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A Novel Dibenzofuran and Two New Xanthones from *Calophyllum panciflorum*

Chihiro ITO,*[,a] Yoshiaki MIYAMOTO,[a] K. Sundar RAO,[b] and Hiroshi FURUKAWA[a]

Faculty of Pharmacy, Meijo University,[a] Tempaku, Nagoya 468, Japan and Department of Chemistry,[b] University of Papua New Guinea, Papua New Guinea.

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Constituents of an EtOH extract of the stem bark of *Calophyllum panciflorum* A. C. SMITH (Guttiferae) collected in Central Province of Papua New Guinea were studied. A novel dibenzofuran named calophyfuran (1) and two new xanthones named pancixanthone-A (3) and pancixanthone-B (4), respectively, were isolated as well as four kinds of known xanthones, and their structures were elucidated by spectrometric methods. Among these components, calophyfuran (1) is the first example of dibenzofuran derivatives having a five-carbon unit (prenyl) as a substituent to be found in nature.

Key words *Calophyllum panciflorum*; dibenzofuran; calophyfuran; xanthone; pancixanthone; Guttiferae

The plant *Calophyllum* sp. (Guttiferae) is a rich source of xanthones,[1)] and various bioactivities of xanthones (for example, cytotoxic and antitumor activities) have been reported.[2)]

During our studies on biologically active natural products from Papua New Guinea medicinal plants, an EtOH extract of the stem bark of *Calophyllum panciflorum* A. C. SMITH (Guttiferae) was found to exhibit an antibacterial activity.[3)] This paper describes the isolation and structural elucidation of a novel dibenzofuran named calophyfuran (**1**), and two new xanthones named pancixanthone-A (**3**) and pancixanthone-B (**4**) from the EtOH extract of *C. panciflorum*.

Results and Discussion

The dried stem bark of *C. panciflorum* was extracted with EtOH under reflux. The EtOAc-soluble portion of the EtOH extract was fractionated by a combination of silica gel column chromatography and preparative TLC to give a novel dibenzofuran and two new xanthones, along with four kinds of known xanthones.

Structure of Calophyfuran (1) Calophyfuran (**1**) was obtained as a colorless oil. The molecular formula was determined as $C_{19}H_{20}O_5$ by high-resolution (HR)-MS. The UV spectrum showed the typical absorption band of the dibenzofuran nucleus.[4)] The IR spectrum exhibited bands at ν_{max} 3590 and 3525 cm^{-1} due to hydroxy groups. The ^{1}H-NMR spectrum revealed signals attributable to two methoxyls [δ 4.05, 3.80 (3H, s)] as well as two hydroxy groups [δ 8.30, 8.10 (1H, br)]. The presence of a prenyl moiety in the molecule was indicated by ^{1}H-NMR signals at δ 3.75 (2H, d, $J=6.2$ Hz), 5.24 (1H, t, $J=6.2$ Hz), 1.88 (3H, s), and 1.68 (3H, s) and by the observation of a base fragment peak at m/z 272 [$M^+ - \cdot CH=C(CH_3)_2 - \cdot H$] in the electron impact (EI)-MS. In the aromatic proton region of the ^{1}H-NMR spectrum, ABC-type protons appeared at δ 7.39 (1H, d, $J=8.8$ Hz), 7.33 (1H, d, $J=2.6$ Hz), and 6.90 (1H, dd, $J=8.8$, 2.6 Hz). Treatment of this dibenzofuran with diazomethane in Et_2O–MeOH gave an *O*,*O*-dimethyl ether (**2**), suggesting the presence of two phenolic hydroxy groups in the original molecule. In differential nuclear Overhauser effect (NOE) experiments, irradiation of the methoxy group at δ 3.80 enhanced the signals at δ 5.24 (H-2′) and 1.88 (3′-CH_3), but there was no NOE enhancement at any proton signal on irradiation of another methoxy group at δ 4.05. In the methyl ether (**2**), irradiation of the newly formed methoxy group at δ 3.95 (3-OCH_3) enhanced the signals at δ 4.11 (4-OCH_3) and 3.82 (2-OCH_3), and irradiation of the other newly formed methoxy group at δ 3.86 (7-OCH_3) enhanced the signals at δ 7.42 (H-8) and 7.05 (H-6). Irradiation of the original methoxy group at δ 4.11 (4-OCH_3) enhanced the signals at δ 3.95 (3-OCH_3), while irradiation of the other original methoxy group at 3.82 (2-OCH_3) enhanced the signals at δ 3.95 (3-OCH_3), 5.23 (H-2′), and 1.90 (3′-CH_3). In the irradiation of the hydrogens at δ 3.80 (H_2-1′), enhancements of the signals at δ 7.42 (H-8) and 1.90 (3′-CH_3) were observed. These results suggested the structure of calophyfuran to be **1**. The structure **1** was confirmed by ^{1}H-detected heteronuclear multiple quantum coherence (HMQC) and ^{1}H-detected heteronuclear multiple bond connectivity (HMBC) spectroscopies, as shown by arrows in Fig. 1. This is the first example of a dibenzofuran derivative having a five-carbon unit (prenyl) to be found in nature.

