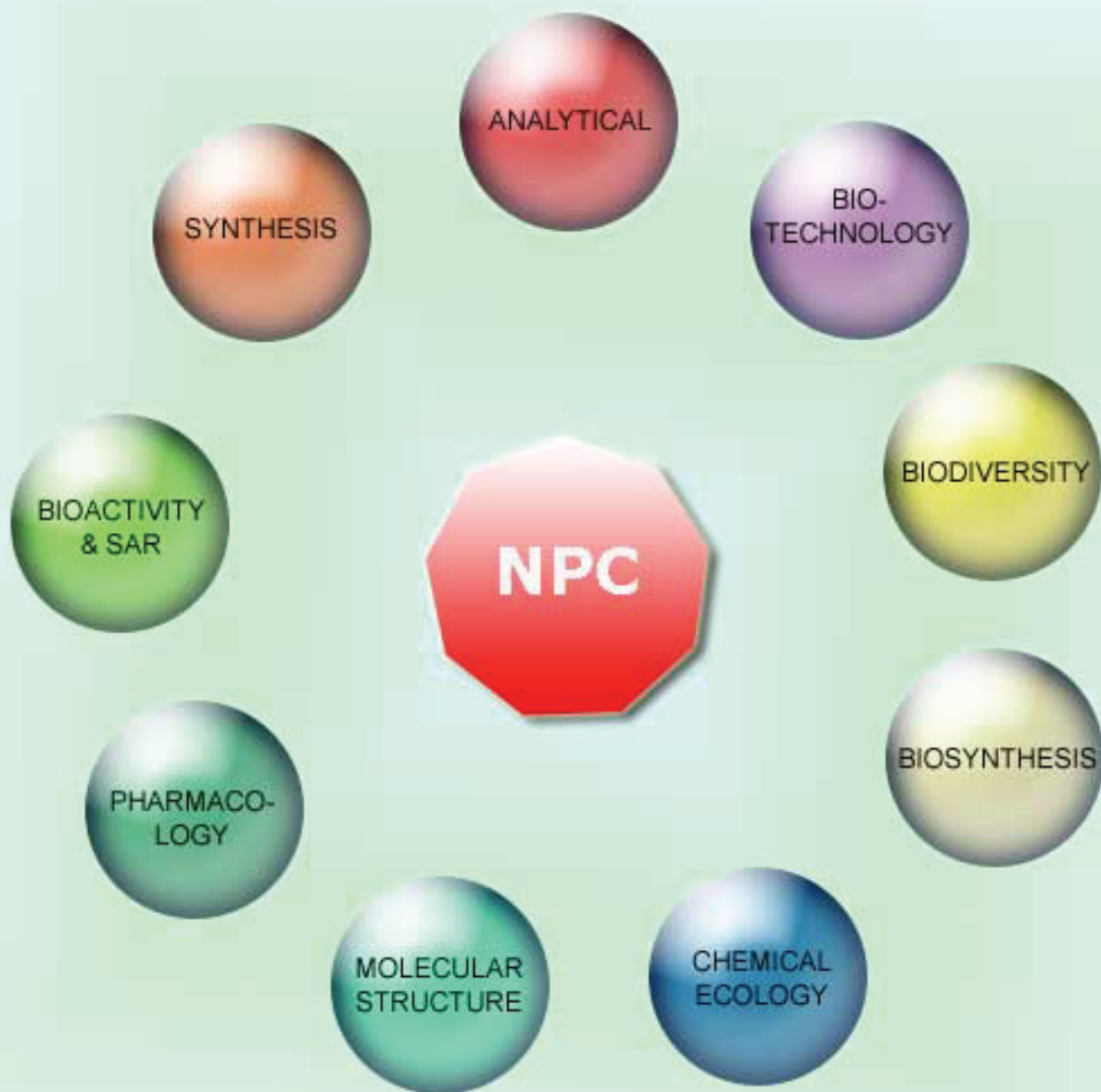


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A New Cytotoxic Polyacetylenic Alcohol from a Sponge *Callyspongia* sp.Walter Balansa<sup>a,b</sup>, Agus Trianto<sup>c</sup>, Nicole J. de Voogd<sup>d</sup> and Junichi Tanaka<sup>a,\*</sup><sup>a</sup>Department of Chemistry, Biology and Marine Science, University of the Ryukyus, Nishihara, Okinawa 903-0213, Japan<sup>b</sup>Department of Fisheries and Marine Science, Nusa Utara Polytechnic, Tahuna Sangihe Islands, North Sulawesi 95812, Indonesia<sup>c</sup>Department of Marine Sciences, Diponegoro University, Tembalang-Semarang, Central Java 50275, Indonesia<sup>d</sup>Naturalis Biodiversity Center, P. O. Box 9517, 2300 RA Leiden, The Netherlands

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A new cytotoxic polyacetylenic alcohol **1** was isolated from an Indonesian sponge *Callyspongia* sp. The structure of compound **1** was elucidated by spectral analyses and by applying modified Mosher's method. Compound **1** killed the cultured NBT-II rat bladder carcinoma cells at 5 and 10 µg/mL.

**Keywords:** *Callyspongia*, Sponge, Polyacetylene, Cytotoxicity.

Polyacetylenes with linear carbon skeletons have been reported from both terrestrial and marine organisms. Among the marine-derived polyacetylenes, compounds vary in the level of unsaturation, carbon chain lengths, oxygenation patterns and terminal such as in polyacetylenic alcohols [1]. Particularly, marine sponges of the genera *Callyspongia*, *Theonella*, *Haliclona* and *Petrosia* produce polyacetylenic alcohols incorporating a 1-yn-3-ol terminal, with antiviral, antimicrobial, antitumor, cytotoxic, neurotogenic and  $\alpha$ -glucosidase inhibiting activities [1-7]. Two reports have described this pharmacophore as playing key roles in antitumor and neurotogenic activities [8-9]. However, despite featuring the 1-yn-3-ol terminal and having been published for nearly two decades, very little is known about biological activity and structural variation of callyspongyne-type polyacetylenes [10].

