

## A Novel Eleven-membered Heterocyclic Compound from Algae *Sargassum Vachell*

Shi Hai XU, Ying Zhou CEN\*, Yao Lan LI, Shao Yu XU

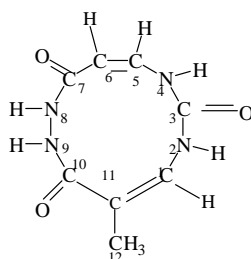
Department of chemistry, Jinan University, Guangzhou 510632

**Abstract:** A novel eleven-membered heterocyclic compound with high nitrogen has been isolated from the marine alga *Sargassum vachell* collected from the South China Sea. Its structure has been established by verity of spectroscopic techniques such as IR, EIMS, 1D NMR,  $^1\text{H}$ - $^1\text{H}$ COSY, HMQC, HMBC.

**Keyword:** Heterocyclic compound; *Sargassum vachell*; structure elucidation.

The algae produced many interesting pharmacological and biological activity metabolites such as antibacterium, antineoplastic, anticancer and antimicrobial activities compound<sup>1-4</sup>. More and more chemist and biologist pay attention to the constituents of the algae. The alga *Sargassum vachell* collected from the South China Sea has been studied and compound **1** was isolated.

**Figure 1** The structure of **1**



The ethanol extract of alga *Sargassum vachell* was eluted by silica gel using petroleum ether with increasing amounts of ethyl acetate as eluent. The fraction obtained with petroleum ether/ethyl acetate 20/80 (v/v) contained compound **1** which was an amorphous powder, m.p.228~230<sup>0</sup>C crystallized from (CH<sub>3</sub>OH). The molecular formula of **1** was shown to be C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>O<sub>3</sub> by EIMS: 210(M<sup>+</sup>), <sup>13</sup>CNMR and element analysis (Found C, 44.91%; H, 4.83%; N, 26.54%. C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>O<sub>3</sub> calculated C, 45.71%; H, 4.71%; N, 26.66%) and its unsaturated degree was 6.

$\delta_{\text{H}}$  at 11.11(1H,m), 11.23(1H,m), 10.81(1H,m) and 10.59(1H,m) disappeared by exchange of deuterated water revealed the presence of four active hydrogens. IR 1720, 1685, 1675, 1648  $\text{cm}^{-1}$  and  $^{13}\text{C}$ NMR signals at 164.8(s), 164.3(s), 151.5(s) shown the existence of three amidated carbonyl groups, which C-3 was carbamido carbon<sup>5</sup>, also the unit of  $-\text{NH}-\text{CO}-\text{NH}-$  contained. Two alkene proton and the structural unit of  $-\text{COCH}=\text{CH}-\text{NH}-$  identified by  $\delta_{\text{H}}$  at 7.38 (1H, d,  $J=7.6\text{Hz}$ ), 5.44 (1H, d,  $J=7.6\text{Hz}$ ) and  $\delta_{\text{C}}$  at 143.0 (d), 100.2 (s). The trisubstituted double bond also confirmed by the presence of  $\delta_{\text{H}}$  7.23 (1H,m),  $\delta_{\text{C}}$  137.6 (d), 107.5 (s), furthermore, the units of  $-\text{CH}=\text{CCH}_3-$  was identified with  $^{13}\text{C}$ NMR and DEPT. The ring was verified by the left unsaturated. The proton coupling certificated by  $^1\text{H}-^1\text{H}$ COSY  $\text{H}_2 \longleftrightarrow \text{H}_1 \longleftrightarrow \text{H}_{12}$ ,  $\text{H}_6 \longleftrightarrow \text{H}_5 \longleftrightarrow \text{H}_4$ ,  $\text{H}_8 \longleftrightarrow \text{H}_9$ , HMBC showed that  $\text{C}_{10}$  related to  $\text{H}_{12}$ ,  $\text{H}_9$  and  $\text{H}_1$ ,  $\text{C}_{11}$  related to  $\text{H}_{12}$  and  $\text{H}_1$ ,  $\text{C}_3$  correspond to  $\text{H}_2$  and  $\text{H}_4$ ,  $\text{C}_5$  correspond to  $\text{H}_6$  and  $\text{H}_4$ ,  $\text{C}_6$  related to  $\text{H}_5$ ,  $\text{C}_7$  related to  $\text{H}_6$  and  $\text{H}_8$ , thus the structure of compound 1 was established.

The data of spectroscopy compound 1, IR ( $\nu$   $\text{cm}^{-1}$  KBr) 3206, 1729, 1658, 1441, 1212, 988, 884, 761.  $^1\text{H}$ NMR ( $\delta_{\text{H}}$ , ppm-DMSO) 11.1 (1H,br), 11.23 (1H,br), 10.59 (1H,br), 7.38 (1H, d,  $J=7.6\text{Hz}$ ), 7.23 (1H,m), 5.44 (1H, d,  $J=7.6\text{Hz}$ ), 1.71 (3H, d,  $J=11.2\text{Hz}$ ).  $^{13}\text{C}$ NMR ( $\delta_{\text{C}}$ , ppm-dmsO) 164.9 (s), 164.3 (s), 151.5 (s), 143.0 (s), 137.7 (d), 107.7 (s), 100.2 (d), 11.8 (q). MS ( $m/z$ ) 210 (5), 126 (100), 112 (80), 83 (20), 55 (62).

### Acknowledgment

The authors are grateful to Ni Yang in Lanzhou University for NMR, MS and element analysis.

### References

1. J. B. Joseph, Jr., E. Richard and M. L. Patterson, *J. Am. Chem. Soc.*, **1984**, 106 (23), 8193.
2. E. Richard, J. Adrian and S. Mynderse, *J. Org. Chem.* **1984**, 49 (12), 2484.
3. E. L. Ghisalber, M. J. Nearbey and C. Y. Rowland, *J. Nat. Prod.* **1990**, 53 (4), 520.
4. C. Carter, E. Moore, S. Mynderse, P. Niemczura and S. Todd, *J. Org. Chem.* **1984**, 29 (2), 236.
5. Q. F. Shen, G. Z. Xu,  *$^{13}\text{C}$ NMR and its application* Beijing Chemical Industry Press, **1986**, p194.

Received 30 November 1998

Revised 8 April 1999