2-PALMITAMIDOETHANESULFONIC ACID, A TAURINE DERIVATIVE FROM THE MARINE SPONGE *Haliclona* SP.

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UDC 547.314

Marine sponges belonging to the genus *Haliclona* have been the subject of extensive chemical studies [1, 2]. Recent investigations of *Haliclona* species have led to the isolation of alkaloids, macrolides, polyacetylenes, polyketides, steroids, and peptides [3–8]. During our search for secondary metabolites from marine sponges, we have isolated a taurine derivative (**1**) from the marine sponge *Haliclona* sp. collected from South China Sea.

The sponge was collected by hand in July 2005, off the coast of Hainan Island, China. The specimen was identified by Dr. Kyung Jin Lee, Hannam University, Daejeon, Korea. A voucher specimen (0507003) was deposited at Key Laboratory of Marine Bio-resources Sustainable Utilization, South China Sea Institute of Oceanology, Chinese Academy of Sciences.

The sponge (20 kg) was extracted with EtOH at room temperature. The EtOH extract was partitioned between $CHCl_3$ and water. The $CHCl_3$ layer was further partitioned between 80% aqueous EtOH and *n*-hexane. The 80% aqueous EtOH fraction was subjected to a reversed-phase flash column chromatography (YMC Gel ODS-A, 60 Å, 230 mesh) with a stepped gradient solvent system of 50 → 95% EtOH/H_2O to afford 11 fractions. Fraction 3 (5.76 g) was separated by a reversed-phase flash column chromatography followed by a gradient of 50 → 75% MeOH to afford 11 subfractions. Fraction 3–6 (2.03 g) was separated by repeated silica flash column chromatography followed by a gradient of 5 → 25% MeOH in $CHCl_3$ to afford 30 subfractions. Compound **1** (5.8 mg) was obtained by separation of the subfraction 3–6–3–6–4–27.

Compound **1** was isolated as colorless crystals. The molecular formula was established as $C_{18}H_{37}NO_4S$ on the basis of negative ESI-MS, EI-MS, and NMR data. The ^1H and ^{13}C NMR, HSQC, and HMBC spectral data indicated the presence of one terminal methyl, 14 aliphatic methylenes, one nitrogenous methylene, one sulfur-bearing methylene, and one amide carbonyl. The ^1H NMR signals at δ_H 3.61 (2H, t, J = 6.85 Hz, H-2) and 2.98 (2H, t, J = 6.85 Hz, H-1) and ^{13}C NMR signals at δ_C 51.5 (C-1) and 36.6 (C-2) indicated the presence of a taurine moiety [9, 10]. The S atom was ascertained by the key fragmentation of **1** in EI-MS (Fig. 1). Thus compound **1** was identified as 2-palmitamidoethanesulfonic acid.

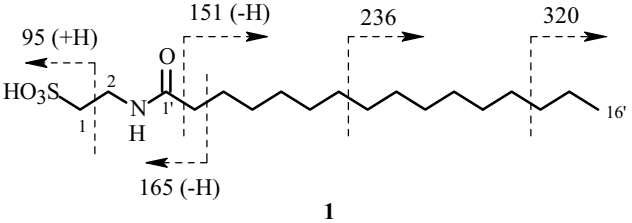

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Fig. 1. Key fragmentation of **1** in EI-MS.

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0009-3130/09/4501-0137 ©2009 Springer Science+Business Media, Inc.

Compound **1**. Colorless cristals, mp 296.5–299.0°C. ^{1}H NMR (500 MHz, CD_3OD, J/Hz): δ_H 3.61 (2H, t, J = 6.85, H-2), 2.98 (2H, t, J = 6.85, H-1), 2.20 (2H, t, J = 7.35, H-2′), 1.61 (2H, m, H-3′), 1.31–1.40 (24H, m, H-4′–H-15′), 0.90 (3H, t, J = 6.55, H-16′); ^{13}C NMR (125 MHz, CD_3OD): δ_C 51.5 (C-1), 36.6 (C-2), 176.1 (C-1′), 37.2 (C-2′), 26.9 (C-3′), 30.4–30.8 (overlap C-4′–C-14′), 23.1 (C-15′), 14.5 (C-16v); ESI-MS (negative mode) *m/z* 362 $[M–H]^-$; EI-MS (Positive mode) *m/z* 364 $[M+H]^+$ (3), 320 (5), 236 (16), 179 (18), 165 (18), 151 (30), 95 (70), 81 (75).

ACKNOWLEDGMENT

The authors gratefully acknowledge the support of National Natural Science Foundation of China (No. 40706046) the Knowledge Innovation Program (LYQY200703) and the Hundred Talents Project of the Chinese Academy of Sciences and Guangdong Key Lab of Marine Materia Medica Foundation.

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