

## Vibsane-type Diterpenes from Taiwanese *Viburnum odoratissimum*

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Two new eleven-membered and three new seven-membered vibsane-type diterpenes, named vibsantin L (1) and 14-hydroxyvibsantin F (2), and vibsantin I (3), 14*R*\*,15-epoxyvibsantin C (4) and 14*S*\*,15-epoxyvibsantin C (5) respectively, have been isolated from *Viburnum odoratissimum* collected in Taiwan. Their structures have been elucidated by spectroscopic analyses and comparison of Nuclear magnetic resonance method (NMR) data with those of the previously reported vibsane-type diterpenes. High performance liquid chromatography (HPLC) analyses indicated that the methanol extract of *V. odoratissimum* collected in Taiwan contained no neovibsantines, characteristic chemicals occurring in *V. awabuki*.

**Key words** Vibsane-type diterpenes; *Viburnum odoratissimum*; Taiwan; Caprifoliaceae

A number of *Viburnum* species are known in the world, and among them fifteen species grow in Japan.<sup>1)</sup> Some *Viburnum* species have elaborated iridoid glycosides, triterpenes, labdane-type diterpenes, and some aromatic compounds.<sup>2–10)</sup> The occurrence of vibsane-type diterpenes, however, are limited to *V. awabuki* and has not been so far isolated from other *Viburnum* species.<sup>11–17)</sup> These diterpenes can be divided into three subtypes such as 11-membered ring, 7-membered ring, and rearranged types. We have examined chemical components in the leaves of *Viburnum odoratissimum* collected in Taiwan to find out some of vibsane-type diterpenes. In a preceding paper,<sup>18)</sup> we already reported the isolation of seven-membered vibsane-type diterpenes, aldovibsantins 6–8, which featured the absence of a  $\beta,\beta$ -dimethylacrylate group at the C-8 position, from the methanol extract of *V. odoratissimum*. Our further chemical studies on this plant have resulted in the isolation of two new eleven-membered and three new seven-membered vibsane-type diterpenes, named vibsantin L (1) and 14-hydroxyvibsantin F (2), and vibsantin I (3), 14*R*\*,15-epoxyvibsantin C (4) and 14*S*\*,15-epoxyvibsantin C (5), respectively. Herein, we report the structures of these new compounds and the chemical difference between *Viburnum* species on the basis of HPLC analyses.

Compound 1 has the molecular formula, C<sub>25</sub>H<sub>36</sub>O<sub>6</sub>, established by high-resolution (HR) fast atom bombardment mass spectrometry (FABMS). Its infrared spectroscopy (IR) spectrum showed the presence of a hydroxy group (3356 cm<sup>-1</sup>) and two carbonyl groups (1700, 1719 cm<sup>-1</sup>). The <sup>1</sup>H-NMR data of 1 showed the signals due to six tertiary methyl groups ( $\delta_{\text{H}}$  1.05, 1.46, 1.57, 1.66, 1.91, 2.15), five olefinic protons [ $\delta_{\text{H}}$  5.05 (ddq,  $J=7.1, 7.1, 0.8$  Hz), 5.34 (dd,  $J=15.9, 9.6$  Hz), 5.66 (d,  $J=15.9$  Hz), 5.71 (qq,  $J=1.4, 1.4$  Hz), 5.84 (dd,  $J=11.0, 6.6$  Hz)], three oxymethines [ $\delta_{\text{H}}$  3.33 (d,  $J=8.7$  Hz), 4.21 (d,  $J=8.7$  Hz), 5.11 (d,  $J=9.6$  Hz)], and an oxymethylene [ $\delta_{\text{H}}$  4.21 (d,  $J=13.2$  Hz), 4.67 (d,  $J=13.2$  Hz)]. Extensive analysis of <sup>1</sup>H–<sup>1</sup>H correlated spectroscopy (COSY) and heteronuclear multiple quantum coherence (HMQC) gave the four partial structures A–F as depicted by bold lines in Fig. 1 and three quaternary carbons at  $\delta_{\text{C}}$  48.0, 62.3 and 201.6. The double bond involved in the B unit should take *E*-geometry due to a large vicinal coupling constant ( $J=15.9$  Hz). In order to determine the connectivities between these six partial structures and three quaternary carbons, heteronuclear

