Three New Isoprenylated 2-Arylbenzofurans from *Artocarpus petelotii*

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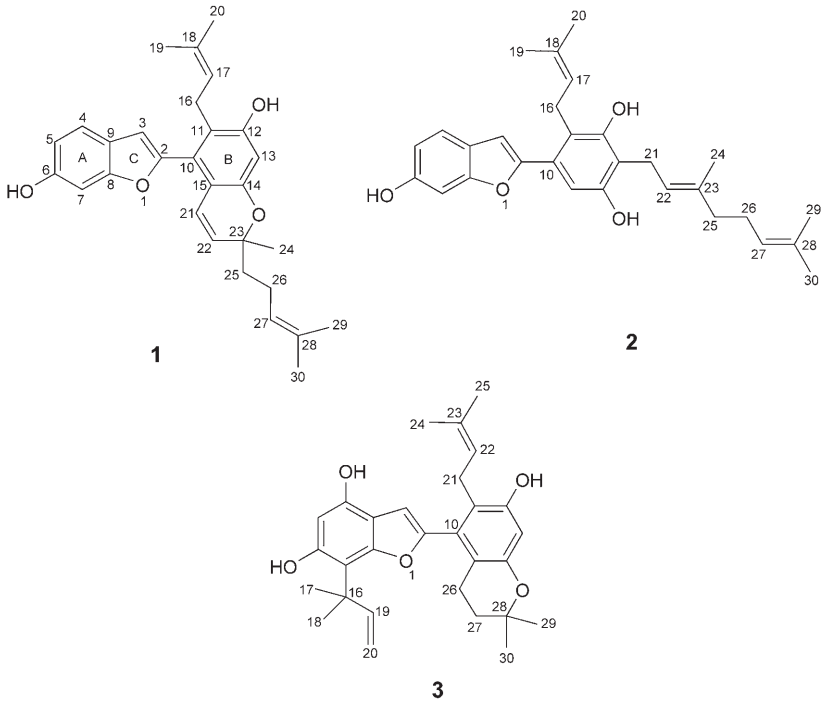
Three new isoprenylated 2-arylbenzofurans, artopetelins A – C (**1** – **3**), were isolated as minor constituents from the EtOH extract of the root barks of *Artocarpus petelotii* GAGNEP. Their structures were elucidated by spectroscopic methods, including HR-EI-MS and 2D-NMR techniques.

Introduction. – Moraceae is a large family comprising 60 genera, with *ca.* 1400 species, including some important groups such as *Artocarpus*, *Morus*, *Ficus*, and *Cudrania*. *Artocarpus* species are evergreen trees distributed over tropical regions of Asia, and some members are used as traditional folk medicines in Indonesia, Thailand, Sri Lanka, and China [1]. Previous research work on this genus has revealed various isoprenylated flavonoids of structural and biological interest [2]. A limited number of biosynthetically related stilbene and 2-arylbenzofuran derivatives were also found in *Artocarpus* plants, with interesting biological activities such as cyclooxygenase- and tyrosinase-inhibitory activities, as well as antimycobacterial effects [3]. Recently, we reported a variety of isoprenylated xanthones, benzophenones, and flavonoids with cytotoxic and antifungal activities from some Chinese *Artocarpus* and *Cudrania* species [4]. As part of our continuing research on Moraceous plants, investigations of the chemical constituents of *Artocarpus petelotii* GAGNEP were carried out. This plant is a 10-m high tree growing in the north of Vietnam and in Yunnan province, China [5] and has been studied neither phytochemically nor pharmacologically so far. In this paper, we describe the isolation and structure elucidation of three new 2-arylbenzofurans, artopetelins A – C (**1** – **3**), which were obtained from the EtOH extract of the root barks of *A. petelotii*.

