9,10-Dihydrophenanthrenes and Phenanthrenes from *Juncus setchuensis*

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Four new 9,10-dihydrophenanthrenes, juncuenins A−D (**1**−**4**), three new phenanthrenes, dehydrojuncuenins A−C (**5**−**7**), and three known compounds were isolated from the whole plants of *Juncus setchuensis*. The structures of the new compounds were established on the basis of detailed 1D and 2D NMR studies.

Juncus setchuensis Buchenau (Juncaceae), a perennial herbaceous plant found in marshes, swamps, and other poorly drained sites, is mainly distributed in South China. The whole plant of *J. setchuensis* and other *Juncus* species such as *J. effusus* L. has long been regarded as an antipyretic and detumescence agent in traditional Chinese medicine.[1] Italian scientists discovered phenanthrenes and dihydrophenanthrenes from *J. effusus*.[2−5] Several dimeric 9,10-dihydrophenanthrenes and benzocoumarins were reported in *J. acutus*.[6−9] Some of these compounds have shown strong antitumor and antialgal activities.

Although the plants of the *Juncus* genus are abundant in China, studies of this genus are restricted to *J. effusus*.[10−16] In this paper, we report the phytochemical investigation of *J. setchuensis*, from which four 9,10-dihydrophenanthrenes, juncuenins A−D (**1**-**4**), and three new phenanthrenes, dehydrojuncuenins A−C (**5**-**7**), were isolated along with three known compounds, effusol,[4] dehydroeffusol,[17] and 2,8-dihydroxy-1,6-dimethyl-5-vinyl-9,10-dihydro phenanthrene.[4] Their structures were determined by extensive analyses of IR, MS, and 1D and 2D NMR data, as well as by comparison with literature data.

Compound **1** was obtained as an amorphous powder. Its molecular formula was established as $C_{18}H_{18}O$ according to the molecular ion at *m*/*z* 250.1358 by HREIMS. The IR peak at 3320 cm^{-1} indicated the presence of an OH group. The ^{13}C NMR spectrum (Table 1) confirmed the presence of 12 aromatic carbons (one bearing an oxygen atom), two olefinic carbons, two methylene carbons, and two methyl carbons. The ^1H NMR spectrum (Table 2) indicated two aromatic methyl groups, two *o*-coupled aromatic protons, two *p*-coupled aromatic protons, and three vinylic protons as an ABX system. These NMR and MS data suggested a 9,10-dihydrophenanthrene skeleton similar to that of effusol isolated from *J. effusus*.[4] The substituents of **1** were determined by a ROESY experiment (see S4 in the Supporting Information). The key correlations of H_3-12 (δ_H 2.35, s)/H-8 (δ_H 7.01, s), H-8/H_2-9, H_2-10/H_3-11 (δ_H 2.24, s), H_3-12/H-13, H_2-14/H-5 (δ_H 7.76, s), H-5/H-4 (δ_H 7.55, d), and H-3 (δ_H 6.74, d)/OH (δ_H 4.84, br s) suggested two methyl groups at C-1 and C-7, the OH group at C-2, and the vinylic substituent at C-6. The substitution pattern of juncuenin A (**1**) was further confirmed by the HMBC spectrum (see S3 in the Supporting Information).

Compound **2**, white needles, showed a molecular formula of $C_{18}H_{18}O_2$ by HREIMS. The IR spectrum showed the presence of two OH groups (3373 and 3240 cm^{-1}). The ^1H and ^{13}C NMR data (Tables 1 and 2) indicated that it is also a 9,10-dihydrophenanthrene derivative substituted by two methyl, one vinyl, and two hydroxy groups. The ROESY spectra (see S7 in the Supporting Information) confirmed its full structure with two methyl groups at C-1 and C-7, a vinyl group at C-8, and two hydroxy groups at C-2 and C-6, thus defining the structure of juncuenin B (**2**) as shown.

Compound **3**, a white, amorphous powder, had a molecular formula of $C_{18}H_{16}O_4$ as determined by HREIMS. The IR absorptions at 3439 and 1641 cm^{-1} showed the presence of an OH and a carbonyl group, which was supported by the resonance at δ_C 174.7 in the ^{13}C NMR spectrum (Table 1). Extensive analysis of its 1D and 2D NMR data revealed that it appeared to be an oxidation product of **2** in which the methyl group at C-7 was oxidized to a carboxylic acid functionality. The ROESY correlations of H_3-11/H_2-10, H-4/H-5, H_2-9/H-13, and H_2-9/H_2-14 and HMBC cross-peaks from H-5 to C-4a, C-8a, C-7, and the carbonyl C-12 and from H-13 to C-7 and C-8a further confirmed the structure of juncuenin C (**3**) as shown (see S10 and S11 in the Supporting Information).

