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Structures of four new alkaloids from *Stemona sessilifolia*

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Abstract—Four new alkaloids, sessilifoliamides E, F, G, and H were isolated from the roots of *Stemona sessilifolia* (Miq.) Miq., together with a known alkaloid, tuberostemonone. The structures of new alkaloids were elucidated by interpretation of the spectral data and X-ray crystallography.

1. Introduction

The plants belonging to the genus *Stemona* (family Stemonaceae) have been used in China and Japan as an insecticide and for cough remedy. A number of alkaloids have been isolated from *Stemona japonica* (Blume) Miq., *Stemona tuberosa* Lour., and *Stemona sessilifolia* (Miq.) Miq.,[1] including sessilifoliamides A–D from the roots of *S. sessilifolia*.[2] In the present study, from the roots of *S. sessilifolia*, we further isolated four new alkaloids, sessilifoliamides E, F, G, and H (**1**–**4**) along with a known alkaloid, tuberostemonone (**5**).[3,4] The structures of the new alkaloids **1**–**4** were determined on the basis of the spectral data and X-ray crystallographic analysis (Fig. 1). This paper describes their isolation and structure elucidation.

2. Results and discussion

From 15 kg of the roots of *S. sessilifolia*, 8 kg of a crude MeOH extract was obtained, from which 250 g of a basic fraction and 300 g of a mixture of neutral and acidic components were prepared. The mixture of neutral and acidic components was subjected to silica gel column chromatography, and the fraction obtained by eluting the column with EtOAc–MeOH (10/1) was then subjected to aminopropyl-bonded silica gel column chromatography, and then to reversed-phase HPLC. Four new alkaloids, **1** (3.6 mg), **2** (4.4 mg), **3** (2.5 mg), and **4** (2.2 mg), and one known alkaloid, **5** (0.7 mg) were obtained. Alkaloid **5** was identified as tuberostemonone by comparison of its spectral data with those reported[3,4] (Fig. 1).

Sessilifoliamide E (**1**) was obtained as colorless prisms. Its molecular formula was determined to be $C_{22}H_{31}NO_6$ from the $[M+H]^+$ peak at *m/z* 406.2228 (calcd for $C_{22}H_{32}NO_6$ 406.2230) in the HRESIMS. The IR spectrum showed the presence of γ-lactone (1774 cm^{-1}), ketone (1698 cm^{-1}), and amide (1678 cm^{-1}) carbonyl groups. Its ^1H NMR spectrum showed the presence of one terminal methyl (δ 0.78) and two secondary methyl (δ 1.25 and 1.30) groups, and two oxymethine protons (δ 4.24 and 4.93) (Table 1). Its ^{13}C NMR spectrum showed twenty-two signals caused by three methyls, seven methylenes, eight methines, and four quaternary carbons. Of the quaternary carbon signals, one at δ 173.4 was assigned to an amide carbonyl carbon, two at δ 177.3 and 178.2 to lactone carbonyl carbons, and one at δ 208.8 to a ketone carbon (Table 2). Analysis of the ^1H–^1H COSY and HMQC spectra revealed the presence

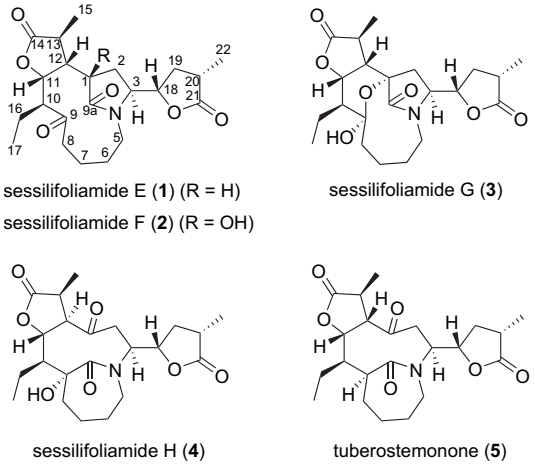

Figure 1. Structures of alkaloids presently isolated from *Stemona sessilifolia*.