Results and Discussion. – Artopetelin A (**1**), an optically active compound ($[\alpha]_D^{20} = -40.9$), was isolated as a pale-yellow, amorphous powder. Its molecular formula was deduced as $C_{29}H_{32}O_4$ by HR-EI-MS (*m/z* 444.2295 (M^+, calc. 444.2301)). The UV data resemble those of 2-arylbenzofuran derivatives [3c]. The IR spectrum of **1** exhibit absorptions for OH groups (3385 cm^{-1}) and aromatic rings (1599, 1489 cm^{-1}). The ^{1}H-NMR spectrum (*Table 1*) show signals of two OH groups at δ(H) 8.66 and 8.51 (2*s*, 1 H each); an aromatic *ABX* spin system (ring *A*) at 7.46 (*d*, *J* = 8.2 Hz, 1 H), 6.98 (br. *d*, *J* = 2.0 Hz, 1 H), and 6.85 (*dd*, *J* = 8.2, 2.0 Hz, 1 H); an olefinic signal at 6.66 (*d*, *J* = 0.8 Hz, 1 H) due to a 'zigzag' coupling in a conjugated system [6], and another aromatic signal at 6.48. Moreover, the following ^{1}H-NMR data were observed: δ(H) 6.13, 5.48 (2*d*, *J* = 10.2 Hz, 1 H each), 5.09 – 5.16 (*m*, 2 H), 3.22 (br. *d*, *J* = 7.0 Hz, 2 H); 2.07 –



2.15, 1.65 – 1.71 (2*m*, 2 H each); 1.64, 1.57, 1.55, 1.40 (4 br. *s*, 3 H each), and 1.36 (*s*, 3 H), which were associated with a prenyl group and a geranyl-derived chromene moiety [7], respectively.

The ^{13}C-NMR and HMQC spectra of **1** revealed the presence of eleven quaternary sp^2, nine methine sp^2, one quaternary sp^3, three methylene sp^3, and five methyl C-atoms. These data suggest that **1** is a doubly isoprenylated, dihydroxylated 2-arylbenzofuran. By means of HMQC, HMBC, and NOESY experiments, we were able to determine the substitution pattern and fully assign all ^{1}H- and ^{13}C-NMR signals (*Tables 1* and *2*, resp.). The HMBC spectrum showed long-range correlations between CH_2(25)[1]) at δ(H) 1.65 – 1.71 and C(22) at δ(C) 127.6, C(23) (78.6), C(24) (26.5), C(26) (23.4), and C(27) (125.1). The CH_2(16) group at δ(H) 3.22 is correlated with C(10) at δ(C) 129.5, C(11) (121.8), C(12) (156.7), C(17) (124.8), and C(18) (130.4). And the OH group at δ(H) 8.66 is correlated with C(11) and C(12) (*Fig. 1*).

These results indicate that a 4-methylpent-3-enyl, a prenyl, and one of the two OH groups are attached at C(23), C(11), and C(12), respectively. The position of the pyranoid ring at C(14) and C(15) with the O-atom connected to C(14) was established by HMBC correlations between H–C(21) at δ(H) 6.13 and C(14) at δ(C) 153.3, between H–C(22) at δ(H) 5.48 and C(15) at δ(C) 114.7, and based on a NOESY cross-peak between H–C(3) at δ(H) 6.66 and H–C(21). The substitution of ring *A* is further supported by the HMBC correlations shown in *Fig. 1.*

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

Table 1. *^{1}H-NMR Data of* **1**–**3**. In (D_6)acetone; δ in ppm, J in Hz. Arbitrary atom numbering.