The molecular formula of **4** was established as $C_{18}H_{18}O_3$ by HRESIMS, with one additional oxygen atom compared to compound **2**. The IR spectrum showed the presence of two OH groups at 3292 and 3234 cm^{-1} and a carbonyl group at 1645 cm^{-1}. The ^1H NMR spectrum (Table 2) displayed signals of two *o*-coupled aromatic protons, four benzylic protons, one low-field singlet, two methyl and one vinylic group proton, and two hydroxylic protons. The ^{13}C NMR spectrum (Table 1) showed one carbonyl (δ_C 185.4) and one oxygenated quaternary carbon (δ_C 66.5) resonance. Comparing the NMR data of **4** with those of **2** (Tables 1 and 2) revealed that the signals of the A ring were similar but not those of ring B. Ring B was deduced to contain a carbonyl carbon and substituted by an OH, a methyl, and a vinyl group. In the HMBC spectrum, the correlation from the low-field proton (δ_H 6.32, s) to C-4 indicated that this proton was likely at C-5. Also from H-5 the correlations to the oxygenated quaternary carbon and the carbon at δ_C 130.1 were observed, suggesting that these two carbons might be C-8a or C-7, respectively. The long-range correlations from the methylene protons of the vinyl group to the carbon at δ_C 152.7 and from the methine proton to the oxygenated carbon and the carbon at δ_C 130.1 suggested that the carbon at δ_C 152.7 was likely to be C-8 and substituted by the vinyl group. In addition, the methyl group protons (δ_H 1.93, 3H, s) showed long-range correlation to the carbonyl carbon, which suggested that the methyl was attached to C-7 and the carbonyl carbon was C-6. Thus, the oxygenated quaternary carbon and the carbon at δ_C 130.1 were designated as C-8a and C-7, respectively, and the OH group was located at C-8a. Therefore, the planar structure of **4** was fully established. The specific rotation of **4** was zero, suggesting that juncuenin D (**4**) is racemic.

Compound **5**, obtained as a white, amorphous solid, had the molecular formula $C_{18}H_{16}O$ as determined by HREIMS. The IR

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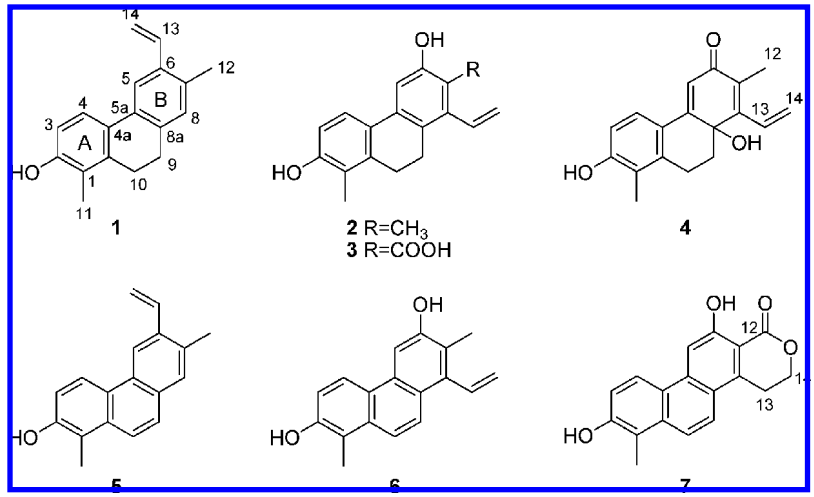

