

### Three New Bisabolocurcumin Ethers from the Rhizomes of *Curcuma longa* L.

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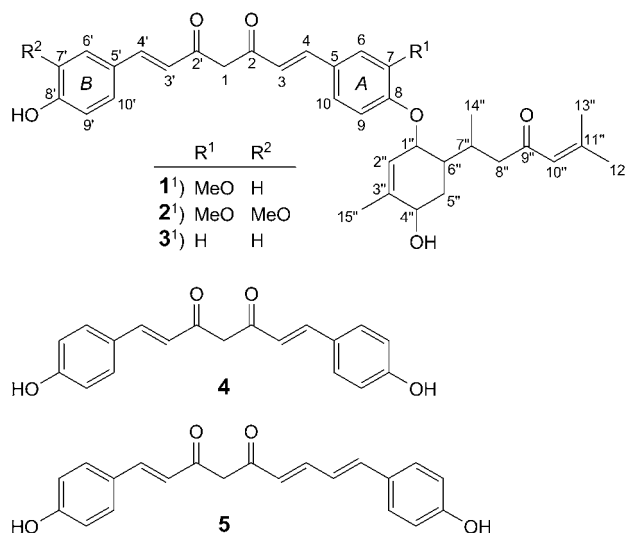
Three new bisabolocurcumin ethers, named demethoxybisabolocurcumin ether (**1**), bisabolocurcumin ether (**2**), and didemethoxybisabolocurcumin ether (**3**), along with two known compounds, **4** and **5**, were isolated from the AcOEt extract of the rhizomes of *Curcuma longa* L. Their structures were established by the analysis of NMR and MS data. The new compounds **1–3**, which possess a new 1,7-diarylheptanoid skeleton linked with a bisabolone-type sesquiterpene substructure by a C–O bond, were found for the first time in a natural source.

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**Introduction.** – *Curcuma longa* L. (Zingiberaceae) is distributed throughout the tropical and subtropical regions of the world and widely cultivated in China and India [1]. The rhizomes of *Curcuma longa* L. (Zingiberaceae) are officially listed in the Chinese Pharmacopoeia as ‘JiangHuang’ [2], and have been used as a medicine in Traditional Chinese, Japanese, and Indian medicines for centuries [3]. The rhizomes have also been used for food, as spices, dyes, and perfumes [3]. Recent pharmacological studies revealed that ‘Jiang Huan’ (*C. longa* L.) possesses antioxidant [4], anti-inflammatory [5], anti-*Alzheimer* [6], anticancer [7], anti-HIV [8], antimicrobial [9], and hypoglycemic properties [10]. Up to now, from *C. longa*, curcuminoids, sesquiterpenoids, and their derivatives were reported as components [11]. In our phytochemical investigation, three new bisabolocurcumin ethers, compounds **1–3**, were isolated from the title plant. In their structures, a bisabolone-type sesquiterpene substructure is combined with a curcuminoid moiety, connected *via* a C–O bond. Two known compounds, (1*E*,4*E*)-1,5-bis(4-hydroxyphenyl)penta-1,4-dien-3-one (**4**) [12] and (1*E*,4*E*,6*E*)-1,7-bis(4-hydroxyphenyl)hepta-1,4,6-trien-3-one (**5**) [13], were also isolated.

In this article, the isolation and structure determination of the three new compounds are described.

**Results and Discussion.** – The dried rhizomes of *C. longa* were extracted with petroleum ether (PE) and AcOEt, respectively. The AcOEt extract was subjected to repeated silica-gel, *Sephadex LH-20*, and *MCI* column chromatography to afford the compounds **1** (50 mg), **2** (50 mg), **3** (100 mg), **4** (2 mg), and **5** (2 mg).



