

## Colossolactones, New Triterpenoid Metabolites from a Vietnamese Mushroom *Ganoderma colossus*<sup>§</sup>

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Seven new triterpenoid metabolites (colossolactones; **1–7**) were isolated from a fruiting body of *Ganoderma colossus*, and their structures were determined by MS and NMR methods.

The fungal family *Ganodermataceae* is represented by more than 200 species, which mostly occur in subtropical and tropical regions.<sup>1</sup> Some members of the *Ganodermataceae*, such as *Ganoderma lucidum* and *Ganoderma applanatum*, are used in Asian folk medicine for treatment of some diseases.<sup>2</sup> Numerous terpenoid compounds have been reported as components of these medically important species.<sup>3–5</sup> However, little information is available on metabolites of other representatives of these genera. In this paper we report the structure of seven new triterpenoid metabolites, colossolactones A–G (**1–7**), isolated from *Ganoderma colossus* Donk (*Ganodermataceae*) (Chart 1).

A fruiting body of *Ganoderma colossus* (ca. 200 g wet weight) was collected on a trunk of *Delonix regia* (Fabaceae) in Hue city, Thua Thien-Hue province, Vietnam. It was characterized taxonomically as a representative of the *Ganoderma* family and species *G. colossus* (synonymous: *Polyporus colossus*, *Dendrophagus colossus*) due to the reticulated cell wall structure and other morphological features.<sup>4</sup>

For the isolation of metabolites **1–7** the lyophilized fruiting body of *G. colossus* was extracted with 1 L of 1:1 CHCl<sub>3</sub>/MeOH and subsequently with 1 L of ethyl acetate. Compounds **1–7** were isolated from the residue of the evaporated extract by several subsequent chromatographic steps. The molecular formulas were determined by HREIMS showing [M]<sup>+</sup> and respective diagnostic fragments. The IR spectra attested to the presence of carbonyl groups due to absorbances in the range 1696–1717 cm<sup>-1</sup>. UV absorbances ( $\lambda_{\max}$  326–328 nm) of compounds **4–7** suggested the occurrence of a triene–lactone chromophore.

The structures of the new sterol-type metabolites **1–7** were determined conclusively by 1D and 2D <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy. The proton broad-band decoupled <sup>13</sup>C NMR and DEPT spectra suggested the number and binding type of the skeleton carbons and the substitution pattern. A prominent feature was the occurrence of lactone carbonyls in **2–7** and conjugated double bonds in **4–7**. The <sup>1</sup>H–<sup>1</sup>H COSY and TOCSY spectra were particularly helpful for the assignment of overlapping proton signals of the individual rings. The sequence of carbon and hydrogen atoms was settled by heteronuclear 2D NMR experiments (HSQC, HMBC). The observable C–H long-range couplings

(HMBC) of the methyl groups at the quaternary ring carbons were attributable to a sterol-type ring system. Assignment of the relative stereochemistry of **1–7** was supported by the observable NOE correlations of the methyl protons with neighboring protons of the rings and other methyl substituents in the NOESY and ROESY spectra. Measurements of optical rotation confirmed the chiral nature of **1–7**. Assignments of NMR signals are given in the Experimental Section.

Structures such as **3–7** containing a seven-membered lactone as the triterpenoid ring A and a  $\delta$ -lactone side chain at C-17 have not been reported previously for fungal metabolites. However, representatives of this structural type such as schisanlactones, kadsulactone A, kadsudilactone, and lancilactones were isolated from the stems and roots of plants such as *Schisandra* sp., *Kadsura heteroclita*, *K. coccinea*, and *K. lancilimba*, used as folk medicines for the treatment of rheumatism, stomachache, and enterogastritis.<sup>6–9</sup>

Colossolactones (**1–7**) displayed no antimicrobial activity against a spectrum of bacteria and fungi but moderate cytotoxicity against L-929, K-562, and HeLa cells with IC<sub>50</sub> values ranging from 15 to 35  $\mu$ g/mL. Moreover, they inhibited 3 $\alpha$ -hydroxysteroid dehydrogenase (3 $\alpha$ -HSD) in concentrations comparable to indomethacin as standard drug, suggesting antiinflammatory properties for **1–7**.<sup>10</sup>

