*Chemistry of Natural Compounds, Vol. 46, No. 2, 2010*

# ANOTHER TWO NOVEL CERAMIDES FROM *Zephyranthes candida*

**Zhiping Wu,[1,2] Yu Chen,[1] Xu Feng,[1*] Bing Xia,[1] Ming Wang,[1] and Yunfa Dong[1]**

UDC 547.29+547.917

*From the the bulbs of* Zephyranthes candida *(Amaryllidaceae), another two novel ceramides have been isolated and identified. The structures of the two novel compounds were established as (2*S*,3*S*,4*R*,21*E*,2′*R*)-2-[*N*-(2′-hydroxynonadecanoyl)-*N*-(1″,2″-dihydroxyethyl)amino]-21-hexacosene-1,3,4-triol, named zephyranamide C (**1**), and 1,3,4,5,6-pentahydroxy-2-(2′-hydroxyhexacosanoyl-amino)-18-(*E*)-tetracosene, named zephyranamide D (**2**). Their structures and stereochemistries were elucidated by spectral techniques including $^1$H NMR, $^{13}$C NMR, as well as HSQC, HMBC, DEPT, and COSY.*

**Keywords**: *Zephyranthes candida*, Amaryllidaceae, ceramide, zephyranamide C, zephyranamide D.

*Zephyranthes candida* (Amaryllidaceae), a member of the genus *Zephyranthes*, is widely distributed in the temperate zone of Western Hemisphere and is also used as an ornamental and medicinal plant in China. Plants of the genus *Zephyranthes* comprise about 60 species, which were reported to be rich sources of Amaryllidaceae alkaloids [1–3]. Previous chemical investigations of *Z. candida* led to the isolation of a flavonoid glycoside, kaempferol-3-*O*-rhamnoglucoside [4], five alkaloids, lycorine, zephyranthine, tazettine, nerinine, and haemanthidine [5], and a cytostatic alkaloid, *trans*-dihydronarciclasine [6].

In previous papers, we reported the structure elucidation of two new ceramides, zephyranamide A and zephyranamide B [7], and two rarely reported ceramides, candidamide A and candidamide B from this plant [8]. Our continuing phytochemical investigation on the constituents of this plant has resulted in the isolation of another two novel ceramides named zephyranamide C (**1**) and zephyranamide D (**2**). In the present study, we describe the characterization of zephyranamide C (**1**) and zephyranamide D (**2**), which belong to the rarely occurring class of ceramides.

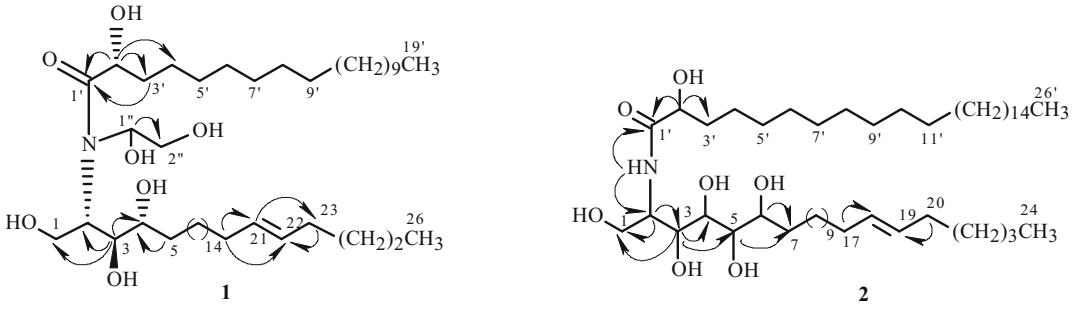
Compound **1** was obtained as a white amorphous powder, $[\alpha]_D^{25}$ –97.4° (*c* 0.073, pyridine). Elemental analysis agrees with the molecular formula $C_{47}H_{93}NO_7$. This molecular formula was confirmed by positive ESI-MS at *m/z* 818 $[M - H + 2H_2O]^+$, 733 $[M - H - CH_2OH - H_2O]^+$, and 806 $[M + Na]^+$. The IR spectrum showed absorption bands at 3345 and 2920, 2850 and 1635, 1530 and 1470, and 718 $cm^{-1}$ indicating the presence of hydroxyl, amide carbonyl functions, and olefinic carbons, respectively, and the $^1$H and $^{13}$C NMR spectra of **1** also indicated the presence of hydroxyl, amide groups, and two aliphatic long chains (Table 1). All this suggested that compound **1** has a ceramide nature [9]. The position and geometry of the double bond were confirmed by $^1$H–$^1$H COSY analysis, ESI-MS fragment analysis, and $^{13}$C NMR data of the carbon next to the double bond.