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Table 1. ^{1}H NMR data for sessilifoliamides E–H (**1**–**4**)[a]

Position	Sessilifoliamide E (**1**)	Sessilifoliamide F (**2**)	Sessilifoliamide G (**3**)	Sessilifoliamide H (**4**)
1	2.83 (ddd, 10.7, 10.2, 5.5)			
2a	1.70 (m)	1.68 (d, 14.8)	1.46 (dd, 12.2, 8.7)	2.67 (dd, 17.4, 6.4)
2b	2.02 (m)	2.28 (ddd, 14.8, 9.1, 1.4)	2.13 (dd, 12.2, 4.8)	3.09 (dd, 17.4, 12.6)
3	3.68 (ddd, 8.7, 8.7, 1.3)	3.63 (ddd, 9.1, 8.9, 1.6)	4.03 (ddd, 8.7, 8.5, 4.8)	5.33 (ddd, 12.6, 6.4, 3.9)
5a	3.29 (dd, 14.2, 6.0)	3.36 (ddd, 14.3, 6.6, 2.5)	3.36 (ddd, 14.9, 11.6, 6.5)	3.39–3.49 (m, 2H)
5b	3.90 (ddd, 14.2, 11.4, 1.6)	3.83 (ddd, 14.3, 10.5, 2.4)	3.75 (ddd, 14.9, 6.1, 1.2)	
6a	1.71 (m)	1.84 (m)	1.81 (m)	1.69–1.76 (m, 2H)
6b	2.15 (m)	2.00 (m)	2.09 (m)	
7a	1.61 (m)	1.75 (m)	1.24 (m)	1.61 (m)
7b	1.84 (m)	1.75 (m)	1.71 (m)	1.92 (m)
8a	2.42 (ddd, 19.7, 5.0, 3.5)	2.41 (m)	1.85 (m)	1.73 (m)
8b	2.71 (ddd, 19.7, 11.7, 3.3)	2.56 (m)	1.90 (m)	1.87 (m)
10	2.47 (m)	2.62 (m)	1.39 (ddd, 10.0, 7.5, 6.0)	1.63 (m)
11	4.93 (dd, 11.9, 6.7)	4.92 (dd, 11.6, 6.6)	4.94 (dd, 10.0, 7.1)	4.38 (dd, 11.0, 1.7)
12	3.04 (ddd, 12.5, 6.7, 5.5)	2.93 (dd, 9.0, 6.6)	2.40 (dd, 12.5, 7.1)	3.41 (dd, 9.4, 1.7)
13	2.33 (dq, 12.5, 7.0)	2.75 (dq, 9.0, 7.2)	2.78 (dq, 12.5, 6.9)	2.79 (dq, 9.4, 7.2)
15	1.25 (d, 7.0, 3H)	1.44 (d, 7.2, 3H)	1.28 (d, 6.9, 3H)	1.11 (d, 7.2, 3H)
16a	1.51 (m)	1.67 (m)	1.55 (m)	1.53 (m)
16b	2.02 (m)	1.95 (m)	1.67 (m)	1.85 (m)
17	0.78 (t, 7.4, 3H)	0.78 (t, 7.5, 3H)	1.04 (t, 7.5, 3H)	1.00 (t, 7.3, 3H)
18	4.24 (ddd, 10.5, 8.7, 5.7)	4.40 (ddd, 10.5, 8.9, 5.5)	4.15 (ddd, 10.7, 8.5, 5.5)	4.48 (ddd, 10.5, 5.7, 3.9)
19a	1.55 (m)	1.58 (m)	1.57 (m)	1.74 (m)
19b	2.51 (m)	2.56 (m)	2.45 (m)	2.53 (m)
20	2.65 (m)	2.65 (m)	2.67 (m)	2.76 (m)
22	1.30 (d, 7.0, 3H)	1.30 (d, 7.0, 3H)	1.31 (d, 7.0, 3H)	1.31 (d, 7.0, 3H)
OH-1		2.91 (d, 1.4)		
OH-9			2.34 (s)	3.81 (d, 1.3)

[a] Recorded at 500 MHz in $CDCl_3$. Chemical shifts in parts per million, relative to the residual $CHCl_3$ resonance at 7.26 ppm. Multiplicity and *J* values are given in Hertz in parentheses.