### Experimental Section

**General Experimental Procedures.** HREIMS were taken with an AMD 402 double-focusing mass spectrometer (AMD Intetra, Harpstedt, Germany). ESIMS was measured with a Quattro triple quadrupole instrument (VG Biotech, Altrincham, England) and HRESIMS with MAT 95 XL (Finnigan, Bremen, Germany). IR spectra were recorded on a Shimadzu IR-470 spectrophotometer. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Bruker AVANCE DRX 500 spectrometer using TMS as internal standard. Optical rotations were measured with a Propol instrument (Dr. Kernchen Optical Works, Seelze, Germany). Melting points are uncorrected.

**Organism.** The fruiting body of *Ganoderma colossus* was characterized by the following morphological features: basidiocarps annual, up to 35 cm diameter, 5–8 cm thickness; context structure spongy, white when fresh, chamoid when dried; basidiospore ovoid, yellow, bitunicate; space well structure has reticulation clearly; basidiospore size 9–17  $\times$  14–20  $\mu$ m. A specimen was deposited in the fungal culture collection of the Mycological Center, University Hanoi, Vietnam.

**Extraction and Isolation.** The fruiting body (200 g wet weight) was extracted with 1 L of CHCl<sub>3</sub>/MeOH and, subse-

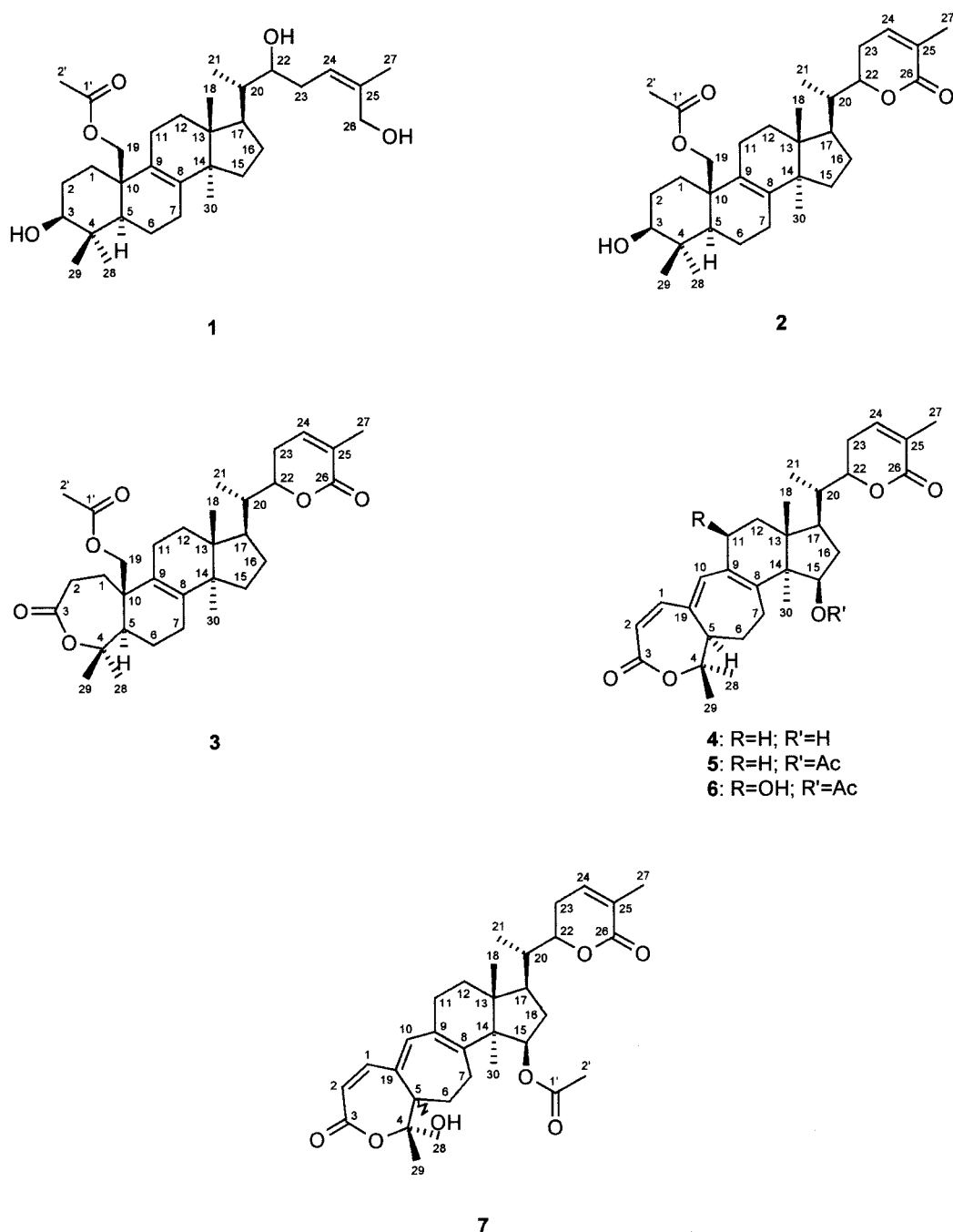
<sup>§</sup> Dedicated to Prof. Günter Adam on the occasion of his 65th birthday.