Structure of Pancixanthone-A (3) Pancixanthone-A (**3**) was obtained as a pale yellow powder. The molecular

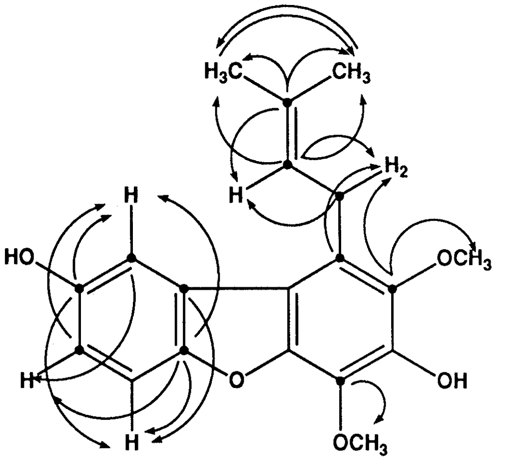

Fig. 1. C–H Long-Range Correlations in the HMBC Spectrum of Calophyfuran (1)

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* To whom correspondence should be addressed.

formula was determined as $C_{18}H_{16}O_5$ by HR-MS. The UV absorptions (see Experimental) and IR bands at ν_{max} 3431 (br) and 1648 cm^{-1} due to hydroxy and carbonyl groups, respectively, were suggestive of a 1,3,5-trioxygenated xanthone derivative.[5] In the ^1H-NMR spectrum, a lone 1H singlet at δ 6.33 and ABC-type protons at δ 7.64 (1H, dd, J=8.1, 1.8 Hz), 7.34 (1H, dd, J=8.1, 1.8 Hz), and 7.25 (1H, t, J=8.1 Hz) appeared in addition to a chelated hydroxyl signal at δ 13.24. The presence of a 1,1-dimethylallyl group at C-4 was indicated by ^1H-NMR signals at δ 6.57 (1H, dd, J=10.6, 17.6 Hz), 5.08 (1H, br d, J=17.6 Hz), 4.92 (1H, dd, J=10.6, 1.5 Hz), and 1.74 (6H, s), and the cross peaks of C-2 (δ_C 100.00) to 1-OH (δ_H 13.24) and C-4 (δ_C 113.16) to H-2 (δ_H 6.33) and to 1'-CH_3 (δ_H 1.74) in the HMBC spectrum. From the aforementioned results and the HMQC and HMBC results shown by arrows in Fig. 2, the structure of pancixanthone-A was concluded to be **3**.

