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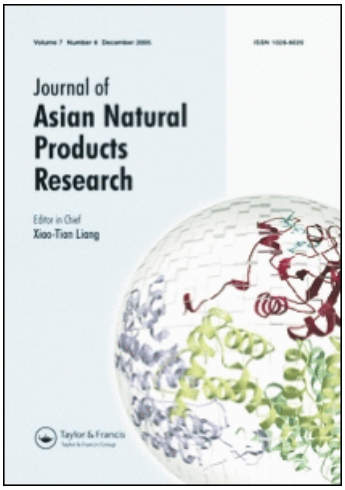

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Two new compounds from marine *Streptomyces* sp. FX-58

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Two new compounds from marine *Streptomyces* sp. FX-58

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Two new compounds, 5-carboxymethyl-2-propylchromone (**1**) and 1,6-dihydroxy-8-propylanthraquinone (**2**), together with a known anthraquinone, 3,8-dihydroxy-1-propylanthraquinone-2-carboxylic acid (**3**), were isolated from the mycelium of an actinomycete, *Streptomyces* sp. FX-58, which was separated from a marine plant collected in Qingdao. Their structures were determined based on spectroscopic methods, especially 2D NMR spectral analysis.

Keywords: Marine actinomycete; Chromone; Anthraquinone

1. Introduction

In recent years, many new biologically active secondary metabolites were found in marine microorganisms, which have become a good source of modern pharmaceutics [1]. During the course of screening bioactive natural compounds from marine microorganisms, we obtained three compounds including 5-carboxymethyl-2-propylchromone (**1**), 1,6-dihydroxy-8-propylanthraquinone (**2**) and 3,8-dihydroxy-1-propylanthraquinone-2-carboxylic acid (**3**). Compound **1** is a new chromone. Compound **2** is a new anthraquinone. Here, we report the isolation and structural elucidation of the two new compounds.

2. Results and discussion

Compound **1** was isolated as colourless fine needles (MeOH), mp 121–126°C. The UV (MeOH) spectrum displayed bands at 300, 245 and 224 nm, which hinted the compound might be a chromone derivative [2]. Its molecular formula was determined as $C_{14}H_{14}O_4$ by HRSI-MS [*m*/*z* 247.0964 $(M + H)^+$]. In the ^1H NMR spectrum of **1**, the signals for three

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aromatic protons, one olefinic proton, three methylenes and a methyl group were observed. Signals of δ 1.05 (3H, t, $J = 7.4$ Hz, H-3′), 1.80 (2H, q-like, $J = 7.4$ Hz, H-2′) and 2.68 (2H, t, $J = 7.4$ Hz, H-1′) were assigned to a propyl function by analysis of ^{1}H–^{1}H COSY and HMBC data (figure 1). In the ^{13}C NMR spectrum of **1**, 14 carbon signals were observed, which were classified into 2 carbonyl carbons, 6 aromatic carbons, 2 olefinic carbons and 4 alkyl carbons. ^{13}C NMR data matched with the basic skeleton of 4-chromone for ring B [2–4]. The signal at δ 174.0 (C-5′) revealed the presence of a carboxylic acid group and it was also supported by the analysis of the EI-MS, which exhibited a prominent peak at *m/z* 202 formed by the loss of CO_2 from the molecular ion at *m/z* 246. Proton signal at δ 4.19 (2H, d, $J = 4.1$ Hz, H-4′) showed a C-H long-range correlation with carbon at δ 174.0 (C-5′), which suggested **1** had a carboxymethyl substitution. In the HMBC spectrum the proton signal at δ 2.68 (2H, t, $J = 7.4$ Hz, H-1′) and 6.16 (1H, s, H-3) showed C-H long-range correlations with olefinic carbon at δ 111.2 (C-3) and aromatic carbon at δ 122.7 (C-10), respectively, so the propyl should be attached to C-2 (δ 171.2). Proton at δ 4.19 (2H, d, $J = 4.1$ Hz, H-4′) showed a C-H long-range correlation with C-10 (δ 122.7) and C-6 (δ 130.1), so the carboxymethyl substitution should be attached to C-5 (δ 136.9). Thus compound **1** was established as 5-carboxymethyl-2-propylchromone.