multiple bond correlation (HMBC) experiments were carried out. The HMBC correlation of H-8 to C-1' linked the  $\beta,\beta$ -acrylate ester group A to C-8 in the partial structure D. The correlation of H-18 to C-3 supported the linkage between C-18 and C-3, thereby assembling the units D and E. The C-4 carbonyl carbon resonated at  $\delta_{\text{C}}$  201.6 showed cross peaks to the H-2, H-18 and H-5 signals, thus making two bonds not only between C-4 and C-3 in the unit E, but also between C-4 and C-5 in the unit C. The quaternary carbon C-11 showed cross peaks to H-1, H<sub>3</sub>-20 and H-12, respectively, and also H-10 correlated to C-11, resulting in the formation of the quaternary carbon C-11 with C-1, C-12, C-10 and C-20. Additionally, the HMBC correlations of H-6, H-8 and H<sub>3</sub>-19 to C-7 ( $\delta_{\text{C}}$  62.3) showed that C-7 connected to both C-6 of the unit C and C-8 of the unit B. These HMBC correlations led to the construction of an eleven membered ring. Taking into consideration eight degrees of unsaturation and high-field <sup>13</sup>C-NMR chemical shifts ( $\delta_{\text{C}}$  63.4 and 62.3) of C-6 and C-7 in comparison with those of normal oxygen-bearing carbons, an epoxide ring must be formed at the C-6 and C-7 positions. Thus, the above spectral data gave the planar structure 1 as shown in Fig. 1.

The relative stereochemistry of 1 was elucidated by nuclear Overhauser exchange spectroscopy (NOESY) as shown in Fig. 2. Namely, the H-9 and H-6 signals showed cross peaks to the H<sub>3</sub>-19 and H<sub>3</sub>-20, and H<sub>3</sub>-19 resonances, respectively, indicating that both the C-19 and C-20 methyl groups took the same  $\beta$ -configuration. The observation of cross peaks from H-5 to H-6, H-9 and H-18 as well as from H-10 to H-12 and H-8, disclosed that the  $\beta,\beta$ -dimethylacryl group on the C-8 position and the C-19 methyl group aligned with the same spatial arrangement, and both the hydroxy group on the C-5 position and the epoxide ring took the same  $\alpha$  orientation. Additionally, the *Z* geometry of  $\Delta^{2,3}$  double bond is evident of NOE between H-18 and H-2. On the basis of the above NOE analyses, the structure of vibsantin L was elucidated as 1.

Compound 2 had the molecular formula C<sub>20</sub>H<sub>32</sub>O<sub>3</sub> as established by HR-FABMS. The spectral data of 2 were very similar to those of an 11-membered vibsane-type diterpene, vibsantin F (2a),<sup>11)</sup> except for the C-12–C-17 side chain. In fact, the <sup>1</sup>H-NMR spectrum of 2 showed the signals were due to an exomethylene group [ $\delta_{\text{H}}$  4.84 (dq,  $J=3.2, 1.6$  Hz), 4.93

