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BONGOSIN: A NEW CHALCONE-DIMER FROM *LOPHIRA ALATA*

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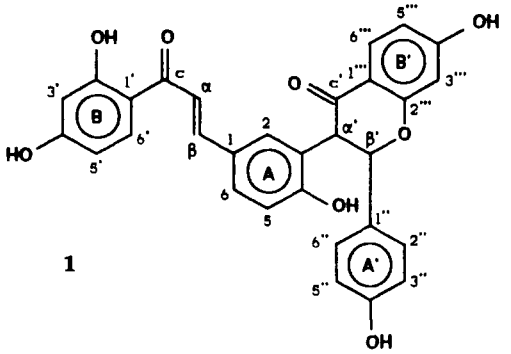
ABSTRACT.—From the stem bark of *Lophira alata*, a new chalcone dimer, bongosin [**2**], has been isolated along with five known biflavonoid compounds. The structure of **2** was established from spectroscopic and chemical evidence.

Lophira alata Banks ex Gaertn. f. (Ochnaceae) is a tall tree widespread in the tropical forests of West Africa and is extensively used in traditional medicine (1,2). Earlier phytochemical studies revealed leaf anthocyanin pigments and phenols (3,4). We recently reported the structure of mbamichalcone, a chalcone dimer, from the Et_2O extract of the stem bark (5). Further purification of the same extract has now furnished six other flavonoid compounds. Five have been identified as lophirones A–E previously described from *Lophira lanceolata* Van Tiegh ex Keay (6–8) by direct comparison of their spectroscopic parameters and with R_f values of authentic samples. The sixth compound, bongosin, is a new chalcone dimer for which structure **2** has been proposed from spectroscopic and chemical evidence.

Bongosin [**2**], $C_{30}H_{22}O_7$, $[\alpha]^{20}D + 1$ ($c = 0.21$, Me_2CO), was obtained as a dark brown amorphous solid. Its ir spectrum had absorption bands of OH groups (3250 cm^{-1}), a conjugated carbonyl group (1635 cm^{-1}), aromatic rings, and double bonds (1605 and 1515 cm^{-1}); the uv spectrum had intense absorptions at λ max 207, 225 sh, 250, and 260 nm, indicating a highly conjugated structure.

Evidence that bongosin [**2**] has five hydroxyl groups came from its complete acetylation (Ac_2O/pyridine), which gave an amorphous solid **3**, $C_{40}H_{32}O_{12}$. The ir spectrum of compound **3** had no residual OH absorption, and the cims showed an $[M+H]^+$ ion at *m/z* 705. Five sharp singlets at δ 2.31, 2.27, 2.21, 2.20, and 2.17 (each 3H) in the ^{1}H-nmr spectrum of **3** were indicative of a pentaacetate derivative.

The ^{1}H-nmr spectrum of bongosin [**2**] had signals for 14 protons and was very similar to that of lophirone B [**1**] (7); both had the same proton systems for the aromatic rings (A, B, A′, and B′) and the trans double bond. Examination of δ values of corresponding aromatic protons showed important differences for protons H-3‴, H-5‴, and H-6‴ (ring B′) that gave signals at δ 6.45, 6.62, and 7.80, respectively in lophirone B [**1**] but were shifted upfield in bongosin [**2**] by 0.22, 0.23, and 0.78 ppm (Table 1). This suggested that ring B′ in bongosin [**2**] had no adjacent carbonyl group. The AB system of aliphatic protons (H-α′ 4.53 ppm and H-β′ 5.93 ppm) in the spectrum of lophirone B was replaced by two proton singlets (each 1H) at 6.47 and 7.07 ppm in the spectrum of bongosin. The signal at 6.47 ppm showed long-range coupling with equivalent protons H-2″ and H-6″ (7.33 ppm, ring A′), while that at 7.07 ppm was correlated with H-6‴ (7.02 ppm, ring B′) suggesting structure **2** for bongosin. The relative down-field shift ($\Delta\delta = 0.23$ ppm) of the proton H-2 (ring A) in bongosin [**2**] may be attrib-



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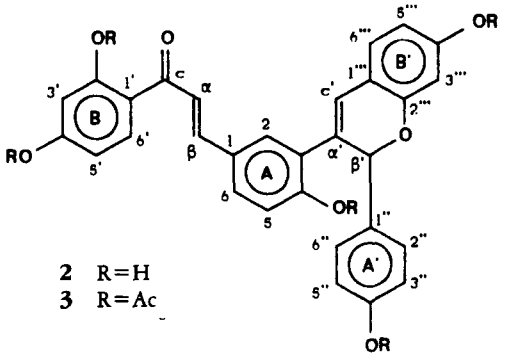
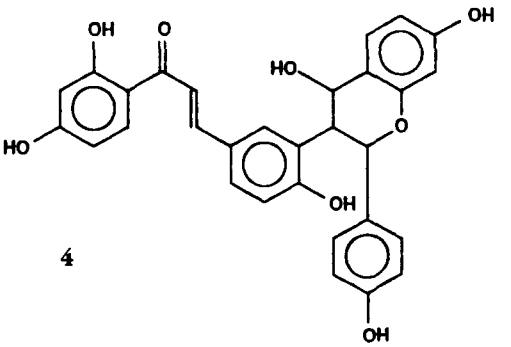


2 R=H
3 R=Ac



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uted to the relief from the anisotropic shielding due to the carbonyl group.

