Isolation and Characterization of a New Compound from Sarcococca saligna

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Abstract: A New Alkaloid named Sracosalgnenone [ (20S)-20- (dimethylamino) -3β-tigoloyl-pregn-5, 16-diene-4-one] was isolated from sarcococca saligna and its structure was established on the basis of spectroscopic techniques including 

Keywords: Sarcococca saligna, Buxaceae, steroidal alkaloids, Sracosalgnenone.

1. Introduction

Sarcococca saligna Muel (syn. Sarcococca prunifomis Lindl.) found in Pakistan [1] generally at an altitude form 4000-9000 ft. above sea level. By the local population it is used in the treatment of rheumatism [2,3]. Many of steroidal alkaloids isolated from aerial parts of plant show cholinesterase inhibition [2-18]. Its alkaloids are antispasmodic, antidiarrheal, antiseptic and calcium antagonist properties. [19-20]. A characteristic feature of the family Buxaceae is its high contents of steroidal alkaloids [21]. Taxonomically it comprises of different genera Pachysandra, Sarcococca, simmonis and Buxus. A number of alkaloids have been isolated from genus Buxus. The extracts of various species of this genus have been used for the treatment of a variety of ailments and skin disease etc. in folk medicine. [22]. A number of compounds have been identified by GCMS technique from aerial parts of sarcococca saligna [23]. The present study describes the isolation of one new pregnane type alkaloid Sracosalgnenone and its structure determination on the basis of spectroscopic techniques.

2. Literature Survey

S. saligna Muel (syn. S. prunifomis Lindl.) is an evergreen shrub abundantly found in the northwest region of Pakistan [1]. Ismat Naem et al, isolated a new alkaloid, sarcococena (3α-dimethylamino-20 α-N-methyl-N-acylaminopregna-5, 16-diene), and two known alkaloids, pachyaxime-A and saracodine were isolated from S. saligna [9]. Recently Atta- ur-Rahman and his research group isolated a number of pregnane type steroidal alkaloids from S. saligna [10] [11] [16] [24]. Three tri-terpenes were also isolated from S. saligna [14] and a number of other compounds were identified by GC MS analysis. [15]

3. Materials and Method

General experimental procedure: IR spectra: JASCO 302-A spectrophotometer; UV spectra: Hitachi U3200 spectrophotometer; EI, FD and HREI MS: JMS 11x100 (with data system) and JMS-DA 500 mass spectrometers; 

4. Results and Discussion

An ethanolic extract of aerial parts of Sarcococca saligna


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after evaporation it was triturated with n-hexane to remove nonpolar compounds. The insoluble content was then partitioned between chloroform and aqueous acid solution at various pH values. The chloroform extract was subjected to repeated column chromatography to afford new alkaloid Sracosalgine. The compound was obtained as a white solid. The HREI mass spectrum of compound reveal a molecular ion peak [M+H]+ at m/z 438. 3226 analyzing for C_{28}H_{33}N_{2}O_{2} with nine degrees of hydrogen (H -2) deficiency. The IR spectrum (CHCl_3) showed absorptions at 3392 (NH), 1620-1660 (C=O) and 1710 cm⁻¹ characteristic of an amide, and α, β-unsaturated ketone functions respectively. The ^1H NMR spectrum of compound showed characteristics chemical shifts of a tigloyl group at C-3 [10]. One proton resonating at δ 2. 35 showed direct correlation to carbon C-3 chemical shift at δ 57. 4 and HMBC connectivities with C-2 (δ 33. 0), a carbonyl carbon at C-4 (δ 196) and C-5 (δ 133. 0). One olefinic proton doublet resonating down field at δ 6. 49 showed COSY 45° interaction with C-7 protons and 1-3 bond removed correlation to carbon atoms with chemical shifts at δ 196. 6, 133. 0, 38. 9, 51. 0 suggesting a double bond at C-5, β to carbonyl function at C-4 [10]. Another one olefinic proton resonating at δ 5. 7 was directly connected to carbon atom with ^13C chemical shift at δ 127. 0 and showed HMBC connectivities to C-17, C-14 and C-13 (δ 156. 0, 22. 8 and 55. 1 respectively). Six proton signals of two methyl groups resonated at δ 0. 83 and 0. 90 and were due to C-18 and C-19 angular methyl groups of pregnane type skeleton.

A three proton doublet at δ 0. 89 (J = 6. 5 Hz) was assigned to secondary C-21 methyl group. The COSY 45° spectrum showed connectivity between C-21 methyl and C-20 methine proton (δ 2. 25). A broad six proton singlet at δ 2. 30 was assigned to a dimethyl amino group at C-20. On the basis of NMR (Table) and 2-D NMR studies (HMQC, HMBC, DEBT etc) the compound was assigned structure ([1]) and was found to be a new alkaloid named sarcosalgine

5. Acknowledgement

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References

5.

