

This article was downloaded by:

On: 22 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Journal of Asian Natural Products Research

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713454007>

Alkaloids from *Anabasis aphylla* L.

Hua Du^a; Ye Wang^b; Chen Yan^b; Li-Gang Zhou^a; Xiao-Jiang Hao^b

^a College of Agronomy and Biotechnology, China Agricultural University, Beijing, China ^b The Key Laboratory of Chemistry for Natural Products of Guizhou Province and Chinese Academy of Science, Guiyang, China

To cite this Article Du, Hua , Wang, Ye , Yan, Chen , Zhou, Li-Gang and Hao, Xiao-Jiang(2008) 'Alkaloids from *Anabasis aphylla* L.', Journal of Asian Natural Products Research, 10: 11, 1093 – 1095

To link to this Article: DOI: 10.1080/10286020802318966

URL: <http://dx.doi.org/10.1080/10286020802318966>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

Alkaloids from *Anabasis aphylla* L.

Hua Du^a, Ye Wang^{b*}, Chen Yan^b, Li-Gang Zhou^a and Xiao-Jiang Hao^b

^aCollege of Agronomy and Biotechnology, China Agricultural University, Beijing, China; ^bThe Key Laboratory of Chemistry for Natural Products of Guizhou Province and Chinese Academy of Science, Guiyang, China

(Received 25 February 2008; final version received 3 June 2008)

A new pyridine alkaloid **1**, together with three known alkaloids *N*-methylanabasine (**2**), anabasamine (**3**), and isonicotene (**4**), was isolated from the aerial part of *Anabasis aphylla* L. Their structures were elucidated by spectroscopic analysis.

Keywords: *Anabasis aphylla* L.; chemical constituents; pyridine alkaloid; spectroscopic analysis

1. Introduction

The family Chenopodiaceae has 38 genera and 184 species, mostly distributed in the desert of Xinjiang province, northwest of China [1]. Since 1930, the chemical constituents from the genus of *Anabasis* have been reported, including alkaloids, steroids, terpenoids, and flavonoids [2]. The plant *Anabasis aphylla* L. was used as pesticide in the local regions [3], and in our continuous study to find antibacterial active components, a new pyridine alkaloid **1**, along with three known alkaloids, *N*-methylanabasine (**2**), anabasamine (**3**), and isonicotene (**4**), was obtained using column chromatography (CC) from the chloroform part of the crude ethanolic extract of this plant. Herein, we describe the isolation and structural elucidation of the new alkaloid.

2. Results and discussion

Compound **1** was obtained as a yellow oil. The molecular formula was determined as C₂₀H₂₀N₄ by positive HR-ESI-MS ion peak at *m/z* 317.1766 [M + H]⁺. ¹H NMR spectrum (Table 1) of **1** showed 3-substituted

pyridine moieties and 1-substituted piperidine group. Based on the ¹H–¹H COSY analysis, the spin coupling system of protons at δ_H 9.17 (1H, s, H-2''), 8.63 (1H, d, *J* = 4.8 Hz, H-6''), 8.31 (1H, d, *J* = 8.0 Hz, H-4''), and 7.40 (1H, dd, *J* = 4.8, 8.0 Hz, H-5''), and δ_H 8.66 (1H, s, H-2'''), 8.50 (1H, d, *J* = 4.8 Hz, H-6'''), 7.83 (1H, d, *J* = 8.0 Hz, H-4'''), and 7.28 (1H, dd, *J* = 4.8, 8.0 Hz, H-5'''), displayed the existence of two 3-substituted pyridyl moieties. Another 2,5-disubstituted pyridyl moiety was observed from the protons at δ_H 8.75 (1H, s, H-6'), 7.95 (1H, d, *J* = 8.0 Hz, H-4'), and 7.30 (1H, d, *J* = 8.0 Hz, H-3'). The piperidine moiety showed the presence of two methine protons at δ_H 3.94 (1H, d, *J* = 10.2 Hz, H-2) and 3.90 (1H, d, *J* = 10.2 Hz, H-6), two overlapped methylene protons at δ_H 1.84 (2H, m, H_a-3,5) and 1.60 (2H, m, H_c-3,5), and one methylene proton at δ_H 2.03 (1H, m, H_a-4) and 1.69 (1H, m, H_c-4). ¹³C NMR (Table 1) and DEPT spectra of **1** exhibited 20 carbons including four aromatic quaternary carbons at δ_C 153.7 (C-2'), 140.4 (C-3'''), 139.8 (C-5'), and 134.7 (C-3''); 11 aromatic tertiary carbons at δ_C 149.7 (C-6''), 148.9 (C-6'), 148.7 (C-6'''), 148.6

