Pergamon

S0031-9422(96)00092-1

Phytochemistry, Vol. 42, No. 4, pp. 1211–1215, 1996

0031-9422/96 $15.00 + 0.00

SPERMINE ALKALOIDS FROM *ALBIZIA SCHIMPERANA*

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(*Received in revised form* 29 *December* 1995)

Key Word Index—*Albizia schimperana*; Leguminosae; macrocyclic spermine alkaloids; budmunchiamines; structural elucidation.

Abstract—The dichloromethane extract of the stem bark of *Albizia schimperana* yielded five macrocyclic spermine alkaloids (budmunchiamines), four of them novel. The structures of these compounds were elucidated by spectral analysis and comparison with literature data.

INTRODUCTION

Albizia schimperana Oliv. (Mimosoidae; Leguminosae) is a tree widely distributed in the highland forests in Kenya, where it is used in the preparation of medicines for the treatment of bacterial and parasitic infections, notably pneumonia, in wound infections and malaria, and more generally against fever and as an analgesic [1]. There is no report on the chemical constituents of *A. schimperana* to date. Related species have been recorded as yielding a wide range of triterpenes [2–6] and flavonoids [7–9]. The reported occurrence of alkaloids in *Albizia* is rare. However, a series of unusual macrocyclic spermine alkaloids (known as budmunchiamines A–I) have been reported from *A. amara* [10, 11] and, more recently, from *A. lebbek* [12]. In our phytochemical screening of some Kenyan *Albizia* species used as medicinal plants, we have isolated from *A. schimperana* five alkaloids containing this macrocyclic spermine nucleus; one was identified as the known budmunchiamine-A (**1**) and the others, which are all new, have been characterized as 6′ξ-hydroxy-budmunchiamine-C (**2**), 5-normethylbudmunchiamine-K (**3**), 6′ξ-hydroxy-5-normethylbudmunchiamine-K (**4**) and 14-normethylbudmunchiamine-K (**5**).

RESULTS AND DISCUSSION

The dichloromethane extract of the powdered bark of *A. schimperana* yielded five budmunchiamines (**1**–**5**). In the characterization of these compounds some common features were noted. They each showed a positive reaction with Dragendorff's reagent and were all isolated as yellow oils. They exhibited no UV absorption, but showed a characteristic IR absorption peak around 1647 cm^{-1}, attributable to a lactam (>5-membered ring) carbonyl group.

The high resolution EI mass spectrum of **1** showed a molecular ion at m/z 452, corresponding to the molecular formula $C_{27}H_{56}N_4O$. The J-modulated ^{13}C NMR spectrum displayed resonances attributable to the following groups: one C–Me (δ 14.3), three N–Me (δ 35.4, 43.0 and 42.7); five *N*-methylene (δ 51.9, 54.7, 56.6, 56.4 and 56.0), two further methylenes, one adjacent to an amide nitrogen atom [CONH–CH_2 (δ 37.9)] and the other next to an amide carbonyl carbon atom [CH_2CONH (δ 37.4)], one amide carbonyl (δ 172.9) and one N–CH (δ 61.5), with the rest of the resonances belonging to *C*-methylenes resonating between δ 22.9 and 33.4. The carbon spectral data (Table 1) thus suggested the existence of a macrocycle with a side chain, typical of the budmunchiamines.

Relationships between ^1H NMR resonances (Table 2) were assigned mainly on the basis of ^1H–^1H COSY and TOCSY experiments; and direct C–H correlations were achieved through the HC-COBI pulse sequence [13]. The HMBC technique [14] provided information on the long range connectivities between various protons and carbon atoms. Thus, the amide proton (δ 8.55) showed ^2J correlations with the amide carbonyl C-2 and to C-17 and ^3J coupling with C-3. The signal at δ 3.32, attributable to H-17, showed ^2J correlation with C-16 and ^3J interactions with C-2 and C-15. The *N*-methyl singlet at δ 2.19 also exhibited ^3J connectivity with C-15 and another methylene which must be C-13. The multiplet at δ 2.84 (δ_c 61.5), attributable to H-4, showed a ^2J connectivity to C-3 and ^3J correlations with C-2′ and the 5-*N*-Me, so fixing the junction between the macrocycle and the aliphatic side chain. The 5-*N*-Me singlet at δ 2.19 displayed ^3J connectivity with carbon signals C-4 and C-6, with the H-6 resonances at δ (2.62 and 2.41) exhibiting in turn, ^3J interactions with 5-*N*-Me, C-4 and C-8. The *N*-