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Chart 1



quently, 1 L of ethyl acetate. The combined extracts were evaporated, and the residue (1.6 g) was subjected to column chromatography on silica gel 60 (Merck, 0.063–0.1 mm;  $\text{CHCl}_3$ ,  $\text{CHCl}_3/\text{MeOH}$ , 95:5;  $\text{CHCl}_3/\text{MeOH}$ , 9:1). The triterpenoid fractions (spotted on a TLC sheet) were detected by bluish-violet staining with 1% vanillin in concentrated  $\text{H}_2\text{SO}_4$ . Further purification was performed by repeated preparative TLC on silica gel aluminum sheets (Merck,  $20 \times 20$  cm, 0.2 mm,  $\text{CHCl}_3/\text{MeOH}$ , 9:1). Compounds 1–7 were obtained as colorless solids in amounts of 15–35 mg.

**Colossolactone A (1):** colorless solid ( $\text{CHCl}_3$ ); mp 135–137 °C;  $[\alpha]_{\text{D}}^{20} +50.2^\circ$  ( $c$  0.25, MeOH); UV–vis (MeCN  $\lambda_{\text{max}}$  222 nm); IR (KBr)  $\nu_{\text{max}}$  3425, 2925, 1705, 1451, 1371, 1250, 1137, 1022  $\text{cm}^{-1}$ ;  $R_f$  (TLC, Si gel) 0.55, eluent  $\text{CHCl}_3/\text{MeOH}$  (95:5);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  5.49 (1H, t,  $J$  = 7.6 Hz, H-24), 4.27 (1H, d,  $J$  = 11.7 Hz, H-26a), 3.99 (1H, d,  $J$  = 11.4 Hz, H-19a), 3.90 (1H, d,  $J$  = 11.7 Hz, H-26b), 3.66 (1H, m, H-22), 3.55 (1H, d,  $J$  = 11.4 Hz, H-19b), 3.26 (1H, dd,  $J$  = 11.7 Hz, 4.4 Hz, H-3), 2.47 (1H, m, H-23a), 2.13 (2H, m, H-7), 2.12 (2H,