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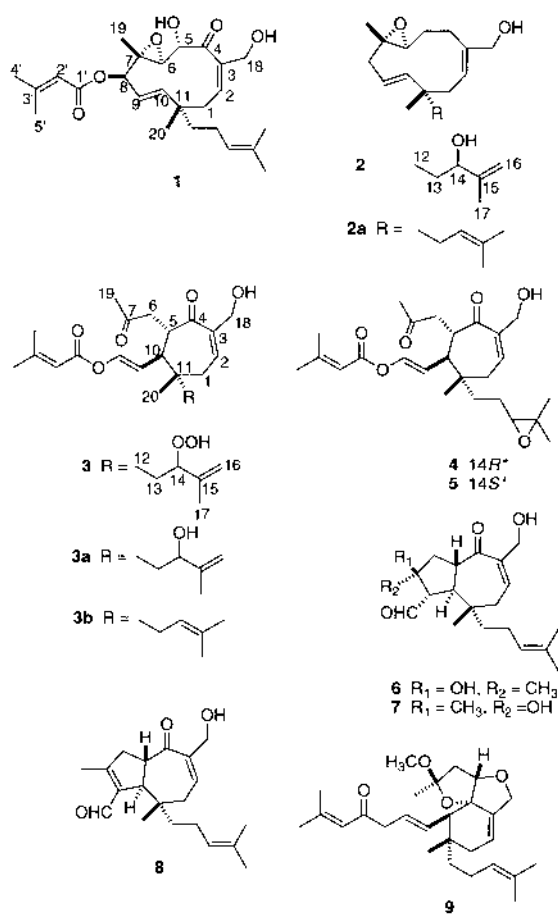
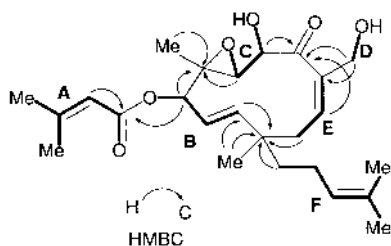
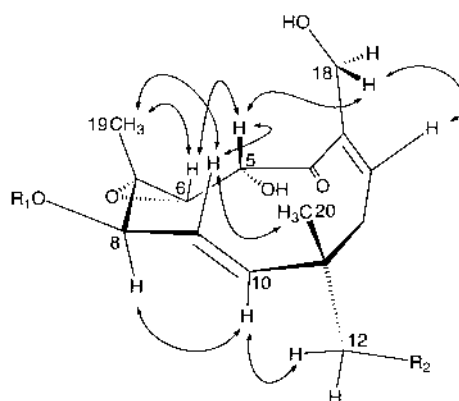


Chart 1

Fig. 1. HMBC Correlations of **1**

(dq,  $J=1.6, 1.1$  Hz)] and oxymethine proton [ $\delta_{\text{H}}$  4.02 (brdd,  $J=12.4, 4.1$  Hz)] instead of the isopropenyl group existing in **2a**. In comparison of  $^{13}\text{C}$ -NMR data between **2** and **2a** (Table 1) the C-12—C-17 side chain of **2** was verified to consist of the  $\Delta^{15,16}$  double bond and the C-14 methine having a hydroxy group. Additionally, the relative stereochemistry of **2** was assigned as the same as that of vibsanin F (**2a**) by the NOESY experiment. The absolute configuration on C-14 has not been determined. Thus, the structure of **2** was represented as 14-hydroxyvibsanin F.

The spectral data of **3** were found to be very similar to those of a seven membered vibsanin-type diterpene, vibsanin G (**3a**).<sup>13</sup> The molecular formula of  $\text{C}_{25}\text{H}_{36}\text{O}_7$  for **3** suggested the presence of one more oxygen atom than **3a**, implying the presence of a hydroperoxy group on the C-14 position. In fact, the  $^{13}\text{C}$ -NMR data of **3** were very similar to

Fig. 2. Conformation of **1** Based on NOE Correlations

those of **3a**, except the low-field appearance of the C-14 carbon signal appeared at  $\delta_{\text{C}}$  87.8 (Table 2). This supported the location of the hydroperoxide group on the C-14 position. The relative stereochemistry of **3** was verified to be the same as that of **3a** by the NOESY experiment.

Compounds **4** and **5** had the same molecular formula  $\text{C}_{25}\text{H}_{36}\text{O}_6$  established by HR-FABMS. Both of the  $^1\text{H}$ -NMR data were not only very similar to each other, but also closely related to those of vibsanin C (**3b**)<sup>14</sup> except for the signals due to H-12—H-17. In fact, the  $^{13}\text{C}$ -NMR data at  $\delta_{\text{C}}$  63.7 and 57.1 for **4** and  $\delta_{\text{C}}$  63.7 and 57.5 for **5** were assignable to C-14 and C-15, indicating the presence of an epoxide ring on the C-14 and C-15 positions in **4** and **5**. These spectral data suggested that **4** and **5** were 14,15-epoxyvibsanin C. However, the absolute stereochemistry regarding C-14 failed to be clarified. Therefore, we tentatively assigned **4** and **5** as 14*R*\*,15-epoxyvibsanin C and 14*S*\*,15-epoxyvibsanin C, respectively.