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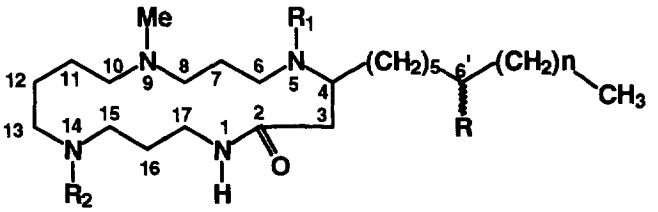

Compound	n	R	R_1	R_2
Budmunchiamine-A (1)	4	H	Me	Me
6ξ'-Hydroxybudmunchiamine-C (2)	6	OH	Me	Me
5-Normethylbudmunchiamine-K (3)	8	H	H	Me
6ξ'-Hydroxy-5-normethylbudmunchiamine-K (4)	8	OH	H	Me
14-Normethylbudmunchiamine-K (5)	8	H	Me	H

Table 1. ^{13}C NMR chemical shift data for compounds **1**–**5**

C	1	2	3	4	5
2	172.9	172.9	171.4	171.6	173.4
3	37.4	37.4	39.0	39.2	38.2
4	61.5	61.4	55.9	55.9	61.4
5-*N*-Me	35.4	35.6	—	—	36.7
6	51.9	51.6	46.7	46.8	51.8
7	26.2	25.8	26.1	28.0	25.8
8	54.7	54.5	57.3	57.4	55.8
9-*N*-Me	43.0	42.5	43.0	43.2	41.4
10	56.6	56.6	56.6	56.7	56.7
11	24.6	24.4	24.7	24.9	25.6
12	23.4	23.3	25.5	24.7	26.3
13	56.4	56.3	57.4	57.5	48.8
14-*N*-Me	42.7	42.4	42.5	42.5	—
15	56.0	55.8	54.4	54.7	27.8
16	27.7	27.5	26.2	26.3	36.9
17	37.6	37.8	37.8	37.9	29.9*
1′	29.5*	29.9*	33.4*	33.3*	27.7
2′	27.6	27.3	26.2	26.1	30.0*
3′	30.0*	29.8*	29.9*	29.7*	29.8*
4′	29.8*	29.7*	29.8*	29.8*	29.8*
5′	29.8*	37.5†	29.8*	37.8†	29.8*
6′	29.8*	72.1	29.8*	72.1	29.8*
7′	29.8*	37.7†	29.7*	37.7†	29.8*
8′	29.7*	29.6*	29.7*	29.5*	29.8*
9′	32.1	29.8*	29.6*	29.5*	29.8*
10′	22.9	29.8*	29.6*	29.5*	29.7*
11′	14.3	32.0	29.5*	29.4*	29.5*
12′		22.9	29.5*	29.5*	29.8*
13′		14.3	32.1	32.2	32.1
14′			22.9	23.0	22.9
15′			14.3	14.3	14.3

*,†Values with the same superscript in each column are interchangeable.

methyl singlet at δ 2.29 (9-*N*-Me) showed ^3J connectivity with carbon signals for C-8 and C-10, while the multiplet at δ 1.51 H-11 also showed ^3J interactions with C-10 and with C-13, so closing the macrocyclic ring.