of two molecular fragments, i.e., a four-carbon chain fragment (C-5–C-6–C-7–C-8) in which all the carbons were methylenes and a ten-carbon chain fragment (C-15–C-13–C-12–C-1–C-2–C-3–C-18–C-19–C-20–C-22) in which C-15 and C-22 were two terminal methyls and C-18 was an oxymethine carbon. The ^{1}H–^{1}H COSY and HMQC data also established the presence of a four-carbon chain (C-11–C-10–C-16–C-17) whose C-17 was a terminal methyl, and C-11 an oxymethine connected to C-12 of the above ten-carbon chain fragment (Fig. 2). The locations of the four carbonyl carbons and connectivity of those two carbon chain fragments were determined on the basis of HMBC experiments. By the correlations from H-1, H-2, and H-3 to the amide carbonyl carbon (δ 173.4), it was placed at position 9a, thus connecting to C-1, and C-3 was connected to the amide nitrogen to form a pyrrolidone ring (Fig. 2). The amide nitrogen was also connected to C-5, as HMBC correlations from H-5b to C-3 and from H_2-5 to C-9a were

Table 2. ^{13}C NMR data for sessilifoliamides E–H (**1**–**4**)[a]

Position	Sessilifoliamide E (**1**)	Sessilifoliamide F (**2**)	Sessilifoliamide G (**3**)	Sessilifoliamide H (**4**)
1	39.0	75.5	77.4	207.8
2	21.6	29.7	32.8	45.0
3	58.7	59.2	64.3	54.6
5	41.7	42.5	45.5	41.7
6	20.9	22.0	25.3	25.0
7	20.2	20.3	14.6	17.9
8	35.0	36.9	33.3[b]	26.3
9	208.8	207.8	101.4	81.8
9a	173.4	173.2	178.2	180.5
10	57.6	54.9	52.6	58.7
11	79.7	77.5	79.0	80.1
12	46.5	52.8	43.9	51.4
13	35.7	35.7	34.5	35.5
14	177.3	178.0	178.2	176.2
15	13.1	16.0	15.2	10.2
16	24.3	23.6	22.0	19.3
17	10.8	9.9	13.2	15.7
18	80.6	81.1	81.8	77.4
19	34.5	34.5	33.4[b]	33.6
20	34.7	34.5	34.5	35.3
21	178.2	178.4	178.3	177.7
22	15.0	14.9	15.0	15.2

[a] Recorded at 125 MHz in $CDCl_3$. Chemical shifts in parts per million, relative to the solvent resonance at 77.03 ppm.
[b] Assignments may be interchanged.

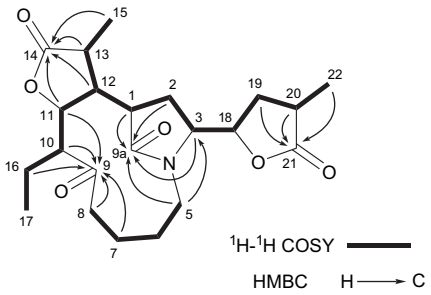

Figure 2. ^{1}H–^{1}H COSY and selected HMBC correlations for sessilifoliamide E (**1**).

observed. NOE correlations between H-3 and H_2-5 and the chemical shift values of C-3 (δ 58.7) and C-5 (δ 41.7) supported connection of those carbons to the amide nitrogen (Fig. 2). The two lactone carbonyl carbons, C-14 and C-21, were shown to form γ-lactones between C-14 and the C-11 oxygen, and between C-21 and the C-18 oxygen, respectively, by HMBC correlations from H-11, H-12, H-13, and H_3-15 to C-14 and from H-19b, H-20, and H_3-22 to C-21, respectively. The ketone carbonyl carbon was placed at position 9 by correlations from H-7a, H_2-8, H-10, H-11, and H_2-16 to C-9. Thus, alkaloid **1** was shown to possess an 11-membered lactam ring structure. The NOESY spectrum gave only limited information about its stereochemistry due to the presence of an 11-membered ring and the rotatable C-3–C-18 single bond. Correlations between the proton signals, H-1/H-18, H-11/H_3-15, and H-18/H-20 indicated that H-1/H-3, H-11/Me-15, and H-18/H-20 were in trans, cis, and cis relationships, respectively. The relative stereochemistry of **1** was finally established by an X-ray crystallographic analysis as shown in Figure 3.