Compound **1** was obtained as a yellow amorphous powder. The IR spectrum indicated the presence of OH ( $3425\text{ cm}^{-1}$ ), CO ( $1674$ ,  $1626\text{ cm}^{-1}$ ), and aromatic moieties ( $1600$ ,  $1581$ ,  $1508\text{ cm}^{-1}$ ). The molecular formula was determined as  $\text{C}_{35}\text{H}_{40}\text{O}_7$  based on the HR-ESI-MS data ( $m/z$  595.2654 ( $[M + \text{Na}]^+$ )), indicating 16 degrees of unsaturation. The  $^1\text{H-NMR}$  data of **1** (Table) exhibited signals of a 1,3,4-trisubstituted aromatic ring ( $\delta(\text{H})$  7.36 (*s*, 1 H), 7.23 and 7.02 (*2d*,  $J = 8.4$ , 1 H each)), a 1,4-disubstituted aromatic ring ( $\delta(\text{H})$  7.57 and 6.83 (*2d*,  $J = 8.4$ , 2 H each)), four olefinic H-atoms ( $\delta(\text{H})$  6.82 and 7.57 (*2d*,  $J = 15.0$ , 1 H each), 6.70 and 7.57 (*2d*,  $J = 15.0$ , 1 H each)), and a MeO group ( $\delta(\text{H})$  3.82 (*s*, 3 H)). The  $^{13}\text{C-NMR}$  data of **1** (Table) also displayed signals of two aromatic rings ( $\delta(\text{C})$  160.3, 150.4, 149.7, 130.8, 130.8, 128.2, 126.3, 123.2, 116.4, 116.4, 114.8, and 111.7), four olefinic C-atoms ( $\delta(\text{C})$  141.1, 140.6, 122.5, and 121.3), a MeO group ( $\delta(\text{C})$  56.2), and two conjugated CO groups ( $\delta(\text{C})$  183.1 and 184.2). The  $^1\text{H-}$  and  $^{13}\text{C-NMR}$  spectra of **1** showed the characteristic signals at  $\delta(\text{H})$  6.08 (*s*, 1 H) and  $\delta(\text{C})$  101.5 for a 1,7-diarylheptanoid skeleton. After comparison of the NMR data of **1** with those of demethoxycurcumin [14], it was concluded that a demethoxycurcumin moiety was present in **1**.

Additionally, in the  $^1\text{H-NMR}$  spectrum of **1**, signals for two olefinic CH groups ( $\delta(\text{H})$  6.11 and 5.38 (*2s*, 1 H each)) and signals for four Me groups ( $\delta(\text{H})$  2.02, 1.82, 1.70 (*3 s*, 3 H each), and 0.74 (*d*,  $J = 6.0$ , 3 H)) were observed. In the  $^{13}\text{C-NMR}$  spectrum of **1**, 15 additional C-atom signals, thereof signals for four olefinic C-atoms ( $\delta(\text{C})$  154.2, 139.6, 124.5, and 123.4), four Me groups ( $\delta(\text{C})$  27.6, 20.7, 15.4, and 20.8), and a CO group ( $\delta(\text{C})$  200.2) were observed. These NMR data indicated that compound **1** contained a bisabolane-type sesquiterpene moiety, which was similar to bisabolon-9-one [15]. Comparing the NMR data with those of bisabolon-9-one, the data for the heptene moiety were almost identical, but the signals for the cyclohexene moiety

<sup>1)</sup> Arbitrary atom numbering. For systematic names, see *Exper. Part*.

Table.  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR Data of **1**, **2**, and **3** in ( $D_6$ )DMSO.  $\delta$  in ppm,  $J$  in Hz.