m, H-11), 2.00 (3H, s, H-2'), 1.99 (1H, m, H-6a, 1H, m, H-16a), 1.96 (1H, m, H-23b), 1.87 (1H, m, H-17), 1.85 (1H, m, H-1a, 3H, s, br, H-27), 1.84 (1H, m, H-4a), 1.72 (1H, m, H-12b), 1.70 (1H, m, H-6b), 1.69 (1H, m, H-2a), 1.66 (1H, m, H-15a), 1.52 (1H, m, H-2b), 1.45 (1H, m, H-20), 1.39 (1H, m, H-16b), 1.31 (1H, m, H-15b), 1.27 (1H, m, H-1b), 1.15 (1H, dd,  $J$  = 13.6 Hz, 2.8 Hz, H-5), 1.03 (3H, s, H-28), 0.97 (3H, s, H-30), 0.93 (3H, s, H-29), 0.92 (3H, d,  $J$  = 6.6 Hz, H-21), 0.77 (3H, s, H-18);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  170.54 (s, C-1'), 137.86 (s, C-8, C-25), 130.37 (s, C-9), 125.38 (s, C-24), 78.86 (d, C-3), 72.73 (d, C-22), 65.83 (t, C-19), 61.39 (t, C-26), 50.42 (s, C-14), 50.11 (d, C-5), 47.08 (d, C-17), 44.38 (s, C-13), 42.18 (s, C-10), 41.62 (d, C-20), 39.04 (s, C-4), 33.98 (t, C-23), 32.59 (t, C-1), 30.96 (t, C-12), 30.71 (t, C-15), 28.36 (q, C-28), 27.96 (t, C-2), 27.46 (t, C-16), 26.20 (t, C-7), 24.65 (q, C-30), 22.35 (q, C-27), 21.97 (t, C-11), 21.09 (q, C-2'), 17.76 (t, C-6), 16.56 (q, C-18), 15.51 (q, C-29), 12.02 (q, C-21); EIMS  $m/z$  516.2  $[\text{M}]^+$  (5), 425.2 (80), 329.2 (100); HREIMS  $m/z$  425.3406 ( $[\text{M} - \text{H}_2\text{O} - \text{C19 side chain}]^+$

(calcd for C<sub>29</sub>H<sub>45</sub>O<sub>2</sub>, 425.3422), 329.2922 (calcd for C<sub>23</sub>H<sub>37</sub>O, 329.2846).

**Colossolactone B (2):** colorless solid (CHCl<sub>3</sub>); mp 116–118 °C; [α]<sub>D</sub><sup>20</sup> +54.4° (c 0.32, MeOH); UV–vis (MeCN) λ<sub>max</sub> 232 nm; IR (KBr) ν<sub>max</sub> 3455, 2945, 1701, 1450, 1373, 1342, 1235, 1136, 1089, 1030 cm<sup>-1</sup>; R<sub>f</sub> (TLC, Si gel) 0.65, eluent CHCl<sub>3</sub>/MeOH (95:5); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 6.60 (1H, m, H-24), 4.48 (1H, dd, *J* = 13.2 Hz, C-14, H-22), 4.34 (1H, d, *J* = 11.3 Hz, H-19a), 4.16 (1H, d, *J* = 11.3 Hz, H-19b), 3.28 (1H, dd, *J* = 11.7 Hz, 4.4 Hz, H-3), 2.56 (1H, m, H-23a), 2.14 (2H, m, H-11), 2.13 (2H, m, H-7), 2.12 (1H, m, H-17), 2.04 (1H, m, H-16a), 2.01 (1H, m, H-1a, 3H, s, H-2'), 1.98 (1H, m, H-23b), 1.91 (3H, s, br, H-27), 1.82 (1H, m, H-12a), 1.71 (1H, m, H-2a), 1.70 (2H, m, H-6), 1.66 (1H, m, H-12b), 1.62 (1H, m, H-15a), 1.59 (1H, m, H-2b), 1.53 (1H, m, H-20), 1.33 (1H, m, H-16b), 1.28 (1H, m, H-1b), 1.25 (1H, m, H-15b), 1.21 (1H, dd, *J* = 12.0 Hz, 4.4 Hz, H-5), 1.03 (3H, d, *J* = 6.6 Hz, H-21), 1.02 (3H, s, H-28), 0.93 (3H, s, H-30), 0.85 (3H, s, H-29), 0.71 (3H, s, H-18); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ 171.07 (s, C-1'), 166.59 (s, C-26), 139.62 (d, C-24), 137.14 (s, C-8), 131.68 (s, C-9), 128.22 (s, C-25), 80.24 (d, C-22), 78.66 (d, C-3), 67.78 (t, C-19), 50.24 (d, C-5, s, C-14), 45.77 (d, C-17), 44.44 (s, C-13), 40.42 (d, C-20), 39.63 (s, C-10), 38.90 (s, C-4), 31.23 (t, C-1), 31.01 (t, C-12), 30.66 (t, C-15), 28.12 (q, C-28), 27.87 (t, C-23), 27.72 (t, C-2), 27.62 (t, C-16), 25.66 (t, C-7), 24.22 (q, C-30), 23.00 (t, C-11), 21.13 (q, C-2'), 17.62 (t, C-6), 17.11 (q, C-27), 15.64 (q, C-18), 15.53 (q, C-29), 13.30 (q, C-21); ESIMS<sup>+</sup> *m/z* 535 [M + Na]<sup>+</sup> (100); ESIMS<sup>-</sup> *m/z* 511 [M - H]<sup>-</sup> (39); HREIMS *m/z* 512.3478 (calcd for C<sub>32</sub>H<sub>48</sub>O<sub>5</sub>, 512.3504).

