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# Two oligostilbenes, *cis*- and *trans*-diptoindonesin B, from *Dryobalanops oblongifolia*

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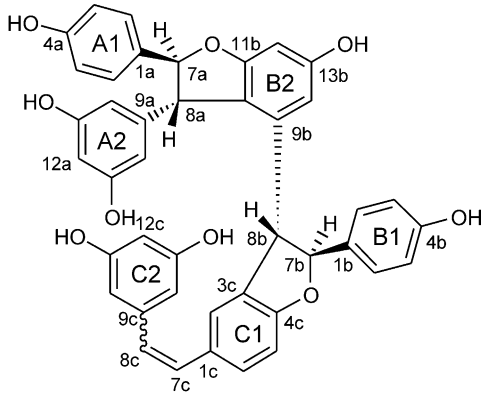
**Abstract**

Two oligostilbenes, *cis*- and *trans*-diptoindonesin B, have been isolated from the tree bark of *Dryobalanops oblongifolia* (Dipterocarpaceae). The structures and relative configurations of both compounds were determined on the basis of spectroscopic evidence, including 2D-NMR spectroscopic analysis.


*Keywords:* Dipterocarpaceae; *cis*- and *trans*-diptoindonesins B; *Dryobalanops oblongifolia*; oligostilbenes

## 1. Introduction

A number of oligostilbenes have been isolated from several plant families: Cyperaceae, Dipterocarpaceae, Gnetaceae, Leguminosae and Vitaceae (Sotheeswaran and Pasupathy, 1993). In dipterocarpaceous plants, a great number of these compounds have been isolated from species belonging to the genera *Balanocarpus* (Sotheeswaran and Pasupathy, 1993), *Hopea* (Sotheeswaran and Pasupathy, 1993; Dai et al., 1998; Tanaka et al., 2000a, 2001a), *Shorea* (Saraswathy et al., 1992; Sotheeswaran and Pasupathy, 1993; Ito et al., 2000a, 2000b; Hirano et al., 2001; Tanaka et al., 2001b), *Vateria* (Sotheeswaran and Pasupathy, 1993; Ito et al., 2001a) and *Vatica* (Sotheeswaran and Pasupathy, 1993; Seo et al., 1999; Tanaka et al., 2000b, 2000c; Ito et al., 2001b; 2001c; Zgoda-Pols et al., 2002). They include stilbene dimers, trimers, tetramers, hexamers, heptamers and octamers with various molecular frameworks resulting from different oxidative condensations of resveratrol monomers. As part of a programme concerned with chemistry of the Indonesian tropical plants (Hakim, 2002; Hakim et al., 2002a, 2002b, Suhartati et al., 2001; Syah et al., 2001, 2002a, 2002b; Ersam et al., 2002), we have examined several species of Dipterocarpaceae, and isolated oligostilbenoids, including a stilbene dimer diptoindonesin A (Aminah et al., 2002). In pursuing our studies on the isolation of oligostilbenoids, we have also examined a rare species of Dipterocarpaceae endemic to Indonesia, *Dryobalanops oblongifolia* Dyer. Previous phytochemical investigation of the species had been restricted to the isolation of terpenoids (Bisset et al., 1967). This paper reports the isolation and structure determination of two stilbene trimers, which we have named *cis*- (**1**) and *trans*-diptoindonesin B (**2**), from the acetone extract of the tree bark of this plant.




**1**: 7c,8c = *cis*
**2**: 7c,8c = *trans*

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## 2. Results and discussion

The dried and powdered tree bark (2.5 kg) of *D. oblongifolia* was macerated in acetone. The acetone-ether soluble fraction of the acetone extract was subjected to vacuum liquid chromatography on silica gel (gradient system of *n*-hexane–EtOAc) to give four major fractions A–D (0.3, 2.0, 1.6 and 30 g, respectively). Repeated purification of fraction C by radial chromatography on silica gel (gradient system of $CHCl_3$–MeOH, *n*-hexane–$CHCl_3$–MeOH or *n*-hexane-acetone) gave *cis*-diptoindonesin B (**1**) (40 mg) and *trans*-diptoindonesin B (**2**) (30 mg).