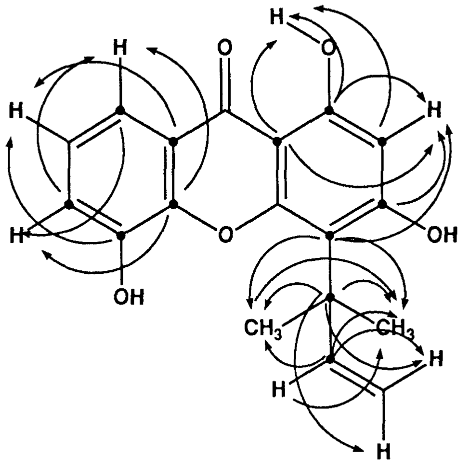
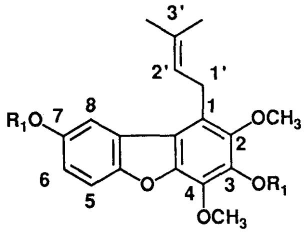


Fig. 2. C–H Long-Range Correlations in the HMBC Spectrum of Pancixanthone-A (**3**)

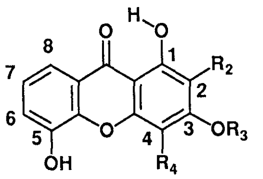
Structure of Pancixanthone-B (4) Pancixanthone-B (**4**) was obtained as a yellow powder having $[\alpha]_D$ +3° ($CHCl_3$). The molecular formula was determined to be $C_{18}H_{16}O_5$ by HR-MS. The UV and IR spectra showed absorptions characteristic of a 1,3,5-trioxygenated xanthone derivative.[5] The IR spectrum also showed a broad band at ν_{max} 3284 and 1649 cm^{-1} due to hydroxy and carbonyl groups. In the ^1H-NMR spectrum, a lone 1H singlet at δ 6.19 and ABC-type protons at δ 7.67 (1H, br d, J=8.1 Hz), 7.38 (1H, dd, J=8.1, 1.5 Hz), and 7.25 (1H, t, J=8.1 Hz) appeared in addition to a chelated hydroxyl signal at δ 13.25, as in **3**. The observation of two 3H-singlets (δ 1.63 and 1.34), a 3H-doublet (δ 1.41, J=6.6 Hz) and a 1H- quartet (δ 4.60, J=6.6 Hz) suggested the presence of 2,3,3-trimethyldihydrofuran ring. An angular orientation of the furan ring in the molecule was indicated by the appearance of C–H long-range correlations in the HMBC spectrum between the oxygenated methine carbon at δ_C 91.85 and two methyl protons at δ_H 1.63 and 1.34, which further correlated to an aromatic carbon at δ_C 114.00 and a quaternary carbon at δ_C 44.49, and also between the C-2 aromatic carbon at δ_C 94.04 and a chelated hydroxy signal at δ_H 13.25. From the above results, the structure of pancixanthone-B was concluded to be **4**.

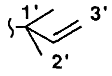
Other xanthones isolated from the EtOH extract were characterized as morusignin I (**5**),[6] cudraxanthone G (**6**),[5] trapezifolixanthone (**7**),[7] and 1,3,7-trihydroxy-2-(3-methylbut-2-enyl)xanthone (**8**)[8,9] by comparisons of the ^1H-NMR, IR, UV, and MS data with those reported in the literature.[5–9] The signal assignments of carbons in the ^{13}C-NMR spectrum (see Experimental) of morusignin I (**5**) and cudraxanthone G (**6**) were made on the basis of the HMQC and HMBC results.