1) Jiangsu Center for Reasearch & Development of Medicinal Plants, Jiangsu Institute of Botany, Chinese Academy of Sciences, Nanjing, 210014, P. R. China, fax: +86 25 84347084, e-mail: fengxu@mail.cnbg.net; 2) College of Chemistry and Bioengineering, Suzhou University of Science and Technology, Suzhou, Jiangsu Province, 215011, P. R. China. Published in Khimiya Prirodnykh Soedinenii, No. 2, pp. 159–162, March–April, 2010. Original article submitted December 9, 2008.

0009-3130/10/4602-0187  

TABLE 1. $^{1}$H NMR (500 MHz), $^{13}$C NMR (125 MHz), and COSY Spectral Data of Compound **1** ($C_5D_5N$, δ, ppm, J/Hz)

| C atom | $\delta_H$ (HSQC) | $\delta_C$ (DEPT) | $^{1}$H–$^{1}$H COSY |
|---|---|---|---|
| 1a | 4.41 (dd, J = 11.0, 5.3) | 61.9 ($CH_2$) | H-2 |
| 1b | 4.46 (dd, J = 11.0, 5.3) | 61.9 | |
| 2 | 5.12 (m) | 53.1 (CH) | H-1, H-3, NH |
| 3 | 4.32 (dd, J = 6.2, 4.7) | 76.8 (CH) | H-2, H-4 |
| 4 | 4.52 (m) | 73.7 (CH) | H-3, H-5 |
| 5a, 5b | 1.97 (m), 2.05 (m) | 33.0 ($CH_2$) | H-4, H-6 |
| 6 | 1.63 (m) | 26.6 ($CH_2$) | H-5, H-7 |
| 7 | 1.92 (m) | 33.9 ($CH_2$) | H-6, H-8 |
| 8 | 1.25 (m) | 30.4 ($CH_2$) | H-7, H-9 |
| 9 | 1.25 (m) | 29.5 ($CH_2$) | H-8, H-10 |
| 10–18 | 1.25–1.38 (br.s) | 29.5–30.4 ($CH_2$) | |
| 19 | 1.63 (m) | 26.6 ($CH_2$) | H-18, H-20 |
| 20 | 1.92 (m) | 33.9 ($CH_2$) | H-19, H-21 |
| 21 | 5.55 (dt, J = 15.3, 9.2) | 130.8 (CH) | H-20, H-22 |
| 22 | 5.50 (dt, J = 15.3, 9.2) | 130.7 (CH) | H-21, H-23 |
| 23 | 2.20 (m) | 33.3 ($CH_2$) | H-22, H-24 |
| 24 | 1.25 (m) | 32.1 ($CH_2$) | |
| 25 | 1.25 (br.s) | 23.0 ($CH_2$) | |
| 26 | 0.86 (t, J = 6.8) | 14.3 ($CH_3$) | |
| 1′ | – | 174.0 (C) | |
| 2′ | 4.73 (dd, J = 7.8, 4.1) | 76.3 (CH) | H-3′ |
| 3′a, 3′b | 2.28 (m), 1.92 (m) | 34.3 ($CH_2$) | H-2′, H-4′ |
| 4′ | 2.04 (m) | 26.7 ($CH_2$) | H-3′, H-(5′–16′) |
| 5′–16′ | 1.24–1.34 (br.s) | 29.5–30.4 ($CH_2$) | |
| 17′ | 1.24 (m) | 32.1 ($CH_2$) | |
| 18′ | 1.24 (m) | 23.0 ($CH_2$) | |
| 19′ | 0.86 (t, J = 6.7) | 14.3 ($CH_3$) | |
| 1″ | 4.28 (m) | 73.0 (CH) | H-2″ |
| 2″a | 4.28 (dd, J = 11.2, 5.3) | 72.9 ($CH_2$) | |
| 2″b | 4.27 (dd, J = 11.2, 5.0) | 72.9 | |




**1** **2**

Fig. 1. Structures and key HMBC correlations (H→C) of **1** and **2**.

