IJIST

International Journal
of
Integrated Sciences & Technology (IJIST)

http://www.cuet.ac.bd/IJIST/index.html

International Journal of Integrated Sciences & Technology **1** (2015) 45-47

Isolation and Structure Elucidation of Alkaloid From *Sida acuta*

R. K. Sutradhar*, R. Karim and R. Khatun

Department of Chemistry, Chittagong University of Engineering and Technology (CUET)
Chittagong-4349, Bangladesh.

Abstract

Alkaloid: 6-Hydroxy-8-(3',4',4'-trimethyl-pentyl)-7,8-dihydro-6H-pyrrolo[1,2-a] pyrimidin-4-one **(1)** along with β-sitosterol **(2)** have been isolated from the aerial parts of *Sida acuta.*

Keywords: Sida acuta; Malvaceae; Isolation; Alkaloid; Characterization.

1. Introduction

Sida acuta of the family Malvaceae is an important medicinal plant of Bangladesh and is used for the treatment of several diseases like chronic dysentery, asthma, gonorrhea and specially for rheumatism [1-2]. Different classes of compounds e.g. glycosides, flavonoids, alkaloids, steroids and amino acids have been reported from the plants of the genus *Sida* [3-6]. Quinazoline alkaloids e.g. vasicine, vasicinone, vasicinol, along with ephedrine, β-phenethylamine and N-methyl tryptophan were reported from *Sida cordifolia* and *Sida rhombifolia* [7-10].

Previous phytochemical studies showed the presence of alkaloids, steroids and terpenoid type compounds in Sida acuta [11-13]. In continuation of studies on medicinal plants, we isolate a new alkaloid along with a known compound β-sitosterol from the aerial parts of *Sida acuta*. The structures of these compounds were established by chemical and spectroscopic methods. In this paper, we report the isolation and structure elucidation of new alkaloid **(1)** along with known compound **(2)**.

2. Materials and Method

2.1 General

The IR spectra were recorded on a Shimadzu FTIR Model 8400 spectrometer. The ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker BPX-200 spectrometer operating at 400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR spectra. The chemical shift values are reported as parts per million (ppm) relative to tetramethylsilane.

* Corresponding author. Mobile: +88-01812149299
E-mail: rksutradhar2002@yahoo.com
For color version visit:http://www.cuet.ac.bd/IJIST/index.html

2.2 Plant material

Matured, fresh and flowering plants of *Sida acuta* were collected from the hilly region of the district of Chittagong situated in the southeastern region of Bangladesh during the month of November 2009. The plant was identified by Prof. Atiqur Rahman, Dept. of Botany, University of Chittagong and a voucher specimen (Accession No. 2009-97) was deposited at the Department of Botany, University of Chittagong .

2.3 Extraction and isolation of compounds

The plant materials were chopped into small pieces, dried at room temperature and ground in to powder. The dried powder (4 kg) of *Sida acuta* was extracted with $CHCl_3$ (3×72 h) and MeOH (3×72 h) respectively. On removal of the solvent under reduced pressure at a temperature < 40˚C the $CHCl_3$ extract gave a green Mass (45 g) and MeOH extract gave a light green Mass (55 g). MeOH extract 7 g was adsorbed on to silica gel and placed over a column of silica gel and eluted with $CHCl_3$-EtOAC (1:1). The eluents were monitored by TLC and only one compound **1** (40 mg) was able to isolate in the pure state from this system. $CHCl_3$ extract 5 g also gave a pure compound **2** (120 mg) after chromatographic separation with solvent system n-hexane-$CHCl_3$ (2:1).

Physical properties of the alkaloid 1

Brown amorphous solid, mp 162-163ºC

IR : ν_{max} (KBr) 3441 (O-H), 1728 (C=O) and 1635 (C=N) cm^{-1}.

ISSN : 0000-0000

^{1}H-NMR ($CDCl_3$) δ: 0.93 (3H, s, H-8′), 0.89 (3H, s, H-7′), 0.89 (3H, s, H-6′), 0.91 (3H, s, H-5′), 1.34 (1H, m, H-3′), 1.25 (2H, m, H-2′), 1.43 (2H, m, H-1′), 1.67 (2H, t, H-7), 2.32 (1H, m, H-8), 4.21 (1H, t, H-6), 7.52 (1H, d, H-3), 7.68 (1H, d, H-2) ^{13}C-NMR (CDC_{l3}) δ: Table 1