**Colossolactone C (3):** colorless solid (CHCl<sub>3</sub>); mp 128–130 °C; [α]<sub>D</sub><sup>20</sup> +64.5° (c 0.64, CHCl<sub>3</sub>); UV–vis (MeCN) λ<sub>max</sub> 233 nm; IR (KBr) ν<sub>max</sub> 3445, 2945, 1709, 1452, 1372, 1341, 1249, 1195, 1139, 1077, 1044 cm<sup>-1</sup>; R<sub>f</sub> (TLC, Si gel) 0.70, eluent CHCl<sub>3</sub>/MeOH (95:5); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 6.60 (1H, m, H-24), 4.48 (1H, dd, *J* = 13.2 Hz, 3.5 Hz, H-22), 4.42 (1H, d, *J* = 12.0 Hz, H-19a), 4.24 (1H, d, *J* = 12.0 Hz, H-19b), 2.59 (1H, m, H-1a), 2.56 (1H, m, H-23a), 2.28 (1H, m, H-2a), 2.21 (1H, m, H-2b), 2.12 (1H, m, H-17), 2.05 (1H, m, H-7a), 2.04 (1H, m, H-16a), 2.00 (3H, s, H-2'), 1.98 (1H, m, H-23b), 1.96 (2H, m, H-11), 1.95 (1H, m, H-7b), 1.90 (3H, s, br, H-27), 1.87 (1H, m, H-12a), 1.70 (1H, m, H-6a), 1.69 (1H, m, H-12b), 1.66 (1H, m, H-1b), 1.64 (1H, m, H-15a), 1.54 (1H, m, H-20), 1.50 (1H, m, H-5, 1H, m, H-6b), 1.35 (1H, m, H-16b), 1.32 (3H, s, H-28), 1.28 (1H, m, H-15b), 1.20 (3H, s, H-29), 1.01 (3H, d, *J* = 6.6 Hz, H-21), 0.98 (3H, s, H-30), 0.74 (3H, s, H-18); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ 178.98 (s, C-3), 170.66 (s, C-1), 166.67 (s, C-26), 143.27 (s, C-8), 139.72 (d, C-24), 128.17 (s, C-25), 126.14 (s, C-9), 80.29 (d, C-22), 75.25 (s, C-4), 67.26 (t, C-19), 51.50 (s, C-14), 47.89 (s, C-5), 45.85 (d, C-17), 45.41 (s, C-10), 44.26 (s, C-13), 40.34 (s, C-20), 33.69 (q, C-28), 31.26 (t, C-15), 30.98 (t, C-12), 28.72 (t, C-1), 28.44 (t, C-2), 27.79 (t, C-23), 27.37 (t, C-16), 26.72 (t, C-7), 26.08 (q, C-29), 24.94 (q, C-30), 23.89 (t, C-6), 21.07 (q, C-2'), 20.84 (t, C-11), 17.08 (q, C-27), 15.91 (q, C-18), 13.39 (q, C-21); ESIMS<sup>+</sup> *m/z* 527 [M + H]<sup>+</sup> (10); HREIMS *m/z* 526.3274 (calcd for C<sub>32</sub>H<sub>46</sub>O<sub>6</sub>, 526.3296).