*Viburnum odoratissimum* is taxonomically placed as the plant identical to *V. awabuki*.<sup>19</sup> Vibsanin-type diterpenes have not been so far found in the terrestrial plant, except for *V. awabuki*. In this study, we isolated a number of vibsanin-type diterpenes from Taiwanese *V. odoratissimum*. As it was thought to be of interest to chemically compare the methanol extract of *V. awabuki* in Tokushima and *V. odoratissimum* in Taiwan, the analyses of them were performed by HPLC. We found that the two plants differed in the subclass of vibsanin-type diterpene as follows; typical 11-membered and 7-membered vibsanin-type diterpenes such as vibsanins A, B and F<sup>11</sup> are present in the *Viburnum* plants in both Tokushima and Taiwan, whereas no neovibsanin-type diterpenes such as **9** has been so far found in *V. odoratissimum*. But aldovibsanins, **6**—**8** which seem to be chemical components characteristic to *V. odoratissimum*, have not been detected from *V. awabuki*.

#### Experimental

Optical rotations were measured with a Jasco DIP-1000 digital polarimeter. UV spectra were recorded on a Shimadzu UV-300 or Hitachi-U-3000 spectrophotometer. IR spectra were recorded on a Jasco FT-IR 5300 or FT-IR 410 infrared spectrophotometer. 1D- and 2D-NMR spectra were recorded on a Varian Unity 600. MS were recorded on a JEOL AX-500 instrument. Silica gel (Merck, 70—230, 230—400 mesh, Wacogel C-300) and octadecylsilica gel (Cosmosil 75C<sub>18</sub>-OPN) were used for column chromatography. Sephadex LH-20 was used for gel filtration chromatography. Precoated Silica gel 60F<sub>254</sub> and RP-8 F<sub>254</sub> plates were used for analytical or preparative thin-layer chromatography, and spots were visualized by UV (254 nm) light and

Table 1.  $^{13}\text{C}$ - (150 MHz) and  $^1\text{H}$ - (600 MHz) NMR Data of **1**<sup>a</sup>, **2**<sup>b</sup>, and **2a**<sup>b</sup>

No.	<b>1</b>		<b>2</b>		<b>2a</b>
	$^{13}\text{C}$	$^1\text{H}$	$^{13}\text{C}$	$^1\text{H}$	$^{13}\text{C}$
1	40.5	2.00 (dd, 12.6, 6.6)	42.1	1.93 (ddd, 13.2, 11.0, 6.6)	42.6
2	132.3	2.55 (dd, 12.6, 1.0)		2.22 (ddd, 13.2, 11.8, 3.0)	
3	145.1	5.84 (dd, 11.0, 6.6)	112.9	5.52 (dd, 11.8, 11.0)	119.8
4	201.6		141.7		142.4
			23.6	2.05 (dd, 12.4, 7.8)	24.1
5	72.7	4.21 (d, 8.7)	26.9	2.33 (dd, 12.4, 11.8)	
				1.08 (m)	27.5
6	63.4	3.33 (d, 8.7)	63.4	2.27 (br dd, 11.0, 10.2)	
7	62.3		60.5	2.72 (d, 11.0)	63.1
8	76.8	5.11 (d, 9.6)	39.0	2.09 (dd, 13.2, 2.7)	59.8
				2.54 (dd, 13.2, 3.4)	39.5
9	123.0	5.34 (dd, 15.9, 9.6)	121.1	5.41 (ddd, 10.7, 3.4, 2.7)	121.4
10	145.7	5.66 (d, 15.9)	143.3	5.44 (d, 10.7)	143.5
11	48.0		40.5		39.9
12	38.6	1.28 (ddd, 13.2, 11.8, 5.2)	35.3	1.36 (ddd, 9.3, 7.1, 3.6)	40.1
		1.38 (ddd, 13.2, 13.2, 4.7)		1.54 (ddd, 9.3, 9.3, 3.0)	
13	23.1	1.78 (m)	29.8	1.32 (m)	23.7
		1.91 (m)		1.51 (m)	
14	124.2	5.05 (br ddq, 7.1, 7.1, 0.8)	76.5	4.02 (dd, 12.4, 4.1)	125.5
15	131.7		147.5		130.9
16	17.5	1.57 (s)	111.0	4.84 (dq, 3.2, 1.6)	25.3
				4.93 (dq, 1.6, 1.1)	
17	25.7	1.66 (s)	17.4	1.71 (dd, 3.2, 1.1)	17.7
18	64.8	4.21 (d, 13.2)	66.2	4.02 (dd, 12.6, 6.6)	65.4
		4.67 (d, 13.2)		4.10 (dd, 12.6, 3.0)	
19	17.6	1.46 (s)	23.3	1.41 (s)	23.4
20	23.4	1.05 (s)	23.7	1.08 (s)	23.8
1'	163.2				
2'	114.9	5.71 (qq, 1.4, 1.4)			
3'	160.3				
4'	20.3	2.15 (d, 1.4)			
5'	27.0	1.91 (d, 1.4)			