Physical properties of the compound 2

White amorphous solid, mp 135-136ºC

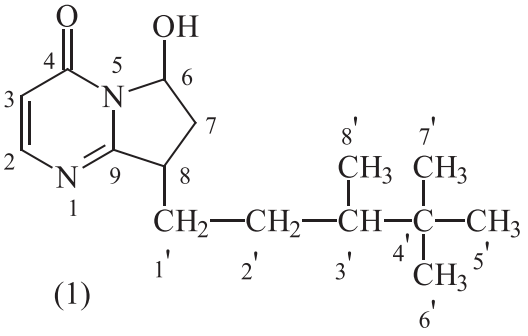
IR: ν_{max} (KBr) 3421 (O-H) cm^{-1}

^{1}H-NMR($CDCl_3$) δ: 0.66 (3H, s, H-29), 0.80 (6H, s, H-27, 26), 0.91(3H, s, H-21), 0.99(3H, s, H-18), 1.10 (3H, s, H-19), 1.48 –1.54 (16H, methylene), 1.81 (2H, d, H-4), 1.98 (2H, m, H-2), 2.25 (2H, m, H-7), 3.50 (1H, m, H-3), 5.13 (1H, t, H-6)

^{13}C-NMR($CDCl_3$) δ: Table 2

3. Results and Discussion

Alkaloid 1 (40.0 mg, 0.0018%) was a brown amorphous solid, m.p 162-163ºC. It gave positive reactions with Mayer's and Dragendorff reagents for an alkaloid [14]. The IR spectrum of alkaloid 1 showed a broad absorption band at 3441 cm^{-1} for hydroxyl (O-H) function, an intense absorption band at 1728 cm^{-1} for carbonyl (C=O) function and absorption band at 1635 cm^{-1} for C=N function. The ^{1}H NMR spectrum revealed the presence of 24 hydrogen atoms in alkaloid 1 (Table 1). The ^{13}C NMR spectrum of alkaloid 1 showed the presence of 15 carbons in the molecule (Table 1). On the basis of carbon and hydrogen content and alkaloidal nature, the molecular formula of alkaloid 1 was computed as $C_{15}H_{24}O_2N_2$. The DEPT spectrum of alkaloid 1 revealed the presence of three methylene carbons at δ 38.78 (C7), 14.04 (C1′) and 10.97 (C2′) and three methine carbons at δ 68.19 (C6), 31.94 (C8) and 23.79 (C3′). Two doublets at δ 7.68 and 7.52 in the ^{1}H NMR spectrum are attributed to two olefinic protons attached to C2 and C3 respectively. Protons attached to C6 and C7 appeared as triplet centered at δ 4.21 (H-6) and 1.67 (H-7) respectively, whereas two methine protons at C8 and C3′appeared as overlapping multiplet at δ 2.32 and 1.34 respectively. The methylene protons of side chain attached to carbon C1′ and C2′appeared as overlapping multiplet at δ 1.43 and 1.25 respectively. The ^{1}H NMR spectrum of alkaloid 1 showed twelve methyl proton absorptions as singlet at δ 0.89 (3×CH_3) and 0.93(1×CH_3). The ^{1}H NMR absorptions along with the carbon content of the molecule suggests the presence of a side chain as –CH_2 – CH_2 – CH(CH_3)–C(CH_3)$_3$ is attached to the nucleus. On the basis of the IR, ^{1}H NMR and ^{13}C NMR spectral analysis the tentative structure **(1)** may be suggested for the alkaloid. Alkaloid 1 is thus characterized as **6-hydroxy-8-(3',4',4'-trimethyl-pentyl)-7,8-dihydro-6H-pyrrolo[1,2-a]pyrimidin-4-one(1)** and it was isolated for the first time from plant body.



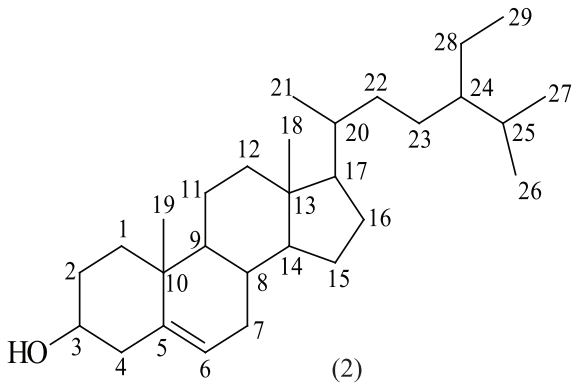
(1)

Table 1: ^{13}C and ^{1}H NMR data of the alkaloid 1

Carbon no.	Alkaloid **1**, ^{13}C, δ	Alkaloid **1** ^{1}H, δ
2	130.88	7.68,d
3	128.83	7.52,d
4	167.76	
6	68.19	4.21,t
7	38.78	1.67,t
8	31.94	2.32,m
9	132.51	
1′	14.04	1.43,m
2′	10.97	1.25,m
3′	23.79	1.34,m
4′	30.40	
5′	28.96	0.89,s
6′	29.71	0.89,s
7′	29.37	0.89,s
8′	22.99	0.93,s