Sessilifoliamide F (**2**) was obtained as colorless prisms. Its molecular formula was determined to be $C_{22}H_{31}NO_7$ from

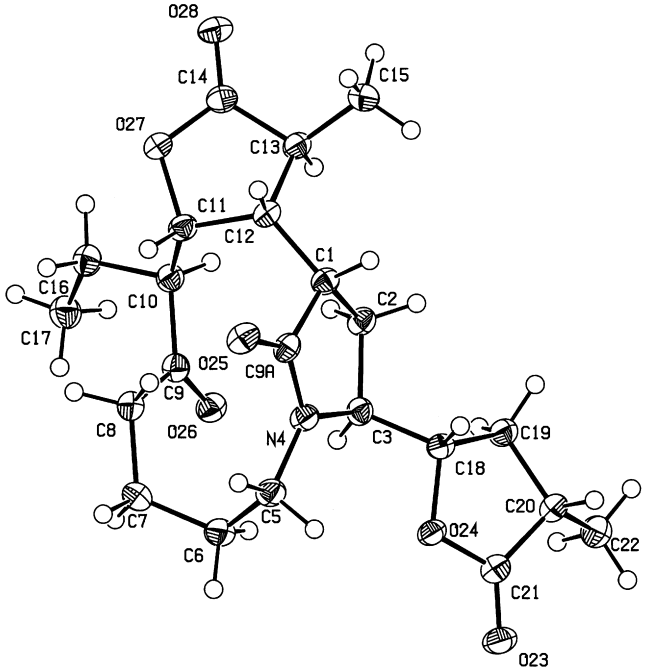

Figure 3. ORTEP drawing of the crystal structure of sessilifoliamide E (**1**).

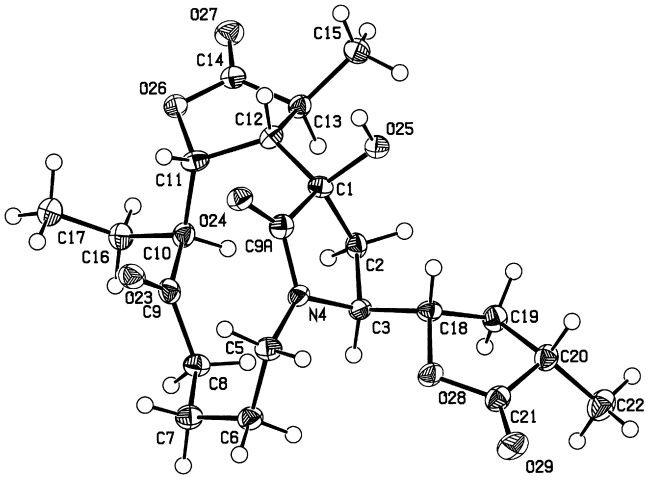

Figure 4. ORTEP drawing of the crystal structure of sessilifoliamide F (**2**).

the $[M+H]^+$ peak at *m/z* 422.2179 (calcd for $C_{22}H_{32}NO_7$ 422.2179) in the HRESIMS. The IR spectrum showed the presence of hydroxyl (3381 cm^{-1}), γ-lactone (1774 cm^{-1}), ketone (1699 cm^{-1}), and amide (1683 cm^{-1}) carbonyl groups. The similarity of the ^{1}H and ^{13}C NMR spectra of **2** to those of **1** and its HMBC spectra revealed that **2** had the same basic structure as **1** (Tables 1 and 2). The major difference between **2** and **1** in the NMR spectra was that the C-1 signal was of an oxygen-bearing quaternary carbon (δ_C 75.5) in **2**, whereas it was of a methine carbon (δ_H 2.83, δ_C 39.0) in **1**, demonstrating that **2** was a C-1 hydroxylated analogue of **1**. The presence of a hydroxyl group at C-1 was demonstrated by the HMBC correlations from OH-1 to C-2 and C-9a. The relative stereochemistry of **2** was established by an X-ray crystallographic analysis, as shown in Figure 4.