a)  $\text{CDCl}_3$ . b)  $\text{C}_6\text{D}_6$ .Table 2.  $^{13}\text{C}$ - (150 MHz,  $\text{C}_6\text{D}_6$ ) and  $^1\text{H}$ - (600 MHz,  $\text{C}_6\text{D}_6$ ) NMR Data of **3**, **4**, and **5**

No.	<b>3</b>		<b>4</b>		<b>5</b>	
	$^{13}\text{C}$	$^1\text{H}$	$^{13}\text{C}$	$^1\text{H}$	$^{13}\text{C}$	$^1\text{H}$
1	35.7	1.86 (dd, 13.2, 4.4)	35.7	1.66 (dd, 16.3, 8.8)	35.6	1.74 (dd, 15.9, 9.1)
		1.63 (dd, 13.2, 9.1)		1.84 (dd, 16.3, 4.9)		1.85 (dd, 15.9, 5.2)
2	38.4	6.06 (dd, 9.1, 4.4)	136.4	6.09 (dd, 8.8, 4.9)	136.5	6.12 (dd, 9.1, 5.2)
3	160.3		143.0		143.2	
4	205.3		205.1		204.9	
5	49.1	2.88 (ddd, 8.5, 4.1, 2.4)	48.5	2.84 (ddd, 9.9, 7.4, 4.4)	48.3	2.88 (ddd, 10.2, 6.7, 4.7)
6	44.3	2.77 (dd, 17.9, 2.2)	44.3	2.50 (dd, 17.9, 4.4)	44.2	2.50 (dd, 17.9, 4.7)
		2.38 (dd, 17.9, 4.1)		2.75 (dd, 17.9, 7.4)		2.78 (dd, 17.9, 6.7)
7	206.4		206.4		206.3	
8	137.6	7.14 (d, 12.4)	137.3	7.17 (d, 12.4)	137.8	7.17 (d, 12.4)
9	113.9	5.08 (dd, 12.4, 11.3)	113.3	5.10 (dd, 12.4, 11.3)	113.3	5.12 (dd, 12.4, 11.3)
10	48.0	1.90 (dd, 12.4, 11.3)	46.4	2.06 (dd, 11.3, 9.9)	46.2	2.09 (dd, 11.3, 10.2)
11	39.9		39.8		39.7	
12	34.5	1.05 (ddd, 12.9, 12.4, 3.0)	36.4	1.20 (m)	36.3	1.12 (m)
		1.28 (ddd, 12.6, 12.4, 3.6)		1.20 (m)		1.27 (m)
13	24.9	1.32 (dddd, 12.9, 12.6, 5.6, 3.6)	24.4	1.16 (m)	24.2	1.12 (m)
		1.43 (dddd, 12.6, 12.6, 5.6, 3.0)		1.28 (m)		1.28 (m)
14	87.8	4.26 (dd, 5.6, 5.6)	63.7	2.39 (dd, 6.0, 5.7)	63.7	2.39 (dd, 6.3, 5.5)
15	143.5		57.1		57.5	
16	113.2	4.90 (br s)	24.9	1.10 (s)	24.9	1.10 (s)
		5.00 (br s)				
17	18.3	1.63 (br s)	18.7	1.12 (s)	18.7	1.08 (s)
18	64.2	4.02 (br d, 12.9)	63.6	4.28 (d, 13.0)	63.5	4.30 (d, 13.1)
		4.37 (br d, 12.9)		4.36 (d, 13.0)		4.41 (d, 13.1)
19	29.5	1.74 (s)	29.5	1.75 (s)	29.5	1.77 (s)
20	29.5	0.61 (s)	24.2	0.60 (s)	23.7	0.60 (s)
1'	163.2		163.1		163.1	
2'	114.9	5.64 (qq, 1.4, 1.4)	114.8	5.64 (qq, 1.4, 1.1)	114.9	5.63 (qq, 1.1, 1.1)
3'	160.3		160.3		160.2	
4'	20.3	1.36 (br d, 1.4)	20.3	2.02 (br d, 1.4)	20.2	2.01 (d, 1.1)
5'	27.0	2.02 (br d, 1.4)	27.0	1.36 (br d, 1.1)	27.0	1.35 (d, 1.1)