*cis*-Diptoindonesin B (**1**), obtained as a white amorphous powder, $[\alpha]_D^{20}-99^\circ$ (c 0.1, MeOH), displayed a molecular formula of $C_{42}H_{32}O_9$ based on HRFABMS ($[M+H]^+$ *m/z* 681.2115) corresponding to a molecular formula of a resveratrol trimer. The UV (204, 230, 289 nm) and IR (3396, 1602, 1517, 1485, 1449, 1002, 833 $cm^{-1}$) spectra of **1** are typical for a phenolic oligostilbene containing a *cis*-stilbene chromophore (Mattifi and Reniero, 1996). The $^1H$ and $^{13}C$ spectral NMR data of **1** (Table 1), together with the $^1H$–$^1H$ COSY and HMQC spectra, showed a series of aromatic signals assignable to two 4-hydroxyphenyl groups (rings A1 and B1), two 3,5-dihydroxyphenyl groups (rings A2 and C2), a 3,5-dihydroxy-1,2-disubstituted benzene ring (ring B2), and a 4-hydroxy-1,3-disubstitutedbenzene ring (ring C1). The NMR spectral data also disclosed the presence of two sets of aliphatic signals characteristic for a 2,3-diaryldihydrobenzofuran moiety (H-7a/H-8a and H-7b/H-8b), in addition to a *cis*-1,2-disubstituted vinyl group ($J=12.2$ Hz, H-7c/8c). These NMR spectrascopic data established the structure of *cis*-diptoindonesin B the arrangement of which resembles those of gnetin E (Boralle et al., 1993), miyabenol C (Kurihara et al., 1991; Ono et al., 1995; Meng et al., 2001) and viniferal (Ito and Niwa, 1996), particularly those involving resveratrol units A and B. Supporting evidence for the proposed structure of **1** was obtained from HMBC measurements (Table 1). The important correlations of the HMBC measurements were found between a broad singlet at $\delta_H$ 6.62 (H-2c) with carbon signals at C-4c, C-6c and C-8b, between a doublet at $\delta_H$ 6.59 (H-5c) with carbon signals at C-1c and C-4c, and between a broad doublet at $\delta_H$ 7.08 (H-6c) with carbon signals at C-2c, C-4c and C-7c, confirming the nature of ring C1 as shown in structure **1**. A complete list of HMBC correlations, in agreement with structure **1**, is shown in Table 1. The relative stereochemistry at C-7a, C-8a, C-7b and C-8b of **1** was determined by NOESY experiments (Fig. 1). Significant NOE interactions between H-7a and H-10a(14a), H-8a and H-2a(6a), H-7b and H-14b, and H-8b and H-2b(6b), confirmed the *trans* relationship of both H-7a/H-8a and H-7b/H-8b. The NOESY spectrum also showed correlations between the signals of H-2c and H-10a(14a), H-8a and H-8b, and H-8b and H-10a(14a). From these data, the relative configurations of H-7a, H-8a, H-7b and H-8b of **1** were assigned to be *rel*-(7a*S*,8a*S*,7b*S*,8b*S*). This configuration of **1** was reinforced by the fact that the protons of H-2c and H-10a(14a) were shielded due to anisotropic effects given by rings A2 to C1 and *vice versa* (Fig. 1).

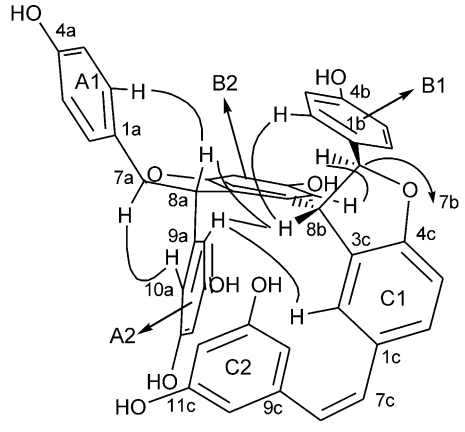


Fig. 1. Selected NOE correlations for compound **1**.