Compound **2** was isolated as a yellow powder, mp 248°C. Its molecular formula was determined as $C_{17}H_{14}O_4$ by HRSI-MS [*m/z* 283.0963 $(M + H)^+$]. The UV (MeOH) spectrum displayed bands at 423 and 280 nm. The ^{13}C NMR spectrum of **2** revealed 12 olefinic carbon and 2 carbonyl carbon signals. All this information suggested **2** had an anthraquinone skeleton. As for substitutions of **2**, its ^{1}H NMR spectrum displayed one methyl and two methylene groups, in addition to two hydroxyl protons, one at δ 13.08 (1H, s, 1-OH) and the other at δ 10.0 (1H, brs, 6-OH). By analysis of ^{1}H NMR, ^{13}C NMR and HMBC data, the alkyl signals were assigned to a propyl function. The chemical shift of the hydroxy protons suggested that the anthraquinone only had one *peri*-OH group (δ 13.08) [5]. The ^{1}H NMR spectrum also suggested the presence of two *meta*-coupled protons at δ 7.62 (1H, d, $J = 2.7$ Hz, H-5) and 7.12 (1H, d, $J = 2.7$ Hz, H-7). In its ^{13}C NMR spectrum the signal at δ 151.4 indicated the carbon (C-8) was attached to the propyl group, and δ 163.3 and 163.1 indicated they (C-1, C-6) were attached to the hydroxyl groups. In the HMBC spectrum the proton signal at δ 7.62 (1H, d, $J = 2.7$ Hz, H-5), 7.12 (1H, d, $J = 2.7$ Hz, H-7) and 3.20 (2H, t, $J = 7.5$ Hz, H-11) showed C-H long-range correlations with C-10 (δ 183.2), C-11 (δ 38.6)

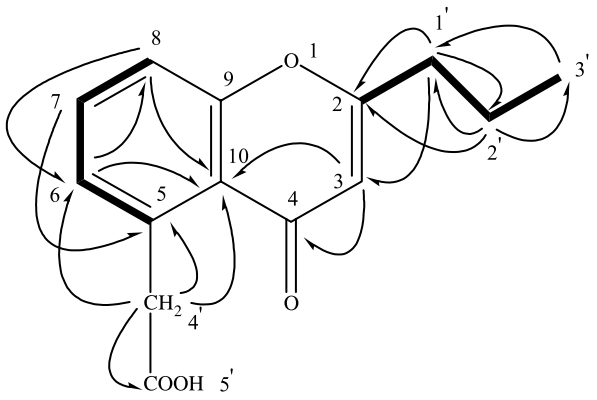

Figure 1. Significant HMBC and ^{1}H–^{1}H COSY correlations of **1**. Bold lines, ^{1}H–^{1}H COSY; curved pointed lines, HMBC.

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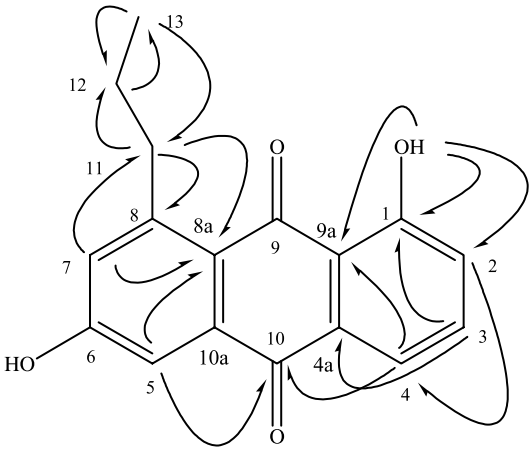

Figure 2. Significant HMBC correlations of **2**.

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and C-8a (δ 123.7), respectively. So on the basis of HMBC data (figure 2), the structure of **2** was elucidated as 1,6-dihydroxy-8-propylanthraquinone.

Compound **3** was identified as 3,8-dihydroxy-1-propylanthraquinone-2-carboxylic acid by comparing its physical and spectral data with literature values [6].

3. Experimental

3.1 General experimental procedures

Melting points were measured on a Yanaco micro-hot-stage apparatus and are uncorrected. UV spectra were measured on a Shimadzu UV-1601. All the NMR spectra were taken on a Bruker-ARX-300 spectrometer (^{1}H at 300 MHz and ^{13}C at 75 MHz). EI-MS spectra were recorded on a DX-300 mass spectrometer; HRSI-MS spectra were measured on an APEX FTICR mass spectrometer. Column chromatography was performed on silica gel G (201–300 mesh, Qingdao Haiyang Chemical Factory), Sephadex LH-20 (Pharmadex) and reversed-phase silica gel (Chromatorex C_{18}).

3.2 Actinomycetic material

The strain FX58-1, which was isolated from a marine plant *Salicornia herbacea* collected in Qingdao, Shandong province, China, in September 2002, was identified as *Streptomyces* sp. by Professor Li Tian. A voucher specimen (No. CAAN03101) is deposited in the Key Laboratory of Marine Biology in the State Oceanic Administration, China.

