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SHORT COMMUNICATION

Crystal structure and configuration revision of 9-hydroxy-7,8-dehydro-sarcotrocheliol and sarcotrocheliol

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\textbf{ABSTRACT}
Herein, we report the isolation and stereo-structure of rare pyrane-based cembranoid diterpenes, 9-hydroxy-7,8-dehydro-sarcotrocheliol (1) and sarcotrocheliol (2), from Sarcophyton trocheliophorum collected from Red Sea. Absolute configurations of both compounds were revised based on single crystal X-ray analyses.

\textbf{1. Introduction}

Sarcophyton sp. is a widespread genus of corals extending from Eastern Africa and the Red Sea in the west to Polynesia in the east (Verseveldt 1965, 1970; Benayahu 1990). Among the 36 known Sarcophyton species, 14 of them are found in the Red Sea (Fabricus & Alderslade 2001), as the most abundant coral reef animals with high cembranoid content (Kobayashi et al. 1983). Cembranoids are 14-membered carbocyclic diterpenes linking one isopropyl and three methyl groups, often oxidized to hydroxymethyls or carboxylic acids, which can also be involved in the formation of pyranosyl or lactone rings (Coll 1992). Undeniably, Sarcophyton trocheliophorum is
so rich in terpenes, cembranoids; probably the most frequently encountered structural type (Anjaneyulu & Venkateswara Rao 1997; Liang & Guo 2013).

In our ongoing program to investigate the structures of novel cembranoids from Sarcophyton trocheliophorum collected from Red Sea at the Hurghada coasts, Egypt (Shaaban et al. 2013, 2015, 2016, 2018), we describe herein the molecular structures and absolute configurations of 9-hydroxy-7,8-dehydro-sarcotracheliol (1), recently published by us (Shaaban et al. 2018), and sarcotracheliol (2) (Al-Footy et al. 2015). Additional investigation of the marine organism afforded cholesterol (Abou-Elwafa et al. 2009), sarcophinone and 7,8-dihydroxydeepoxy-sarcophine (Shaaban et al. 2017).

2. Results and discussion

Compounds 1 and 2 were afforded after a re-investigation and precise purification of the coral extract showing colorless solid natures, in contrast to their afore-reported oily appearance (Al-Footy et al. 2015; Shaaban et al. 2018). The two compounds 1 and 2 were consequently obtained as colorless orthorhombic crystals from chloroform, and dichloromethane: methanol 1:1, respectively. The molecular and planar structural formulas of 1 and 2 were assigned on the bases of NMR and HR-ESI-MS data and single crystal X-ray analysis (CCDC 1852835 and 1852836 contain the supplementary crystallographic data for compounds 1 and 2. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/conts/retrieving.html).

2.1. 9-Hydroxy-7,8-dehydro-sarcotracheliol

The absolute configuration of 9-hydroxy-7,8-dehydro-sarcotracheliol (1) was determined by single crystal X-ray analysis of a co-crystal with CHCl₃ refining to a Flack parameter value of −0.013(8). Chirality of the latter is (S) at C-1 and C-5 and (R) at C-4, C-9 and C-14 (Figure 2). In accordance, results have shown that the absolute configuration of the five asymmetric centers for compound 1 were 1S, 4R, 5S, 9R and 14R, exhibiting a reverse configuration at the three asymmetric centers: 1, 4 and 9 of the reported one (1a: 1R, 4S and 9S) determined by relative configuration studies (Shaaban et al. 2018) (Figures 1 and 2).

Figure 1. Revised (1, 2) and reported (1a, 2a) structures for 9-hydroxy-7,8-dehydro-sarcotracheliol and sarcotracheliol.
2.2. Sarcotrocheliol

Sarcotrocheliol (2) crystalizes in the space group P2₁2₁2₁ with two molecules per asymmetric unit, the absolute structure was refined to a Flack parameter value of -0.05(8). The two molecules are different conformers by a rotation around C7–C8 and C9–C10 resp. C27–C28 and C29–C30, but both ones show the same configuration: Chirality is (S) at C1 and C5 resp. C21 and C25 and (R) at C4 and C14 resp. C24 and C34 (Figure 3). Accordingly, results exhibited that the absolute configuration of the four asymmetric centers for compound 2 were identical to those of 1 (1S, 4R, 5S, 14R), displaying a reverse configuration at the two asymmetric centers: 5, and 14 in the previously reported one by relative configuration studies (Al-Footy et al. 2015) (2a: 5R, 14S).

3. Experimental (see supplementary data)
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Disclosure statement

No potential conflict of interest was reported by the authors.

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