Sessilifoliamide G (**3**) was obtained as colorless prisms. Its molecular formula was determined to be $C_{22}H_{31}NO_7$ from the $[M+H]^+$ peak at *m/z* 422.2186 (calcd for $C_{22}H_{32}NO_7$ 422.2179) in the HRESIMS, being the same as that of **2**. The IR spectrum showed the presence of hydroxyl (3463 cm^{-1}), γ-lactone (1773 cm^{-1}), and carbonyl (1702 cm^{-1}) groups. The ^{1}H and ^{13}C NMR spectra of **3** were generally similar to those of **2**, indicating the presence of one terminal methyl (δ_H 1.04, δ_C 13.2) and two secondary methyl groups (δ_H 1.28, δ_C 15.2; δ_H 1.31, δ_C 15.0), and two oxymethine carbons (δ_H 4.15, δ_C 81.8; δ_H 4.94, δ_C 79.0) (Tables 1 and 2). The major difference between the NMR spectra of **3** and **2** was that the C-9 signal of **3** was at δ 101.4, whereas the corresponding C-9 signal of **2** was at δ 207.8. The chemical shift value of C-9 and the HMBC correlations from the hydroxyl proton (δ_H 2.34) to C-8, C-9, and C-10 showed that C-9 was to be assigned to a hemiacetal carbon. Since the molecule has been shown to have eight degrees of unsaturation, the hemiacetal ether linkage was placed between C-1 and C-9. Its relative stereochemistry was determined as shown in Figure 5 by an X-ray crystallographic analysis.

Sessilifoliamide H (**4**) was obtained as colorless prisms. Its molecular formula was determined to be $C_{22}H_{31}NO_7$ from the $[M+H]^+$ peak at *m/z* 422.2179 (calcd for $C_{22}H_{32}NO_7$

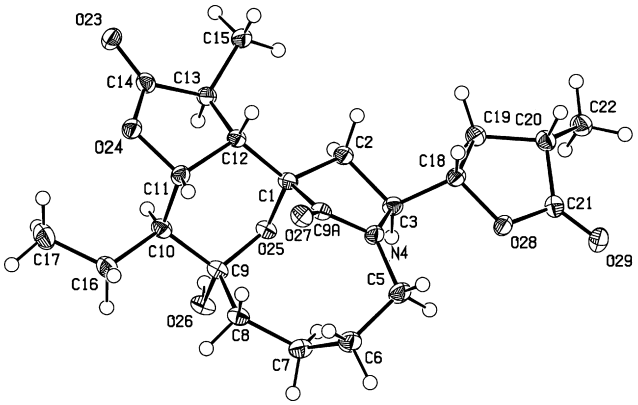

Figure 5. ORTEP drawing of the crystal structure of sessilifoliamide G (**3**).

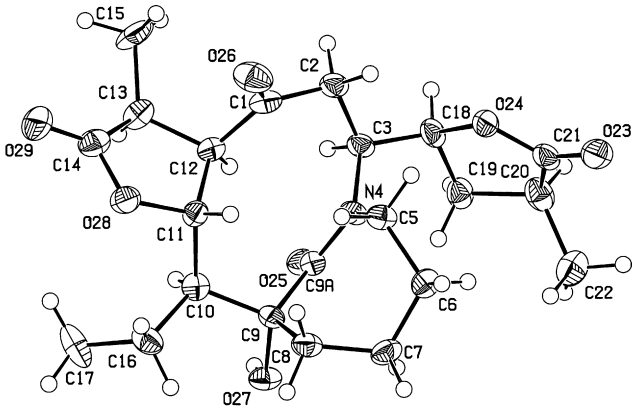

Figure 7. ORTEP drawing of the crystal structure of sessilifoliamide H (**4**).