2% CeSO<sub>4</sub> in H<sub>2</sub>SO<sub>4</sub> after heating.

**Plant Material** The leaves of *Viburnum odoratissimum* were collected in Taiwan in March, 1999. A voucher specimen was deposited in our institute.

**Extraction and Isolation** Air-dried and powdered leaves (1.5 kg) of *V. odoratissimum* were immersed in MeOH at room temperature for 30 d. The MeOH extract was concentrated *in vacuo* to give a gummy extract (600 g). The MeOH extract (384 g) was mixed with silica gel (Merck, 70–230 mesh, 220 g) and then the MeOH was removed under reduced pressure. The obtained solids were pulverized, packed into a glass column, and eluted in order with CH<sub>2</sub>Cl<sub>2</sub> (2 l), CH<sub>2</sub>Cl<sub>2</sub>:EtOAc=9:1 (2 l), CH<sub>2</sub>Cl<sub>2</sub>:EtOAc=3:2 (2 l), CH<sub>2</sub>Cl<sub>2</sub>:EtOAc=1:1 (2 l), CH<sub>2</sub>Cl<sub>2</sub>:EtOAc=2:3 (2 l), EtOAc (2 l), EtOAc:MeOH=9:1 (2 l), EtOAc:MeOH=3:2 (2 l), EtOAc:MeOH=1:1 (2 l), EtOAc:MeOH=2:3 (2 l), and MeOH (2 l) to give fractions 1–7. Fraction 5 (2.8 g) was purified by a combination of silica gel column chromatography (C-300, CHCl<sub>3</sub>:EtOAc=1:1) and gel filtration (LH-20, hexane:CHCl<sub>3</sub>=3:2) and finally by preparative TLC (Benzen:EtOAc=7:3) to give vibsananin L (**1**) (3.4 mg). Fraction 6 was subjected to reversed-phase chromatography using Cosmosil 75C<sub>18</sub>-OPN (150 g) and eluted with MeOH:H<sub>2</sub>O=2:1 to give fractions 8–15. Fraction 14 was purified by column chromatography over silica gel (Merck, 230–400 mesh, benzene:EtOAc=1:1) to give 14-hydroxyvibsananin F (**2**) (5 mg). Fraction 11 was subjected to medium-pressure chromatography (Lobar RP-18, MeOH:H<sub>2</sub>O=3:1) and finally purified by HPLC [Cosmosil 5C<sub>18</sub>-AR, i.d. 10×250 mm; MeOH:H<sub>2</sub>O:CH<sub>3</sub>CN=2:1:1; 2 ml/min; 254 nm] to give vibsananin I (**3**) (3.6 mg). Fraction 12 (1.5 g) was purified by column chromatography over silica gel (C-300, benzene:EtOAc=7:3) and then by HPLC [Cosmosil 5C<sub>18</sub>-AR, i.d. 10×250 mm; MeOH:H<sub>2</sub>O:CH<sub>3</sub>CN=34:33:33; 2 ml/min; 254 nm] to give 14R\*,15-epoxyvibsananin C (**4**) (3 mg) and 14S\*,15-epoxyvibsananin C (**5**) (2 mg). The previously known vibsananin B (818 mg), vibsananin F (**2a**) (18 mg) and vibsananin A (15 mg) were obtained from fraction 18.