3.3 Cultivation, extraction and isolation

The strain was cultured on seed medium in 12 1-L flasks at room temperature for 1–8 days. The culture medium contained starch 20 g, KNO_3 1 g, K_2HPO_4 0.5 g, $MgSO_4$ 0.5 g, $FeSO_4$ 0.01 g, NaCl 3 g, KCl 0.03 g, $MgCl_2$ 0.23 g, and leaching liquor of soil 200 ml (pH 7.0). On the eighth day, the fermentation broth, including cells, was harvested and then centrifuged to

separate mycelial mass from the aqueous layer. The mycelial mass was exhaustively extracted with acetone to obtain a crude extract (9 g). The extract was chromatographed on a silica gel column using a gradient of acetone in petroleum ether. Compound **2** (10 mg) was obtained with petroleum ether/acetone (100:4) as eluent and was further purified on a Sephadex LH-20 column (Pharmadex, petroleum ether/$CHCl_3$/MeOH 5:4:1). Compound **3** (8 mg) was obtained with petroleum ether/acetone (100:15) as eluent and was further purified on a column of reversed-phase silica gel (Chromatorex C_{18}, MeOH/H_2O 3:2). Compound **1** (7 mg) was obtained with petroleum ether/acetone (100:20) as eluent and was further purified on a column of reversed-phase silica gel (Chromatorex C_{18}, MeOH/H_2O 3:2).

3.3.1 5-Carboxymethyl-2-propylchromone (1). Colourless fine needles from MeOH, mp 121–126°C; UV (MeOH) λ_{max} nm: 224, 250, 300; ^{1}H NMR (300 MHz, methanol-d_4): 7.67 (1H, t, $J = 7.2$ Hz, H-7), 7.53 (1H, dd, $J = 7.2$ Hz, 2.3 Hz, H-8), 7.25 (1H, dd, $J = 7.2$ Hz, 2.3 Hz, H-6), 6.16 (1H, s, H-3), 4.19 (2H, d, $J = 4.1$ Hz, H-4′), 2.68 (2H, t, $J = 7.4$ Hz, H-1′) 1.80 (2H, q-like, $J = 7.4$ Hz, H-2′) 1.05 (3H, t, $J = 7.4$ Hz, H-3′); ^{13}C NMR (75 MHz, methanol-d_4): 171.2 (C-2), 111.2 (C-3), 182.0 (C-4), 136.9 (C-5), 130.1 (C-6), 134.5 (C-7), 119.0 (C-8), 159.4 (C-9), 122.7 (C-10), 36.6 (C-1′), 21.2 (C-2′), 13.8 (C-3′), 41.6 (C-4′), 174.0 (C-5′); EI-MS *m/z*: 246.2 $[M]^+$(18), 202.3 (100), 172.1 (98), 145.1 (44), 115.1 (92); HRSI-MS *m/z*: 247.0964 $(M + H)^+$ (calcd for $C_{14}H_{15}O_4$, *m/z* 247.0970).

3.3.2 1,6-Dihydroxy-8-propylanthraquinone (2). A yellow powder from MeOH, mp 248°C, UV (MeOH) λ_{max} nm: 423, 280, 217, 202; ^{1}H NMR (300 MHz, acetone-d_6): 13.08 (1H, s, 1-OH), 7.30 (1H, dd, $J = 6.6$ Hz, $J = 2.9$ Hz, H-2), 7.72 (2H, m, H-3 and H-4), 7.62 (1H, d, $J = 2.7$ Hz, H-5), 10.0 (1H, brs, 6-OH), 7.12 (1H, d, $J = 2.7$ Hz, H-7), 3.20 (2H, t, $J = 7.5$ Hz, H-11), 1.69 (2H, m, H-12), 1.04 (3H, t, $J = 7.2$ Hz, H-13); ^{13}C NMR (75 MHz, acetone-d_6): 163.1 (C-1or C-6), 125.1 (C-2 or C-7), 136.7 (C-3), 119.1 (C-4), 133.9 (C-4a), 113.3 (C-5), 163.3 (C-6 or C-1), 125.1 (C-7 or C-2), 151.3 (C-8), 123.7 (C-8a), 190.8 (C-9), 117.9 (C-9a), 183.2 (C-10), 138.8 (C-10a), 38.6 (C-11), 24.6 (C-12), 14.6 (C-13); EI-MS *m/z*: 282.1 $[M]^+$(85), 264.1 (100), 247.1 (36), 221.1 (13); HRSI-MS *m/z*: 283.0963 $(M + H)^+$ (calcd for $C_{17}H_{15}O_4$, *m/z* 283.0970).

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