Ms data for bongosin is consistent with structure **2**. The base peak at *m/z* 121 was assigned to the para hydroxybenzaldehyde anion, while cleavage of the interflavonoid bond gave ions at *m/z* 239 and 255. Important fragments at *m/z* 155, 199, and 243 were rearrangement peaks. Biosynthetically, **2** could have been derived from **1** by a selective reduction of the c′ carbonyl to obtain **4**, which further undergoes dehydration.

EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—Melting points are uncorrected and were taken on a Kofler microscope. Nmr spectra (^1H and ^{13}C) were recorded in Me_2CO-d_6 solutions on a Bruker NM250 spectrometer. Fabms were obtained on a VG.NM.2AB.HF spectrometer, while cims were obtained on a Riber Nermag U30 spectrometer with NH_3 as ionizing gas. Cc was conducted on Merck Si gel of particle size 0.04–0.063 mm.

PLANT MATERIAL.—The stem bark of *L. alata* was harvested in Balamba, Mbam, Cameroon, in April 1988. A voucher specimen was deposited at the National Herbarium in Yaounde, Cameroon.

EXTRACTION AND PURIFICATION.—The powdered, sun-dried stem bark (5 kg), extracted with cold Et_2O, gave a dark brown gum (42 g) after removal of solvent. This was first fraction-

TABLE 1. ^{1}H-nmr Data for Compounds **1**, **2**, and **3** (250 MHz, Me_2CO-d_6, TMS).

Carbon	Compound					
	1		**2**		**3**	
	δ (ppm)	*J* (Hz)	δ (ppm)	*J* (Hz)	δ (ppm)	*J* (Hz)
C-3′	6.35 d	2.2	6.36 d	2.4	7.08 d	2.3
C-5′	6.43 dd	9.0, 2.3	6.48 dd	8.9, 2.4	7.21 dd	8.4, 2.3
C-6′	8.00 d	9.0	8.02 d	8.9	7.87 d	8.4
C-α	7.64 d	15.4	7.68 d	15.4	7.38 d	15.9
C-β	7.71 d	15.4	7.77 d	15.4	7.55 d	15.9
C-2	7.51 d	2.1	7.74 d	2.1	7.88 d	2.3
C-5	6.83 d	8.1	6.97 d	8.4	7.74 dd	8.5, 2.3
C-6	7.50 dd	8.1, 2.1	7.58 dd	8.4, 2.1	7.24 d	8.5
C-3‴	6.45 d	2.2	6.23 d	2.4	6.56 d	1.9
C-5‴	6.62 dd	8.6, 2.2	6.39 dd	8.2, 2.4	6.69 dd	8.4, 1.9
C-6‴	7.80 d	8.6	7.02 d	8.2	7.27 d	8.4
C-c′	—		7.07 s		7.00 s	
C-α′	4.53 d	12.2	—		—	
C-β′	5.93 d	12.2	6.47 s		6.41 s	
C-2′	7.30 m		7.33 m		7.50 m	
C-3″	6.72 m		6.72 m		7.04 m	
C-5″	6.72 m		6.72 m		7.04 m	
C-6″	7.30 m		7.33 m		7.50 m	
Ac					2.31 s	
					2.27 s	
					2.21 s	
					2.20 s	
					2.17 s	

ated by cc with a gradient mixture of hexane-EtOAc (3:2). Fractions obtained were then purified by cc and finally by repeated preparative tlc eluted with solvent mixture CH_2Cl_2-MeOH (10:1) to give six pure compounds, five of which were identified ([α]D, mp, ir, ^{1}H- and ^{13}C nmr, and co-tlc) as lophirones A–E (6–8).

Lophirone B [**1**].—Pale yellow crystals: mp 251–253° (Me_2CO); $[\alpha]^{25}D$ +7° (*c* = 0.4, Me_2CO); uv λ max (EtOH) nm (log ε) 215 (4.55), 230 (4.43), 273 (4.23), 312 (4.20), 378 (4.49).

Lophirone C.—Yellow crystals: mp 191–193° (Me_2CO); uv λ max (EtOH) nm (log ε) 207 (4.51), 225 (4.34), 285 (4.27), 335 (4.36), 372 (4.49).

Lophirone D.—Yellow crystals: mp 270–276° (Me_2CO); uv λ max (EtOH) nm (log ε) 207 (4.34), 215 (4.25), 275 (4.30), 315 (4.56), 365 (4.81).

Lophirone E.—Yellow crystals: mp 266–268° (Me_2CO); uv λ max (EtOH) nm (log ε) 207 (4.47), 215 (4.38), 311 (4.64), 363 (4.54).

Bongosin [**2**].—Amorphous brown solid: $C_{30}H_{12}O_7$ from hrms; uv λ max (EtOH) nm (log ε) 207 (4.63), 225 (4.50), 250 (4.28); ir (KBr) ν cm^{-1} 3250, 1627, 1515, 1228, 849; ^{1}H nmr (250 MHz, acetone-d_6) see Table 1; fabms *m/z* (%) $[M-H]^-$ 493 (5), 243 (70), 199 (68), 155 (25), 154 (11), 153 (16), 121 (100), 119 (27), 102 (14).

Acetylation of **2**.—Bongosin [**2**] (5 mg) was dissolved in pyridine (1 ml), and Ac_2O (1 ml) was added. The mixture was left overnight at 50°, after which it was concentrated under vacuum, giving a powder which was purified by cc on Si gel with *n*-C_6H_{14}–EtOAc (1:1) *to give* **3** (3 *mg*): $C_{40}H_{32}O_{12}$ from hrms; cims *m/z* $[M+H]^+$ 705; ^{1}H nmr see Table 1.

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