# Three Novel Compounds from the Flowers of *Bombax malabaricum*

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Two unusual 9′-norneolignans, bombasin (**1**) and bombasin 4-*O*-*β*-glucoside (**2**), and a novel D-gulono-*γ*-lactone derivative, bombalin (**3**), were isolated from the flowers of *Bombax malabaricum*, together with the three known compounds dihydrodehydrodiconiferyl alcohol 4-*O*-*β*-D-glucopyranoside (**4**), *trans*-3-(*p*-coumaroyl)quinic acid (**5**), and neochlorogenic acid (**6**). Their structures were elucidated by extensive spectroscopic methods as well as chemical transformation. Compounds **1**–**3** were evaluated against the HGC-27 gastrointestinal and Hela cervical human cancer cell lines, but all were inactive in both lines ($IC_{50} > 50$ μM).

**Introduction.** – *Bombax malabaricum* DC. (Bombacaceae), a medium-sized deciduous tree, mainly grows in tropical areas such as Southern China, India and Northern Australia [1]. The whole plant of *B. malabaricum* has been used versatilely in both Chinese and Indian traditional medicine for the treatment of diarrhea, fever, chronic inflammation, and catarrhal affection [1–3]. Previous chemical investigations on this plant have led to the isolation and structural elucidation of flavonoids [3][4], terpenoids [5], and polysaccharide [6]. In our recent chemical research on the flowers of *B. malabaricum*, three new compounds, bombasin (**1**), bombasin 4-*O*-*β*-glucoside (**2**), and bombalin (**3**), were isolated. Also isolated were three known compounds **4**–**6**. Herein, we describe the isolation and structural elucidation of the new compounds.

**Results and Discussion.** – Compound **1** was obtained as a colorless gum. The molecular formula was determined as $C_{19}H_{20}O_6$, based on the HR-ESI-MS peak at *m*/*z* 367.1159 ($[M + Na]^+$; calc. 367.1158) and NMR data (*Table 1*). The IR absorption bands implied the presence of aromatic moieties (1591 and 1517 $cm^{-1}$), OH groups (3423 $cm^{-1}$), and a conjugated C=O group (1658 $cm^{-1}$).

Detailed analysis of the HMQC, $^1$H,$^1$H-COSY, HMBC, and NOESY data (*Fig. 1*) established the structure of **1** as (7*R*,8*S*)-1′-acetyl-7,8-dihydro-7-(4-hydroxy-3-methoxyphenyl)-8-(hydroxymethyl)-3′-methoxybenzofuran[1]), a novel 9′-norneolignan given the trivial name bombasin.

The $^1$H-NMR spectrum of **1** exhibited the signals of two benzene moieties, of which one was an *AX* system ($\delta$(H) 7.51 (*d*, *J* = 1.4, 1 H), and 7.64 (*d*, *J* = 1.4, 1 H)), and the other one an *ABX* system ($\delta$(H) 6.83 (*d*, *J* = 8.1, 1 H), 6.89 (*dd*, *J* = 8.1, 1.9, 1 H), and