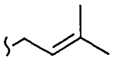


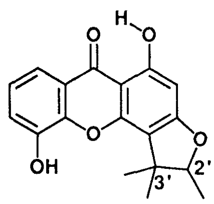
1 R_1 = H

2 R_1 = CH_3



3 R_2 = R_3 = H, R_4 = 

6 R_2 = R_4 =  R_3 = CH_3



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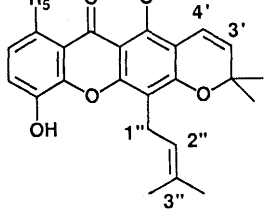
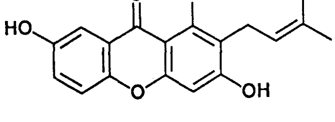


5 R_5 = OH

7 R_5 = H



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Experimental

Melting points were measured on a micromelting point hot-stage apparatus (Yanagimoto). ^{1}H- and ^{13}C-NMR, HMQC, and HMBC ($J=8$ Hz) spectra were recorded on an A-400 or A-600 (JEOL) spectrometer, in acetone-d_6. Chemical shifts are shown in δ values (ppm) with tetramethylsilane (TMS) as an internal reference. All mass spectra were taken under electron impact (EI) conditions, unless otherwise stated, using an M-80 (Hitachi) spectrometer having a direct inlet system. UV spectra were recorded on a UVIDEC-610C double-beam spectrophotometer (JASCO) in MeOH, IR spectra on an IR-230 (JASCO) in CHCl$_3$, and optical rotations on a DIP-370 (JASCO) in CHCl$_3$ at 25 °C. Preparative TLC was done on Kieselgel 60 F$_{254}$ (Merck).

Extraction and Isolation The plant material used in this study, *Calophyllum panciflorum* A. C. Smith (Guttiferae) was collected in Central Province of Papua New Guinea. A voucher specimen has been deposited at the herbarium of the University of Papua New Guinea. The dried stem bark (600 g) was extracted with EtOH under reflux. The EtOH extract was evaporated under reduced pressure to give an oily residue, which was extracted with hexane, CH$_2$Cl$_2$, EtOAc, CH$_2$Cl$_2$–MeOH (3 : 1), and MeOH, successively. The EtOAc-soluble portion was further subjected to silica gel chromatography with iso-Pr$_2$O–acetone (4 : 1, 7 : 3, 1 : 1, 3 : 7), acetone, CH$_2$Cl$_2$–MeOH (3 : 1), and MeOH, successively. The iso-Pr$_2$O–acetone (4 : 1) eluate was further subjected repeatedly to silica gel column and preparative thin layer chromatographies (prep. TLC) with appropriate combinations of hexane, CH$_2$Cl$_2$, acetone, EtOAc, CHCl$_3$, and MeOH as developing solvents to obtain calophyfuran (**1**) (1.4 mg), pancixanthone-A (**3**) (1.9 mg), pancixanthone-B (**4**) (1.7 mg), morusignin I (**5**) (4.2 mg),[6] cudraxanthone G (**6**) (2.2 mg),[5] trapezifolixanthone (**7**) (1.2 mg),[7] and 1,3,7-trihydroxy-2-(3-methylbut-2-enyl)-xanthone (**8**) (4.6 mg).[8,9]