	<b>1</b> <sup>1)</sup>		<b>2</b> <sup>1)</sup>		<b>3</b> <sup>1)</sup>	
	$\delta(\text{H})^{\text{a}}$	$\delta(\text{C})^{\text{b}}$	$\delta(\text{H})^{\text{c}}$	$\delta(\text{C})^{\text{d}}$	$\delta(\text{H})^{\text{c}}$	$\delta(\text{C})^{\text{d}}$
CH <sub>2</sub> (1)	6.08 (s)	101.5	6.09 (s)	101.2	6.07 (s)	101.4
C(2)	–	183.1	–	182.9	–	182.9
H–C(3)	6.82 (d, $J = 15.0$ )	122.5	6.83 (d, $J = 16.0$ )	122.3	6.77 (d, $J = 16.0$ )	121.9
H–C(4)	7.57 (d, $J = 15.0$ )	140.6	7.57 (d, $J = 16.0$ )	140.4	7.56 (d, $J = 16.0$ )	140.0
C(5)	–	128.2	–	128.0	–	127.5
H–C(6)	7.36 (s)	111.7	7.36 (s)	111.5	7.67 (d, $J = 8.4$ )	130.5
C(7) or H–C(7)	–	150.4	–	150.1	7.00 (d, $J = 8.4$ )	116.0
C(8)	–	149.7	–	149.4	–	159.9
H–C(9)	7.02 (d, $J = 8.4$ )	114.8	7.04 (d, $J = 8.0$ )	114.5	7.00 (d, $J = 8.4$ )	116.0
H–C(10)	7.23 (d, $J = 8.4$ )	123.2	7.24 (d, $J = 8.0$ )	123.0	7.67 (d, $J = 8.4$ )	130.5
MeO–C(7)	3.82 (s)	56.2	3.84 (s)	55.9	–	–
C(2')	–	184.2	–	183.9	–	183.9
H–C(3')	6.70 (d, $J = 15.0$ )	121.3	6.77 (d, $J = 16.0$ )	121.3	6.71 (d, $J = 16.0$ )	121.0
H–C(4')	7.57 (d, $J = 15.0$ )	141.1	7.56 (d, $J = 16.0$ )	141.2	7.58 (d, $J = 16.0$ )	140.8
C(5')	–	126.3	–	126.5	–	126.0
H–C(6')	7.57 (d, $J = 8.4$ )	130.8	7.32 (s)	111.6	7.57 (d, $J = 8.0$ )	130.6
H–C(7') or C(7')	6.83 (d, $J = 8.4$ )	116.4	–	148.2	6.82 (d, $J = 8.0$ )	116.2
C(8')	–	160.3	–	149.6	–	160.1
H–C(9')	6.83 (d, $J = 8.4$ )	116.4	6.82 (d, $J = 8.0$ )	115.9	6.82 (d, $J = 8.0$ )	116.2
H–C(10')	7.57 (d, $J = 8.4$ )	130.8	7.16 (d, $J = 8.0$ )	123.4	7.57 (d, $J = 8.0$ )	130.6
MeO–C(7')	–	–	3.83 (s)	55.9	–	–
H–C(1'')	4.70 (d, $J = 9.0$ )	75.5	4.73 (d, $J = 8.8$ )	75.2	4.75 (d, $J = 8.8$ )	74.6
H–C(2'')	5.38 (s)	123.4	5.38 (s)	123.1	5.41 (s)	123.0
C(3'')	–	139.6	–	139.3	–	139.6
H–C(4'')	3.82 ( $m_c$ ) <sup>e</sup>	66.1	3.83 ( $m_c$ ) <sup>e</sup>	65.8	3.85 ( $m_c$ )	65.8
CH <sub>2</sub> (5'')	1.66–1.70 ( $m$ ), 1.47 ( $dt$ , $J = 13.2, 1.8$ )	30.9	1.66–1.70 ( $m$ ), 1.45 ( $dt$ , $J = 12.8, 2.8$ )	30.7	1.65–1.71 ( $m$ ), 1.49 ( $dt$ , $J = 13.2, 2.8$ )	30.6
H–C(6'')	2.02–2.04 ( $m$ )	38.6	2.01–2.03 ( $m$ )	38.3	1.97–2.03 ( $m$ )	38.0
H–C(7'')	2.30–2.39 ( $m$ )	28.2	2.30–2.37 ( $m$ ) <sup>e</sup>	27.9	2.30–2.41 ( $m$ ) <sup>e</sup>	28.0
CH <sub>2</sub> (8'')	2.40 (d, $J = 13.8$ ), 2.30 ( $dd$ , $J = 13.8, 9.0$ )	49.8	2.41 (d, $J = 11.2$ ), 2.30–2.37 ( $m$ ) <sup>e</sup>	49.5	2.30–2.41 ( $m$ ) <sup>e</sup>	49.3
C(9'')	–	200.2	–	199.9	–	199.8
H–C(10'')	6.11 (s)	124.5	6.12 (s)	124.2	6.13 (s)	124.3
C(11'')	–	154.2	–	154.1	–	154.1
Me(12'')	2.02 (s)	20.7	2.02 (s)	20.4	2.03 (s)	20.5
Me(13'')	1.82 (s)	27.6	1.83 (s)	27.3	1.79 (s)	27.3
Me(14'')	0.74 (d, $J = 6.0$ )	15.4	0.74 (d, $J = 6.0$ )	15.1	0.74 (d, $J = 6.0$ )	15.1
Me(15'')	1.70 (s)	20.8	1.70 (s)	20.6	1.71 (s)	20.6

<sup>a)</sup> Measured at 600 MHz. <sup>b)</sup> Measured at 150 MHz. <sup>c)</sup> Measured at 400 MHz. <sup>d)</sup> Measured at 100 MHz.  
<sup>e)</sup> Overlapping signals.