Table 1. ^{13}C NMR Data of Compounds **1**−**7** (100 MHz, δ in ppm)

no.	**1**[a]	**2**[b]	**3**[c]	**4**[d]	**5**[a]	**6**[b]	**7**[d]
1	121.2 qC	121.5 qC	122.9 qC	123.6 qC	117.5 qC	117.5 qC	117.3 qC
2	153.0 qC	155.1 qC	157.7 qC	156.7 qC	151.4 qC	155.1 qC	155.2 qC
3	113.1 CH	113.4 CH	114.6 CH	113.1 CH	116.2 CH	116.5 CH	116.7 CH
4	122.0 CH	122.4 CH	124.8 CH	124.4 CH	121.5 CH	125.0 CH	123.2 CH
5	120.3 CH	109.3 CH	111.9 CH	116.9 CH	119.1 CH	105.0 CH	106.4 CH
6	135.4 qC	154.2 qC	160.3 qC	185.4 qC	136.3 qC	153.5 qC	157.5 qC
7	133.7 qC	120.7 qC	114.3 qC	130.1 qC	133.4 qC	124.0 qC	108.2 qC
8	129.5 CH	137.9 qC	140.2 qC	152.7 qC	129.0 CH	131.2 qC	140.3 qC
9	28.5 CH_2	25.9 CH_2	27.0 CH_2	33.7 CH_2	126.7 CH	119.7 CH	121.2 CH
10	25.3 CH_2	26.1 CH_2	27.5 CH_2	23.1 CH_2	122.5 CH	121.9 CH	122.5 CH
1a	137.6 qC	138.4 qC	142.8 qC	138.3 qC	132.1 qC	132.8 qC	133.4 qC
4a	127.6 qC	125.4 qC	127.3 qC	121.3 qC	125.0 qC	123.1 qC	121.9 qC
5a	132.9 qC	134.3 qC	140.7 qC	158.5 qC	129.2 qC	137.9 qC	136.9 qC
8a	135.8 qC	127.4 qC	127.4 qC	66.5 qC	130.2 qC	122.9 qC	120.0 qC
11	11.5 CH_3	12.7 CH_3	12.0 CH_3	12.0 CH_3	10.9 CH_3	13.3 CH_3	10.8 CH_3
12	19.3 CH_3	11.2 CH_3	174.7 qC	10.9 CH_3	19.9 CH_3	10.7 CH_3	169.3 qC
13	135.1 CH	137.9 CH	138.2 CH	132.8 CH	135.7 CH	135.5 CH	23.8 CH_2
14	114.4 CH_2	119.8 CH_2	119.1 CH_2	123.1 CH_2	115.8 CH_2	121.9 CH_2	62.6 CH_2

[a] In $CDCl_3$. [b] In acetone-d_6. [c] In methanol-d_4. [d] In DMSO-d_6.

Table 2. ^{1}H NMR Data of Compounds **1**−**4** (300 MHz, δ in ppm, J in Hz)

no.	**1**[a]	**2**[a]	**3**[b]	**4**[c]
3	6.74, d (8.3)	6.72, d (8.4)	6.73, d (8.3)	6.75, d (8.8)
4	7.55, d (8.3)	7.40, d (8.4)	7.41, d (8.3)	7.37, d (8.8)
5	7.76, s	7.06, s	7.13, s	6.32, s
8	7.01, s			
9	2.80, 2H, s	2.77, 2H, m	2.73, 2H, m	1.54, m; 2.40, m
10	2.80, 2H, s	2.77, 2H, m	2.73, 2H, m	2.68, m; 2.86, m
11	2.24, 3H, s	2.01, 3H, s	2.17, 3H, s	2.02, 3H, s
12	2.35, 3H, s	2.01, 3H, s		1.93, 3H, s
13	6.95, dd (17.4, 10.9)	6.75, dd (16.7, 11.3)	7.03, dd (17.8, 11.0)	6.70, dd (18.1, 11.9)
14	5.69, dd (17.4, 1.5)	5.61, dd (16.7, 2.1)	5.04, dd (17.8, 1.4)	5.75, d (18.1, 2.1)
	5.29, dd (10.9, 1.5)	5.20, dd (11.3, 2.1)	5.42, dd (11.0, 1.4)	5.70, d (11.9, 2.1)
OH-2	4.84, br s	4.67, br s		5.54, br s
OH		4.78, br s		9.69, br s

[a] In $CDCl_3$. [b] In methanol-d_4. [c] In DMSO-d_6.

spectrum indicated the presence of an OH group (3284 cm^{-1}). The ^{1}H and ^{13}C NMR data (Tables 1 and 3) were similar to those of **1** except that signals of four benzylic protons at δ 2.80 (4H) in the ^{1}H NMR spectrum of **1** were replaced by two *o*-coupled protons (δ_H 7.68, d, J = 9.2 Hz and 7.86, d, J = 9.2 Hz; δ_C 126.7 and 122.5) in compound **5**. These data strongly suggested that **5** was a 9,10-dehydro derivative of **1**. Further confirmation of the structure as 9,10-dehydrojuncuenin A was provided by 2D NMR experiments such as the key ROESY and HMBC spectra shown in the Supporting Information (S18 and S19).