	1[a])	**2**[b])	**3**[b])
H–C(3)	6.66 (*d*, *J* = 0.8)	6.73 (*d*, *J* = 0.8)	6.66 (*s*)
H–C(4)	7.46 (*d*, *J* = 8.2)	7.41 (*d*, *J* = 8.4)	–
H–C(5)	6.85 (*dd*, *J* = 2.0, 8.2)	6.81 (*dd*, *J* = 2.0, 8.4)	6.33 (*s*)
H–C(7)	6.98 (br. *d*, *J* = 2.0)	6.96 (br. *d*, *J* = 2.0)	–
H–C(13)	6.48 (*s*)	–	6.40 (*s*)
H–C(15)	–	6.84 (*s*)	–
CH_2(16)	3.22 (br. *d*, *J* = 7.0)	3.54 (br. *d*, *J* = 6.2)	–
H–C(17)	5.09–5.16 (*m*)	5.18 (br. *t*, *J* = 6.2)	–
Me(17)	–	–	1.64 (*s*)
Me(18)	–	–	1.64 (*s*)
Me(19)	1.40 (br. *s*)	1.68 (br. *s*)	–
H–C(19)	–	–	6.43 (*dd*, *J* = 10.6, 17.5)
H_a–C(20)	–	–	5.02 (*dd*, *J* = 1.3, 17.5)
H_b–C(20)	–	–	4.91 (*dd*, *J* = 1.3, 10.6)
Me(20)	1.55 (br. *s*)	1.66 (br. *s*)	–
H–C(21)	6.13 (*d*, *J* = 10.2)	–	–
CH_2(21)	–	3.48 (br. *d*, *J* = 7.0)	3.27 (br. *d*, *J* = 6.8)
H–C(22)	5.48 (*d*, *J* = 10.2)	5.30 (br. *t*, *J* = 7.0)	5.11 (br. *t*, *J* = 6.8)
Me(24)	1.36 (*s*)	1.81 (br. *s*)	1.42 (br. *s*)
Me(25)	–	–	1.55 (br. *s*)
CH_2(25)	1.65–1.71 (*m*)	1.99–2.02 (*m*)	–
CH_2(26)	2.07–2.15 (*m*)	2.08–2.11 (*m*)	2.48 (*t*, *J* = 6.8)
CH_2(27)	–	–	1.69 (*t*, *J* = 6.8)
H–C(27)	5.09–5.16 (*m*)	5.10 (br. *t*, *J* = 7.0)	–
Me(29)	1.57 (br. *s*)	1.58 (br. *s*)	1.28 (*s*)
Me(30)	1.64 (br. *s*)	1.63 (br. *s*)	1.28 (*s*)
4-HO	–	–	8.46 (*s*)
6-HO	8.51 (*s*)	8.40 (*s*)	7.63 (*s*)
12-HO	8.66 (*s*)	6.79 (*s*)	8.17 (*s*)
14-HO	–	8.16 (*s*)	–

[a]) At 400 MHz. [b]) At 500 MHz.

Table 2. *^{13}C-NMR Data of* **1**–**3**. At 125 MHz in (D_6)acetone; δ in ppm. Arbitrary atom numbering.

	1	**2**	**3**		**1**	**2**	**3**
C(2)	152.6	156.1	151.3	C(16)	26.8	27.3	41.2
C(3)	107.9	105.6	103.5	C(17)	124.8	125.4[a])	29.1
C(4)	121.8	122.2	149.6	C(18)	130.4	132.3	29.1
C(5)	113.0	113.4	100.1	C(19)	17.8	18.5	150.2
C(6)	156.4	156.9	154.4	C(20)	25.8	26.2	109.6
C(7)	98.5	98.7	110.0	C(21)	121.8	23.8	26.9
C(8)	156.9	156.8	156.2	C(22)	127.6	123.9	125.3
C(9)	122.1	123.0	113.0	C(23)	78.6	136.5	130.1
C(10)	129.5	130.0	132.8	C(24)	26.5	16.7	17.8
C(11)	121.8	119.5	121.8	C(25)	41.7	40.9	25.9
C(12)	156.7	155.5	155.0	C(26)	23.4	27.8	21.6
C(13)	105.2	117.4	105.4	C(27)	125.1	125.5[a])	33.6
C(14)	153.3	154.9	153.5	C(28)	131.9	132.1	74.2
C(15)	114.7	108.8	113.6	C(29)	17.6	18.1	26.9
				C(30)	25.8	26.2	26.9

[a]) Signals may be exchangeable.

From all these data, the structure of planar artopetelin A (**1**) was deduced as 5-(6-hydroxybenzofuran-2-yl)-2-methyl-6-(3-methylbut-2-enyl)-2-(4-methylpent-3-enyl)-2*H*-1-benzopyran-7-ol. The configuration at C(23) remains to be determined.