**Colossolactone D (4):** colorless solid (CHCl<sub>3</sub>); mp 122–125 °C; [α]<sub>D</sub><sup>20</sup> +72.5° (c 0.20, MeOH); UV–vis (MeOH) λ<sub>max</sub> 220, 328 nm; IR (KBr) ν<sub>max</sub> 3435, 2925, 1707, 1682, 1597, 1569, 1446, 1379, 1341, 1286, 1237, 1206, 1181, 1131, 1047 cm<sup>-1</sup>; R<sub>f</sub> (TLC, Si gel) 0.85, eluent CHCl<sub>3</sub>/MeOH (95:5); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 6.66 (1H, d, *J* = 12.2 Hz, H-1), 6.62 (1H, m, H-24), 6.23 (1H, s, H-19), 5.82 (1H, d, *J* = 12.2 Hz, H-2), 4.49 (1H, dd, *J* = 13.2 Hz, 2.8 Hz, H-22), 4.05 (1H, d, *J* = 7.2 Hz, H-15), 2.64 (1H, ddd, *J* = 15.1 Hz, 9.1 Hz, 7.9 Hz, H-16a), 2.59 (1H, m, H-23a), 2.56 (1H, m, H-5), 2.42 (1H, m, H-6a), 2.34 (1H, m, H-7a), 2.30 (1H, m, H-6b), 2.26 (1H, m, H-11a), 2.14 (1H, m, H-7b, 1H, m, H-17), 2.05 (1H, m, H-11b), 2.02 (1H, m, H-23b), 1.92 (3H, s, br, H-27), 1.90 (1H, m, H-12a), 1.76 (1H, m, H-12b), 1.73 (1H, m, H-20), 1.55 (3H, s, H-29), 1.46 (1H, m, H-16b), 1.42 (3H, s, H-28), 1.09 (3H, s, H-18, 3H, d, *J* = 6.6 Hz, H-21), 1.08 (3H, s, H-30); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ 167.10 (s, C-3), 166.42 (s, C-26), 147.56 (s, C-8), 143.69 (d, C-1), 142.98 (d, C-19), 139.60 (d, C-24), 139.10 (s, C-10), 130.52 (s, C-9), 128.25 (s, C-25), 118.00 (d, C-2), 80.53 (s, C-4), 80.00 (d, C-22), 76.21 (d, C-15), 56.43 (s, C-14), 49.00 (d, C-5), 45.59 (d, C-17),

43.68 (s, C-13), 40.56 (t, C-16), 40.00 (d, C-20), 38.65 (t, C-6), 31.41 (t, C-12), 28.97 (q, C-28), 27.81 (t, C-23), 27.63 (t, C-7), 26.87 (t, C-11), 26.61 (q, C-30), 26.33 (q, C-29), 17.25 (q, C-18), 17.11 (q, C-27), 13.41 (q, C-21); ESIMS<sup>+</sup> *m/z* 503 [M + Na]<sup>+</sup> (100); ESIMS<sup>-</sup> *m/z* 479 [M - H]<sup>-</sup> (100); HREIMS *m/z* 480.2871 (calcd for C<sub>30</sub>H<sub>40</sub>O<sub>5</sub>, 480.2878).