Compound **6** was obtained as a brown, amorphous powder and had the molecular formula $C_{18}H_{16}O_2$ as determined by HREIMS. The IR spectrum showed an OH absorption band at 3267 cm^{-1}. The detailed analysis of its NMR data (Tables 1 and 3) indicated that **6** was a 9,10-dehydro derivative of **2**, and such elucidation was further supported by its ROESY spectrum, hence establishing the structure of dehydrojuncuenin B as shown.

Compound **7** was obtained as yellow crystals (EtOAc). Its molecular formula $C_{18}H_{14}O_4$ was established by HREIMS. The IR spectrum showed two hydroxy groups at 3471 and 3282 cm^{-1} and a carbonyl group at 1616 cm^{-1}. Its ^{1}H and ^{13}C NMR data (Tables 1 and 3) suggested that **7** was also a phenanthrene analogue (δ_H 7.76, d, J = 9.2 Hz, H-10; δ_H 7.84, d, J = 9.2 Hz, H-9) and contained the same A ring as compounds **1**−**6**. In the ^{1}H NMR spectrum, an oxyethylene group (δ_H 3.54, 2H, t, J = 5.0 Hz and 4.74, 2H, t, J = 5.0 Hz) instead of a vinyl group was observed.

Table 3. ^{1}H NMR Data of Compounds **5**−**7** (300 MHz, δ in ppm, J in Hz)

no.	**5**[a]	**6**[b]	**7**[c]
3	7.15, d (8.9)	7.20, d (9.1)	7.26, d (9.1)
4	8.50, d (8.9)	8.20, d (9.1)	8.50, d (9.1)
5	8.67, s	8.01, s	8.02, s
8	7.62, s		
9	7.68, d (9.2)	7.96, d (9.6)	7.84, d (9.2)
10	7.86, d (9.2)	7.70, d (9.6)	7.76, d (9.2)
11	2.60, 3H, s	2.57, 3H, s	2.47, 3H, s
12	2.53, 3H, s	2.38, 3H, s	
13	7.15, dd (17.4, 10.9)	7.10, dd (15.9, 9.0)	3.54, t (5.0)
14	5.87, dd (17.4, 1.5)	5.40, dd (15.9, 2.5)	4.74, t (5.0)
	5.43, dd (10.9, 1.5)	5.80, dd (9.0, 2.5)	
OH	4.93, br s	8.42, br s	9.97, br s
OH		8.66, br s	10.94, br s

[a] In $CDCl_3$. [b] In acetone-d_6. [c] In DMSO-d_6.

The ROESY correlations of H-3/H-4, H-4/H-5, H_3-11/H-10, H-9/H_2-13, and H_2-13/H_2-14 revealed that C-6, C-7, and C-8 were substituted and the oxyethylene group was attached to C-8. The key HMBC cross-peaks from H-5 and H_2-13 to C-7 (δ_C 108.2) and from H_2-14 to the C-12 carbonyl function suggested that a lactone ring fused at C-7 and C-8 was formed. Thus, the remaining OH group was located at C-6. Thus, the structure of dehydrojuncuenin C was established as shown.

In summary, a total of 10 phenanthrenes and dihydrophenanthrenes were isolated from *J. setchuensis* in the first phytochemical investigation of this species. Characteristic for the new compounds were a methyl group at C-1, an OH group at C-2, and a vinyl functionality at C-6 or C-8, showing that compound **7** is considered to have a modified vinyl functionality at C-8. This was somewhat different from the known compounds reported from *J. setchuensis*, in which a vinyl group was observed only at C-5.