The $^{13}$C NMR (Table 1) and DEPT spectral data of compound **1** showed one quaternary carbon at $\delta_C$ 174.0 (C-1′), six carbon atom signals bearing hydroxyl groups ($\delta_C$ 61.9, 76.8, 73.7, 76.3, 73.0 and 72.9) and one double bond ($\delta_C$ 130.8, 130.7), as well as aliphatic methylenes between $\delta_C$ 29.5–30.4, and two terminal methyls at $\delta_C$ 14.3 (C-26 and C-19′). The signal at $\delta_H$ 5.12 (H-2) showed a cross-peak with the signals at $\delta_H$ 4.41, 4.46 (H-1) and 4.32 (H-3) in the $^{1}$H–$^{1}$H COSY spectrum of **1**, and the latter correlated with the signal at $\delta_H$ 4.52 (H-4). The correlations were also observed in $^{1}$H–$^{1}$H COSY spectrum between $\delta_H$ 4.28, 4.27 (H-2″) and 4.28 (H-1″), $\delta_H$ 2.28, 1.92 (H-3′) and 4.73 (H-2′), $\delta_H$ 2.05, 1.97 (H-5) and 4.52 (H-4), $\delta_H$ 1.63 (H-6) and 2.05, 1.97 (H-5), and $\delta_H$ 1.92 (H-7) and 1.63 (H-6). The structure of **1** was further illustrated by combined analysis of HSQC and HMBC spectra (Fig. 1). In the $^{1}$H NMR spectrum of compound **1**, however, there were no signals between the $\delta_H$ 8–9, which indicated a tertiary amide may be present [10].

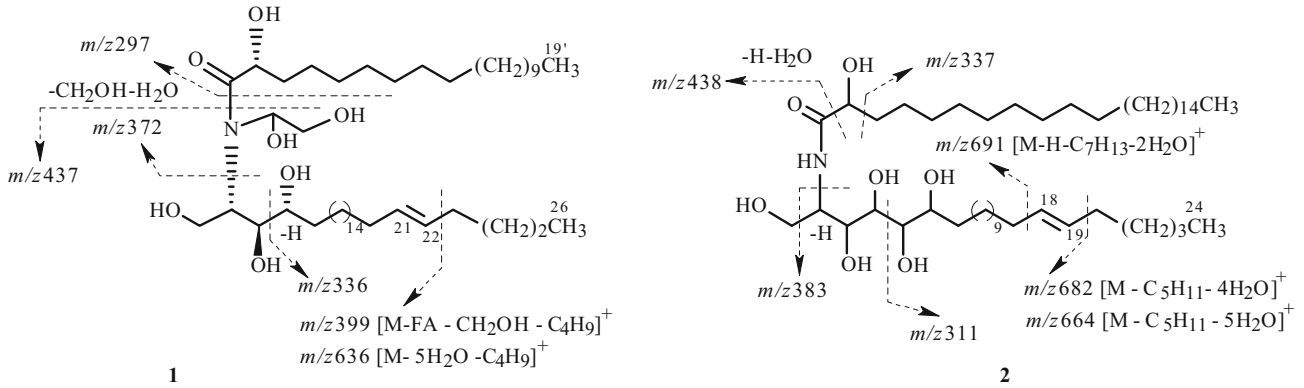
TABLE 2. $^{1}$H NMR (500 MHz), $^{13}$C NMR (125 MHz), and COSY Spectral Data of Compound **2** ($C_5D_5N$, δ, ppm, J/Hz)