The EI mass spectral fragmentation pattern of **1** exhibited a significant peak at *m/z* 297 (36.9%) corresponding to $C_{16}H_{33}N_4O$ [$M^+ - C_{11}H_{23}$] attributable to the macrocycle. The loss of the side chain was further demonstrated by a series of fragment ions with a systematic loss of an additional 14 mass units after the initial loss of the terminal methyl group; this is characteristic of long chain aliphatic compounds [15]. Thus the mass spectrum confirmed the presence of the 11-carbon aliphatic side chain linked to the macrocycle, establishing **1** as the known compound budmunchiamine-A. The above data are given in some detail as they proved critical to the identification of the much less abundant new alkaloids.

The high resolution EI mass spectrum of **2** showed a molecular ion at *m/z* 496, corresponding to the formula $C_{29}H_{60}N_4O_2$, again with a significant fragment at *m/z* 297 (41.0%), attributable to the macrocycle as in **1**. The *J*-modulated ^{13}C NMR spectrum (Table 1) showed resonances which were, for the most part, comparable to those of **1**, the main difference being the presence of a carbinolic resonance at δ 72.1 and two methylene resonances at δ 37.5 and 37.7, assumed to be for the carbons adjacent to the carbinol. Thus, these data indicated the existence of the same macrocycle, but differing in the presence of a secondary alcohol in a side chain that was two carbons longer than **1** (corresponding to the known compound budmunchiamine-C [10]). The ^1H NMR spectrum (Table 2) also showed resonances comparable to those of **1**, except that in **2** there were additional signals at δ 3.56 (*m*) and 1.45

Table 2. ^{1}H NMR chemical shift data for compounds **1–5**

H	1	2	3	4	5
1-*N*-H	8.55 *bs*	8.43 *bs*	8.42 *bs*	8.34 *bs*	8.35 *bs*
3	2.23, 2.37 *m*	2.23, 2.27 *m*	2.19, 2.30 *m*	2.20, 2.35 *m*	2.25, 2.45 *m*
4	2.84 *m*	2.85 *m*	2.95 *m*	3.00 *m*	2.06 *m*
5-*N*-Me	2.19 *s*	2.22 *s*			2.28 *s*
6	2.62 *dt* (7)	2.65 *dt* (7)	2.82 *m*	2.89 *m*	2.65 *m*
	2.42 *m*	2.45 *m*	2.82 *m*	2.82 *m*	2.45 *m*
7	1.63 *m*	1.69 *m*	1.72 *m*	1.73 *m*	1.65 *m*
8	2.39 *m*	2.52–2.62 *m*	2.48–2.53 *m*	2.48–2.56 *m*	2.53–2.58 *m*
9-*N*-Me	2.29 *s*	2.37 *s*	2.26 *s*	2.26 *s*	2.29 *s*
10	2.30–2.40 *m*	2.39–2.50 *m*	2.38–2.41 *m*	2.39–2.42 *m*	2.38–2.43 *m*
11	1.51 *m*	1.53 *m*	1.53 *m*	1.53 *m*	1.75 *m*
12	1.55 *m*	1.58 *m*	1.54 *m*	1.54 *m*	1.85 *m*
13	2.40–2.52 *m*	2.50–2.55 *m*	2.34–2.40 *m*	2.35–2.42 *m*	2.95, 2.72 *m*
14-*N*-Me	2.19 *s*	2.25 *s*	2.23 *s*	2.24 *s*	
15	2.35–2.43 *m*	2.42–2.48 *m*	2.40–2.48 *m*	2.42–2.48 *m*	2.87 *m*
16	1.66 *m*	1.67 *m*	1.67 *m*	1.66 *m*	1.93 *m*
17	3.32 *dt* (11,5)	3.36 *dt* (13,6)	3.34 *m*	3.34 *m*	3.64 *m*
		3.27 *dt* (13,6)			3.15 *m*
1′	1.12 *m*	1.15 *m*	1.20 *m*	1.26 *m*	1.25 *m*
	1.53 *m*	1.52 *m*	1.20 *m*	1.26 *m*	1.25 *m*
2′	1.26 *m*	1.25 *m*	1.26 *m*	1.26 *m*	1.25 *m*
3′	1.26 *m*	1.25 *m*	1.26 *m*	1.26 *m*	1.25 *m*
4′	1.26 *m*	1.25 *m*	1.26 *m*	1.26 *m*	1.25 *m*
5′	1.26 *m*	1.45 *m*	1.26 *m*	1.43 *m*	1.25 *m*
6′	1.26 *m*	3.56 *m*	1.26 *m*	3.58 *m*	1.25 *m*
7′	1.26 *m*	1.45 *m*	1.26 *m*	1.43 *m*	1.25 *m*
8′	1.26 *m*	1.25 *m*	1.26 *m*	1.26 *m*	1.25 *m*
9′	1.26 *m*	1.25 *m*	1.26 *m*	1.26 *m*	1.25 *m*
10′	1.30 *m*	1.25 *m*	1.26 *m*	1.26 *m*	1.25 *m*
11′	0.88 *t* (7)	1.25 *m*	1.26 *m*	1.26 *m*	1.25 *m*
12′		1.30 *mm*	1.26 *m*	1.26 *m*	1.25 *m*
13′		0.88 *t* (7)	1.26 *m*	1.26 *m*	1.25 *m*
14′			1.30 *m*	1.30 *m*	1.30 *m*
15′			0.88 *t* (7)	0.89 *t* (7)	0.88 *t* (7)