Experimental Section

General Experimental Procedures. The optical rotation was measured on a Perkin-Elmer 341 polarimeter. The melting points were determined by a XT-4 binocular microscope (Beijing Tech Instrument Co., China) and were not corrected. IR spectra were recorded on a Nicolet Magna FT-IR 750 spectrophotometer using KBr disks. NMR spectra were recorded on Bruker AM-300, AM-400, and INVOR-600 NMR spectrometers. The chemical shifts (δ) are given in ppm with TMS as internal standard, and coupling constants (J) are in Hz. EIMS and HREIMS spectra were recorded on the Finnigan MAT-95 mass spectrometer. ESIMS and HRESIMS spectra were recorded on a Micromass LC-MS-MS mass spectrometer. Column chromatographic separations were carried out by using silica gel (200−300 mesh, 300−400 mesh and H60, QingdaoHaiyang Chemical Group Corporation, People's Republic of China), MCI gel CHP20P (75−150 μm, Mitsubishi Chemical Industries, Japan), and Sephadex LH20 (Pharmacia Biotech AB, Uppsala, Sweden) as packing material. TLC was carried out on precoated silica gel GF_{254} plates (Yantai Chemical Industrials), and the TLC spots were viewed at 254 nm and visualized by 5% sulfuric acid in EtOH containing 10 mg/mL vanillin. Analytical HPLC was performed on a Waters 2690 instrument with a 996 PAD (photodiode array detector) and coupled with an Alltech ELSD 2000 detector. Chromatographic separation was carried out on a C-18 column (250 × 10.0 mm, 5 μm, Waters), using a solvent system comprised of H_2O and CH_3CN, with a flow rate of 3 mL/min.

Plant Material. The whole plants of *J. setchuensis* were collected in Jinxiu County, Guangxi Province, China, and identified by Professor Jing-Gui Shen of Shanghai Institute of Material Medica. A voucher specimen (20071024) was deposited at the herbarium of Shanghai Institute of Materia Medica, Chinese Academy of Sciences.

Extraction and Isolation. Air-dried whole plants (9.5 kg) of *J. setchuensis* were ground into a powder and extracted with EtOH (35 L) at room temperature three times (three days each). After filtration and evaporation of the percolate under reduced pressure, the combined EtOH extract (415 g) was suspended in H_2O (1 L) and then partitioned successively with EtOAc (3 × 1 L) and *n*-BuOH (3 × 1 L), affording EtOAc (216.6 g) and *n*-BuOH (61.9 g) extracts.

The EtOAc extract (216.6 g) was subjected to column chromatography (CC) over silica gel (2.3 kg) eluted with petroleum ether (PE)/acetone (from 6:1 to 1:1) to give eight fractions 1−8. Fraction 3 (16.3 g) was further separated with silica gel eluting with PE/isopropyl ether gradients (10:1 to 1:1). The PE/isopropyl ether (5:1) fraction gave **1** as an amorphous powder (52 mg). The PE/isopropyl ether (2:1) fraction was subjected to a Sephadex LH-20 column eluted with $CHCl_3$/MeOH (1:1) to give **5** (333 mg). Fraction 4 (70.5 g) was dissolved in $CHCl_3$ and recrystallized to give white needles of **2** (21 g). The mother liquor (34 g) was subjected to CC over MCI gel eluting with EtOH/H_2O (30% to 100%). The subfraction obtained by eluting with 80% EtOH was resolved on Sephadex LH-20 ($CHCl_3$/MeOH, 1:1) and then purified by preparative TLC, giving **6** (19 mg). Fraction 5 (10.2 g) was subjected to CC over MCI gel eluting with EtOH/H_2O (40% to 100%). The 60% EtOH subfraction gave small yellow cubic crystals of **4** (76 mg) crystallized from EtOAc. The 80% EtOH subfraction was subjected to silica gel column (200−300 mesh, 80 g) chromatography eluting with PE/acetone (7:2) and gave yellow needles of **7** (28 mg) from EtOAc. Fraction 6 (12.1 g) was subjected to MCI gel chromatography eluting with EtOH/H_2O (40% to 100%). The 60% EtOH subfraction was then separated with a Sephadex LH-20 column ($CHCl_3$:MeOH, 1:1) and afforded **3** (42 mg).

Juncuenin A (1): amorphous powder; IR (KBr) ν_{max} 3320, 2949, 2889, 1626, 1591, 1554, 1481, 1337, 1279, 1072, 889, 820 cm^{-1}; ^{13}C and ^{1}H NMR data see Tables 1 and 2; ESIMS m/z 251.1 $[M + H]^+$; EIMS m/z 250 $[M]^+$ (100), 249 (10), 235 (22), 234 (11), 220 (14), 202 (10), 189 (9), 178 (5), 165 (6), 149 (50), 101 (7); HREIMS m/z 250.1358 $[M]^+$ (calcd for $C_{18}H_{18}O$, 250.1358).