**Vibsananin L (1)** Colorless oil;  $[\alpha]_D^{20}$  -61.6° (*c*=0.20, CHCl<sub>3</sub>); IR  $\nu_{\max}^{\text{FT}}$ : 3356 (OH), 1719, 1700 (C=O), 1649 (C=C) cm<sup>-1</sup>; UV  $\lambda_{\max}^{\text{EtOH}}$  nm: 218 ( $\epsilon$  14000); FAB-MS: *m/z* 455 [M+Na]<sup>+</sup>, 471 [M+K]<sup>+</sup>; HR-FAB-MS: Calcd 455.2410 for C<sub>25</sub>H<sub>36</sub>O<sub>6</sub>Na. Found 455.2431.; <sup>1</sup>H- and <sup>13</sup>C-NMR: Table 1.

**14-Hydroxyvibsananin F (2)** Colorless oil;  $[\alpha]_D^{20}$  +9.1° (*c*=0.18, CHCl<sub>3</sub>); IR  $\nu_{\max}^{\text{FT}}$ : 3376 (OH) cm<sup>-1</sup>; FAB-MS: *m/z* 343 [M+Na]<sup>+</sup>, 359 [M+K]<sup>+</sup>; HR-FAB-MS: Calcd 343.2250 for C<sub>20</sub>H<sub>32</sub>O<sub>3</sub>Na. Found 343.2252.; <sup>1</sup>H and <sup>13</sup>C-NMR: Table 1.

**Vibsananin I (3)** Colorless oil;  $[\alpha]_D^{20}$  +73.6° (*c*=0.56, CHCl<sub>3</sub>); IR  $\nu_{\max}^{\text{FT}}$ : 3420 (OH), 1715 (C=O), 1645 (C=C) cm<sup>-1</sup>; UV  $\lambda_{\max}^{\text{EtOH}}$  nm: 252 ( $\epsilon$  4300); FAB-MS: *m/z* 471 [M+Na]<sup>+</sup>, 487 [M+K]<sup>+</sup>; HR-FAB-MS: Calcd 471.2359 for C<sub>25</sub>H<sub>36</sub>O<sub>7</sub>Na. Found 471.2357.; <sup>1</sup>H- and <sup>13</sup>C-NMR: Table 2.

**14R\*,15-Epoxyvibsananin C (4)** Colorless oil;  $[\alpha]_D^{20}$  +97.0° (*c*=0.12, CHCl<sub>3</sub>); IR  $\nu_{\max}^{\text{FT}}$ : 3445 (OH), 1728 (C=O), 1649 (C=C) cm<sup>-1</sup>; UV  $\lambda_{\max}^{\text{EtOH}}$  nm: 242 ( $\epsilon$  6700); FAB-MS: *m/z* 455 [M+Na]<sup>+</sup>, 471 [M+K]<sup>+</sup>; HR-FAB-

MS: Calcd 455.2410 for C<sub>25</sub>H<sub>36</sub>O<sub>6</sub>Na. Found 455.2407.; <sup>1</sup>H- and <sup>13</sup>C-NMR: Table 2.

**14S\*,15-Epoxyvibsananin C (5)** Colorless oil;  $[\alpha]_D^{20}$  +86.0° (*c*=0.12, CHCl<sub>3</sub>); IR  $\nu_{\max}^{\text{FT}}$ : 3491 (OH), 1728 (C=O), 1649 (C=C) cm<sup>-1</sup>; UV  $\lambda_{\max}^{\text{EtOH}}$  nm: 241 ( $\epsilon$  7200); FAB-MS: *m/z* 455 [M+Na]<sup>+</sup>, 471 [M+K]<sup>+</sup>; HR-FAB-MS: Calcd 455.2410 for C<sub>25</sub>H<sub>36</sub>O<sub>6</sub>Na. Found 455.2403.; <sup>1</sup>H and <sup>13</sup>C-NMR: Table 2.

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