| C atom | $\delta_H$ (HSQC) | $\delta_C$ (DEPT) | $^{1}$H–$^{1}$H COSY |
|---|---|---|---|
| 1a | 4.42 (dd, J = 10.8, 4.4) | 62.1 ($CH_2$) | H-2 |
| 1b | 4.50 (dd, J = 10.6, 4.9) | 62.1 | |
| 2 | 5.09 (m) | 53.0 (CH) | H-1, H-3, NH |
| 3 | 4.33 (dd, J = 5.2, 3.7) | 76.9 (CH) | H-2, H-4 |
| 4 | 4.25 (m) | 76.8 (CH) | H-3, H-5 |
| 5 | 4.27 (m) | 73.0 (CH) | H-4, H-6 |
| 6 | 4.27 (m) | 72.9 (CH) | H-5, H-7 |
| 7a, 7b | 1.92 (m), 2.25 (m) | 34.2 ($CH_2$) | H-6, H-8 |
| 8 | 1.92 (m) | 29.5 ($CH_2$) | H-7, H-9 |
| 9–16 | 1.25–1.38 (br.s) | 29.5–30.3 ($CH_2$) | |
| 17 | 2.15 (m) | 33.8 ($CH_2$) | H-16, H-18 |
| 18 | 5.56 (dt, J = 15.1, 6.1) | 130.8 (CH) | H-17, H-19 |
| 19 | 5.50 (dt, J = 15.3, 5.9) | 130.7 (CH) | H-18, H-20 |
| 20 | 1.98 (m) | 33.3 ($CH_2$) | H-19, H-21 |
| 21 | 1.91 (m) | 32.9 ($CH_2$) | H-20, H-22 |
| 22 | 1.25 (m) | 32.1 ($CH_2$) | |
| 23 | 1.25 (br.s) | 22.9 ($CH_2$) | |
| 24 | 0.86 (t, J = 6.5) | 14.2 ($CH_3$) | |
| NH | 8.55 (d, J = 8.9) | – | H-2 |
| 1′ | – | 175.2 (C) | |
| 2′ | 4.60 (m) | 72.5 (CH) | H-3′ |
| 3′a, 3′b | 2.21 (m), 2.02 (m) | 35.7 ($CH_2$) | H-2′, H-4′ |
| 4′ | 1.78 (m) | 26.6 ($CH_2$) | H-3′, H-5′ |
| 5′ | 1.71 (m) | 25.8 ($CH_2$) | H-4′, H-(6′–24′) |
| 6′–24′ | 1.25–1.38 (br.s) | 29.5–30.3 ($CH_2$) | |
| 25′ | 1.25 (br.s) | 22.9 ($CH_2$) | |
| 26′ | 0.86 (t, J = 6.5) | 14.2 ($CH_3$) | H-(6′–24′) |

Furthermore, the lengths of the long-chain base (LCB) and the fatty acid (FA), together with the presence of 2-amino and 1,2′,3,4-tetrahydroxyl groups, as well as the C-21 and C-22 double bonds, were established by ESI-MS fragment pattern analysis (Fig. 2). The investigation of the ESI-MS showed two important fragmentions at *m/z* 636 $[M - 5H_2O - C_4H_9]^+$ and 399 $[M - FA - CH_2OH - C_4H_9]^+$, suggesting the C=C bond to be located at C-21 and C-22. Additionally, the fragments at *m/z* 437 $[M - FA - CH_2OH - H_2O]^+$ and 372 $[M - LCB]^+$ suggested compound **1** was a tertiary amide. Fragments at *m/z* 636, 437, 399, 372, 336, and 297 were the six significant ions of compound **1**, predicting 26 and 19 carbons in the LCB and FA, respectively, and that the double bond must be located in the LCB (Fig. 2). Moreover, the C-21, C-22 double bond was assigned on the basis of the typical fragment ions at *m/z* 705 $[M - 2H_2O - C_3H_6]^+$ and 697 $[M - H_2O - C_3H_6 - C_2H_2]^+$, which was formed by elimination of propene through a McLafferty rearrangement [11]. The 21,22 alkenyl bond should be *trans* on the basis of the coupling constant of H-21 and H-22 (15.3 Hz) and the chemical shifts of C-20 and C-23 ($\delta_C$ 33.9 and 33.3) [12].

Regarding the stereochemistry, the formulated relative configuration of compound **1** was based on the carbon chemical shifts at $\delta_C$ 61.9 (C-1), 53.1 (C-2), 76.8 (C-3), 73.7 (C-4), 174.0 (C-1′), and 76.3 (C-2′), which happened to be fairly close to those previously reported for (2*S*, 3*S*, 4*R*, 2′*R*) sphingosine moieties [13, 14]. Including biogenetic considerations, the structure of compound **1** was therefore concluded to be (2*S*,3*S*,4*R*,21*E*,2′*R*)-2-[*N*-(2′-hydroxynonadecanoyl)-*N*-(1′′,2′′-dihydroxyethyl)amino]-21-hexacosene-1,3,4-triol, and we have named compound **1** zephyranamide C.