**Colossolactone E (5):** colorless solid (CHCl<sub>3</sub>); mp 141–146 °C; [α]<sub>D</sub><sup>20</sup> +80.6° (c 0.40, MeOH); UV–vis (MeOH) λ<sub>max</sub> 232, 326 nm; IR (KBr) ν<sub>max</sub> 3430, 2935, 1708, 1684, 1597, 1569, 1446, 1372, 1341, 1284, 1250, 1207, 1130, 1043 cm<sup>-1</sup>; R<sub>f</sub> (TLC, Si gel) 0.80, eluent CHCl<sub>3</sub>/MeOH (95:5); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 6.66 (1H, d, *J* = 12.1 Hz, H-1), 6.60 (1H, m, H-24), 6.22 (1H, s, H-19), 5.82 (1H, d, *J* = 12.1 Hz, H-2), 4.87 (1H, dd, *J* = 7.4 Hz, 1.3 Hz, H-15), 4.43 (1H, ddd, *J* = 13.6 Hz, 3.1 Hz, 1.3 Hz, H-22), 2.72 (1H, ddd, *J* = 15.4 Hz, 8.8 Hz, 7.4 Hz, H-16a), 2.56 (1H, m, H-23a), 2.52 (1H, m, H-5), 2.35 (1H, m, H-6a), 2.26 (1H, m, H-11a), 2.20 (1H, m, H-6b), 2.17 (1H, m, H-17), 2.11 (1H, m, H-11b), 2.02 (1H, m, H-7a, 1H, m, H-23b), 1.96 (1H, m, H-7b), 1.95 (3H, s, H-2'), 1.91 (1H, m, H-12a, 3H, s, br, H-27), 1.79 (1H, m, H-12b), 1.64 (1H, m, H-20), 1.54 (3H, s, H-29), 1.40 (3H, s, H-28), 1.38 (1H, m, H-16b), 1.14 (3H, s, H-30), 1.08 (3H, d, *J* = 6.6 Hz, H-21), 1.01 (3H, s, H-18); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ 170.36 (s, C-1'), 166.98 (s, C-3), 166.26 (s, C-26), 147.03 (s, C-8), 143.63 (d, C-1), 142.76 (d, C-19), 139.35 (d, C-24), 139.26 (d, C-10), 129.87 (s, C-9), 128.36 (s, C-25), 118.13 (d, C-2), 80.52 (s, C-4), 79.77 (d, C-22), 78.01 (d, C-15), 54.89 (s, C-14), 49.02 (d, C-5), 45.30 (d, C-17), 44.29 (s, C-13), 39.94 (d, C-20), 38.63 (t, C-16), 38.39 (t, C-6), 31.11 (t, C-12), 28.83 (q, C-28), 27.76 (t, C-23), 27.22 (t, C-7), 26.79 (t, C-11), 26.35 (q, C-29, C-30), 21.42 (q, C-2'), 17.06 (q, C-27), 16.74 (q, C-18), 13.31 (q, C-21); ESIMS<sup>+</sup> *m/z* 545 [M + Na]<sup>+</sup> (100); ESIMS<sup>-</sup> *m/z* 521 [M - H]<sup>-</sup> (12); HREIMS *m/z* 522.2976 (calcd for C<sub>32</sub>H<sub>42</sub>O<sub>6</sub>, 522.2983).

**Colossolactone F (6):** colorless solid (CHCl<sub>3</sub>); mp 134–136 °C; [α]<sub>D</sub><sup>20</sup> +26.5° (c 0.20, MeOH); UV–vis (MeOH) λ<sub>max</sub> 233, 326 nm; IR (KBr) ν<sub>max</sub> 3440, 2930, 1717, 1705, 1686, 1572, 1447, 1379, 1342, 1283, 1245, 1209, 1181, 1130, 1084, 1043, 1024 cm<sup>-1</sup>; R<sub>f</sub> (TLC Si gel) 0.92, eluent CHCl<sub>3</sub>/MeOH (95:5); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 6.72 (1H, d, *J* = 12.1 Hz, H-1), 6.70 (1H, s, H-19), 6.61 (1H, m, H-24), 5.85 (1H, d, *J* = 12.1 Hz, H-2), 4.88 (1H, d, *J* = 7.2 Hz, H-15), 4.42 (1H, dd, *J* = 13.2 Hz, 3.2 Hz, H-22), 4.24 (1H, d, *J* = 6.9 Hz, H-11), 2.76 (1H, ddd, *J* = 15.4 Hz, 8.8 Hz, 7.6 Hz, H-16a), 2.58 (1H, m, H-5), 2.57 (1H, m, H-23a), 2.35 (1H, m, H-6a), 2.33 (1H, dd, *J* = 15.1 Hz, 7.2 Hz, H-12a), 2.19 (1H, m, H-17), 2.17 (1H, m, H-6b), 2.06 (2H, m, H-7), 2.03 (1H, m, H-12b), 2.00 (1H, m, H-23b), 1.97 (3H, s, H-2'), 1.91 (3H, s, br, H-27), 1.68 (1H, m, H-20), 1.53 (3H, s, H-29), 1.43 (1H, m, H-16b), 1.39 (3H, s, H-28), 1.19 (3H, s, H-18), 1.11 (3H, d, *J* = 6.6 Hz, H-21), 1.10 (3H, s, H-30); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ 170.26 (s, C-1'), 166.79 (s, C-3), 166.24 (s, C-26), 149.29 (s, C-8), 144.24 (d, C-1), 141.03 (d, C-19), 140.11 (d, C-10), 139.38 (d, C-24), 131.79 (s, C-9), 128.36 (s, C-25), 118.50 (d, C-2), 80.64 (s, C-4), 79.62 (d, C-22), 77.57 (d, C-15), 67.68 (d, C-11), 55.58 (s, C-14), 49.18 (d, C-5), 45.13 (d, C-17), 43.17 (s, C-13), 42.46 (t, C-12), 39.86 (d, C-20), 38.50 (t, C-16), 37.28 (t, C-6), 28.13 (q, C-28), 27.72 (t, C-23), 27.21 (t, C-7), 26.71 (q, C-29), 25.40 (q, C-30), 21.38 (q, C-2'), 18.87 (q, C-18), 17.07 (q, C-27), 13.24 (q, C-21); ESIMS<sup>+</sup> *m/z* 561 [M + Na]<sup>+</sup> (100); HREIMS *m/z* 538.2891 (calcd for C<sub>32</sub>H<sub>42</sub>O<sub>7</sub>, 538.2932).