[1]) Arbitrary numbering. For systematic names, see *Exper. Part.*

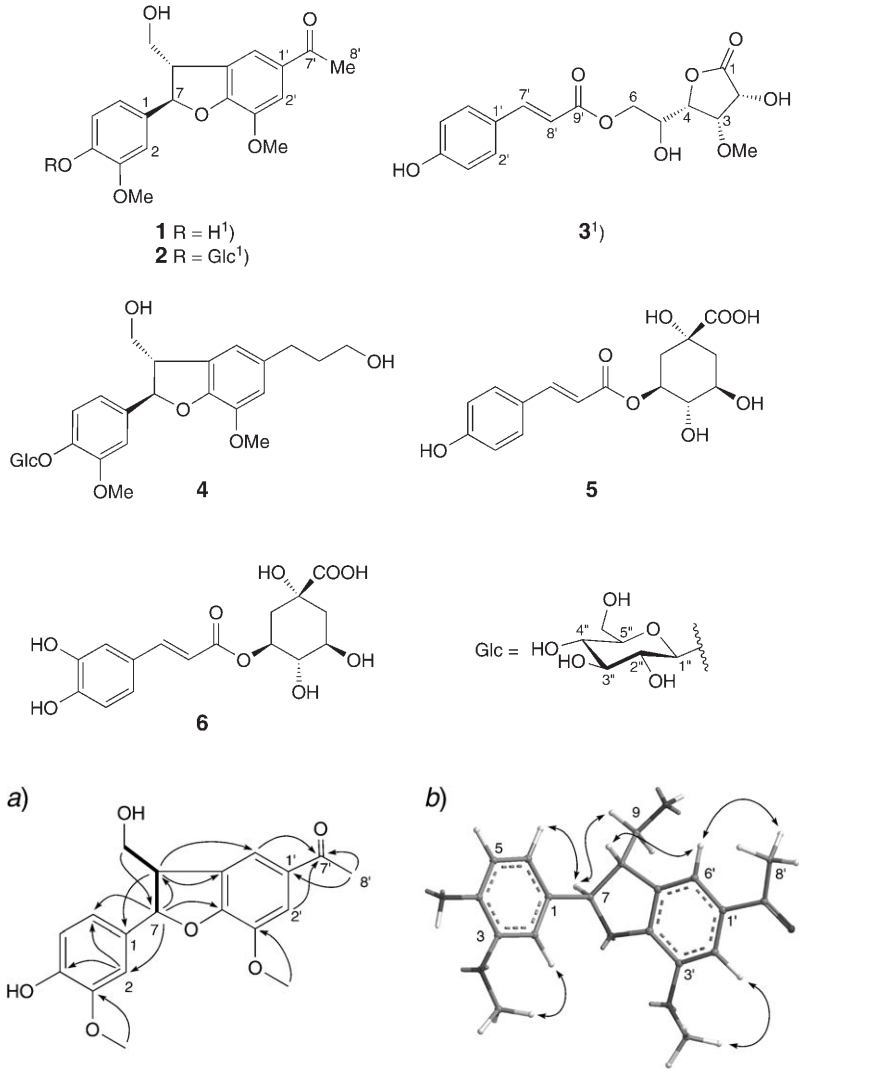


Fig. 1. a) *Selected HMBC* (H → C) *and* $^1H,^1H$*-COSY* (—) *correlations of* **1**. b) *Key NOESY* (↔) *correlations of* **1**[1])

7.05 (*d*, $J = 1.9$, 1 H)). Additionally, the $^1$H- and $^{13}$C-NMR data (*Table 1*) clearly indicated the existence of one conjugated C=O group ($\delta$(C) 196.5), two aromatic MeO groups ($\delta$(H) 3.82 (*s*, 3 H) and 3.90 (*s*, 3 H)), and one Me group ($\delta$(H) 2.52 (*s*, 3 H)) probably linked to an sp$^2$-C-atom. The $^{13}$C-NMR data (*Table 1*) and the molecular formula led to the attribution of the remaining two CH groups (one oxygenated) and one oxygenated $CH_2$ group to C(7) (89.6), C(8) (54.1), and C(9) (64.2), resp.[1])

In the $^1$H,$^1$H-COSY spectrum, the spin system CH(7)–CH(8)–$CH_2$(9) was deduced by correlations from H–C(8) to both H–C(7) and $CH_2$(9) (*Fig. 1,a*). Then,

Table 1. *$^1$H- and $^{13}$C-NMR Data of* **1** *and* **2**[a]). $\delta$ in ppm, $J$ in Hz.