Juncuenin B (2): white needles ($CHCl_3$); mp 154.0−155.5 °C; IR (KBr) ν_{max} 3373, 3240, 2955, 2829, 1632, 1591, 1468, 1281, 810 cm^{-1}; ^{13}C and ^{1}H NMR data see Tables 1 and 2; ESIMS m/z 267.1 $[M + 1]^+$; EIMS m/z 266 $[M]^+$ (100), 265 (11), 251 (83), 237 (16), 223 (10), 208 (9), 189 (9), 178 (8), 165 (9), 109 (9), 71 (5); HREIMS m/z 266.1302 $[M]^+$ (calcd for $C_{18}H_{18}O_2$, 266.1307).

Juncuenin C (3): white, amorphous powder; IR (KBr) ν_{max} 3439, 2945, 2567, 1641, 1583, 1562, 1454, 1273, 812 cm^{-1}; ^{13}C and ^{1}H NMR data see Tables 1 and 2; ESIMS m/z 297.1 $[M + H]^+$; EIMS m/z 296 $[M]^+$ (26), 295 (6), 278 (18), 252 (100), 237 (50), 236 (14), 207 (17), 178 (9), 165 (11); HREIMS m/z 296.1050 $[M]^+$ (calcd for $C_{18}H_{16}O_4$, 296.1048).

Juncuenin D (4): yellow cubic crystals (EtOH); $[\alpha]^{23}_D$ 0 (c 0.16, MeOH); IR (KBr) ν_{max} 3292, 3234, 2953, 2920, 1645, 1614, 1581, 1456, 1256, 816 cm^{-1}; ^{13}C and ^{1}H NMR data see Tables 1 and 2; ESIMS m/z 305.1 $[M + Na]^+$; HRESIMS m/z 305.1143 $[M + Na]^+$ (calcd for $C_{18}H_{18}O_3Na$, 305.1154).

Dehydrojuncuenin A (5): white, amorphous powder; IR (KBr) ν_{max} 3284, 3084, 2945, 1618, 1574, 1510, 1406, 1059, 887, 806 cm^{-1}; ^{13}C and ^{1}H NMR data see Tables 1 and 3; ESIMS m/z 249.1 $[M + H]^+$; EIMS m/z 248 $[M]^+$ (100), 233 (18), 202 (16), 189 (11), 149 (6), 101 (13), 57 (11); HREIMS m/z 248.1210 $[M]^+$ (calcd for $C_{18}H_{16}O$, 248.1202).

Dehydrojuncuenin B (6): brown, amorphous powder; IR (KBr) ν_{max} 3267, 2963, 2922, 1616, 1599, 1581, 1475, 1261, 822 cm^{-1}; ^{13}C and ^{1}H NMR data see Tables 1 and 3; ESIMS m/z 265.0 $[M + H]^+$; EIMS m/z 264 $[M]^+$ (100), 249 (77), 234 (28), 202 (8), 149 (12), 101 (11), 57 (10); HREIMS m/z 264.1155 $[M]^+$ (calcd $C_{18}H_{16}O_2$, 264.1150).

Dehydrojuncuenin C (7): yellow needles (EtOAc); IR (KBr) ν_{max} 3471, 3282, 2945, 2893, 1616, 1595, 1473, 1390, 1059, 820 cm^{-1}; ^{13}C and ^{1}H NMR date see Tables 1 and 3; ESIMS m/z 611.1 $[2M + Na]^+$; EIMS m/z 294 $[M]^+$ (69), 264 (100), 249 (70), 234 (24), 177 (16), 149 (12), 101 (11), 71 (6); HREIMS m/z 294.0910 $[M]^+$ (calcd for $C_{18}H_{14}O_4$, 294.0892).

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Supporting Information Available: ^{1}H and ^{13}C NMR spectra for **1**−**7**, HMQC spectrum for **4**, HMBC spectra for **1**, **3**, **4**, **5**, and **7**, ROESY spectra for **1**, **2**, **3**, **5**, **6**, and **7**, HMBC correlations indicated for **1**, **3**, **4**, **5**, and **7**, ROESY correlations indicated for **1**, **2**, **3**, **5**, **6**, and **7**, and chemical structures of three known compounds are available free of charge via the Internet at http://pubs.acs.org.

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