**Colossolactone G (7):** colorless solid (CHCl<sub>3</sub>); mp 143–145 °C; [α]<sub>D</sub><sup>20</sup> +23.5° (c 0.10, MeOH); UV–vis (MeOH) λ<sub>max</sub> 235, 326 nm; IR (KBr) ν<sub>max</sub> 3430, 2935, 1696, 1685, 1576, 1435, 1378, 1250, 1206, 1181, 1134, 1044, 1023 cm<sup>-1</sup>; R<sub>f</sub> (TLC Si gel) 0.85, eluent CHCl<sub>3</sub>/MeOH (95:5); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 6.93 (1H, d, *J* = 9.8 Hz, H-1), 6.60 (1H, m, H-24), 6.24 (1H, s, H-19), 5.89 (1H, d, *J* = 9.8 Hz, H-2), 4.85 (1H, d, *J* = 7.2 Hz, H-15), 4.43 (1H, dd, *J* = 13.2 Hz, 2.5 Hz, H-22), 2.74 (1H, ddd, *J* = 15.4 Hz, 8.5 Hz, 7.6 Hz, H-16a), 2.55 (1H, m, H-23a), 2.44 (1H, m, H-6a), 2.29 (1H, m, H-6b), 2.27 (2H, m, H-11), 2.17 (1H, m, H-17), 2.10 (2H, m, H-7), 2.03 (3H, s, H-2'), 2.00 (1H, m, H-23b), 1.94 (1H, m, H-12a), 1.92 (3H, s, br, H-27), 1.80 (1H, m, H-12b), 1.66 (1H, m, H-20), 1.41 (1H, m, H-16b), 1.24 (3H, s, H-29), 1.18 (3H, s, H-28), 1.08 (3H, d, *J* = 6.0 Hz, H-21), 1.07 (3H, s, H-18), 0.99 (3H, s, H-30); <sup>13</sup>C NMR (CDCl<sub>3</sub>,

125 MHz)  $\delta$  170.06 (s, C-1'), 166.20 (s, C-26), 163.91 (s, C-3), 149.46 (s, C-8), 147.89 (d, C-1), 139.42 (d, C-19), 139.31 (d, C-24), 132.85 (d, C-10), 128.38 (s, C-25), 127.62 (s, C-9), 116.71 (d, C-2), 92.64 (s, C-5), 79.70 (d, C-22), 78.39 (d, C-15), 77.52 (s, C-4), 55.14 (s, C-14), 45.73 (d, C-17), 44.27 (s, C-13), 44.09 (t, C-6), 39.80 (d, C-20), 38.37 (t, C-16), 31.09 (t, C-12), 28.01 (t, C-11), 27.70 (t, C-23), 26.79 (t, C-7), 24.92 (q, C-28), 24.81 (q, C-29), 24.41 (q, C-30), 21.31 (q, C-2'), 17.07 (q, C-27), 16.81 (q, C-18), 13.29 (q, C-21); ESIMS<sup>+</sup>  $m/z$  561 [M + Na]<sup>+</sup> (100); ESIMS<sup>-</sup>  $m/z$  537 [M - H]<sup>-</sup> (48); HRESIMS  $m/z$  561.2812 (calcd for C<sub>32</sub>H<sub>42</sub>O<sub>7</sub>Na, 561.2830).

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