Calophyfuran (1) Colorless oil. UV λ_{max} nm: 221, 240 (sh), 264, 297, 322. IR ν_{max} cm^{-1}: 3590 (br), 3525. ^{1}H-NMR δ: 8.30 (1H, br), 8.10 (1H, br), 7.39 (1H, d, $J=8.8$ Hz, H-5), 7.33 (1H, d, $J=2.6$ Hz, H-8), 6.90 (1H, dd, $J=8.8$, 2.6 Hz, H-6), 5.24 (1H, t, $J=6.2$ Hz, H-2′), 4.05 (3H, s, 4-OCH$_3$), 3.80 (3H, s, 2-OCH$_3$), 3.75 (2H, d, $J=6.2$ Hz, H-1′), 1.88 (3H, s, 3′-CH$_3$), 1.68 (3H, s, 3′-CH3). ^{13}C-NMR δ: 154.26 (s, C-7), 151.18 (s, C-9a), 146.63 (s, C-4a), 143.52 (s, C-2), 143.46 (s, C-3), 132.92 (s, C-3′), 132.33 (s, C-4), 126.39 (s, C-8a), 124.50 (s, C-8b), 123.24 (d, C-2′), 115.66 (s, C-1), 114.13 (d, C-6), 112.13 (d, C-5), 108.13 (d, C-8), 61.39 (q, 2-OCH$_3$), 61.12 (q, 4-OCH$_3$), 26.11 (t, C-1′), 25.76 (q, 3′-CH$_3$), 18.29 (q, 3′-CH$_3$). MS *m/z* (%): 328 (M$^+$, 100), 313 (45), 297 (42), 281 (53), 272 (63), 260 (37), 257 (34), 239 (33), 229 (38). HR-MS Calcd for C$_{19}$H$_{20}$O$_5$: 328.1309. Found: 328.1301. Differential NOE : irradiation of the 2-OCH$_3$ (δ 3.80) gave 2% NOE at H-2′ (δ 5.24) and 2% NOE at 3′-CH$_3$ (δ 1.88); irradiation of the 4-OCH$_3$ (δ 4.05) gave no NOE at any proton signal.

***O*-Methylation of 1 with Diazomethane** A large excess of ethereal diazomethane was added to a methanolic solution (20 ml) of **1** (1.4 mg), and the mixture was left overnight at room temperature. The solvent was evaporated, and the residue was purified by preparative TLC to give **2** almost quantitatively: Colorless oil. UV λ_{max} nm: 210 (sh), 222, 240 (sh), 263, 294, 310, 324. ^{1}H-NMR δ: 7.52 (1H, d, $J=8.8$ Hz, H-5), 7.42 (1H, d, $J=2.6$ Hz, H-8), 7.05 (1H, dd, $J=8.8$, 2.6 Hz, H-6), 5.23 (1H, t, $J=6.2$ Hz, H-2′), 4.11 (3H, s, 4-OCH$_3$), 3.95 (3H, s, 3-OCH$_3$), 3.86 (3H, s, 7-OCH$_3$), 3.82 (3H, s, 2-OCH$_3$), 3.80 (2H, d, $J=6.2$ Hz, H-1′), 1.90 (3H, s, 3′-CH$_3$), 1.70 (3H, s, 3′-CH$_3$). MS *m/z* (%): 356 (M$^+$, 79), 334 (18), 325 (13), 310 (9), 301 (7), 288 (6), 273 (7), 243 (6), 223 (6), 205 (6). Differential NOE: irradiation of the 3-OCH$_3$ (δ 3.95) gave 2% NOE at 4-OCH$_3$ (δ 4.11) and 2% NOE at 2-OCH$_3$ (δ 3.82); irradiation of the 7-OCH$_3$ (δ 3.86) gave 8% NOE at H-8 (δ 7.42) and 5% NOE at H-6 (δ 7.05); irradiation of the 4-OCH$_3$ (δ 4.11) gave 2% NOE at 3-OCH$_3$ (δ 3.95); irradiation of the 2-OCH$_3$ (δ 3.82) gave 3, 2, and 3% NOE at 3-OCH$_3$ (δ 3.95), H-2′ (δ 5.23), and 3′-CH$_3$ (δ 1.90), respectively; irradiation of the H$_2$-1′ (δ 3.80) gave 12% NOE at H-8 (δ 7.42) and 4% NOE at 3′-CH$_3$ (δ 1.90).