Table 3: D NMR assignments of compound (Sracosalgnenone)

<table>
<thead>
<tr>
<th>No</th>
<th>Multiplicity</th>
<th>13C Shift (δ)</th>
<th>1H Shift (δ)</th>
<th>Important HMBC</th>
<th>No</th>
<th>Multiplicity</th>
<th>13C Shift (δ)</th>
<th>1H Shift (δ)</th>
<th>Important HMBC</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>CH₂</td>
<td>34.3</td>
<td>1.84/1.92</td>
<td>CH</td>
<td>14.</td>
<td>C</td>
<td>196.6</td>
<td>54.4</td>
<td>1.13 (1H, m)</td>
</tr>
<tr>
<td>2.</td>
<td>CH₂</td>
<td>33.2</td>
<td>2.17/1.14(1H, m)</td>
<td>C₂, C₄, C₅</td>
<td>15.</td>
<td>CH₂</td>
<td>20.8</td>
<td>1.24 (2H, m)</td>
<td>C15, C16</td>
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<tr>
<td>3.</td>
<td>CH</td>
<td>57.4</td>
<td>2.35 (1H, m)</td>
<td>CH</td>
<td>16.</td>
<td>CH</td>
<td>127.0</td>
<td>5.7 (1H, s)</td>
<td>C16, C17, C14, C13</td>
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<tr>
<td>4.</td>
<td>C</td>
<td>133.0</td>
<td>-</td>
<td>C</td>
<td>17.</td>
<td>C</td>
<td>156.0</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>5.</td>
<td>C</td>
<td>133.0</td>
<td>-</td>
<td>CH₂</td>
<td>18.</td>
<td>CH₂</td>
<td>12.2</td>
<td>0.83 (3H, s)</td>
<td>C13, C14, C17</td>
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<tr>
<td>6.</td>
<td>CH</td>
<td>131.8</td>
<td>6.49 (d, J = 6.9 Hz)</td>
<td>C₄, C₅</td>
<td>19.</td>
<td>CH₃</td>
<td>13.3</td>
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<td>C1, C5</td>
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<tr>
<td>7.</td>
<td>CH₂</td>
<td>38.9</td>
<td>2.55</td>
<td>(dd, J = 18.0, 2.5 Hz)</td>
<td>20.</td>
<td>CH</td>
<td>55.1</td>
<td>2.23 (1H, q, J = 6.5 Hz)</td>
<td>-</td>
</tr>
<tr>
<td>8.</td>
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<td>24.1</td>
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<td>CH₂</td>
<td>21.</td>
<td>CH₂</td>
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<td>0.88 (3H, d, J = 6.5 Hz)</td>
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<td>2.30 (1H, m)</td>
<td>NCH₃</td>
<td>22.</td>
<td>NCH₃</td>
<td>42.3</td>
<td>2.30 (3H, bs)</td>
<td>C20</td>
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<tr>
<td>10.</td>
<td>C</td>
<td>40.1</td>
<td>-</td>
<td>NCH₃</td>
<td>23.</td>
<td>NCH₃</td>
<td>42.3</td>
<td>2.30 (3H, bs)</td>
<td>C20</td>
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<tr>
<td>11.</td>
<td>CH₃</td>
<td>20.6</td>
<td>1.42/1.60 (2H, m)</td>
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<td>24.</td>
<td>C₁’</td>
<td>167.7</td>
<td>-</td>
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<tr>
<td>12.</td>
<td>CH₃</td>
<td>30.8</td>
<td>1.87/1.92 (2H, m)</td>
<td>C₉, C₁₂</td>
<td>25.</td>
<td>C₂’</td>
<td>131.8</td>
<td>-</td>
<td></td>
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<tr>
<td>13.</td>
<td>C</td>
<td>46.0</td>
<td>-</td>
<td>C₉</td>
<td>26.</td>
<td>C₃’</td>
<td>133.5</td>
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<td>-</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>NH</td>
<td>27.</td>
<td>C₄’</td>
<td>12.0</td>
<td>1.78 (3H, d, J = 6.9 Hz)</td>
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</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>C₅’</td>
<td>28.</td>
<td>C₅’</td>
<td>14.0</td>
<td>1.87 (3H, s)</td>
<td>C2’, C5’</td>
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</tbody>
</table>

Structure of compound (Sracosalgnenone)

Showing some HMBC Connectivity’s of Compound