| Atom[1]) | **1** | | **2** | |
|---|---|---|---|---|
| | $\delta$(H) | $\delta$(C) | $\delta$(H) | $\delta$(C) |
| C(1) | | 133.6 | | 134.6 |
| H–C(2) | 7.05 (*d*, *J* = 1.9) | 110.6 | 6.95 (*d*, *J* = 1.5) | 110.5 |
| C(3) | | 148.5 | | 149.1 |
| C(4) | | 147.5 | | 146.4 |
| H–C(5) | 6.83 (*d*, *J* = 8.1) | 115.7 | 7.04 (*d*, *J* = 8.5) | 115.5 |
| H–C(6) | 6.89 (*dd*, *J* = 8.1, 1.9) | 118.7 | 6.82 (*dd*, *J* = 8.5, 1.5) | 118.2 |
| H–C(7) | 5.69 (*d*, *J* = 6.4) | 89.6 | 5.62 (*d*, *J* = 6.3) | 88.0 |
| H–C(8) | 3.63 (*dd*, *J* = 12.3, 6.4) | 54.1 | 3.35 (*dd*, *J* = 12.0, 6.3) | 52.5 |
| $CH_2$(9) | 3.87–3.93 (*m*) | 64.2 | 3.75–3.78 (*m*) | 62.6 |
| C(1′) | | 132.4 | | 131.1 |
| H–C(2′) | 7.51 (*d*, *J* = 1.4) | 113.3 | 7.44 (*s*) | 112.2 |
| C(3′) | | 145.0 | | 143.6 |
| C(4′) | | 153.7 | | 152.0 |
| H–C(5′) | | 130.4 | | 129.4 |
| H–C(6′) | 7.64 (*d*, *J* = 1.4) | 119.7 | 7.58 (*s*) | 118.8 |
| C(7′) | | 196.5 | | 196.2 |
| Me–C(7′) | 2.52 (*s*) | 26.4 | 2.44 (*s*) | 26.5 |
| MeO–C(3) | 3.82 (*s*) | 56.3 | 3.75 (*s*) | 55.8[b]) |
| MeO–C(3′) | 3.90 (*s*) | 56.4 | 3.82 (*s*) | 55.8[b]) |
| H–C(1″) | | | 4.85 (*d*, *J* = 7.5) | 100.0 |
| H–C(2″) | | | 3.24–3.26 (*m*) | 73.1 |
| H–C(3″) | | | 3.24–3.30 (*m*)[b]) | 76.6 |
| H–C(4″) | | | 3.14–3.17 (*m*) | 69.6 |
| H–C(5″) | | | 3.24–3.30 (*m*)[b]) | 76.9 |
| H–C(6″) | | | 3.65 (*dd*, *J* = 12.2, 2.0), 3.43 (*d*, *J* = 12.2) | 60.6 |

[a]) Recorded at 400 ($^1$H) and 100 ($^{13}$C) MHz in $CD_3COCD_3$ and ($D_6$)DMSO for **1** and **2**, resp. [b]) Overlapped signals.

the HMBC correlations of H–C(7)/C(4′), H–C(8)/C(5′) and C(6′) (*Fig. 1*,*a*) allowed the linkage of C(8)–C(5′) and a C(7),C(4′)-epoxy bridge to outline the dihydrobenzofuran fragment. The basic skeleton was thus constructed through the C(7)–C(1) linkage confirmed by the multiple HMBC cross-peaks of H–C(7) to C(1), C(2), and C(6). Moreover, the HMBC correlations of Me(8′) to C(7′) and C(1′) unambiguously revealed the presence of an unusual Ac group at C(1′) as the side chain. Finally, two MeO groups were positioned at C(3) and C(3′) according to the HMBC correlations between MeO–C(3) and C(3), and between MeO–C(3′) and C(3′), respectively, which was further confirmed by the NOE interactions of MeO–C(3)/H–C(2) and MeO–C(3′)/H–C(2′) (*Fig. 1*,*b*). Therefore, the planar structure of **1** was established as depicted in *Fig. 1*.

The relative orientation of the substituents at C(7) and C(8) was elucidated to be *trans* mainly on the basis of the coupling constant of 6.4 Hz between H–C(7) and H–C(8) in the $^1$H-NMR spectrum [7], which was further confirmed by the NOE correlation of H–C(7) to $CH_2$(9). In addition, **1** showed an identical *Cotton* effect at 299, 245, 233 nm as (7*R*,8*S*)-4-*O*-methyldihydrodehydrodiconiferyl alcohol [8] and an

opposite *Cotton* effect at *ca.* 230 nm as (2*S*,3*R*)-2,3-dihydro-7-hydroxy-2-(4-hydroxy-3-methoxyphenyl)-3-(hydroxymethyl)benzofuran-5-ylpropanol 4′-*O*-*β*-D-glucopyranoside [9]. Therefore, the absolute configuration of **1** was determined as (7*R*,8*S*)[1]).