*Corresponding author. Email: wangyeqiang517@163.com

Table 1. ^1H and ^{13}C NMR spectral data and HMBC correlations of compound **1** (in CDCl_3 , J in Hz)^a.

| Position | δ_{H} | δ_{C} | HMBC |
|----------|---|---------------------|------------------------|
| 2 | 3.94 (1H _a , $J = 10.2$) | 59.6 | C-6 |
| 3 | 1.84 (1H _a , m) 1.60 (1H _c , m) | 34.4 | C-5, C-4 |
| 4 | 2.03 (1H _a , m) 1.69 (1H _c , m) | 25.3 | C-2, C-6 |
| 5 | 1.84 (1H _a , m) 1.60 (1H _c , m) | 34.4 | C-3, C-4 |
| 6 | 3.90 (1H _a , $J = 10.2$) | 59.9 | C-6' |
| 2' | – | 153.7 | |
| 3' | 7.30 (1H, d, $J = 8.0$) | 120.4 | C-2', C-5', C-3'' |
| 4' | 7.95 (1H, d, $J = 8.0$) | 135.4 | C-2', C-6', C-6 |
| 5' | – | 139.8 | |
| 6' | 8.75 (1H, s) | 148.9 | C-2', C-4', C-6 |
| 2'' | 9.17 (1H, s) | 148.0 | C-2', C-6'', C-4'' |
| 3'' | – | 134.7 | |
| 4'' | 8.31 (1H, d, $J = 8.0$) | 134.2 | C-2', C-6'', C-2''' |
| 5'' | 7.40 (1H, dd, $J = 4.8, 8.0$) | 123.5 | C-6'', C-3'' |
| 6'' | 8.63 (1H, d, $J = 4.8$) | 149.7 | C-2'', C-4'' |
| 2''' | 8.66 (1H, s) | 148.6 | C-6''', C-4''', C-2 |
| 3''' | – | 140.4 | |
| 4''' | 7.83 (1H, d, $J = 8.0$) | 134.4 | C-2''', C-6''', C-2 |
| 5''' | 7.28 (1H, dd, $J = 4.8, 8.0$) | 123.4 | C-3''', C-6''' |
| 6''' | 8.50 (1H, d, $J = 4.8$) | 148.7 | C-2''', C-4''', C-5''' |

^a Experiments were run at 400 MHz for ^1H and 100 MHz for ^{13}C .

(C-2'''), 148.0 (C-2''), 135.4 (C-4'), 134.4 (C-4'''), 134.2 (C-4''), 123.5 (C-5''), 123.4 (C-5'''), and 120.4 (C-3'); two tertiary carbons at δ_{C} 59.6 (C-2) and 59.9 (C-6); and three methylene carbons at δ_{C} 34.3 (overlapped, C-3, C-5) and 25.3 (C-4). It was easily recognized that the 2- and 6-positions of the piperidine ring were substituted by pyridine moieties, respectively, which was further supported by HMBC analysis. The HMBC spectrum (Figure 1) showed correlations between the protons at $\delta 7.83$ (H-4''') and 8.66 (H-2''') and the carbon at $\delta 59.6$ (C-2), which indicated that the 2-position of piperidine was connected to the 3'''-position of pyridine. The correlations between the protons at $\delta 7.95$ (H-4') and 8.75 (H-6') and the carbon at $\delta 59.6$ (C-6), and

between the protons at $\delta 9.17$ (H-2'') and 8.31 (H-4'') and the carbon at $\delta 153.7$ (C-2'), were also readily observed, which indicated that the 6-position of the piperidine ring was adjacent to the 5'-position of 3''-2' bipyridyl moiety. Thus, the structure of **1** was elucidated as 2-(pyridin-3-yl)-6-(2-(pyridin-3-yl)pyridin-5-yl)piperidine.