Pancixanthone-A (3) Pale yellow powder. UV λ_{max} nm: 218, 245, 256 (sh), 320, 363. IR ν_{max} cm^{-1}: 3431 (br), 1648, 1588. ^{1}H-NMR δ: 13.24 (1H, s, 1-OH), 7.64 (1H, dd, $J=8.1$, 1.8 Hz, H-8), 7.34 (1H, dd, $J=8.1$, 1.8 Hz, H-6), 7.25 (1H, t, $J=8.1$ Hz, H-7), 6.57 (1H, dd, $J=10.6$, 17.6 Hz, H-2′), 6.33 (1H, s, H-2), 5.08 (1H, br d, $J=17.6$ Hz, H-3′), 4.92 (1H, dd, $J=10.6$, 1.5 Hz, H-3′), 1.74 (6H, s, 1′-CH$_3$). ^{13}C-NMR δ: 182.04 (s, C=O), 165.03 (s, C-3), 162.47 (s, C-1), 156.59 (s, C-4a), 152.46 (d, C-2′), 147.04 (s, C-5), 145.90 (s, C-10a), 124.74 (d, C-7), 121.76 (s, C-8a), 120.66 (d, C-6), 116.02 (d, C-8), 113.16 (s, C-4), 107.88 (t, C-3′), 104.18 (s, C-9a), 100.00 (d, C-2), 41.89 (s, C-1′), 29.90 (q × 2, 1′-CH$_3$). MS *m/z* (%): 312 (M$^+$, 81), 297 (100), 282 (21), 271 (11), 269 (23), 257 (39), 241 (13), 207 (30), 203 (27). HR-MS Calcd for C$_{18}$H$_{16}$O$_5$: 312.0996. Found: 312.0990.

Pancixanthone-B (4) Yellow powder. $[\alpha]_D$ +3° ($c=0.117$). UV λ_{max} nm: 215, 238 (sh), 246, 256 (sh), 320, 362. IR ν_{max} cm^{-1}: 3284 (br), 1649. ^{1}H-NMR δ: 13.25 (1H, s, 1-OH), 9.30 (1H, br, OH), 7.67 (1H, br d, $J=8.1$ Hz, H-8), 7.38 (1H, dd, $J=8.1$, 1.5 Hz, H-6), 7.25 (1H, t, $J=8.1$ Hz, H-7), 6.19 (1H, s, H-2), 4.60 (1H, q, $J=6.6$ Hz, H-2′), 1.63 (3H, s, 3′-CH$_3$), 1.41 (3H, d, $J=6.6$ Hz, 2′-CH$_3$), 1.34 (3H, s, 3′-CH$_3$). ^{13}C-NMR δ: 181.57 (s, C=O), 165.24 (s, C-1), 147.17 (s, C-5), 124.73 (d, C-7), 121.32 (d, C-6), 116.33 (d, C-8), 114.00 (s, C-4), 94.04 (d, C-2), 91.85 (d, C-2′), 44.49 (s, C-3′), 25.74 (q, 3′-CH$_3$), 21.33 (q, 3′-CH$_3$), 14.51 (q, 2′-CH$_3$). Other signals were difficult to detect because of the small quantity of the sample available. MS *m/z* (%): 312 (M$^+$, 37), 297 (100), 282 (17), 269 (23), 241 (13). HR-MS Calcd for C$_{18}$H$_{16}$O$_5$: 312.0996. Found: 312.0993.