Compound **2**, obtained as a white amorphous powder, had a molecular formula of $C_{25}H_{30}O_{11}$ on the basis of the pseudomolecular-ion peak at *m*/*z* 529.1671 ($[M+Na]^+$; calc. 529.1686) in the HR-ESI-MS. The IR spectrum showed absorptions for a conjugated C=O group (1651 $cm^{-1}$), aromatic moieties (1601 and 1514 $cm^{-1}$), and OH groups (3367 $cm^{-1}$). The $^1$H- and $^{13}$C-NMR data of **2** were similar to those of **1** (*Table 1*) except signals for a glucose unit, which was consistent with the increase in the molecular mass of 162 amu (glucosyl) for **2**. The above-mentioned information clearly suggested compound **2** being a glucosidic derivative of **1**. Extensive 2D-NMR experiments including HMQC, $^1$H,$^1$H-COSY, and HMBC techniques further confirmed **2** as bombasin 4-*O*-*β*-glucoside.

From the $^1$H-NMR spectrum (*Table 1*), the coupling constant ($J = 7.5$) of the signal for the anomeric H-atom at $\delta$(H) 4.85 indicated the glucosidic linkage to have a *β*-configuration. The glucosidic linkage was positioned at C(4) due to a HMBC correlation of H–C(1″) to C(4)[1]) (*Fig. 2*). Further evidence came from the slight upfield shift for C(4) and the slight downfield shifts of C(3) and H–C(5) (*Table 1*). The same absolute configuration of (7*R*,8*S*)[1]) for **2** was considered by comparison of the CD spectrum with that of **1**. Finally, enzymatic hydrolysis (by *Trichoderma viride*) of **2** yielded the liberated aglycone, compound **1**, and glucose (identified by co-TLC with authentic samples), which further confirmed our structural elucidation of compounds **1** and **2**. Consequently, compound **2** was identified as (7*R*,8*S*)-7,8-dihydro-7-(4-*β*-glucopyranosyloxy-3-methoxyphenyl)-8-(hydroxymethyl)-1′-acetyl-3′-methoxybenzofuran[1]), named bombasin 4-*O*-*β*-glucoside after **1**.

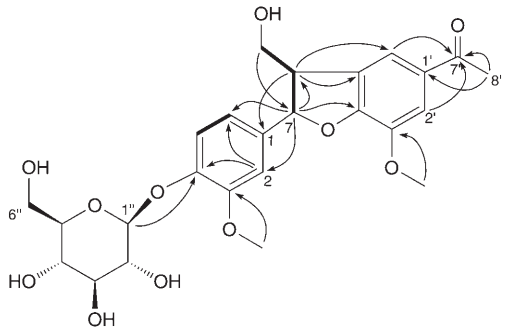


Fig. 2. *Selected HMBC* (H → C) *and* $^1$*H,*$^1$*H-COSY* (—) *correlations of* **2**[1])

Both compounds **1** and **2** apparently derive from a 8–5′ linked neolignan [10], featuring an unusual Ac group at C(1′). Neolignans with this structural character are rare in nature with only one example ever reported in the literature [11].

Compound **3** was isolated as an optically active, white amorphous powder with $[\alpha]_D^{20} = -20$ ($c = 0.55$, MeOH). Its molecular formula of $C_{16}H_{18}O_8$ was determined from the HR-EI-MS signal at *m*/*z* 338.1001 ($M^+$; calc. 338.1002). The IR spectrum displayed absorptions of a *γ*-lactone C=O group (1774 $cm^{-1}$) [12], an *α*,*β*-unsaturated C=O group (1704 $cm^{-1}$, 1637 $cm^{-1}$), as well as a benzene moiety (1606 and 1515 $cm^{-1}$).

Analysis of further spectral data, such as HMQC, $^1$H,$^1$H-COSY, HMBC, and NOESY spectra, established the structure of **3** as 6-*O*-4′-(hydroxycinnamoyl)-3-methyl-D-gulono-$\gamma$-lactone[1]), named bombalin.