422.2179) in the HRESIMS. The IR spectrum showed the presence of hydroxyl (3458 cm^{-1}), γ-lactone (1770 cm^{-1}), and carbonyl (1703 cm^{-1}) groups. Its ^{1}H NMR spectrum showed characteristic signals of one terminal (δ 1.00) and two secondary methyl groups (δ 1.11 and 1.31), and two oxymethine protons (δ 4.38 and 4.48), as observed in alkaloids **1**–**3** (Table 1). Its ^{13}C NMR spectrum showed twenty-two signals caused by three methyls, seven methylenes, seven methines, and five quaternary carbons, four of which were carbonyl carbons (Table 2). Analogous studies on its ^{1}H–^{1}H COSY and HMQC data as for **1** revealed that the molecule had the following three chain fragments, i.e., a six-carbon chain fragment (C-2–C-3–C-18–C-19–C-20–C-22) in which C-22 was a terminal methyl and C-18 was an oxymethine carbon, an all methylene four-carbon chain fragment (C-5–C-6–C-7–C-8), and a seven-carbon chain fragment (C-15–C-13–C-12–C-11–C-10–C-16–C-17) in which C-15 and C-17 were both terminal methyls and C-11 was an oxymethine carbon (Fig. 6). The locations of the five quaternary carbons and connectivity of those carbon chain fragments were determined by the HMBC experiments. The carbons resonated at δ 176.2 and 177.7 were assigned to lactone carbonyl carbons at positions 14 and 21, respectively, as in **1**, by the observed correlations from H-11, H-12, H-13, and H_3-15 to C-14, and from H-19b, H-20, and H_3-22 to C-21, respectively. The carbon resonated at δ 180.5 was assigned to a lactam carbonyl carbon at position 9a, as correlations were observed from H-3, H_2-5, and H-8b to this carbon, and the carbon resonated at δ 207.8 was assigned to a ketone carbon and placed at position 1, thus connecting to C-2 and C-12, because of the correlations to C-1 from H_2-2, H-11, H-12, and H-13 (Fig. 6). Analogously, the quaternary carbon at δ 81.8 was assigned to a hydroxyl-bearing carbon and placed at position 9 because of the correlations observed from H_2-7, H-8b, H-10, H-11, and H-16b to C-9 and from OH-9 to C-8, C-9, and C-9a. HMBC correlations from H-2b to C-12, H-3 to C-5, and H-10 to C-8 also supported the carbon chain fragments being connected as shown in Figure 6. These facts suggested that alkaloid **4** possessed a nine-membered lactam ring structure as observed in tuberostemonone (**5**). NOESY experiments were not useful for giving decisive information about the stereochemistry of **4** as it had a nine-membered ring structure. Its relative stereochemistry was unequivocally determined by an X-ray crystallographic analysis as shown in Figure 7.

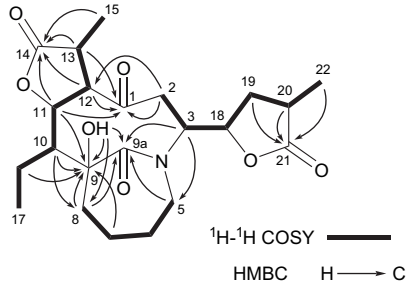

Figure 6. ^{1}H–^{1}H COSY and selected HMBC correlations for sessilifoliamide H (**4**).

Sessilifoliamides E and F (**1** and **2**) are the first examples of natural *Stemona* alkaloids bearing an 11-membered macrolactam ring. Biogenetically, these alkaloids are considered to be derived from those alkaloids having a tuberostemonine-type skeleton by oxidative cleavage of the C-9–C-9a bond. Sessilifoliamide G (**3**) is structurally closely related to **1** and **2**. Biogenetically, it may be produced from the C-1 epimer of **2** by forming a transannular hemiacetal linkage between the C-1 hydroxyl group and the C-9 ketone carbonyl group. Sessilifoliamide H (**4**) is the second *Stemona* alkaloid isolated and reported, having a rare tuberostemonone-type skeleton.[3,4]

3. Experimental

3.1. General

Optical rotations were determined on a JASCO P-1030 digital polarimeter, and IR spectra on a JASCO FT/IR 620 spectrophotometer. Mass spectra were obtained with a Micromass LCT spectrometer. NMR spectra were obtained on a Bruker DRX-500 spectrometer at 300 K. In ^{1}H NMR spectra, the chemical shifts (δ) are given in parts per million relative to the resonance of residual $CHCl_3$ at 7.26 ppm, and in ^{13}C NMR spectra, the chemical shifts are given in parts per million relative to the resonance at 77.03 ppm for $CDCl_3$. Preparative HPLC was carried out on a Shimadzu LC-6AD system equipped with a SPD-10A UV detector (220 nm) and a reversed-phase column, Wakosil-II 5C18HG

prep (5 μm, 20×250 mm), using a MeOH–H_2O or a MeCN–H_2O mixture as mobile phase, at a flow rate of 10 mL/min. Single-crystal X-ray analysis was carried out on a Mac Science DIP diffractometer with Mo Kα radiation (λ=0.71073 Å).