(4H, *m*) attributable to oxymethine proton, and the methylenes adjacent to it. The high resolution EI mass spectrum of **2** demonstrated the normal systematic fragmentation of the aliphatic chain, giving rise to a series of fragments with a difference of 14 mass units. This pattern of fragmentation proceeded until the ion *m/z* 397 (39.0%), corresponding to $[M^+ - C_7H_{15}]$. Subsequent to this, fragments showed loss of an oxygen atom with the accompanying methylene unit {(next ion *m/z* 367 (29.0%) $[M^+ - C_8H_{17}O]$}. The fragments at *m/z* 397 (39.0%) and 367 (29.0%) were significant as they resulted from α-fission of the C–C bonds adjacent to the carbinolic carbon [16]. These considerations permitted the placement of the hydroxyl group on C-6′ in the aliphatic side chain (stereochemistry not assigned), Compound **2** is, therefore, 6′*ξ*-hydroxybudmunchiamine-C, which is novel.

The high resolution EI mass spectrum of **3** gave a molecular formula of $C_{30}H_{62}N_4O$ and the *J*-modulated ^{13}C NMR spectrum (Table 1) revealed all the typical resonances, except that there were only two *N*-methyl signals (δ 43.0 and 42.5). This suggested that **3** had a macrocycle similar to that of **1** and **2**, with the principal difference being that **3** had only two *N*-methyl groups. The absence of a *N*-methyl resonance at *ca* δ 35 suggested that **3** had a 5-NH, and the shifts of the methine C-4 and methylene C-6 resonances by *ca* 5 ppm upfield was supportive of this hypothesis. The HMBC spectrum provided further confirmation of the substitution on the macrocycle. The *N*-Me singlet at δ 2.23 showed ^3J correlations with δ 54.4 (C-15) and 57.4 (C-13), with the δ 1.67 multiplet attributable to H-16 also correlating with C-15 and identifying C-17 (δ 37.8). The second methyl singlet at δ 2.26 showed ^3J connectivities with C-8 (δ 57.3) and C-10 (δ 56.6). The multiplet at δ 2.82, assignable to H-6, also showed coupling to C-8 (^3J), as well as to C-7 (δ 26.1) and with the *N*-methine resonance at δ 55.9 (C-4). These correlations confirm the presence of *N*-Me at N-9 and N-14.