In our collaborative project for finding new bioactive molecules from Indonesian marine organisms [11-12], we became interested in the extract of a *Callyspongia* sponge collected off Flores Island. The <sup>1</sup>H NMR of the extract showed characteristics of the structurally uncommon callyspongyne-type polyacetylenes [10]. The data prompted us to further examine the structure and bioactivity of the major fraction of the extract, leading to the discovery of a new and cytotoxic polyacetylenic alcohol **1** [13]. This note describes the structure elucidation and cytotoxicity of compound **1** (Figure 1).

Since ESIMS of compound **1** showed a sodiated ion at *m/z* 459.3605, the molecular formula was deduced as C<sub>31</sub>H<sub>48</sub>O ( $\Delta +0.4$  ppm) with eight degrees of unsaturation. Spectral data indicated the presence of the following functional groups: two terminal and one internal acetylenes at  $\delta_H$  2.57, 3.07;  $\delta_C$  73.9, 79.5, 80.3, 80.8, 83.3, 81.4; 3280, 2115 cm<sup>-1</sup>, two double bonds at  $\delta_H$  5.48, 5.61, 5.92, 6.01;  $\delta_C$  108.6, 128.3, 134.8, 145.2, one secondary alcohol at  $\delta_H$  4.84;  $\delta_C$  62.8; 3359 cm<sup>-1</sup>, eight methylenes at  $\delta_H$  1.35-2.42, and a methylene chain at  $\delta_H$  1.27 brs;  $\delta_C$  29.4-29.7. As three acetylenes and two double bonds satisfied all the unsaturation degrees, the compound was suggested to be a new member of linear callyspongyne-type polyacetylenes.

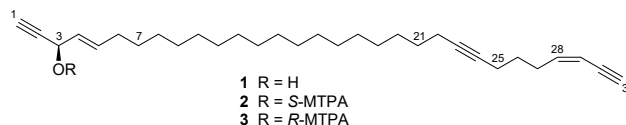


Figure 1: Structures for compounds 1-3.