Morusignin I (5) Yellow needles, mp 146—148 °C . UV λ_{max} nm: 226, 272, 298, 341, 405. IR ν_{max} cm^{-1}: 3591 (br), 1625, 1588. ^{1}H-NMR δ: 12.30 (1H, s, 1-OH), 11.21 (1H, s, 8-OH), 8.44 (1H, br, 5-OH), 7.31 (1H, d, $J=8.8$ Hz, H-6), 6.68 (1H, d, $J=9.9$ Hz, H-4′), 6.62 (1H, d, $J=8.8$ Hz, H-7), 5.77 (1H, d, $J=9.9$ Hz, H-3′), 5.27 (1H, t, $J=7.3$ Hz, H-2″), 3.52 (2H, d, $J=7.3$ Hz, H-1″), 1.84 (3H, s, 3″-CH$_3$), 1.64 (3H, s, 3″-CH$_3$), 1.50 (6H, s, 2′-CH$_3$). ^{13}C-NMR δ: 186.03 (s, C=O), 159.98 (s, C-3), 155.90 (s, C-1), 154.96 (s, C-4a), 154.15 (s, C-8), 144.92 (s, C-10a), 138.14 (s, C-5), 131.95 (s, C-3″), 128.95 (d, C-3′), 124.96 (d, C-6), 123.05 (d, C-2″), 115.75 (d, C-4′), 110.26 (d, C-7), 108.90 (s, C-8a), 105.34 (s, C-2), 102.93 (s, C-9a), 79.36 (s, C-2′), 28.50 (q × 2, 2′-CH$_3$), 25.92 (q, 3″-CH$_3$), 21.85 (t, C-1″), 18.10 (q, 3″-CH$_3$). MS *m/z* (%): 394 (M$^+$, 78), 379 (100), 363 (14), 351 (22), 339 (62), 321 (11), 311 (12), 162 (30). HR-MS Calcd for C$_{23}$H$_{22}$O$_6$: 394.1415. Found: 394.1415.

Cudraxanthone G (6) Yellow prisms, mp 127—129 °C. UV λ_{max} nm: 205, 242, 260, 315, 378. IR ν_{max} cm^{-1}: 3572 (br), 1639, 1596. ^{1}H-NMR δ: 13.15 (1H, s, 1-OH), 7.71 (1H, dd, $J=8.1$, 1.5 Hz, H-8), 7.40 (1H, dd, $J=8.1$, 1.5 Hz, H-6), 7.28 (1H, t, $J=8.1$ Hz, H-7), 5.33 (1H, t, $J=6.9$ Hz, H-2″), 5.27 (1H, t, $J=6.9$ Hz, H-2′), 3.85 (3H, s, 3-OCH$_3$), 3.62 (2H, d, $J=6.9$ Hz, H-1″), 3.40 (2H, d, $J=6.9$ Hz, H-1′), 1.84 (3H, s, 3″-CH$_3$), 1.79 (3H, s, 3′-CH$_3$), 1.66 (3H, s, 3″-CH$_3$), 1.65 (3H, s, 3′-CH$_3$). ^{13}C-NMR δ: 182.87 (s, C=O), 164.82 (s, C-3), 159.75 (s, C-1), 153.70 (s, C-4a), 147.31 (s, C-5), 146.63 (s, C-10a), 132.00 (s, C-3′ or C-3″), 131.90 (s, C-3″ or C-3′), 124.77 (d, C-7), 123.87 (d, C-2′ or C-2″), 123.57 (d, C-2″ or C-2′), 122.06 (s, C-8a), 121.64 (d, C-6), 117.76 (s, C-2), 116.34 (d, C-8), 114.38 (s, C-4), 106.48 (s, C-9a), 62.23 (q, 3-OCH$_3$), 25.86 (q, 3′-CH$_3$ or 3″-CH$_3$), 25.79 (q, 3″-CH$_3$ or 3′-CH$_3$), 23.15 (t, C-1′ or C-1″), 23.05 (t, C-1″ or C-1′), 18.03 (q, 3′-CH$_3$ or 3″-CH$_3$), 17.95 (q, 3″-CH$_3$ or 3′-CH$_3$). MS *m/z* (%): 394 (M$^+$, 95), 379 (15), 351 (100), 339 (97), 323 (28), 309 (10), 305 (10), 295 (17), 283 (11), 281 (16), 271 (18), 269 (35). HR-MS Calcd for C$_{24}$H$_{26}$O$_5$: 394.1778. Found: 394.1771. Differential NOE: Irradiation of 3-OCH$_3$ (δ 3.85) gave 3, 1, 3, and 1% NOE at H-1′ (δ 3.40), H-2′ (δ 5.27), H-1″ (δ 3.62), and H-2″ (δ 5.33) respectively.

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