differed. The chemical shifts ( $\delta(\text{H})$  3.82 ( $m_c$ , 1 H) and  $\delta(\text{C})$  66.1) and the correlations in the HMBC ( $\delta(\text{H})$  3.82 with  $\delta(\text{C})$  123.4 (C(2'')), 139.6 (C(3'')), and 38.6 (C(6'')) indicated that there was a OH substituent located at C(4'') (Fig. 1). In bisabolon-9-one, there was a keto group at C(1) ( $\delta(\text{C})$  200.5) [15]. However, in **1** this C-atom signal for C(1'') was not observed. Instead, an O-bearing CH group ( $\delta(\text{H})$  4.70 ( $d$ ,  $J=9.0$ , 1 H) and  $\delta(\text{C})$  75.5) was found. The assignment of these signals to H–C(1'') was supported by the HMBCs of the signal at  $\delta(\text{H})$  4.70 with those at  $\delta(\text{C})$  123.4 (C(2'')), 139.6 (C(3'')), 38.6 (C(6'')), and 28.2 (C(7'')). Based on this evidence, the bisabolane-type sesquiterpene moiety in **1** was elucidated as a 1-O-bearing 4-hydroxy-3-methyl 6-(6-methyl-4-oxohept-5-en-2-yl)cyclohex-2-ene moiety.

The bisabolane-type sesquiterpene moiety was linked at C(8) of demethoxycurcumin, which was determined by the correlation of H–C(1'') ( $\delta(\text{H})$  4.70) with C(8) ( $\delta(\text{C})$  149.7) in the HMBC spectrum of **1** (Fig. 1). Based on all above evidences, the structure of compound **1** was elucidated as (1*E*,6*E*)-1-(4-[[4-hydroxy-3-methyl-6-(6-methyl-4-oxohept-5-en-2-yl)cyclohex-2-en-1-yl]oxy]-3-methoxyphenyl)-7-(4-hydroxyphenyl)-hepta-1,6-diene-3,5-dione, and named demethoxybisabolocurcumin ether.

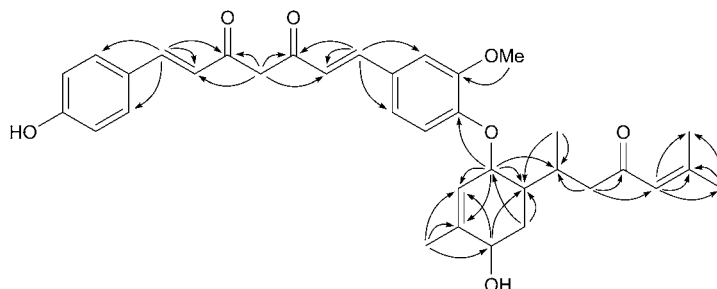


Fig. 1. Key HMBCs of demethoxybisabolocurcumin ether (**1**)

Compound **2** was obtained as a yellow amorphous powder. The IR spectrum indicated the presence of OH ( $3417\text{ cm}^{-1}$ ), CO ( $1678$ ,  $1625\text{ cm}^{-1}$ ), and aromatic moieties ( $1585$ ,  $1509\text{ cm}^{-1}$ ). The molecular formula was determined to be  $\text{C}_{36}\text{H}_{42}\text{O}_8$ , based on the HR-ESI-MS data ( $m/z$  625.2754 ( $[M + \text{Na}]^+$ )), indicating that compound **2** had 30 atomic mass units more than compound **1**. The  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR spectra of **2** showed also signals of a 1,7-diarylheptanoid moiety and a bisabolone-type sesquiterpene moiety, indicating that the structure of **2** was similar to the one of **1** (Table). Comparison of the NMR data and the molecular formula of **2** with the data of **1** revealed that compound **2** contained an additional MeO group ( $\delta(\text{H})$  3.83 ( $s$ , 3 H);  $\delta(\text{C})$  55.9) compared to compound **1**. The MeO group was located at C(7') as determined by the  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR chemical shifts of the B-ring and the correlations ( $\delta(\text{H})$  3.83 with  $\delta(\text{C})$  148.2 (C(7'));  $\delta(\text{H})$  7.32 (H–C(6')) with  $\delta(\text{C})$  148.2 (C(7')), 123.4 (C(10')), 149.6 (C(8')), and 141.2 (C(4'));  $\delta(\text{H})$  6.82 (H–C(9')) with  $\delta(\text{C})$  148.2 (C(7')) and 126.5 (C(5'));  $\delta(\text{H})$  7.16 (H–C(10')) with  $\delta(\text{C})$  149.6 (C(8')), 141.2 (C(4')), and 111.6 (C(6'))); see Fig. 2). Accordingly, the structure of **2** was elucidated as (1*E*,6*E*)-1-(4-hydroxy-3-methoxyphenyl)-7-{4-[4-hydroxy-3-methyl-6-(6-methyl-4-oxohept-5-en-2-yl)cyclohex-2-en-1-yl]-3-methoxyphenoxy}hepta-1,6-diene-3,5-dione, and named bisabolocurcumin ether.