The $^1$H-NMR data (*Table 2*) showed a *trans-p*-coumaroyl (4-hydroxycinnamoyl) moiety due to a symmetrical 1,4-disubstitued benzene moiety at $\delta$(H) 6.82 (*d*, $J$ = 9.0, 2 H) and 7.50 (*d*, $J$ = 9.0, 2 H), and *trans*-coupled olefinic H-atoms at $\delta$(H) 6.40 (*d*, $J$ = 15.5, 1 H) and 7.70 (*d*, $J$ = 15.5, 1 H). The $^1$H- and $^{13}$C-NMR signals in the aliphatic region at $\delta$(H) 4.15 (*dd*, $J$ = 4.5, 3.5, 1 H), 4.23 (*dd*, $J$ = 8.1, 4.2, 1 H), 4.33–4.36 (*m*, 2 H), 4.52 (*dd*, $J$ = 8.1, 3.5, 1 H), 4.70 (*d*, $J$ = 4.5, 1 H), and 3.60 (*s*, 3 H), as well as $\delta$(C) at 65.9, 69.6, 73.1, 80.2, 81.7, and 177.7 pointed out the skeleton of a methylated hexose unit, which was further confirmed by the $CH_2$(6)–CH(5)–CH(4)–CH(3)–CH(2) chain distinguished from the mutually coupled signals in the $^1$H,$^1$H-COSY spectrum (*Fig. 3*). Furthermore, the remarkably downfield shifted C(4) ($\delta$(C) 81.7) and the IR absorption band at 1774 cm$^{-1}$ suggested that the sugar moiety is cyclized to a $\gamma$-lactone between C(4) and C(1). The single MeO group was placed at C(3) from the HMBC cross-peak of MeO to C(3). The *cis*-orientation of H–C(3) with respect to both H–C(2) and H–C(4) was deduced by the relatively small coupling constants $J$(2,3) = 4.5 and $J$(3,4) = 3.5 Hz (in the literature $J$(2,3) = 4.6 and $J$(3,4) = 2.7 Hz) [13] and further confirmed by the NOE correlations of H–C(4) to H–C(2) and H–C(5) to MeO–C(3) (*Fig. 4*). These spectral data together

Table 2. *$^1$H-, $^{13}$C-, and 2D-NMR Data of* **3**[a]). $\delta$ in ppm, $J$ in Hz.

| Atom[1]) | $\delta$(H) | $\delta$(C) | HMBC |
|---|---|---|---|
| C(1) | | 177.7 | |
| H–C(2) | 4.70 (*d*, $J$ = 4.5) | 73.1 | 1, 3 |
| H–C(3) | 4.15 (*dd*, $J$ = 4.5, 3.5) | 80.2 | 1, 4, MeO |
| H–C(4) | 4.52 (*dd*, $J$ = 8.1, 3.5) | 81.7 | 2, 3, 5, 6 |
| H–C(5) | 4.23 (*dd*, $J$ = 8.1, 4.2) | 69.6 | 4, 6 |
| $CH_2$(6) | 4.33–4.36 (*m*) | 65.9 | 4, 5, 9′ |
| C(1′) | | 127.4 | |
| H–C(2′)/(6′) | 7.50 (*d*, $J$ = 9.0) | 131.5 | 3′/5′, 4′, 7′ |
| H–C(3′)/(5′) | 6.82 (*d*, $J$ = 9.0) | 117.1 | 1′, 4′ |
| C(4′) | | 161.5 | |
| H–C(7′) | 7.70 (*d*, $J$ = 15.5) | 147.3 | 1′, 2′, 6′, 8′, 9′ |
| H–C(8′) | 6.40 (*d*, $J$ = 15.5) | 115.0 | 1′, 7′, 9′ |
| C(9′) | | 169.2 | |
| MeO–C(3) | 3.60 (*s*) | 61.1 | 3 |

[a]) Recorded at 400 ($^1$H) and 100 ($^{13}$C) MHz in $CD_3OD$.

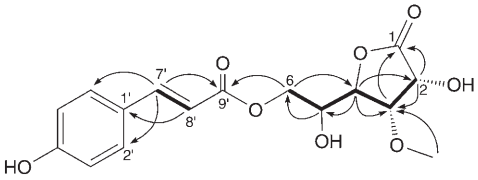


Fig. 3. *Selected HMBC* (H → C) *and $^1$H,$^1$H-COSY* (—) *correlations of* **3**[1])

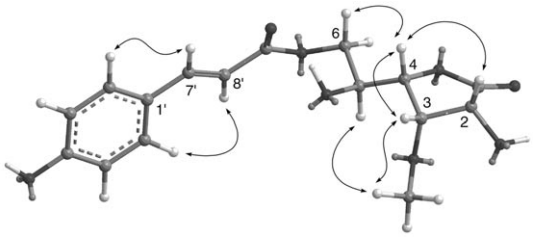


Fig. 4. *Key NOESY* (↔) *correlations of* **3**

with the negative optical rotation indicated that the hexose moiety was 3-methyl-D-gulono-$\gamma$-lactone [13][14]. Finally, the coumaroyl group and the hexose unit were connected through an ester bridge by the key HMBC correlation of H–C(6) to C(9′) (*Fig. 3*). From these data, the structure of **3** was fully elucidated.

