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## A NEW NAPHTHOQUINONE FROM *BOMBAX MALABARICUM*

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A new naphthoquinone **1** together with 7-hydroxycadalene (**2**) and 8-formyl-7-hydroxy-5-isopropyl-2-methoxy-3-methyl-1,4-naphthoquinone (**3**) were isolated from the heartwood of *Bombax malabaricum*. The new naphthoquinone was characterized as 7-hydroxy-5-isopropyl-2-methoxy-3-methyl-1,4-naphthoquinone (**1**) based on spectral and chemical studies.

*Keywords:* *Bombax malabaricum*; Bombacaceae; Naphthoquinone

### INTRODUCTION

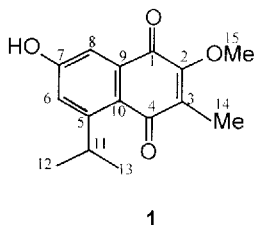
*Bombax malabaricum* DC (Bombacaceae), syn. *Salmalia malabaricum* DC, is a medium sized deciduous tree found throughout the hotter regions of India and Andamans [1,2]. It is widely used in indigenous medicine as demulcent, diuretic, aphrodisiac, emetic and for curing impotence [3]. Previous investigations on the root bark of this species has resulted in the isolation of several new sesquiterpenoids [4–6]. In the present investigation we have examined the heartwood of this species and report here the isolation and characterization of a new naphthoquinone 7-hydroxy-5-isopropyl-2-methoxy-3-methyl-1,4-naphthoquinone (**1**), besides 7-hydroxycadalene (**2**) and 8-formyl-7-hydroxy-5-isopropyl-2-methoxy-3-methyl-1,4-naphthoquinone (**3**).

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## RESULTS AND DISCUSSION

Compound **1** was crystallized from methanol as yellow crystals mp 158–159°C. The EIMS of **1** showed a molecular ion at  $m/z$  260 and the HRFABMS exhibited the protonated molecular ion at  $m/z$  261.1115 consistent with the molecular formula  $C_{15}H_{16}O_4$ . The UV absorption maxima at 206, 267, 286 and 343 nm and a positive response to sodium dithionite reagent suggested that **1** could be a quinonoid derivative [7]. An *ortho* quinonoid structure in **1** has been ruled out since it did not form a quinoxaline derivative with *o*-phenylenediamine [8]. The IR absorption bands at 3410 and 1665 and 1630  $cm^{-1}$  were attributed to hydroxyl and two quinonoid carbonyls, respectively. The presence of quinonoid carbonyls in **1** was further substantiated by the appearance of two downfield carbon resonances at 187.03 and 181.77 ppm in its  $^{13}C$  NMR spectrum.



The  $^1H$  NMR spectrum of **1**, showed the presence of an isopropyl group ( $\delta$  1.25, *d*, 6H,  $J$  = 6.8 Hz;  $\delta$  4.34, sept, 1H,  $J$  = 6.8 Hz), a methyl ( $\delta$  2.08, *s*, 3H) and a methoxyl ( $\delta$  4.03, *s*, 3H) group. A broad  $D_2O$  exchangeable signal at  $\delta$  6.70 was assigned to a phenolic hydroxyl group whose presence was supported by the formation of a monoacetate ( $M^+$ , 302). It also showed the presence of two *meta* coupled ( $J$  = 2.7 Hz) aromatic protons at  $\delta$  7.51 and 7.17.

In the HMBC spectrum of **1** (Fig. 1), the correlation of methine proton signal ( $\delta$  4.34) of isopropyl group with the carbon signals at 156.63 ppm

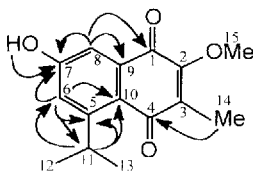


FIGURE 1 Significant HMBC correlations observed in **1**.

(C-5), 119.09 ppm (C-6) and 122.19 ppm (C-10), and the reverse correlation of the proton signal at  $\delta$  7.17 (H-6) with the carbon signals at 156.63 ppm (C-5), 122.18 ppm (C-10) and 29.20 ppm (C-11) located the isopropyl group at C-5 position. The non-chelated hydroxyl at  $\delta$  6.70 was placed at C-7 as it correlated with this carbon at 161.83 ppm which in turn showed cross correlation with the *meta* coupled aromatic protons which fixes the attachment of aromatic protons at  $\delta$  7.17 and 7.51 to C-6 and C-8 positions, respectively. The foregoing spectral studies suggested that both the methoxyl and methyl groups should be present in the quinonoid ring. The HMBC correlation of the H-8 signal with the carbon at 187.03 ppm (C-1) and the correlation of the methyl protons signal with the carbon signal at 181.77 ppm (C-4) fixes the attachment of the methyl group at C-3 position. Therefore the methoxy group should be present at C-2 position. The presence of a methoxyl and a methyl group at 2 and 3 positions, respectively of quinonoid ring in **1** was further evidenced by the appearance of a fragment at  $m/z$  228  $[\text{M}-\text{CH}_3\text{OH}]^+$  which could have resulted due to an *ortho* effect [6]. Thus compound **1** was characterized as 7-hydroxy-5-isopropyl-2-methoxy-3-methyl-1,4-naphthoquinone (**1**).