Compound **2** was also obtained as a white amorphous powder, $[\alpha]_D^{25}$ 0° (*c* 0.09, pyridine). Elemental analysis agrees with the molecular $C_{50}H_{99}NO_7$, mp 146–147°C. This molecular formula was again analyzed by positive ESI-MS at *m/z* 718 $[M + H - 6H_2O]^+$ and 848 $[M + Na]^+$. The IR absorption bands data at 3330, 2920, 1620, 1465, and 720 $cm^{-1}$ indicated the presence of hydroxyls, amide groups, and long aliphatic chains, respectively.




**1** **2**

Fig. 2. ESI-MS fragment analysis of compounds **1** and **2**.

The structure of **2** was deduced by combined analysis of DEPT, HSQC, and HMBC spectra. The NMR spectra of **2** (Table 2) showed an amide NH signal (1H, $\delta_H$ 8.55, d, J = 8.9 Hz), a carbonyl ($\delta_C$ 175.2, C-1′) and a group of proton signals ($\delta_H$ 1.25–1.38, br.s), and two terminal methyls (6H, $\delta_H$ 0.86, t, J = 6.5), which indicated that **2** might belong to the ceramide class; in addition, another signal at low field was observed at $\delta_H$ 5.09 (1H, m) for a methine proton vicinal to the nitrogen atom of an amide linkage, and the data indicated a phytosphingolipid structure [15]. In the $^1$H NMR spectrum of **2** (Table 2), signals of a *trans*-olefinic bond at $\delta_H$ 5.56 (1H, dt, J = 15.1, 6.1 Hz) and 5.50 (1H, dt, J = 15.3, 5.9 Hz) and seven characteristic signals of geminal carbon protons to hydroxyl groups were also observed at $\delta_H$ 4.60 (1H, m), 4.50 (1H, dd, J = 10.6, 4.9 Hz), 4.42 (1H, dd, J = 10.8, 4.4 Hz), 4.33 (1H, dd, J = 5.2, 3.7 Hz), 4.25 (1H, m), 4.27 (1H, m), and 4.27 (1H, m). The $^{13}$C NMR spectral data of **2** (Table 2) showed one quaternary carbon at $\delta_C$ 175.2 (CONH), two olefinic methine carbon atoms at $\delta_C$ 130.8 and 130.7 (C=C), as well as six methines at $\delta_C$ 53.0 (CHNH), 76.9 (CHOH), 76.8 (CHOH), 73.0 (CHOH), 72.9 (CHOH), and 72.5 (CHOH) and one methylene at $\delta_C$ 62.1 ($CH_2$OH). The C-18, C-19 alkenyl bond was found to be *trans*, as evidenced by the *E*-geometry of the double bond in the unsaturated LCB determined according to the $^{13}$C NMR chemical shifts of C-17 ($\delta_C$ 33.8) and C-20 ($\delta_C$ 33.3) of the methylene carbon next to the olefinic carbon, which usually appears at $\delta \approx 27$ in the (*Z*) isomers and at $\delta \approx 33$ in the (*E*) isomers [16], and the coupling constant of H-18 and H-19 was 15.1 Hz. All these spectral data indicated that compound **2** possessed two aliphatic long chains containing one double bond and six hydroxyl groups, and suggested that it was a phytosphingosine type ceramide.