*trans*-Diptoindonesin B (**2**) was obtained as a white amorphous powder, $[\alpha]_D^{20}-192$ (c 0.1, MeOH). HRFABMS ($[M+H]^+$ *m/z* 681.2054) showed a molecular formula of $C_{42}H_{32}O_9$, corresponding to a resveratrol trimer. The UV (204, 231, 310 nm in MeOH) and IR (3366, 1599, 1516, 1486, 1450, 999, 832 $cm^{-1}$) spectra of **2** were characteristic of an oligostilbene containing a *trans*-stilbene chromophore (Mattifi and Reniero, 1996). The NMR spectral data of **2** (Table 2) were found to be very similar to those of **1**, except for the presence of a *trans*-, instead of a *cis*, 1,2-disubstituted vinyl group in the $^1H$ NMR spectrum ($\delta_H$ 6.89 and 6.76, each 1H, *d*, $J=16.2$ Hz). The HMQC and HMBC spectra (Table 1) confirmed the structure of **2**, while the relative stereochemistry was determined by NOESY spectrum as shown in Fig. 2. By the same arguments as those described for the stereostructure of **1**, the relative sterochemistry of **2** was found to be *rel*-(7a*S*,8a*S*,7b*S*,8b*S*).

A number of stilbene trimers have been isolated from several *Shorea* and *Vatica* species (Hakim, 2002). They represent a complex arrangement of stilbene oligomers containing five-, seven-, eight- and nine-membered rings, in addition to dihydrofuran rings. The trimers represented by *cis*- (**1**) and *trans*-diptoindonesin B (**2**) contain only dihydrofuran rings and, thus, inconsistent with the trimers found in the dipterocarpaceaeous plants. However, a trimer that was structurally comparable to that of **1** and **2** has recently been isolated from a *Shorea* species, i.e. ampelopsin E from *S. pinanga* (Jayuska et al., 2001). Therefore, the occurrence of these compounds in *D. oblongifolia* and *S. pinanga*

Table 1
NMR spectroscopic data of compounds **1** and **2**[a]