3.2. Plant material

S. sessilifolia (Miq.) Miq. was cultivated and harvested in Shandong Province, China in 2000. The botanical origin of the plant was identified by Prof. Z. W. Xie of the China Academy of Traditional Chinese Medicine.

3.3. Extraction and isolation

The air-dried roots (15 kg) were extracted with hot MeOH (3×35 L). The solvent was removed to give a crude MeOH extract (8 kg), which was, after acidifications with 3% aqueous tartaric acid (8 L), treated with EtOAc (3×8 L). The combined EtOAc layers were evaporated in vacuo to give a residue (mixture of neutral and acidic components, 300 g). The aqueous layer was then made to pH 9 with solid Na_2CO_3 and extracted with $CHCl_3$ (3×8 L). The combined $CHCl_3$ extracts were evaporated in vacuo to give a residue (basic fraction, 250 g).

The mixture of neutral and acidic components (300 g) was subjected to silica gel (1700 g) column chromatography eluting sequentially with hexane–EtOAc (3/1, 5 L), hexane–EtOAc (1/1, 5 L), EtOAc (5 L), EtOAc–MeOH (10/1, 8 L), and MeOH (8 L) to afford six fractions. The fourth fraction (43.6 g), a part of the EtOAc–MeOH (10/1) eluate, was subjected to aminopropyl-bonded silica gel (570 g) column chromatography eluting sequentially with hexane–EtOAc (1/0, 3/1, 1/1, 1/3, and 0/1, 4 L each), EtOAc–MeOH (10/1, 8 L), and MeOH (8 L) to give seven fractions (fractions 1–7). After removal of the solvent to dryness, fraction 4 (0.72 g) was subjected to HPLC using MeOH–H_2O (35/65, 65/35, then 100/0) to afford 13 fractions (fractions 4A–4M). Fractions 4D (7.5 mg), 4F (6.5 mg), and 4I (5.2 mg) were each subsequently purified by HPLC using MeCN–H_2O (22/78, 23/77, and 27/73, respectively) to give **5** (0.7 mg), **1** (3.6 mg), and **3** (2.5 mg), respectively.

Fraction 5 (0.33 g) was subjected to HPLC using MeOH–H_2O (40/60, then 100/0) to afford nine fractions. The second fraction (37.2 mg) was subsequently purified by HPLC using MeCN–H_2O (28/72) to give **4** (2.2 mg).

Fraction 6 (5.59 g) was subjected to silica gel (170 g) column chromatography eluting sequentially with $CHCl_3$–MeOH (30/1, 20/1, 10/1, 5/1, and 0/1) to afford 11 fractions. The second fraction (0.32 g), a part of the $CHCl_3$–MeOH (30/1) eluate, was then subjected to HPLC using MeOH–H_2O (45/55) to give eleven fractions, the second fraction (23.1 mg) of which was subsequently separated by HPLC using MeCN–H_2O (23/77) to afford **2** (4.4 mg).

3.4. Characteristics of each alkaloid

3.4.1. Sessilifoliamide E (1). Colorless prisms, mp 197–200 °C; $[\alpha]_D$ −18 (*c* 0.10, $CHCl_3$); IR ν_{max} (film) 2972, 2935, 2878, 1774, 1698, 1678 cm^{-1}; ^1H and ^{13}C NMR data, given in Tables 1 and 2; HRESIMS *m/z* 406.2228 ($[M+H]^+$, calcd for $C_{22}H_{32}NO_6$, 406.2230).

3.4.2. Sessilifoliamide F (2). Colorless prisms, mp 200–202 °C; $[\alpha]_D$ −42 (*c* 0.08, $CHCl_3$); IR ν_{max} (film) 3381, 3017, 2974, 2937, 2879, 1774, 1699, 1683 cm^{-1}; ^1H and ^{13}C NMR data, given in Tables 1 and 2; HRESIMS *m/z* 422.2179 ($[M+H]^+$, calcd for $C_{22}H_{32}NO_7$, 422.2179).

