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A New Sesquiterpene from the Alga *Caloglossa Leprieurii*

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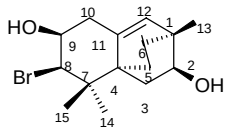
Abstract: A new bromo-sesquiterpene **1**, has been isolated from the alga *Caloglossa leprieurii* collected from the XiSha islands in south china sea. Its structure has been determined on the basis of spectral analysis, and The relative configuration of **1** was assigned on the basis of difference spectrum of NOE.

Keywords: *Caloglossa leprieuriii*, hydroclathrol, spectral analysis.

The alga *Caloglossa leprieurii* is a chinese traditional drug having been used as anthelmintic, from the alga *Caloglossa leprieurii*, we had isolated two novel metabolites and three known compounds[1-2]. Further efforts to study this genus systematically made us to obtain a novel bromo-sesquiterpene **1** from *Caloglossa leprieurii* collected from the XiSha islands in south China sea.

The EtOH extract of the alga *Caloglossa leprieuri* was partitioned between EtOAc and H_2O, and the resulted organic layer was eluted with EtOAc-hexane. The fracation (25:100) was purified by PTLC on a silica gel plate to yield compound **1**(8mg).



compound **1**

Compound **1**, colorless needle crystals, mp. 104-105°C, gave a pair of molecular ion peak at *m/z*: 316(24) and314(24) in a 1 : 1 ratio, which demonstrates that compound **1** contains one bromine atom, and its ^{13}C-NMR DEPT spectrum was assigned to three methyls, four methenes, four methines and four quaternal carbons, corresponding to the molecular formula $C_{15}H_{23}BrO_2$, which required four degrees of the unsaturation. Its IR (KBr, ν) 1669 cm^{-1} and ^{1}H-NMR signals at δ 5.65(brs) supported the existence of one tri-substitute double bond, therefore, compound **1** must be a three cyclic compound. The IR spectra of **1** exhibiting an absorption band at 3420 cm^{-1} indicated the presence of the hydroxyl group, and the significant ion *m/z:* 235 $(M\text{-}Br)^+$, 217$(M\text{-}Br\text{-}H_2O)^+$ and

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199(M-Br-$2H_2O$)$^+$ also confirmed the existence of one bromine and two hydroxyl group. ^{1}H-NMR showed three methyl group signals at δ_H (1.00, 1.12, 1.23), corresponding to 13-Me,14-Me,15-Me,respectively.threeseparated spin-systems "=CHCH(X)CH(X)="(a), "-CH_2CH_2-" (b), and "=CH(X)CH_2-" (c) were identified by means of ^{1}H-^{1}H COSY and ^{1}H-^{13}C COSY spectra, X maybe is either a bromine or one hydroxyl group, furthermore it was found that the ^{13}CNMR data of compound **1** are in good agreement with those of the known compound[3-4], one bromine atom was assigned for C-8 position, and another two hydroxyl group for C-2 and C-9 position. Based on the HMBC correlations of compound **1**. It revealed the presence of the gemdimethylcyclohexane ring with vicinal hydroxy and bromine groups as the skeleton of some charmigrene sesquiterpenes[5-7], and the partial structure "a-c" could be connected as structure **1** (see **Table** 1).

The relative configuration of **1** was assigned on the basis of NOE correlation between H-8, H-9 and H-15. The sterochemistry at C-2 and C-1 could be deduced from the NOE correlation between H-2 and H-15, and H-2and H-1.

Table 1 NMR data of compound **1** (500 MHz, $CDCl_3$)

Position	δ_C	δ_H	Key HMBC (C-H)
1	39.8		C-1, H-2, 3, 12, 13
2	72.1	4.21 (dd, 3.0,8.2Hz,1H)	
3	42.7	2.37 (dd,8.2,13.8Hz, 1H),1.90(dd,3.00,13.8Hz,1H)	
4	44.5		C-4, H-2,3,5,6,, 10,12
5	27.8	1.40(m,1H),1.22(m.1H)	
6	32.7	1.3 (m, 2H)	
7	39.3		C-7,H-8,9,14,15
8	72.4	4.39(d,4.8,1H)	
9	66.8	4.08 (ddd,4.8,8.0,8.5Hz, 1H)	
10	35.4	2.94(dd, 8.5,17.0Hz , 1H), 2.84 (dd, 8.0,17.1Hz , 1H)	
11	139.3		C-11,H-3,5,9,10,12
12	130.1	5.64 (brs, 1H)	
13	26.9	1.00 (s,3H)	
14	22.7	1.12 (s, 3H)	
15	23.2	1.25 (s, 3H)	

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