| No. | Compound **1** | | | Compound **2** | | |
|---|---|---|---|---|---|---|
| | $\delta_H$ (*mult.*, *J* in Hz) | $\delta_C$ | HMBC (H⟺C) | $\delta_H$ (*mult.*, *J* in Hz) | $\delta_C$ | HMBC (H⟺C) |
| **1a** | – | 133.9 | – | – | 134.0 | – |
| **2a/6a** | 6.91 (*d*, 8.6) | 128.1 | C-4a, C-3a/5a, C-6a/2a, C-7a | 6.96 (*d*, 8.5) | 128.1 | C-3a/5a, C4a, C-6a/2a, C-7a |
| **3a/5a** | 6.74 (*d*, 8.6) | 116.3 | C-1a, C-2a/6a, C-4a, C-5a/3a | 6.75 (*d*, 8.5) | 116.3 | C-1a, C-4a, C-5a/3a |
| **4a** | – | 158.4 | – | – | 158.4 | – |
| **7a** | 5.14 (*d*, 5.2) | 94.5 | C-1a, C-2a/6a, C-8a, C-9a, C-10b, C-11b | 5.23 (*d*, 4.9) | 94.5 | C-1a, C-2a/6a, C-8a, C-9a, C-10b, C-11b |
| **8a** | 3.49 (*d*, 5.2) | 56.8 | C-1a, C-7a, C-9a, C-10a/14a, C-9b, C-10b, C-11b | 3.68 (*d*, 4.9) | 56.9 | C-1a, C-7a, C-10a/14a, C-9b, C-10b |
| **9a** | – | 147.6 | – | – | 147.9 | – |
| **10a/14a** | 5.84 (*d*, 2.1) | 107.1 | C-8a, C-11a/13a, C-12a, C-14a/10a | 5.93 (*d*, 2.1) | 107.1 | C-8a, C-11a/13a, C-12a, C-14a./10a |
| **11a/13a** | - | 159.8 | – | – | 160.0 | – |
| **12a** | 6.09 (*t*, 2.1) | 102.2 | C-10a/14a, C-11a/13a | 6.13 (*t*, 2.1) | 102.3 | C-10a/14a, C-11a/13a |
| **1b** | – | 131.9 | – | – | 132.4 | – |
| **2b/6b** | 6.98 (*d*, 8.5) | 128.9 | C-4b, C-6b/2b, C-7b | 7.01 (*d*, 8.5) | 128.6 | C-4b, C-6b/2b, C-7b |
| **3b/5b** | 6.67 (*d*, 8.5) | 116.4 | C-1b, C-4b, C-5b/3b | 6.68 (*d*, 8.5) | 116.4 | C-1b, C-4b, C-5b/3b |
| **4b** | – | 158.8 | – | – | 158.7 | – |
| **7b** | 5.13 (*d*, 10.0) | 95.0 | C-1b, C-2b/6b, C-8b, C-9b | 5.27 (*d*, 9.1) | 95.0 | C-1b, C-2b/6b, C-9b |
| **8b** | 4.28 (*d*, 10.0) | 55.5 | C-1b, C-7b, C-9b, C-10b, C-14b, C-2c, C-3c, C-4c | 4.33 (*d*, 9.1) | 55.3 | C-1b, C-7b, C-9b, C-10b, C-14b, C-4c |
| **9b** | – | 140.3 | – | – | 141.0 | – |
| **10b** | – | 122.0 | – | – | 122.2 | – |
| **11b** | – | 162.6 | – | – | 162.5 | – |
| **12b** | 6.25 (*d*, 2.1) | 96.8 | C-10b, C-11b, C-13b, C-14b | 6.27 (*d*, 2.1) | 96.9 | C-10b, C-11b, C-13b, C-14b |
| **13b** | – | 160.2 | – | – | 160.5 | – |
| **14b** | 6.18 (*d*, 2.1) | 108.1 | C-8b, C-10b, C-12b, C-13b | 6.19 (*d*, 2.1) | 108.2 | C-10b, C-12b, C-13b |
| **1c** | – | 131.7 | – | – | 132.4 | – |
| **2c** | 6.62 (*brs*) | 127.2 | C-8b, C-4c, C-6c | 6.84 (*brs*) | 123.6 | C-8b, C-4c, C-7c |
| **3c** | – | 131.1 | – | – | 132.2 | – |
| **4c** | – | 160.5 | – | – | 161.1 | – |
| **5c** | 6.59 (*d*, 8.3) | 109.7 | C-1c, C-3c, C-4c | 6.75 (*d*, 8.2) | 110.2 | C-1c, C-3c, C-4c |
| **6c** | 7.08 (*brd*, 8.3) | 130.3 | C-2c, C-4c, C-7c | 7.25 (*brd*, 8.2) | 128.8 | C-2c, C-4c, C-7c |
| **7c** | 6.36 (*d*, 12.2) | 131.1 | C-1c, C-2c, C-6c, C-8c, C-9c | 6.89 (*d*, 16.2) | 129.3 | C-1c, C-2c, C-6c, C-8c, C-9c |
| **8c** | 6.29 (*d*, 12.2) | 129.5 | C-1c, C-7c, C-9c, C-10/14c | 6.76 (*d*, 16.2) | 127.4 | C-1c, C-7c, C-9c, C-10c/14c |
| **9c** | – | 140.9 | – | – | 141.3 | – |
| **10c/14c** | 6.22 (*d*, 2.1) | 108.6 | C-8c, C-11c/13c, C-12c, C-14c/10c | 6.45 (*d*, 2.1) | 105.9 | C-8c, C-11c/13c, C-12c, C-14c/10c |
| **11c/13c** | – | 159.1 | – | – | 159.6 | – |
| **12c** | 6.12 (*t*, 2.1) | 102.6 | C-10c/14c, C-11c/13c | 6.15 (*t*, 2.1) | 102.7 | C-10c/14c, C-11c/13c |

[a] Measured in methanol-$d_4$ at 500 MHz ($^1$H) and 125 MHz ($^{13}$C).

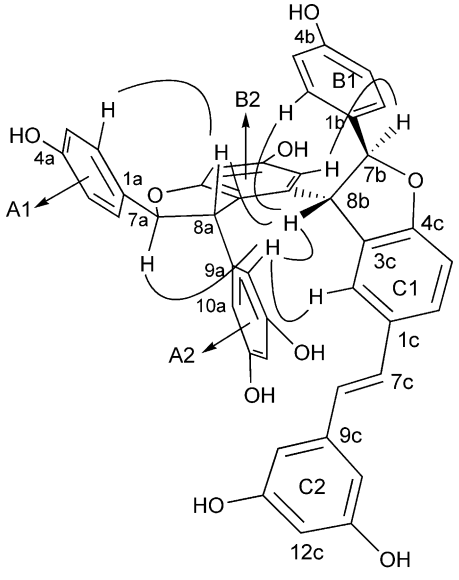


Fig. 2. Selected NOE correlations for compound **2**.

are of significance as regards their taxonomic relationships. Moreover, stilbene oligomers containing structures of similar arrangement to those **1**–**2** and ampelopsin E, are frequently isolated from the species of Gnetaceae, Cyperaceae (Sotheeswaran and Pasupathy, 1993), and Vitaceae (Masatake and Yoshiaki, 2001).