3.4.3. Sessilifoliamide G (3). Colorless prisms, mp 213–215 °C; $[\alpha]_D$ −85 (*c* 0.06, MeOH); IR ν_{max} (film) 3463, 2934, 2878, 1773, 1702 cm^{-1}; ^1H and ^{13}C NMR data, given in Tables 1 and 2; HRESIMS *m/z* 422.2186 ($[M+H]^+$, calcd for $C_{22}H_{32}NO_7$, 422.2179).

3.4.4. Sessilifoliamide H (4). Colorless prisms, mp 240–243 °C; $[\alpha]_D$ −42 (*c* 0.10, MeOH); IR ν_{max} 3458, 2931, 2876, 1770, 1703, 1620 cm^{-1}; ^1H and ^{13}C NMR data, given in Tables 1 and 2; HRESIMS *m/z* 422.2179 ($[M+H]^+$, calcd for $C_{22}H_{32}NO_7$, 422.2179).

3.5. X-ray crystallographic study

3.5.1. Sessilifoliamide E (1). $C_{22}H_{31}NO_6$, *M*=405.48, 0.38×0.25×0.20 mm, monoclinic, $P2_1$, *a*=9.7640(3) Å, *b*=10.2060(6) Å, *c*=10.5100(7) Å, β=96.686(3)°, *V*=1040.21(10) $Å^3$, *Z*=2, D_x=1.295 Mg m^{-3}, μ(Mo Kα)=0.094 mm^{-1}, 2305 measured reflections, 2305 unique reflections, 2118 observed reflections [$I>2\sigma(I)$], *R*1=0.0401, *wR*2=0.1039 (observed data), GOF=1.024; *R*1=0.0429, *wR*2=0.1039 (all data).

3.5.2. Sessilifoliamide F (2). $C_{22}H_{31}NO_7$, *M*=421.48, 0.43×0.43×0.20 mm, triclinic, *P*1, *a*=9.5090(16) Å, *b*=9.9970(8) Å, *c*=11.707(2) Å, α=97.505(8)°, β=103.350(7)°, γ=95.584(8)°, *V*=1064.1(3) $Å^3$, *Z*=2, D_x=1.315 Mg m^{-3}, μ(Mo Kα)=0.098 mm^{-1}, 4295 measured reflections, 4295 unique reflections, 2988 observed reflections [$I>2\sigma(I)$], *R*1=0.0375, *wR*2=0.0808 (observed data), GOF=0.896; *R*1=0.0527, *wR*2=0.0834 (all data).

3.5.3. Sessilifoliamide G (3). $C_{22}H_{31}NO_7$, *M*=421.48, 0.38×0.35×0.30 mm, orthorhombic, $P2_12_12_1$, *a*=9.7840(2) Å, *b*=10.2500(3) Å, *c*=21.3050(7) Å, *V*=2136.59(10) $Å^3$, *Z*=4, D_x=1.310 Mg m^{-3}, μ(Mo Kα)=0.097 mm^{-1}, 2638 measured reflections, 2638 unique reflections, 2319 observed reflections [$I>2\sigma(I)$], *R*1=0.0325, *wR*2=0.0883 (observed data), GOF=1.020; *R*1=0.0388, *wR*2=0.0895 (all data).

3.5.4. Sessilifoliamide H (4). $C_{22}H_{31}NO_7$, *M*=421.48, 0.48×0.20×0.20 mm, monoclinic, $P2_1$, *a*=9.6640(18) Å, *b*=11.6990(9) Å, *c*=9.5870(16) Å, β=99.372(7)°, *V*=1069.4(3) $Å^3$, *Z*=2, D_x=1.309 Mg m^{-3}, μ(Mo Kα)=0.097 mm^{-1}, 2271 measured reflections, 2271 unique reflections, 1315 observed reflections [$I>2\sigma(I)$], *R*1=0.0461, *wR*2=0.1010 (observed data), GOF=0.812; *R*1=0.0791, *wR*2=0.1082 (all data).

The structures were solved by direct methods using the maXus crystallographic software package,[5] and refined by full-matrix least-squares on F^2 using the program SHELXL-97.[6] The absolute structures could not be determined crystallographically.

CCDC 619827, 619828, 619829, and 619830 contain the supplementary crystallographic data for compounds **1**–**4**, respectively, studied in this paper. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/data_request/cif, by e-mailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

References and notes

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