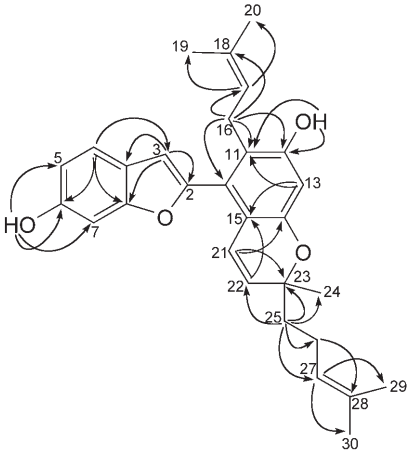

Fig. 1. *Selected HMBC correlations of compound* **1**

Artopetelin B (**2**), an orange, amorphous powder, had the molecular formula $C_{29}H_{34}O_4$, as determined by HR-EI-MS (*m/z* 446.2475 (M^+, calc. 446.2457)). The UV and IR properties of **2** were very similar to those of **1**. A comparison of the NMR data of these two compounds (*Tables 1* and *2*) showed that they shared the same ring-*A* moiety, but had different substitution patterns on ring *B*. The latter contained two OH, one prenyl, and one geranyl group(s) in the case of **2**. In the HMBC spectrum, the following correlations appeared (*Fig. 2*): the CH_2(16) group at δ(H) 3.54 was correlated with C(10) at δ(C) 130.0, C(11) (119.5), and C(12) (155.5); CH_2(21) at δ(H) 3.48 was correlated with C(12) (155.5), C(13) (117.4), and C(14) (154.9); the 12-OH group at δ(H) 6.79 was correlated with C(11) and C(13); and the 14-OH group at δ(H) 8.16 was correlated with C(13) and C(15). Hence, the prenyl, geranyl, and two OH substituents had to be located in the 11-, 13-, 12-, and 14-position, respectively.

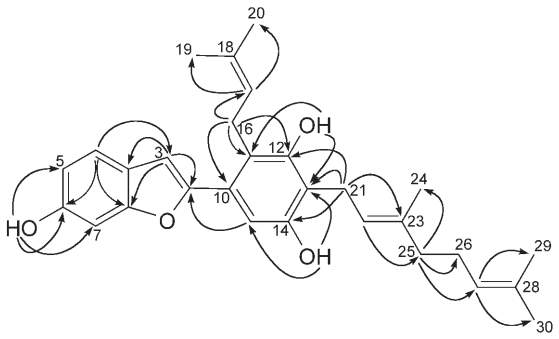

Fig. 2. *Selected HMBC correlations of compound* **2**

The (*E*)-configuration of the C(22)=C(23) bond in **2** was indicated by a NOESY cross-peak between H–C(22) and CH_2(25). From all these data, the structure of artopetelin B (**2**) was elucidated as 2-[(*E*)-3,7-dimethylocta-2,6-dienyl]-5-(6-hydroxy-1-benzofuran-2-yl)-4-(3-methylbut-2-enyl)benzene-1,3-diol.

Artopetelin C (**3**), a pale-yellow, amorphous powder, had the molecular formula $C_{29}H_{34}O_5$, as deduced by HR-EI-MS (*m/z* 462.2410 (M^+, calc. 462.2406)). Its UV and IR spectra also suggested the presence of a 2-arylbenzofuran skeleton. The ^{1}H-NMR spectrum exhibited signals of three OH groups at δ(H) 8.46, 8.17, and 7.63 (3*s*, 1 H each), three downfield resonances at δ(H) 6.66, 6.40, and 6.33 (3*s*, 1 H each), a prenyl group at δ(H) 5.11 (br. *t*, *J* = 6.8 Hz, 1 H), 3.27 (br. *d*, *J* = 6.8 Hz, 2 H), and 1.55 and 1.42 (2 br. *s*, 3 H each), as well as signals of a 1,1-dimethylallyl group at δ(H) 6.43 (*dd*, *J* = 10.6, 17.5 Hz, 1 H), 5.02 (*dd*, *J* = 1.3, 17.5 Hz, 1 H), 4.91 (*dd*, *J* = 1.3, 10.6 Hz, 1 H), and 1.64 (*s*, 6 H). In addition, a 3,4-dihydro-2,2-dimethylpyran moiety was identified from the following ^{1}H- and ^{13}C-NMR (HMQC) data: δ(H) 2.48, 1.69 (2*t*, *J* = 6.8 Hz, 2 H each); 1.28 (*s*, 6 H)); δ(C) 21.6 (C(26)), 33.6 (C(27)), 74.2 (C(28)), and 26.9 (C(29,30)). This latter moiety was fused at C(14) and C(15), as established by HMBC correlations between H–C(26) at δ(H) 2.48 and C(10) at δ(C) 132.8, C(14) (153.5), and C(15) (113.6), as well as by those between H–C(27) at δ(H) 1.69 and C(15) at δ(C) 113.6 (*Fig. 3*).