Compound 2 (120.0 mg, 0.0148%) was a white amorphous solid, m.p 135-136ºC. Compound 2 readily responded to the Salkowski and Liebermann-Burchard colour tests for steroids exhibiting its steroidal nature [14]. The IR spectrum of compound 2 showed a strong absorption band at 3421 cm^{-1} which indicated the presence of hydroxyl (O-H) function in the molecule. One proton absorptions at δ 5.01 in ^{1}H NMR spectrum, suggested that the compound 2 contains one olefinic proton. The 13C NMR spectrum of compound 2 showed the presence of 29 carbons in the molecule (Table 2). By analyzing its IR, ^{1}H NMR, and ^{13}C NMR spectra, it appeared that the molecular formula

of the compound 2 is $C_{29}H_{50}O$. It is thus presume that compound 2 is ***β*-Sitosterol (2).** The ^{13}C NMR peaks of compound 2 (Table 2) corresponded to that of *β*-Sitosterol [15-16].



(2)

Table 2: Comparison of ^{13}C NMR data of Compound 2 with those of the published data of *β*-Sitosterol.

Carbon no.	Compd. 2, ^{13}C, δ	*β*-Sitosterol, ^{13}C, δ
1	37.29	36.54
2	31.68	29.61
3	71.81	72.52
4	42.33	41.14
5	140.79	141.56
6	121.71	125.93
7	31.92	30.56
8	31.95	31.04
9	51.26	48.66
10	36.53	36.25
11	21.08	21.78
12	39.81	37.02
13	42.25	45.95
14	56.80	52.95
15	24.32	26.10
16	28.26	28.81
17	56.01	49.47
18	11.87	14.67
19	19.40	20.67
20	36.16	33.56
21	21.11	18.70
22	24.32	26.70
23	24.38	26.39
24	45.88	47.78
25	28.91	27.15
26	18.80	19.96
27	19.07	18.90
28	26.16	25.61
29	12.24	18.31

4. Conclusion

The principal objective of this project was to isolate alkaloids along with other secondary metabolites from the aerial parts of Sida acuta as well as to determine the molecular architecture of the isolated compounds. We are successful to finding out some of them. Alkaloid 1 is a new natural product isolated from any plant body.

Acknowledgements

We are grateful to CUET Authority for providing necessary research fund to complete this research work. Prof. Dr. M. Atiqur Rahman, Department of Botany, University of Chittagong is thanked for identification of plant materials.

References

1. K.R. Kiritker, B.D. Basu, Indian medicinal plants, 2nd. Edition. Vol.1 International book distributors publication. Dehradun, India, pp.312 (1981).
2. M. Yusuf, M. Kabir, Medicinal plants of Bangladesh, Bangladesh Council of Scientific and Industrial Research, Dhaka, Bangladesh .pp 226 (1999).
3. L. V. Ligai, V. A. Bandyukova, Constituents of aerial parts of Sida hermaphrodita. Khim. Prir. Soedin 2, 269 (1990).
4. I. Matlawska, Herba pol. 36(3), 65 (1990).
5. M. M. Goyal and K. K. Rani, Fitoterapia 60(2), 163 (1989).
6. D. J. Bhaltt, A. J. Baxi, A. R. Parikh, Journal of Indian Chem. Soc. 60(1), 98 (1983).
7. B. Asha and N. R. Bannerjee, Current science 54(14), 690 (1985).
8. S. Ghosal, R. B. P. S. Chauhan and R. Mehta, Phytochemistry 14(3), 830 (1975).
9. A. A. L. Gunatilaka, S. Sotheeswaran, S. Balasubramaniam, A. I. Chandrasekara and H. T. Badrasriyani, Studies on medicinal plants of Sri Lnaka. Planta Med. 39(1), 66 (1980).
10. S. Ghosh and A. Dutt, J of Indian Chem. Soc. 7, 825 (1930).
11. B. Coa, J. Qi, Zhongguo Zhongyao Zazhi 18(11), 681 (1993).
12. R. V. K. Rao, T. Satyanarayan, Fitoterapia 55(4), 249 (1984).
13. S. Ghosal, A. Prakash and R. K. Varma, Planta Med. 43, 384 (1981).
14. I. L. Finar, Organic chemistry, Vol. 2, 5th Edition, ELBS, Longman publishers, Singapore, pp.784 (1975).
15. I. Rubinstein, L. J. Gold, A. D. H. Clague and L. J. Mulheirn, Phytochemistry, 15, 195 (1976).
16. J. Sakakibara, T. Kaiya, H. Fukuda and T. Ohki, Phytochemistry, 22(11), 2553 (1983).