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The Isolation and Characterisation of Seco-phthalide Isoquinoline Alkaloid from *Corydalis* species

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Abstract

Chromatographic resolution of crude base fraction of the methanolic extract of the whole plant of Corydalis longipes on Silca-gel column yielded one Seco-phthalide isoquinoline alkaloid, N-methylhydrasteine hydroxyl lactam. Its structure was established on the basis of extensive spectroscopic data analysis and comparison with spectroscopic data reported.

Keywords: *Corydalis longipes , Fumariaceae, secophthalide alkaloid.*

Introduction

Corydalis longipes DC.Prodr.(Fumariaceae) a perennial herb, grows at an altitude of 2290-2350 m in the Himalayan region [1,2]. The extract of various *corydalis* species is reported to be efficacious in many ailments in the Indian Ayurvedic system and Chinese system of medicine[3,4].There have been no medicinal use has been reported from this plant. Very few alkaloids have been isolated from *Corydalis longipes*; viz. adlumidine, bicuculline, cheilanthifoline, protopine, soulerine, sibirine etc.[5,6]. In view of the above observations, the isolation of further alkaloids from the plant *Corydalis longipes* has resulted in the isolation and characterization of one seco-phthalide isoquinoline alkaloid , not earlier been reported from this plant.

Experimental methods

The melting point was determined on a Toshniwal apparatus and was uncorrected. UV spectrum was recorded with Perkin-Elmer Lambde spectrophotometer using spectral methanol. An IR spectrum was recorded in KBr pellets. ^{1}HNMR and ^{13} CNMR spectra were recorded in 500 MHz and 100 MHz respectively in $CDCl_3$ and CD_3OD using tetramethylsilane as internal reference. A mass spectrum was recorded on Kratos M-50 mass spectrometer operating 70 Ev. The purity of the was checked on TLC plates.

The whole plants of *Corydalis longipes* was collected and identified by comparison with the authentic herbarium specimen. Air dried powdered whole plant of the *C.longipes* (1kg) was extracted with methanol in a Soxhlet extracter which gave a brown semi solid mass(120 g). The methanolic extract was treated with 7% citric acid and separated to alkaloidal fraction according to the known method[7].

The chloroform extract(9 g) was chromatographed over silica-gel column using solvents of increasing polarity. The elluates from $CHCl_3$:MeOH (95:5) on crystallization from methanol yielded 41 mg of seco-phthalide isoquinoline alkaloid(1) identified by spectral analysis.

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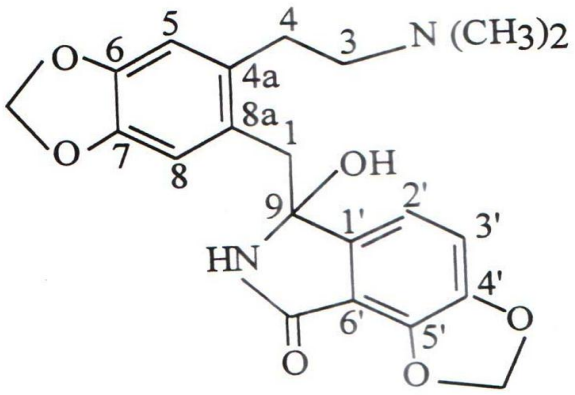

Structure: 1

Seco-phthalide alkaloid

Colourless granules, m.p.110-113^{o}C, uv, λmax (MeOH,nm); 216,(logε 4.24), 293 (log ε 4.10) and 315 (logε 3.18); ir (νmax, cm-1); 3320,1705, 1490, 1460, 1430,1270,; ^{1}HNMR ($CDCl_3$,δ); 1.85 (6 H,S), 2.35 (2H, t,J= 8 Hz), 2.70 (2H,m), 3.15(1H, d,J= 14Hz), 3.52(1H,d,J= 14), 5.75(1H,S), 5.80 (1H,S), 3.87 (3 H,S), 3.95 (3 H,S), 6.10(1H,S), 6.48(1 H, S), 7.15(1H, d, J=8Hz), 7.35 (1H,d, J= 8Hz), 8.0(1H,S),; 100 Mz.[13] CNMR($CDCl_3$,δ); 41.8(C-1),60.2(C-3),30.8(C-4),126.6(C-4a),190.4(C-5),146.4(C-6),145.4(C-7),111.0(C-8),132.6(C-8a),87.0(C-9),143.0(C-1$^{|}$),117.4(C-2$^{|}$),116.4(C-3$^{|}$),153.4(C-4$^{|}$),147.0(C-5$^{|}$),123.8(C-6$^{|}$),44.4(N-Me),44.4(N-Me),167.0(C=O),100.0(6,7-O-CH_2O),56.4(4$^{|}$-OMe),62.6(5$^{|}$-OMe),; MS(m/z, relative intensity %) 414(M+,10), 396 (70), 208(25),204(60),58(100).