Compounds **1**–**3** were screened against the HGC-27 gastrointestinal and Hela cervical human cancer cell lines, using a 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl-2*H*-tetrazolium bromide (MTT) assay [15]. However, no interesting bioactive results for all new compounds in the two cell lines ($IC_{50} > 50$ μM) were found. The results are summarized in *Table 3*.

Table 3. In vitro *Proliferation Inhibition of Human Tumor Cell Lines.* Average values are given (± SEM) for $n = 3$. For details, see *Exper. Part.*

| Drug | $IC_{50}$ [μM][a]) | |
|---|---|---|
| | HGC-27 | Hela |
| Taxol[b]) | $777.8 \times 10^{-3} \pm 102.4 \times 10^{-3}$ | $5.6 \times 10^{-3} \pm 1.8 \times 10^{-3}$ |
| **1** | $200.5 \pm 6.8$ | $180.8 \pm 8.8$ |
| **2** | $124.3 \pm 5.6$ | $150.4 \pm 12.4$ |
| **3** | $253.7 \pm 9.0$ | $121.5 \pm 10.8$ |

[a]) Concentration required to reduce the number of viable cells by 50% ($n = 3$). [b]) Positive control.

The three known compounds were identified as dihydrodehydrodiconiferyl alcohol 4-*O*-$\beta$-D-glucopyranoside (**4**) [16], *trans*-3-(*p*-coumaroyl)quinic acid (**5**) [17], and neochlorogenic acid (**6**) [18] by comparison of their $^1$H- and $^{13}$C-NMR as well as ESI-MS data with those reported in the literature. All of them were isolated for the first time from this specific plant.

## Experimental Part

*General.* Reverse-phase column chromatography (CC): *Sephadex LH-20* (*Amersham Pharmacia Biotech AB*), *MCI-CHP20P* gel (75–150 μm; *Mitsubishi Chemical Industries Co., Ltd.*), *HW-40F* (30–60 μm; *Tosoh Co., Ltd.*), and *Cosmosil 75 $C_{18}$-OPN* (40–105 μm; *Nacalai Tesque Inc.*). TLC: precoated silica-gel $GF_{254}$; visualization under UV light, with $I_2$ vapor, or by spraying anisaldehyde/$H_2SO_4$ reagent. Optical rotation: *Perkin-Elmer 341* polarimeter. UV Spectra: *Shimadzu UV-2450* spectrophotometer; $\lambda_{max}$ (log $\varepsilon$) in nm. CD Spectra: *JASCO J-810* instrument. IR Spectra: *Hitachi 275–50* spectrometer; in $cm^{-1}$. $^1$H- and $^{13}$C-NMR, $^1$H,$^1$H-COSY, HMQC, HMBC, NOESY: *Bruker DRX-400* spectrometer; $\delta$ in

ppm, *J* in Hz. ESI-MS and HR-ESI-MS: *Finnigan LCQ-DECA* spectrometer; in *m*/*z*. EI-MS and HR-EI-MS: *Finnigan MAT-95* mass spectrometer; in *m*/*z*.

*Plant Material.* The flowers of *Bombax malabaricum* were collected at Guangxi, China, in October 2006, and identified by Prof. *Heming Yang*. A voucher specimen (No. BM002) has been deposited in the herbarium of Shanghai Institute of Materia Medica, Chinese Academy of Sciences, P. R. of China.

*Extraction and Isolation.* The air-dried flowers of *B. malabaricum* (5.0 kg) were crushed and extracted three times with 70% (*v*/*v*) aq. EtOH at r.t. for 3 d each. The combined EtOH soln. was concentrated to give a crude syrup, which was extracted with BuOH five times (2 l each). The BuOH-soluble fraction was then separated by CC (*Sephadex LH-20*; MeOH/$H_2O$ gradient) to give *Fractions A–E*. *Fr. A* was purified by CC (*MCI*; MeOH/$H_2O$ gradient) to afford **3** (40 mg), *trans*-3-(*p*-coumaroyl)quinic acid (**5**, 15 mg), and neochlorogenic acid (**6**, 11 mg). *Fr. B* was subjected to CC (*RP-18*; MeOH/$H_2O$ gradient) first, and the major component was subsequently purified by CC (*HW-40F*; 10% aq. MeOH) to give **1** (16 mg) and dihydrodehydrodiconiferyl alcohol 4-*O*-*β*-D-glucopyranoside (**4**, 18 mg). By the analogous separation and purification procedures as for *Fr. B*, *Fr. C* gave **2** (15 mg).