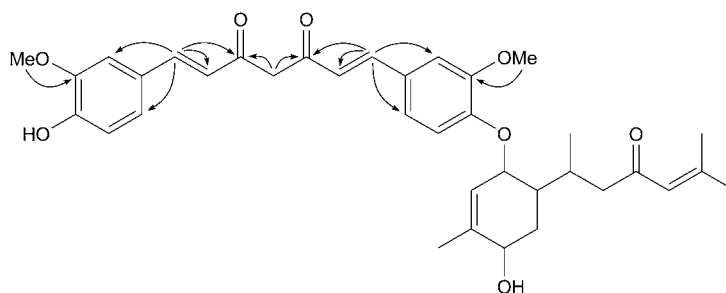


Fig. 2. Key HMBCs of bisabololcurcumin ether (**2**)

Compound **3** was obtained as a yellow amorphous powder. The IR spectrum indicated the presence of OH ( $3382\text{ cm}^{-1}$ ), CO ( $1675$ ,  $1626\text{ cm}^{-1}$ ), and aromatic moieties ( $1600$ ,  $1578$ ,  $1509\text{ cm}^{-1}$ ). The molecular formula was determined to be  $\text{C}_{34}\text{H}_{38}\text{O}_6$ , based on the HR-ESI-MS data ( $m/z$  565.2559 ( $[M + \text{Na}]^+$ )), indicating that compound **3** had 30 atomic mass units less than compound **1**. The  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR spectra of **3** showed also resonances of a 1,7-diarylheptanoid moiety and a bisabolone-type sesquiterpene moiety, indicating that the structure of **3** was similar to the one of **1** (Table). Comparison of the NMR data and the molecular formula of **3** with those of **1**, revealed that compound **3** did not contain any MeO groups. The  $^1\text{H}$ -NMR spectrum of **3** showed signals of two 1,4-disubstituted aromatic rings ( $\delta(\text{H})$  7.67, 7.00 ( $2d$ ,  $J = 8.4$ , 2 H each) and  $\delta(\text{H})$  7.57, 6.82 ( $2d$ ,  $J = 8.0$ , 2 H each)). Accordingly, the structure of **3** was elucidated as (1*E*,6*E*)-1-[4-[4-hydroxy-3-methyl-6-(6-methyl-4-oxohept-5-en-2-yl)cyclohex-2-en-1-yl]phenoxy]-7-(4-hydroxyphenyl)hepta-1,6-diene-3,5-dione, and named didemethoxybisabololcurcumin ether.

#### Experimental Part

**General.** All solvents used were of anal. grade (Tianjin Chemical Plant, Tianjin, P. R. China). Column chromatography (CC): Silica gel ( $\text{SiO}_2$ , 200–300 mesh; Qingdao Ocean Chemical Industry Co., P. R. China), MCI gel (75–150  $\mu\text{m}$ ; Mitsubishi Chemical Corporation, Tokyo), and Sephadex LH-20 (Amersham Biosciences). TLC Spots were visualized under UV light and by spraying 10%  $\text{H}_2\text{SO}_4$  in alcohol followed by heating. Optical rotation: Perkin-Elmer-241 polarimeter. UV Spectra: Shimadzu UV-2210-UV/VIS spectrometer; MeOH solns.;  $\lambda_{\text{max}}$  in nm. IR Spectra: Vector 22-FTIR spectrometer with KBr pellets; in  $\text{cm}^{-1}$ . NMR Spectra: Bruker AV-400 spectrometer, at 400 ( $^1\text{H}$ ), and 100 ( $^{13}\text{C}$ ) MHz, resp.; Bruker AV-600 spectrometer, at 600 ( $^1\text{H}$ ), and 150 ( $^{13}\text{C}$ ) MHz, resp.; ( $\text{D}_6$ )DMSO solns.;  $\delta$  in ppm,  $J$  in Hz. HR-ESI-MS: Bruker micrOTOF-Q mass spectrometers; in  $m/z$ .