Table 1: <sup>1</sup>H and <sup>13</sup>C NMR Data for Compound **1** in CDCl<sub>3</sub>.

Position	<sup>13</sup> C	<sup>1</sup> H	COSY	HMBC
1	73.9 CH	2.57 d, <i>J</i> = 2.4 Hz		C-3
2	83.3 C	-		
3	62.8 CH	4.84 brs	H-4	C-2,4,5
OH		1.83 brd, <i>J</i> = 6.0 Hz		C-2,3,4
4	128.3 CH	5.61 ddt, <i>J</i> = 15.2, 6.1, 1.4 Hz	H-3,5	C-2,3,6
5	134.8 CH	5.92 brdt, <i>J</i> = 15.2, 6.7 Hz	H-4,6	C-3,6,7
6	31.9 CH <sub>2</sub>	2.06 brq, <i>J</i> = 7.0 Hz	H-5,7	C-4,5,7
7	28.8 CH <sub>2</sub> <sup>a</sup>	1.38 m	H-6,8	C-5,6,8
8	29.1 CH <sub>2</sub> <sup>b</sup>	1.27 brs		
9-19	29.4-7 CH <sub>2</sub>	1.27 brs		
20	28.9 CH <sub>2</sub> <sup>a</sup>	1.35 m		C-21
21	29.2 CH <sub>2</sub> <sup>b</sup>	1.48 quint, <i>J</i> = 7.3 Hz	H-20,22	C-20,22,23
22	18.7 CH <sub>2</sub>	2.13 tt, <i>J</i> = 7.3, 2.1 Hz	H-21,25	C-23,24
23	80.8 C	-		
24	79.5 C	-		
25	18.4 CH <sub>2</sub>	2.18 tt, <i>J</i> = 7.3, 2.1 Hz	H-22,26	C-23,24,26,27
26	28.3 CH <sub>2</sub>	1.61 quint, <i>J</i> = 7.3 Hz	H-25,27	C-24,25,27,28
27	29.7 CH <sub>2</sub>	2.42 brq, <i>J</i> = 7.3 Hz	H-26,28	C-25,26,28,29,30
28	145.2 CH	6.01 dt, <i>J</i> = 11.0, 7.3 Hz	H-27,29	C-30
29	108.6 CH	5.48 brd, <i>J</i> = 11.0 Hz	H-28,31	C-27
30	80.3 C	-		
31	81.4 CH	3.07 d, <i>J</i> = 2.1 Hz	H-29	C-30

<sup>a</sup> and <sup>b</sup> indicate that the signals are exchangeable.

One of acetylenic protons at  $\delta_H$  2.57 showed an HMBC cross peak with the oxymethine at  $\delta_C$  62.8 (C-3), which proton at  $\delta_H$  4.84 showed correlations to C-2, 4 and 5 indicating that the alcohol is flanked by a terminal acetylene and a *trans* double bond (*J* = 15.2 Hz). The double bond is connected to methylenes at  $\delta_C$  31.9 and 28.8 (C-6 and 7). This moiety has been found in polyacetylenes from marine origins such as petrosynol [3], and the data was consistent with those reported for analogs [10].

Another terminal acetylenic signal at  $\delta_H$  3.07 (H-31) showed a COSY cross peak to one of *cis* olefinic protons at  $\delta_H$  5.48 (*J* = 11.0 Hz, H-29). The other *cis* vinyl proton at  $\delta_H$  6.01 showed a

correlation with a methylene at  $\delta_{\text{H}}$  2.42 (H-27), which in turn coupled to a characteristic methylene at  $\delta_{\text{H}}$  1.61 appearing as a quintet. This methylene showed COSY cross peaks to the neighbor methylenes at C-25 and 27, and also HMBC cross peaks to one of internal acetylenic carbons at  $\delta_{\text{C}}$  79.5 (C-24) in addition to methylenes at C-25 and 27, and an olefin at C-28. The proton signal at H-25 showed two coupling constants: one with  $J = 7.3$  Hz for vicinal coupling to H-26 and the other  $J = 2.1$  Hz for long range coupling with H-22. The signal at H-22 appeared similar to that of H-25, and COSY and HMBC supported the presence of H-20 and 21. The data for C21-C31 are consistent with those reported for analogous moieties [14,15].