The structural elucidation and assignments of complete proton and carbon signals were achieved by 2D NMR (HSQC, HMBC, and $^1$H–$^1$H COSY) techniques and chemical methods. The $^1$H–$^1$H COSY spectrum of **2** showed a pair of double doublets of oxygenated methylene at $\delta_H$ 4.50 (1H, dd, J = 10.6, 4.9 Hz) and 4.42 (1H,dd, J = 10.8, 4.4 Hz) coupled to the nitrogen-bearing methine signal at $\delta_H$ 5.09 (1H, m), which was coupled further to the signal at $\delta_H$ 4.33 (1H, dd, J = 5.2, 3.7). Olefinic protons at $\delta_H$ 5.56 (1H, dt, J = 15.1, 6.1 Hz) and 5.50 (1H, dt, J = 15.3, 5.9 Hz) showed coupling with methylene protons resonating at $\delta_H$ 2.15 (1H, m) and 1.98 (1H, m). In the HMBC spectrum of **2**, the proton at $\delta_H$ 5.09 (H-2) showed correlation with oxygenated methylene ($\delta_C$ 62.1) and oxygenated methines ($\delta_C$ 76.9); the signal at $\delta_H$ 4.60 (H-2′) correlated with the quaternary carbon and methylene carbon resonating at $\delta_C$ 175.2 (C-1′) and 35.7 (C-3′), respectively.

According to the analysis of positively charged ESI-MS of **2**, the number of carbons in the LCB and the FA were determined to be 24 and 26, respectively. The positively charged ESI-MS of **2** showed significant fragments at *m/z* 682 $[M - C_5H_{11} - 4H_2O]^+$, 664 $[M - C_5H_{11} - 5H_2O]^+$, and 691 $[M - 1 - C_7H_{13} - 2H_2O]^+$, suggesting the double bond to be located at C-18 and C-19 (Fig. 2). This was confirmed on the basis of the typical fragment ions at *m/z* 746 $[M - H - C_3H_6 - 2H_2O]^+$ and 772 $[M - H - C_3H_6 - C_2H_2 - 2H_2O]^+$ in the positively charged ESI-MS, which were formed by elimination of propene through a McLafferty rearrangement [17, 18].

Based on the above information and compared with the literature [19], the structure of compound **2** was assigned as 1,3,4,5,6-pentahydroxy-2-(2′-hydroxyhexacosanoyl amino)-18-(*E*)-tetracosene, and designated zephyranamide D.

## EXPERIMENTAL

**General Methods**. $^1$H NMR and $^{13}$C NMR, COSY, HSQC, and HMBC spectra: Bruker spectrometers operating at 500 MHz; ESI-MS: Agilent 1100 LC/MSD SL; IR spectra: IMPACT 400 (KBr); JASCO P-1020 optical rotation apparatus; Melting points: SGW X-4 micromelting point apparatus.

**Plant Material**. The bulbs of *Zephyranthes candida* were collected from Nanjing Agricultural University, Jiangsu province, People′s Republic of China, in August, 2006. A voucher specimen (No. 61018/JSH) documenting the collection was authenticated at the Herbarium by Prof. Yun-fa Dong, phytochemist, Institute of Botany (Nanjing Botanical Garden Mem. Sun Yat-Sen), Jiangsu province and the Chinese Academy of Sciences, where it was deposited.

**Extraction and Purification**. The air-dried and powdered bulbs (ground parts cut off) of *Zephyranthes candida* (3.5 kg) were extracted three times with 95% EtOH (10 L × 3) at room temperature for one week. The aqueous ethanol phase was evaporated under reduced pressure and the initial residue dried in *vacuo*. The resulting residue was dissolved in $H_2O$ and re-extracted with petroleum ether, $CHCl_3$ (3×), and then $CHCl_3$–MeOH (3:2, v/v). The extract portions of $CHCl_3$ and $CHCl_3$–MeOH (3:2) were combined and evaporated, and the crude residue (105 g) was subjected to silica gel column chromatography eluting with $CHCl_3$–MeOH (99:1, 97:3, 95:5, 9:1, 8:2, 7:3, 6:4, and 1:1, v/v, 800 mL each) to yield six fractions (Fr. 1 to 6). Fraction 3 (22.0 g) was then chromatographed on silica gel eluting with petroleum ether–EtOAc (100:0, 9:1, 8:2, 7:3, v/v, 250 mL each) to furnish four subfractions (Fr. 3-1 to Fr. 3-4). Fraction 3-2 (7.8 g) was further separated by CC using $CHCl_3$–MeOH with gradient polarity as eluent (8:1, 7:1, 6:1, v/v, 250 mL each) and then purified by Sephadex LH-20 eluted with $CHCl_3$–MeOH (1:1, v/v, 180 mL each) to afford compound **1** (30.0 mg) and **2** (25.0 mg).