*Enzymatic Hydrolysis.* Compound **2** (2 mg) was dispersed in 10 ml of $H_2O$ and then treated with *Trichoderma Viride* (25 mg). The suspension was left in $H_2O$ at 50° for 24 h. Extraction with BuOH of the mixture resulted in two portions. The BuOH portion and the $H_2O$ portion were analyzed by TLC, co-eluting with isolated compound **1** and commercially purchased D-glucose, resp.

*Cytotoxicity Assay.* Compounds **1**–**3** were evaluated for cytotoxic activity according to previously described protocols [5e]. The results are summarized in *Table 3*.

*Bombasin* (=*1-[(2*R,*3*S*)-2,3-Dihydro-2-(4-hydroxy-3-methoxyphenyl)-3-(hydroxymethyl)-7-methoxybenzofuran-5-yl]ethanone*; **1**). Colorless gum. $[\alpha]_D^{20} = -61.8$ (*c* = 7.57, MeOH). UV (MeOH): 231 (4.40), 288 (4.18). CD (*c* = 0.1, MeOH): $\Delta\varepsilon$ ($\lambda_{299}$) −4.92, $\Delta\varepsilon$ ($\lambda_{245}$) −3.77, $\Delta\varepsilon$ ($\lambda_{233}$) +7.72. IR (KBr): 3423, 2937, 1658, 1591, 1517, 1492, 1361, 1324, 1178, 1031, 592. $^1$H- and $^{13}$C-NMR: see *Table 1*. ESI-MS (pos.): 711.2 ($[2M + Na]^+$), 367.1 ($[M + Na]^+$), 345.1 ($[M + H]^+$). ESI-MS (neg.): 709.1 ($[2M + Na - 2H]^-$). HR-ESI-MS (pos.): 367.1159 ($[M + Na]^+$, $C_{19}H_{20}NaO_6^+$; calc. 367.1158).

*Bombasin 4-O-β-Glucoside* (=*1-[(2*R,*3*S*)-2,3-Dihydro-2-(4-β-glucopyranosyloxy-3-methoxyphenyl)-3-(hydroxymethyl)-7-methoxybenzofuran-5-yl]ethanone*; **2**): White amorphous powder. $[\alpha]_D^{20} = -74$ (*c* = 1.31, MeOH). UV (MeOH): 231 (4.23), 283 (4.17). CD (*c* = 0.08, MeOH): $\Delta\varepsilon$ ($\lambda_{291}$) −3.96, $\Delta\varepsilon$ ($\lambda_{246}$) −2.45, $\Delta\varepsilon$ ($\lambda_{233}$) +8.06. IR (KBr): 3367, 2920, 1651, 1601, 1514, 1464, 1427, 1265, 1223, 1159, 1126, 1070, 1032, 814. $^1$H- and $^{13}$C-NMR: see *Table 1*. ESI-MS (pos.): 1035.4 ($[2M + Na]^+$), 529.2 ($[M + Na]^+$), 345.1 ($[M - Glc + H]^+$). ESI-MS (neg.): 1011.4 ($[2M - H]^-$), 551.5 ($[M + COOH]^-$). HR-ESI-MS (pos.): 529.1671 ($[M + Na]^+$, $C_{25}H_{30}NaO_{11}^+$; calc. 529.1686).