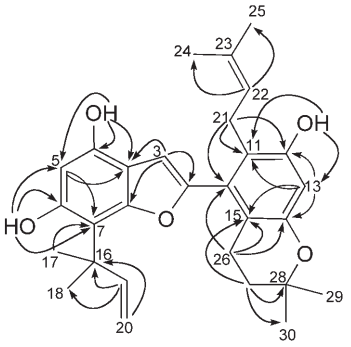

Fig. 3. *Selected HMBC correlations of compound* **3**

The prenyl group in **3** was located at C(11), on the basis of HMBC couplings between CH_2(21) at δ(H) 3.27 and C(10) at δ(C) 132.8, C(11) (121.8), and C(12) (155.0). The signal at δ(H) 8.17 was assigned to the 12-OH group, as shown in *Fig. 3*. The attachment of the remaining substituents on ring *A* was corroborated by HMBC cross-peaks between 4-OH at δ(H) 8.46 and C(4) at δ(C) 149.6, C(5) (100.1), and C(9) (113.0), between 6-OH at δ(H) 7.63 and C(5) at δ(C) 100.1, C(6) (154.4), and C(7) (110.0), as well as between Me(17,18) at δ(H) 1.64 and C(7). From these data, the structure of artopetelin C (**3**) was elucidated as 7-(1,1-dimethylprop-2-enyl)-2-[3,4-dihydro-7-hydroxy-2,2-dimethyl-6-(3-methylbut-2-enyl)-2*H*-1-benzopyran-5-yl]-1-benzofuran-4,6-diol.

Compounds **1**–**3** were found to be minor phenolic constituents of *A. petelotii*. It is interesting that, so far, we have not found any isoprenylated 2-arylbenzofurans in *A. chama*, which is rich in prenylated flavones [4e], both plants being collected at the same

time in Xishuangbanna, Yunnan province, China. Only a few 2-arylbenzofuran derivatives have been isolated from some *Artocarpus* species, with isoprenoid substituents in the form of prenyl, geranyl [3b], and 3,3-dimethylpropenyl groups [3d], or fused in the form of a 2,2-dimethylpyran ring [3a]. Therefore, artopetelin A (**1**) is the first 2-arylbenzofuran bearing a geranyl-derived 2-methyl-2-(4-methylpent-3-enyl)pyran ring from *Artocarpus*, although similar compounds have been found in *Morus* species [8]. Moreover, although 1,1-dimethylallyl groups appear quite commonly in xanthones [4a–d], artopetelin C (**3**) is the first 2-arylbenzofuran with this side chain isolated from Moraceous plants. Another such compound is burttinol D, which has been isolated from *Erythrina burttii* (Leguminosae) [9]. To the best of our knowledge, compound **3** is also the first natural 2-arylbenzofuran containing a 3,4-dihydro-2,2-dimethylpyran moiety.

Experimental Part

General. Column chromatography (CC): silica gel *H* (10–40 μm and 200–300 mesh; *Yantai Institute of Chemical Technology*, China) and *Chromatorex RP-18* gel (20–45 μm; *Fuji Silysia Chemical, Ltd.*, Kasugai, Japan). Prep. and anal. TLC: precoated silica-gel GF_{254} plates (10–40 μm; *Yantai Institute of Chemical Technology*, China). Optical rotation: *Jasco P1030* polarimeter. UV spectra: *Shimadzu UV-2401PC* spectrophotometer; λ_{max} (log ε) in nm. IR spectra: *Nicolet Avatar-360* spectrometer, KBr pellets; in cm^{-1}. NMR spectra: *Bruker DRX-400* and *-500* instruments; chemical shifts δ in ppm rel. to residual solvent peaks of (D_6)acetone (δ(H) 2.04, δ(C) 206.0). EI-MS (70 eV): *Finnigan MAT-95* mass spectrometer; in *m/z* (rel. %).

Plant Material. The root barks of *A. petelotii* GAGNEP were collected in Xishuangbanna, Yunnan, P. R. China, in July 1998, and air-dried. The plant was identified by Prof. *Han-Dong Sun*, Kunming Institute of Botany, and a voucher specimen (TCM 98-07-02 Hou) was deposited at the Herbarium of the Department of Pharmacognosy, School of Pharmacy, Fudan University.