The high resolution EI mass spectrum of **3** showed a significant peak at *m/z* 283 (100%) corresponding to $[M^+ - C_{15}H_{31}]$ for a macrocycle 14 mass units less than in **1** and **2** (*m/z* 297). The rest of the mass fragmentation resembled that of **1** starting with loss of terminal methyl followed by progressive loss of an additional 14 mu. Compound **3** is unusual in having a 15-C side chain and appears to be novel. We have

assigned the trivial name 5-normethylbudmunchiamine-K, based on a hypothetical parent compound (budmunchiamine-K) where N-5 is methylated.

The high resolution EI mass spectrum of **4** showed a molecular ion at m/z 520 ($C_{30}H_{62}N_4O_2$) and a significant fragment ion at m/z 283 (67.0%) similar to **3**. The J-modulated ^{13}C NMR spectral data (Table 1) displayed resonances comparable to those of **3**, except that there were signals at δ 72.1 and at 37.8 and 37.7 attributable to the carbinolic carbon and the neighbouring methylene carbons. The ^1H NMR spectrum likewise showed resonances for an oxymethine (δ 3.58, m), and 1.43 (4H, m). Thus, spectral data suggested **4** was a direct parallel with **2** and this was substantiated by EI mass spectral fragmentation of the side chain, which again revealed the oxygen to be at C-6′. Compound **4** is a novel alkaloid given the trivial name 6′ξ-hydroxy-5-normethylbudmunchiamine-K.

The high resolution EI mass spectrum of **5** gave a molecular ion at m/z 494 ($C_{30}H_{62}N_4O$) and a significant fragment at m/z 283 (62.6%), suggesting it was an isomer of **3**. The ^{13}C NMR spectrum displayed all the anticipated resonances with two N-Me signals at δ 36.7 and 41.4. This therefore implied that either 9-N-Me or 14-N-Me was replaced by an N–H. This was further supported by the upfield shift of two N-methylene carbon atoms to δ 48.8 and 46.6 resulting from the shielding effect provided by the N–H group between them. In the HMBC spectrum the methyl resonance at δ 2.28 revealed ^3J couplings with the methylene C-6 (δ 51.8) and the methine C-4 (δ 61.4), so establishing the presence of a 5-N-Me substituent. This permitted identification of H-6 which showed a further ^3J correlation to another N-methylene at δ 55.8, which must be C-8. As the second N-Me resonance (δ 2.29) also correlated with C-8 there must be a 9-N-Me and it must be N-14, which is demethylated. On this basis **5** must be 14-normethylbudmunchiamine-K, which is again novel.

Budmunchiamines are unusual compounds whose biosynthetic route has been postulated [17] as originating from spermine and long chain fatty acids. Until now their occurrence had been limited to *A. amara* [10, 11], *A. lebbek* [12]; and related alkaloids (pithecolobines) are known from *Pithecellobium saman* (Mimosoidae) [18].

EXPERIMENTAL

IR spectra were recorded on a Mattson Genesis Series FT-IR spectrophotometer; $[\alpha]_D$ were obtained on a Perkin-Elmer 241 polarimeter. EIMS were recorded using an AEI-MS 902 double-focusing instrument (direct probe insert at 70 eV). NMR spectra were recorded on a Bruker AMX-400 instrument in $CDCl_3$ (δ_H 7.27, δ_C 77.23). Petrol refers to petroleum ether (bp 60–80°).

Plant material. The stem bark of *A. schimperana* was collected in June 1992 from Karura forest, Kiambu District, Kenya. The plant was identified by the staff of the Botany Department, University of Nairobi, and voucher specimens are deposited at the Herbarium of that Department.