*Bombalin* (=*(2*S*)-2-Hydroxy-3-[(2*S*,*3*S*,*4*R*)-4-hydroxy-3-methoxy-5-oxotetrahydrofuran-2-yl]ethyl (2*E*)-3-(4-Hydroxyphenyl)prop-2-enoate*; **3**): White powder. $[\alpha]_D^{20} = -20$ (*c* = 0.55, MeOH). UV (MeOH): 228 (4.12), 313 (4.43). IR (KBr): 3480, 2952, 1774, 1704, 1637, 1606, 1515, 1444, 1278, 1155, 1037, 979, 829, 773. $^1$H- and $^{13}$C-NMR: see *Table 2*. ESI-MS (neg.): 337.2 ($[M - H]^-$). EI-MS: 338 (16), 207 (2), 164 (76), 147 (100), 119 (24), 91 (16), 65 (9), 55 (4). HR-EI-MS: 338.1001 ($M^+$, $C_{16}H_{18}O_8^+$; calc. 338.1002).

## REFERENCES


[1] Editorial Committee for Flora of the Chinese Academy of Science, 'Flora of China', Science Press, Beijing, 1984, Vol. 49, p. 106.

[2] S. Faizi, M. Ali, *Planta Med*. **1999**, *65*, 383; R. Saleem, M. Ahmad, S. A. Hussain, A. M. Qazi, S. I. Ahmad, M. H. Qazi, M. Ali, S. Faizi, S. Akhtar, S. N. Hussain, *Planta Med*. **1999**, *65*, 331.

[3] A. A. Shahat, R. A. Hassan, N. M. Nazif, S. V. Miert, L. Pieters, F. M. Hammuda, A. J. Vlietinck, *Planta Med*. **2003**, *69*, 1068.

[4] G. S. Niranjan, P. C. Gupta, *Planta Med*. **1973**, *24*, 196.

[5] a) A. V. B. Sankaram, N. S. Reddy, J. N. Shoolery, *Phytochemistry* **1981**, *20*, 1877; b) R. P. Sood, K. A. Suri, O. P. Suri, K. L. Dhar, C. K. Atal, *Phytochemistry* **1982**, *21*, 2125; c) L. S. Puckhaber, R. D. Stipanovic, *J. Nat. Prod*. **2001**, *64*, 260; d) J. Bucking, 'Dictionary of Natural Products', Chapman and

Hall, London, 1994, Vol. 7, p. 446; e) X. H. Zhang, H. L. Zhu, S. W. Zhang, Q. Yu, L. J. Xuan, *J. Nat. Prod*. **2007**, *70*, 1526; f) Y. J. You, N. H. Nam, Y. Kim, K. H. Bae, B. Z. Ahn, *Phytother. Res.* **2003**, *17*, 341.
[6] G. D. Agrawal, S. A. I. Rizvi, P. C. Gupta, J. D. Tewari, *Planta Med*. **1972**, *21*, 293.
[7] P. K. Agrawal, R. P. Rastogi, B. G. Osterdhal, *Org. Magn. Res.* **1983**, *21*, 119.
[8] G. Lemiere, M. Gao, A. D. Groot, R. Dommisse, J. Lepoivre, L. Pieters, V. Buss, *J. Chem. Soc., Perkin Trans. 1* **1995**, 1775.
[9] K. Machida, M. Takano, R. Kakuda, Y. Yaota, M. Kikuchi, *Chem. Pharm. Bull.* **2002**, *50*, 669.
[10] L. B. Davin, N. G. Lewis, *Phytochem. Rev.* **2003**, *2*, 257.
[11] Y. F. Bi, X. K. Zheng, W. S. Feng, S. P. Shi, *Acta Pharm. Sin.* **2004**, *39*, 41.
[12] C. F. Chyu, Y. H. Kuo, *Helv. Chim. Acta* **2007**, *90*, 738.
[13] Z. Walaszek, D. Horton, *Carbohydr. Res.* **1982**, *105*, 131.
[14] R. Kosler, P. Idelmann, W. V. Dahlhoff, *Angew. Chem., Int. Ed.* **1980**, *19*, 547.
[15] D. T. Vistica, P. Skehan, D. Scudiero, A. Monks, A. Pittman, M. R. Boyd, *Cancer Res.* **1991**, *51*, 2515.
[16] F. Abe, T. Yamauchi, *Chem. Pharm. Bull.* **1986**, *34*, 4340.
[17] V. Ossipov, K. Nurmi, J. Loponen, E. Haukioja, K. Pihlaja, *J. Chromatogr., A* **1996**, *721*, 59.
[18] N. Nakatani, S. Kayano, H. Kikuzaki, K. Sumino, K. Katagiri, T. Mitani, *J. Agric. Food Chem.* **2000**, *48*, 5512.

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