**Plant Material.** The dried rhizomes of *C. longa* were collected from Sichuan province, P. R. China, in July 2009, and identified by Prof. Jing Huang (West China School of Pharmacy, Sichuan University, Chengdu, P. R. China) and a voucher specimen (No. 20090711) was deposited at West China School of Pharmacy, Sichuan University, Chengdu, P. R. China.

**Extraction and Isolation.** The dried rhizomes of *C. longa* (2.8 kg) were extracted with petroleum ether (PE) and AcOEt, respectively. The AcOEt extract (180 g) was subjected to CC ( $\text{SiO}_2$ ;  $\text{CH}_2\text{Cl}_2/\text{MeOH}$ , 100:0  $\rightarrow$  100:1  $\rightarrow$  50:1  $\rightarrow$  20:1  $\rightarrow$  10:1  $\rightarrow$  0:1) to afford five fractions. Fr. 3 ( $\text{CH}_2\text{Cl}_2/\text{MeOH}$ , 50:1) was purified by repeated CC over MCI gel (MeOH/ $\text{H}_2\text{O}$  9:1),  $\text{SiO}_2$  (PE/AcOEt 2:1, then  $\text{CH}_2\text{Cl}_2/\text{MeOH}$  100:1), and Sephadex LH-20 (MeOH/ $\text{H}_2\text{O}$  9:1), to afford **1** (50 mg), **2** (50 mg), **3** (100 mg), (1*E*,4*E*)-1,5-bis(4-hydroxyphenyl)penta-1,4-dien-3-one (**4**; 2 mg), and (1*E*,4*E*,6*E*)-1,7-bis(4-hydroxyphenyl)hepta-1,4,6-trien-3-one (**5**; 2 mg).

*Demethoxybisabolocurcumin Ether* (= (1E,6E)-1-(4-([4-Hydroxy-3-methyl-6-(6-methyl-4-oxohept-5-en-2-yl)cyclohex-2-en-1-yl]oxy)-3-methoxyphenyl)-7-(4-hydroxyphenyl)hepta-1,6-diene-3,5-dione; **1**). Yellow amorphous powder.  $[\alpha]_{\text{D}}^{20} = +0.5$  ( $c = 0.45$ , MeOH). UV: 420 (4.78). IR: 3425, 2960, 2920, 1674, 1626, 1600, 1581, 1508, 1444, 1380, 1256, 1167, 1134, 1033, 1015.  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR: see Table. HR-ESI-MS: 595.2654 ( $[M + \text{Na}]^+$ ,  $\text{C}_{35}\text{H}_{40}\text{NaO}_7^+$ ; calc. 595.2672).

*Bisabolocurcumin Ether* (= (1E,6E)-1-(4-Hydroxy-3-methoxyphenyl)-7-[4-[4-hydroxy-3-methyl-6-(6-methyl-4-oxohept-5-en-2-yl)cyclohex-2-en-1-yl]-3-methoxyphenoxy]hepta-1,6-diene-3,5-dione; **2**). Yellow amorphous powder.  $[\alpha]_{\text{D}}^{20} = +0.6$  ( $c = 0.61$ , MeOH). UV: 423 (4.72). IR: 3417, 2960, 2920, 1678, 1625, 1585, 1509, 1449, 1427, 1382, 1258, 1162, 1131, 1031.  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR: see Table. HR-ESI-MS: 625.2754 ( $[M + \text{Na}]^+$ ,  $\text{C}_{36}\text{H}_{42}\text{NaO}_8^+$ ; calc. 625.2777).

*Didemethoxybisabolocurcumin Ether* (= (1E,6E)-1-[4-[4-Hydroxy-3-methyl-6-(6-methyl-4-oxohept-5-en-2-yl)cyclohex-2-en-1-yl]phenoxy]-7-(4-hydroxyphenyl)hepta-1,6-diene-3,5-dione; **3**). Yellow amorphous powder.  $[\alpha]_{\text{D}}^{20} = +0.4$  ( $c = 0.46$ , MeOH). UV: 416 (4.74), 241 (4.43). IR: 3382, 2960, 2917, 1675, 1626, 1600, 1578, 1509, 1442, 1379, 1246, 1169, 1137, 1016.  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR: see Table. HR-ESI-MS: 565.2559 ( $[M + \text{Na}]^+$ ,  $\text{C}_{34}\text{H}_{38}\text{NaO}_6^+$ ; calc. 565.2566).

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