Both the methylene groups at H-7 and H-20 showed HMBC correlations to overlapped methylenes at  $\delta_{\text{C}}$  28-29 suggesting that the remaining methylenes connect the above two terminal units.

For the sole chiral center at C-3, we applied modified Mosher's method [16] to form esters **2** and **3**. As results,  $\Delta\delta_{2,3}$  values were +0.11, +0.05 and +0.04 for H-4, 5 and 6, while -0.04 for H-1 as shown (Figure 2). Therefore, C-3 was concluded to be *R* configuration and the whole structure of **1** and the MTPA esters (**2**, **3**) can be depicted as in Figure 1.

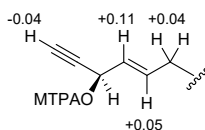


Figure 2:  $\Delta\delta_{2,3}$  values of MTPA esters.

Compound **1** killed the cultured NBT-II rat bladder carcinoma cells at 5 and 10  $\mu\text{g}/\text{mL}$  (Supplementary material).

## Experimental

**General:** Reagent-grade solvents were used. HPLC separation was carried out on a unit of Hitachi HPLC instruments with a Cosmosil 5SL-II column. NMR spectra were measured on a Jeol Alpha 500 NMR spectrometer. ESI-MS were measured on a Micromass LCT instrument. FTIR spectra were obtained on a Jasco FTIR-300 spectrophotometer, respectively. Optical rotation was measured on a Jasco DIP-1000 polarimeter.

**Sponge:** A specimen of the purple soft sponge *Callyspongia* sp. was collected by hand using SCUBA at a depth of 43 m off Labuhan Bajo, Flores, Indonesia in August, 2001. The specimen (01Z25) was examined by one of us (NJdV).

**Extraction and Isolation:** The specimen (0.95 g after drying) was frozen at the collection site. After brought back to laboratory, it was extracted with acetone (150 mL) three times. The lipophilic portion (0.23 g) was subjected to a silica gel column to give seven fractions. The first fraction (38.6 mg) was purified on a silica HPLC column (Cosmosil 5SL-II, 6 x 250 mm) eluted with *n*-hexane-EtOAc (3-1) to give five subfractions. The third subfraction (22.8 mg) was further purified by silica HPLC (Cosmosil 5SL-II, 6 x 250 mm) with *n*-hexane-EtOAc (6-1, 4.0 mL/min) to give compound **1** (7.3 mg, eluted at 7.02 min).

## Compound 1

White powder.

## References and note

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$[\alpha]_{\text{D}}^{25}$ : -7.9 (*c* 0.046,  $\text{CHCl}_3$ ).

IR (KBr): 3359, 3035, 2921, 2354, 1668  $\text{cm}^{-1}$ .

$^1\text{H}$  and  $^{13}\text{C}$  NMR (500 and 125 MHz,  $\text{CDCl}_3$ ): Table 1.

ESIMS:  $m/z$  675, 490, 413, 360, 304, 241, 185.

HRESIMS:  $m/z$  459.3605  $[\text{M}+\text{Na}]^+$ ; calcd for  $\text{C}_{31}\text{H}_{48}\text{ONa}$ : 459.3603.