Extraction and Isolation. The dried and powdered root barks (6.4 kg) of *A. petelotii* were percolated with 95% EtOH (60 l) at r.t. The filtrate was evaporated *in vacuo* to give a residue (800 g), which was suspended in H_2O (2 l), and extracted successively with petroleum ether (4 × 800 ml) and AcOEt (4 × 800 ml). The AcOEt extract (110 g after evaporation) was subjected to CC (SiO_2; petroleum ether/acetone 8:2 → 2:8): fractions *Fr. A–I. Fr. C* (3.7 g) was separated by CC (SiO_2; petroleum ether/i-PrOH 50:1 → 18:1): *Fr. C1–11. Fr. C5* was purified by CC (1. *RP-18*, MeOH/H_2O 4:1; 2. SiO_2, petroleum ether/AcOEt 8:1) to afford **1** (7 mg). *Fr. C6* was fractionated by CC (1. *RP-18*, MeOH/H_2O 7:3 → 17:3; 2. SiO_2, $CHCl_3$/AcOEt 14:1), followed by prep. TLC (SiO_2; $CHCl_3$/acetone 15:1), to afford **2** (10 mg). *Fr. D* (4.5 g) was subjected to CC (SiO_2; petroleum ether/i-PrOH 30:1 → 8:1) to give *Fr. D1–9. Fr. D3* was further purified by CC (1. *RP-18*, MeOH/H_2O 3:1; 2. SiO_2, $CHCl_3$/AcOEt 50:3) to provide **3** (50 mg).

Artopetelin A (*= 5-(6-Hydroxybenzofuran-2-yl)-2-methyl-6-(3-methylbut-2-enyl)-2-(4-methylpent-3-enyl)-2*H*-1-benzopyran-7-ol*; **1**). Yield: 7 mg. Pale-yellow, amorphous powder. $[\alpha]_D^{20} = -40.9$ ($c = 0.23$, acetone). UV (MeOH): 215 (4.52), 299 (4.20). IR (KBr): 3385, 2925, 2854, 1624, 1599, 1489, 1456, 1382, 1265, 1144, 1113, 840, 738. ^{1}H- and ^{13}C-NMR: see *Tables 1* and *2*, resp. EI-MS: 444 (12, M^+), 361 (100), 317 (8), 305 (13), 277 (5), 256 (10), 192 (8), 149 (9), 101 (13), 71 (15), 58 (84). HR-EI-MS: 444.2295 (M^+, $C_{29}H_{32}O_4^+$; calc. 444.2301).

Artopetelin B (*= 2-[(*E*)-3,7-Dimethylocta-2,6-dienyl]-5-(6-hydroxy-1-benzofuran-2-yl)-4-(3-methylbut-2-enyl)benzene-1,3-diol*; **2**). Yield: 10 mg. Orange, amorphous powder. UV (MeOH): 219 (4.44), 281 (sh, 4.15), 312 (4.36). IR (KBr): 3423, 2967, 2918, 2851, 1621, 1600, 1489, 1442, 1375, 1289, 1223, 1145, 1114, 1052, 976, 829. ^{1}H- and ^{13}C-NMR: see *Tables 1* and *2*, resp. EI-MS: 446 (100, M^+), 431 (12), 403 (16), 392 (10), 377 (5), 361 (27), 321 (62), 307 (50), 293 (15), 279 (45), 267 (95), 201 (29), 163(19), 69(37), 55(20). HR-EI-MS: 446.2475 (M^+, $C_{29}H_{34}O_4^+$; calc. 446.2457).

Artopetelin C (*= 7-(1,1-Dimethylprop-2-enyl)-2-[3,4-dihydro-7-hydroxy-2,2-dimethyl-6-(3-methylbut-2-enyl)-2*H*-1-benzopyran-5-yl]-1-benzofuran-4,6-diol*; **3**). Yield: 50 mg. Pale-yellow, amorphous powder. UV (MeOH): 222 (4.48), 291 (4.14). IR (KBr): 3423, 2973, 2927, 1609, 1418, 1370, 1325, 1266, 1160, 1147, 1120, 1051, 1029, 966, 738. ^{1}H- and ^{13}C-NMR: see *Tables 1* and *2*, resp. EI-MS: 462 (100, M^+), 447 (31), 419 (33), 407 (29), 391 (17), 351 (26), 256 (28), 213 (23), 149 (10), 69 (26), 57 (23). HR-EI-MS: 462.2410 (M^+, $C_{29}H_{34}O_5^+$; calc. 462.2406).

Financial support from the *Key Pharmaceutical Subjects of Shanghai Educational Committee* is gratefully acknowledged. We thank Prof. *Han-Dong Sun*, Kunming Institute of Botany, Academia Sinica, Kunming, P. R. China, for plant collection and authentication.

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Received April 21, 2005