**Zephyranamide C (1)**, white amorphous powder, mp 150–151°C ($CHCl_3$–MeOH), $[\alpha]_D^{25}$ –7.4° (*c* 0.073, pyridine). IR (KBr, $\nu_{max}$, $cm^{-1}$): 3345, 2920, 2850, 1635, 1560, 1530, 1470, 1070, 960 and 718; ESI-MS: *m/z* 806 $[M + Na]^+$.

**Zephyranamide D (2)**, white amorphous powder, mp 146–147°C ($CHCl_3$–MeOH), $[\alpha]_D^{25}$ 0° (*c* 0.09, pyridine). IR (KBr, $\nu_{max}$, $cm^{-1}$): 3330, 3220, 2920, 2850, 1620, 1540, 1465, 1370, 1270, 1070, 1065, 1020, 960, 870 and 720; ESI-MS: *m/z* 848 $[M + Na]^+$.

For $^1H$ NMR, $^{13}C$ NMR, HSQC, HMBC, and COSY spectral data of **1** and **2**, see Tables 1 and 2 and Fig. 1.

## REFERENCES


1. K. Kojima, M. Mutsuga, M. Inoue, and Y. Ogihara, *Phytochemistry*, **48**, 1199 (1998).
2. M. Mutsuga, K. Kojima, M. Nose, M. Inoue, and Y. Ogihara, *Nat. Med.*, **55**, 201 (2001).
3. M. R. Herrera, A. K. Machocho, R. Brun, F. Viladomat, C. Codina, and J. Bastida, *Planta Med*., **67**, 191 (2001).
4. N. Mitsuru, H. Tokunaru, T. Masao, M. Mitsuo, and H. Shuichi, *J. Biosciences*, **33C**, 587 (1978).
5. S. Ozeki, *Yakugaku Zasshi*, **84**, 1194 (1964).
6. G. R. Pettit, G. M. Cragg, S. B. Singh, J. A. Duke, and D. L. Doubek, *J. Nat. Prod*., **53**, 176 (1990).
7. Zh. P. Wu, Y. Chen, X. Feng, B. Xia, M. Wang, and Y. F. Dong, *Chem. Nat. Comp*., **45**, 829 (2009).
8. Zh. P. Wu, Y. Chen, B. Xia, M. Wang, Y. F. Dong, and X. Feng, *Lipids*, **44**, 63 (2008).
9. B. N. Su, R. Misico, E. J. Park, B. D. Santarsiero, A. D. Mesecar, H. H. S. Fong, J. M. Pezzuto, and A. D. Kinghorn, *Tetrahedron*, **58**, 3453 (2002).
10. X. S. Chen, D. H. Chen, J. Y. Si, and Z. T. Guang, *J. Asian Nat. Prod. Res*., **3**, 277 (2001).
11. M. A. Ouyang, R. Liu, and Y. H. Kuo, *J. Asian Nat. Prod. Res*., **7**, 761 (2005).
12. H. Shibuya, K. Kawashima, M. Sakagami, H. Kawanishi, M. Shimomura, K. Ohashi, and I. Kitagawa, *Chem. Pharm. Bull*., **38**, 2933 (1990).
13. A. Liu, Z. M. Zou, L. Z. Xu, and S. L. Yang, *J. Asian Nat. Prod. Res*., **7**, 861 (2005).
14. M. H. Oueslati, Z. Mighri, H. B. Jannet, and P. M. Abreu, *Lipids*, **40**, 1075 (2005).
15. T. Natori, M. Morita, K. Akimoto, and Y. Koezuka, *Tetrahedron*, **50**, 2771 (1994).
16. S. Sugiyama, M. Honda, R. Higuchi, and T. Komori, *Liebigs Ann. Chem*., 349 (1991).
17. X. Li, D. D. Sun, J. W. Chen, L. W. He, H. Q. Zhang, and H. Q. Xu, *Fitoterapia*, **78**, 490 (2007).
18. C. C. F. Simo, S. F. Kouam, H. M. P. Poumale, I. K. Simo, B. T. Ngadjui, I. R. Green, and K. Krohn, *Biochem. Syst. Ecol*., **36**, 238 (2008).
19. N. Kumar, B. Singh, A. P. Gupta, and V. K. Kaul, *Tetrahedron*, **62**, 4317 (2006).