Extraction and isolation of compounds. The stem bark was air dried, powdered and a sample (500 g) exhaustively extracted in a Soxhlet with, successively, petrol, CH_2Cl_2 and MeOH. The frs were dried *in vacuo* and analysed by TLC, alkaloids being detected with Dragendorff's reagent. The CH_2Cl_2 fr. (4.0 g), containing most of the alkaloids, was subjected to CC on silica gel, eluting with $CHCl_3$ containing increasing amounts of Et_2NH (to a max. of 2%). Frs (100–150 ml) were collected and their content monitored using precoated analyt. silica gel plates (solvent: $CHCl_3$–Et_2NH, 19:1). Pure frs were pooled while impure ones were further subjected to prep. TLC, with multiple developments using solvent systems composed of $CHCl_3$–Et_2NH (125:1, 100:1 and 49:1). The major compound, **1** (150 mg, 0.03%), was obtained pure from CC, while **2** (15 mg, 0.003%), **3** (15 mg, 0.003%), **4** (10 mg, 0.002%) and **5** (11 mg, 0.0022%) were obtained after PTLC.

Budmunchiamine-A (**1**). $[\alpha]_D$ −9.3° (*c* 0.08, $CHCl_3$). IR ($CHCl_3$) ν_{max} cm^{-1}: 3400, 2980, 2800, 1647. EIMS m/z (rel. int): 452 (4.2), 437 (11.9), 423 (36.1), 409 (27.5), 395 (7.0), 381 (11.3), 367 (20.2), 353 (17.1), 339 (36.2), 325 (8.6), 311 (8.8), 297 (36.9).

6ξ-Hydroxybudmunchiamine-C (**2**). $[\alpha]_D$ −6.4° (*c* 0.08, $CHCl_3$). IR ($CHCl_3$) ν_{max} cm^{-1}: 3400, 2928, 2800, 1647. EIMs m/z (rel. int): 496 (5.0), 481 (10.1), 467 (5.9), 453 (15.7), 439 (36.2), 425 (18.0), 411 (11.5), 397 (39.0), 367 (29.0), 353 (9.0), 339 (7.0), 325 (3.1), 311 (4.7), 297 (41.0).

5-Normethylbudmunchiamine-K (**3**). $[\alpha]_D$ −5.1° (*c* 0.08, $CHCl_3$). IR ($CHCl_3$) ν_{max} cm^{-1}: 3200, 2928, 2855, 1647. EIMS m/z (rel. int): 494 (3.5), 479 (4.4), 465 (10.0), 451 (32.2), 437 (38.4), 423 (14.0), 409 (13.0), 395 (10.3), 382 (11.2), 368 (13.2), 367 (9.8), 366 (9.0), 354 (8.9), 353 (9.8), 352 (11.0), 340 (12.9), 339 (11.2), 325 (12.6), 311 (14.9), 297 (23.4), 283 (100).

6ξ-Hydroxy-5-normethylbudmunchiamine-A (**4**). $[\alpha]_D$ −2.3° (*c* 0.01, $CHCl_3$). IR ($CHCl_3$) ν_{max} cm^{-1}: 3400, 2928, 2855, 1650. EIMS m/z (rel. int): 510 (4.8), 495 (2.7), 482 (2.3), 467 (10.0), 453 (20.0), 439 (38.4), 425 (14.0), 411 (4.1), 397 (7.6), 383 (30.5), 366 (9.0), 354 (8.9), 353 (33.7), 340 (12.9), 339 (5.4), 325 (6.5), 311 (5.2), 297 (13.4), 283 (67.0).

14-Normethylbudmunchiamine-A (**5**). $[\alpha]_D$ −1.8° (*c* 0.09, $CHCl_3$). IR ($CHCl_3$) ν_{max} cm^{-1}: 3200, 2928, 2855, 2799, 1647. EIMS m/z (rel. int): 494 (5.4), 479 (6.4), 465 (13.0), 451 (36.2), 437 (45.4), 423 (23.0), 409 (13.5), 395 (13.1), 381 (20.8), 367 (11.8), 353 (11.3), 352 (11.0), 339 (10.0), 325 (11.5), 311 (10.6), 297 (25.6), 283 (62.6).

Acknowledgements—One of us (G. M. R.) thanks the Association of Commonwealth Universities for granting

the scholarship. NMR spectra were run in the University of Strathclyde NMR facility.

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