**MTPA esters 2 and 3 from compound 1:** A solution of compound **1** (0.6 mg, 0.0013 mmol) in 50  $\mu\text{L}$  of dry  $\text{CH}_2\text{Cl}_2$  was added to a stirring solution of (-)- $\alpha$ -methoxy- $\alpha$ -(trifluoromethyl)phenyl acetic acid (*S*-MTPA) (1.0 mg, 0.0042 mmol), dicyclohexylcarbodiimide (DCC) (0.5 mg, 0.0024 mmol) and 4-dimethylaminopyridine (DMAP) (0.1 mg, 0.0008 mmol) in  $\text{CH}_2\text{Cl}_2$ . The mixture was allowed to stand for 12 h at room temperature. The reaction mixture was filtered off and the filtrate was purified by HPLC (*n*-hexane-EtOAc, 3-1) to give *S*-MTPA ester **2** (0.7 mg, 0.0010 mmol, 78%). Using (+)- $\alpha$ -methoxy- $\alpha$ -(trifluoromethyl)phenyl acetic acid (*R*-MTPA) in place of *S*-MTPA, *R*-MTPA ester was also similarly prepared from 0.6 mg of compound **1**, resulting in **3** (1.0 mg, 0.0015 mmol, 89%).

## S-MTPA ester 2

White powder.

$[\alpha]_{\text{D}}^{25}$ : -196 (*c* 0.007,  $\text{CHCl}_3$ ).

$^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  7.26-7.45 (5H, m), 6.07 (1H, m, H-5), 6.02 (1H, m, H-3), 6.01 (1H, m, H-28), 5.61 (1H, dd,  $J = 16.5, 6.5$  Hz, H-4), 5.48 (1H, m, H-29), 3.07 (1H, d,  $J = 2.5$  Hz, H-31), 2.59 (1H, d,  $J = 2.0$  Hz, H-1), 2.42 (2H, brq,  $J = 15.0, 8.0$  Hz, H-27), 2.18 (2H, m, H-25), 2.13 (2H, m, H-22), 2.08 (2H, q,  $J = 7.0$  Hz, H-6), 1.61 (2H, quint,  $J = 7.0$  Hz, H-21), 1.48 (2H, m, H-21), 1.38 (2H, m, H-7), 1.26 (26H, m, H-8~H-20).

HRESIMS:  $m/z$  675.4028  $[\text{M}+\text{Na}]^+$ ; calcd for  $\text{C}_{41}\text{H}_{55}\text{F}_3\text{O}_3\text{Na}$ : 675.4001.

## R-MTPA ester 3

White powder.

$[\alpha]_{\text{D}}^{25}$ : -116 (*c* 0.010,  $\text{CHCl}_3$ ).

$^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  7.26-7.45 (5H, m), 6.03 (1H, m, H-3), 6.02 (1H, m, H-5), 6.01 (1H, m, H-28), 5.50 (1H, m, H-4), 5.48 (1H, m, H-29), 3.07 (1H, d,  $J = 2.5$  Hz, H-31), 2.63 (1H, d,  $J = 2.0$  Hz, H-1), 2.42 (2H, brq,  $J = 13.5, 7.5$  Hz, H-27), 2.18 (2H, m, H-25), 2.13 (2H, m, H-22), 2.04 (2H, q,  $J = 7.0$  Hz, H-6), 1.61 (2H, quint,  $J = 7.0$  Hz, H-26), 1.48 (2H, m, H-21), 1.38 (2H, m, H-7), 1.26 (26H, m, H-8~H-20).

HRESIMS:  $m/z$  675.4025  $[\text{M}+\text{Na}]^+$ ; calcd for  $\text{C}_{41}\text{H}_{55}\text{F}_3\text{O}_3\text{Na}$ : 675.4001.

**Preliminary cytotoxicity test:** Suspensions of NBT-II cells in 1 mL DMEM (Dulbecco's Modified Eagle Medium) were dispensed into 24 wells. After preincubation for 24 h, DMSO (Dimethylsulfoxide) solution of compound **1** was added to each well to adjust the final concentration at 5 and 10  $\mu\text{g}/\text{mL}$ . After 48 h incubation, each well was observed under a microscope.

**Supplementary data:**  $^1\text{H}$ ,  $^{13}\text{C}$  and 2D NMR spectra of compound **1** and cell assay images are available.

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