## 3. Experimental

### 3.1. General experimental procedure

All melting points were determined on a micro-melting point apparatus and are uncorrected. UV and IR spectra were measured with UV/VIS Varian Cary 100 Conc and One Perkin-Elmer spectrophotometers, respectively. $^1$H and $^{13}$C NMR spectra were recorded with JEOL JNM A500 spectrometer, operating at 500.1

MHz ($^1$H) and 125.65 MHz ($^{13}$C) using TMS as an internal standard. MS were obtained with a JEOL JMS-AM20 mass spectrometer, using the FAB mode. VLC was carried out using Merck Si-gel 60 GF254, flash chromatography with Merck Si-gel 60 (230–400 mesh), and TLC analysis on precoated Si-gel plates (Merck Kieselgel 60 $F_{254}$, 0.25 mm). All solvents were of technical grades and were distilled before use.

### 3.2. Plant material

Samples of *D. oblongifolia* were collected in November 2001 from the experimental garden of Haurbentes, Bogor, West Java, Indonesia. The plant was identified by the staff at the Herbarium Bogoriense, Bogor Botanical Garden, Bogor, Indonesia, and a voucher specimen had been deposited at the herbarium.

### 3.3. Extraction and Isolation

The dried powdered tree bark (2.5 kg) of *D. oblongifolia* was macerated with acetone (3×8 L) and the acetone extract was concentrated to a volume of ca. 250 mL. Diethyl ether was added to the concentrated acetone extract to obtain ether-soluble (40 g) and-insoluble (60 g) fractions. The ether-soluble fraction was fractionated by VLC (silica gel, *n*-hexane–EtOAc of increasing polarity) into four major fractions (A–D). From TLC analysis, fraction C was chosen for further analysis. Fraction C (1.6 g), therefore, was further fractionated by radial chromatography (silica gel, $CHCl_3$–MeOH = 9:1) to give two fractions C1 (70 mg) and C2 (70 mg). Fraction C1, on repeated purification using radial (silica gel, *n*-hexane-acetone = 65:35) and flash (silica gel, $CHCl_3$–MeOH = 8:2), gave compound **1** (40 mg). Using the same method (silica gel, *n*-hexane–$CHCl_3$–MeOH = 1:8:1; silica gel, $CHCl_3$–MeOH = 8:2, respectively) from fraction C2 afforded compound **2** (30 mg).

#### 3.3.1. cis-Diptoindonesin B (1)

White amorphous powder; $[\alpha]_D^{20}$ −99° (MeOH; *c* 0.1); UV $\lambda_{max}$ (MeOH) (log ϵ): 204 (3.08), 230 (3.56), 289 (3.11) nm; IR $\nu_{max}$ (KBr): 3396 (OH), 1602, 1517, 1485 (aromatic), 1449, 1002, 833 cm$^{-1}$; $^1$H NMR ($d_4$-MeOH, 500 MHz) see Table 1. $^{13}$C NMR ($d_4$-MeOH, 125 MHz) see Table 1; HRFABMS *m*/*z*: $[M+H]^+$ 681.2115 (calc. for $C_{42}H_{33}O_9$, 681.2125).

#### 3.3.2. trans-Diptoindonesin B (2)

White amorphous powder; $[\alpha]_D^{20}$ −192° (MeOH; *c* 0.1); UV $\lambda_{max}$ (MeOH) (log ϵ): 204 (3.67), 231 (3.45), 310 (3.16) nm; IR $\nu_{max}$ (KBr): 3366 (OH), 1599, 1516, 1486 (aromatic), 1450, 999, 832 cm$^{-1}$; $^1$H NMR ($d_4$-MeOH, 500 MHz) see Table 1; $^{13}$C NMR ($d_4$-MeOH, 125 MHz) see Table 1; HRFABMS *m*/*z*: $[M+H]^+$ 681.2054 (calc. for $C_{42}H_{32}O_9$, 681.2125).

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