## EXPERIMENTAL SECTION

### General Experimental Procedures

Mps were determined on a Koffler hot-stage apparatus and are uncorr. UV spectra was taken in MeOH on a Shimadzu 240 double beam spectrophotometer. IR spectra was recorded in KBr on a Perkin-Elmer 283B double beam spectrophotometer.  $^1\text{H}$  and  $^{13}\text{C}$  NMR experiments were performed on a Bruker AC-300 spectrometer using  $\text{CDCl}_3$  with TMS as int. standard. HMBC was recorded on a Bruker AC-300 spectrometer using standard pulse sequences. EIMS was obtained on a VG Micromass 7070F instrument at 70 eV. HRFABMS was obtained on a 700 JEOL mass spectrometer.

### Plant Material

The heartwood of *Bombax malabaricum* was collected from Tirumala Hills, S. India in January, 1993. A voucher specimen (DG-931) documenting its collection was deposited in the herbarium of the Department of Botany, Sri Venkateswara University, Tirupati.

### Extraction and Isolation

The air dried and powdered heartwood of *B. malabaricum* (1 kg) was successively extracted with *n*-hexane, C<sub>6</sub>H<sub>6</sub> and CHCl<sub>3</sub> at room temp. The concentrated C<sub>6</sub>H<sub>6</sub> extract (50 g) was triturated with hexane and the hexane-soluble part after concentration followed by recrystallization from CH<sub>2</sub>Cl<sub>2</sub> afforded **2** (100 mg). The CHCl<sub>3</sub> extract was found to be a mixture of two compounds and was subjected to column chromatography over silica gel using C<sub>6</sub>H<sub>6</sub>-EtOAc step gradients to yield **1** (80 mg) and **3** (110 mg).

### 7-Hydroxy-5-isopropyl-2-methoxy-3-methyl-1,4-naphthoquinone

(1) Yellow crystals from MeOH, 80 mg, mp 158–159°C; UV (MeOH)  $\lambda_{\max}$  (log  $\epsilon$ ) 206 (4.52), 267 (4.41), 286 (4.12), 343 (3.50) nm; IR (KBr)  $\nu_{\max}$  3410 (OH), 2925, 1665 (>C=O), 1630 (>C=O), 1605 (C=C), 1560 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.51 (*d*, 1H, *J*=2.7 Hz, H-8), 7.17 (*d*, 1H, *J*=2.7 Hz, H-6), 6.70 (*br s*, 1H, OH-7), 4.34 [Sept. 1H, *J*=6.8 Hz, —CH(CH<sub>3</sub>)<sub>2</sub>], 4.03 (*s*, 3H, OCH<sub>3</sub>-2), 2.08 (*s*, 3H, CH<sub>3</sub>-3), 1.25 [*d*, 6H, *J*=6.8 Hz, —CH(CH<sub>3</sub>)<sub>2</sub>]; <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  187.03 (C-1), 181.77 (C-4), 161.83 (C-7), 156.63 (C-5), 155.38 (C-2), 136.32 (C-9), 133.72 (C-3), 122.18 (C-10), 119.09 (C-6), 118.82 (C-8), 60.68 (C-15), 29.20 (C-11), 23.68 (C-12 and C-13), 9.70 (C-14); EIMS *m/z* 260 [M]<sup>+</sup> (100), 245 (54), 243 (9), 231 (14), 229 (28), 228 (33), 227 (12), 217 (16), 202 (12), 201 (9), 199 (9); HRFABMS *m/z* 261.1115 [M+H]<sup>+</sup> (calcd. for C<sub>15</sub>H<sub>17</sub>O<sub>4</sub> 261.1103).

### Acetylation of 1

A mixture of compound **1** (5 mg), Ac<sub>2</sub>O (1.5 ml) and C<sub>5</sub>H<sub>5</sub>N (1 ml) was kept at room temp. for 72 h, and poured into crushed ice to yield the monoacetate as colourless crystals (5 mg) from Me<sub>2</sub>CO, mp 107–108°C; IR (KBr)  $\nu_{\max}$  2923, 1756 (>C=O of OAc) 1658 (>C=O) 1625 (>C=O), 1610, 1560 cm<sup>-1</sup>; <sup>1</sup>H NMR. (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.69 (*d*, 1H, *J*=2.4 Hz, H-8), 7.39 (*d*, 1H, *J*=2.4 Hz, H-6), 4.36 [Sept. 1H, *J*=6.8 Hz, —CH(CH<sub>3</sub>)<sub>2</sub>] 4.06 (*s*, 3H, OCH<sub>3</sub>-2), 2.44 (*s*, 3H, CH<sub>3</sub>-3), 2.06 (*s*, 3H, OAc-7), 1.26 [*d*, 6H, *J*=6.8 Hz, —CH(CH<sub>3</sub>)<sub>2</sub>]; EIMS *m/z* 302 [M]<sup>+</sup> (2), 260 [M-42] (100), 245 (60), 229 (28), 217 (16), 202 (12).

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