3. Experimental

3.1 General experimental procedures

The optical rotations were measured with a Horiba SEPA-300 spectropolarimeter. IR spectrum was obtained on a Bruker Vector-22 infrared spectrophotometer with KBr pellets. UV spectrum was taken on a Hewlett

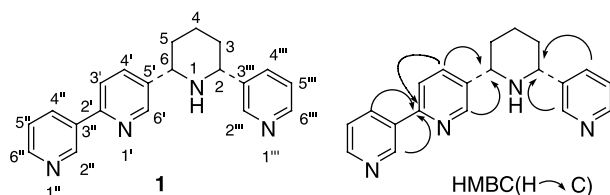


Figure 1. Structure and key HMBC correlations of **1**.

Packard 8453 spectrophotometer. NMR spectra (^1H NMR, ^{13}C NMR, and DEPT) were recorded on Varian Inova-400 instrument with TMS as an internal standard: δ in ppm and J in Hz. EI-MS and HR-ESI-MS were recorded on VG Autospec-3000 spectrometer.

Thin layer chromatography and CC were performed on plates precoated with silica gels F₂₅₄ and H (Qingdao Haiyang Chemical Co., Ltd., Qingdao, China), respectively. Solvents were distilled before use.

3.2 Plant material

The plants of *A. aphylla* were collected from Xinjiang in July 2005 and authenticated by Dr Y. Ping, Xinjiang Shihezi University. A voucher specimen (XJ20050710) has been deposited in Xinjiang Shihezi University.

3.3 Extraction and isolation

The air-dried aerial parts of *A. aphylla* were extracted with EtOH (95%) for three times under reflux (each process lasting 3 h). The EtOH extract was concentrated under reduced pressure, the residue was suspended in H₂O, and successively extracted with EtOAc and CHCl₃. The solvent was removed to give the EtOAc and CHCl₃ extracts. The CHCl₃ extracts were subjected to CC on silica gel; five fractions were obtained by gradient elution with petroleum ether–diethylamine (100:1 to 50:1) and fraction 4 was further

purified by CC on RP C₁₈ silica gel column eluting with CH₃OH–H₂O (2:8 to 4:6) and on Sephadex LH-20 eluting with CHCl₃–CH₃OH (1:1) to afford compounds **1** (30 mg), **2** (1 g), **3** (20 mg), and **4** (15 mg).

3.3.1 2-(Pyridin-3-yl)-6-(2-(pyridin-3-yl)pyridin-5-yl) piperidine (**1**)

Yellow oil, C₂₀H₂₀N₄. $[\alpha]_{\text{D}}^{20} + 60$ (c 1.0, CHCl₃). UV $\lambda_{\text{max}}^{\text{CHCl}_3}$ (nm) (log ϵ): 263 (3.66), 269 (3.59). IR (KBr) ν_{max} (cm⁻¹): 3415, 3036, 2939, 2863, 1665, 1578, 1479, 1428, 1249, 995, 715. For ^1H and ^{13}C NMR spectral data, see Table 1. EI-MS m/z : 316 [M⁺], 273, 210, 182. HR-ESI-MS m/z : 317.1766 [M + H]⁺ (calcd for C₂₀H₂₁N₄, 317.6555).

Acknowledgements

This study was supported by Foundation of Talents in Western China of CAS (2006) 378, Specialized Foundation of Prophase of National Major Basic Research (973) (2006CB708518) and Bingtuan Doctoral Foundation of China (2006JC09).

References

- [1] Editorial Committee of Flora of China, *Flora of China*, Vol. 43 (Sciences Press, Beijing, 1979), pp. 114–150.
- [2] A. Sadykov, *Izvest. Akad. Nau.* 524 (1953).
- [3] Z.M. Mao, *Flora of Xinjiang*, Vol. 2 (Xinjiang Health Sciences and Technology Press, Urumchi, 1994), pp. 73–76.