Result and Discussion

The seco-phthalide isoquiniline alkaloid isolated from *Corydalis longipes* was characterized using spectroscopic analysis . The molecular formula of compound based on the high resolution mass spectrum was found to be $C_{22}H_{26}N_2O_6$; ms, m/z 414.1426 (M^+), 396,208,204,58(base peak). The base peak at m/z 58 suggests the fragment ion $CH_2=N^+(Me)_2$. Therefore, the structural unit $CH_2=N(Me)_2$ is present in compound. An intense peak at m/z 396 in the high mass region is not the M^+ but a fragment produced by loss of H_2O either thermally or by the electron impact from the M^+ m/z 414. This is characteristic of seco-phthalide isoquinoline alkaloid[8]. The peaks at m/z 204 and 208 in the spectrum originates by rupture of bond between 1 and 9 and provided the clue to the substitution pattern in ring A and B(Mass fragmentation Fig. 1). The ultraviolet spectrum in MeOH showed absorption maxima at 216, 293, and 315 nm like that of secothalide isoquinoline alkaloids[5]. The infra-red spectrum in KBr showed NH group at 3220cm-1 and lactam at 1705 cm-1 [6]. It also clearly indicated that the isolated alkaloid may be of seco-phthalide isoquinoline alkaloid having lactam group. 400MHz ^{1}HNMR in $CDCl_3$ exhibited a number of signals. The chemical shift of each signals together with there splitting pattern and probable assignment were compared with known seco-phthalide isoquinoline having lactam group [7] with only difference in attachment at position C-4$^{|}$ and C-5$^{|}$, two methoxy groups are attached at 4$^{|}$ and5$^{|}$ positions whereas known seco-phthalide contain methylene dioxy group at 4$^{|}$ and 5$^{|}$positions. All the other proton signals found to be similar. Hence , the structure of compound must be N-methylhydrasteine hydroxyl lactam(1). Further the structure of compound was supported by the comparison of the ^{13}CNMR data of compound with that of reported data[7,8]

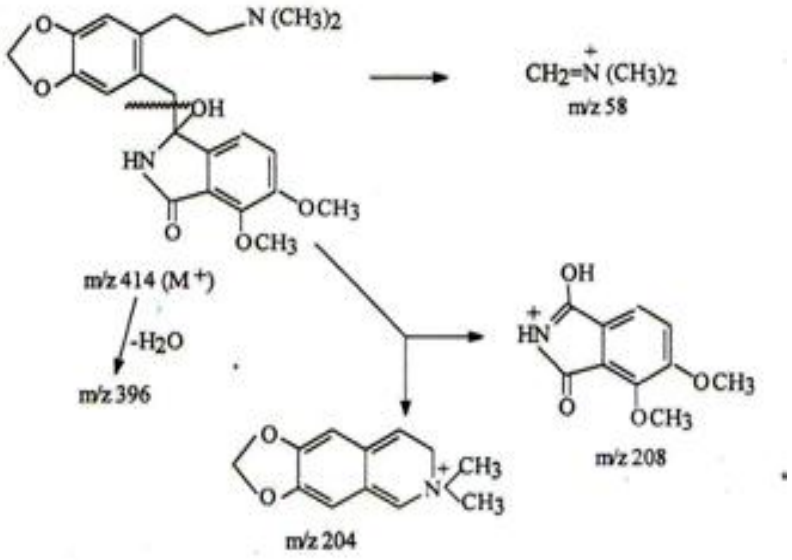

Fig. 1: *Mass fragmentation pattern*

Conclusion

The structure of isolated seco-phthalide isoquinoline alkaloid was determined by physical and spectroscopic method and comparison of its spectral data with those in the literature as N-methylhydrasteine hydroxyl lactam. This is the first report of the occurrence of this alkaloid in *Corydalis longipes.*

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