Acta Crystallographica Section E
**Structure Reports**
**Online**

ISSN 1600-5368

# 3,6,9-Trimethyl-2,3-dihydrobenzo[*de*]-chromene-7,8-dione

**Hoong-Kun Fun,[a]* Sompong Boonsri,[b] Suchada Chantrapromma,[b]‡ Nawong Boonnak[b] and Chatchanok Karalai[b]**

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand

‡ Additional correspondence author, email: suchada.c@psu.ac.th

Correspondence e-mail: hkfun@usm.my

Received 22 March 2007
Accepted 26 March 2007

The title sesquiterpene *ortho*-naphthoquinone compound, $C_{15}H_{14}O_3$, was isolated from *Thespesia populnea*. There are four independent molecules (*A*, *B*, *C* and *D*) in the asymmetric unit. The conformations of molecules *A* and *B* differ from those observed for *C* and *D*. In all four molecules, the pyran ring adopts an envelope conformation, with the methylene C atom as the flap atom. The molecules are arranged in layers parallel to the (100) plane, and are interconnected into a three-dimensional network by C—H⋯O interactions.

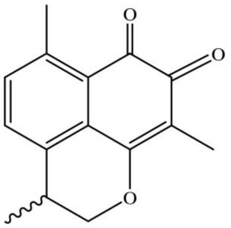
**Key indicators**

Single-crystal X-ray study
$T$ = 100 K
Mean $\sigma$(C–C) = 0.005 Å
$R$ factor = 0.061
$wR$ factor = 0.166
Data-to-parameter ratio = 10.8

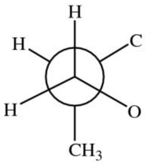
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

## Comment

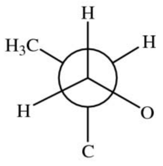
*Thespesia populnea*, or Po-ta-lae in Thai, is a plant in the *Malvaceae* with mainly tropical and subtropical worldwide distribution. The heartwood of *T. populnea* is a rich source of sesquiterpenoid quinines (Milbrodt *et al.*, 1997). The title compound, (I), also known as mansonone E (Marini Bettolo *et al.*, 1965; Tanaka *et al.*, 1966; Kim *et al.*, 1996), was isolated from the heartwood of *T. populnea* which was collected from Suratthani province in the southern part of Thailand. As part of our ongoing studies on the structure and biological activities of natural products from medicinal plants (Boonnak *et al.*, 2005; Chantrapromma *et al.*, 2005; Chantrapromma, Boonnak *et al.*, 2006; Chantrapromma, Boonsri *et al.*, 2006; Chantrapromma, Fun *et al.*, 2006; Fun *et al.*, 2005), we have undertaken the X-ray crystal structure determination of (I). The results of our biological activity investigation of (I) show strong cytotoxic activity and moderate antibacterial activity against *B. substilis*.



(I)



(II*a*)



(II*b*)

Compound (I) crystallizes with four independent molecules (*A*, *B*, *C* and *D*) per asymmetric unit (Fig. 1). The dimensions

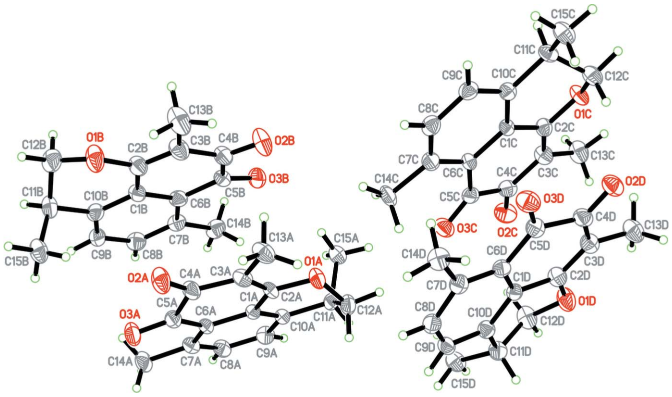


**Figure 1**
The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering scheme.

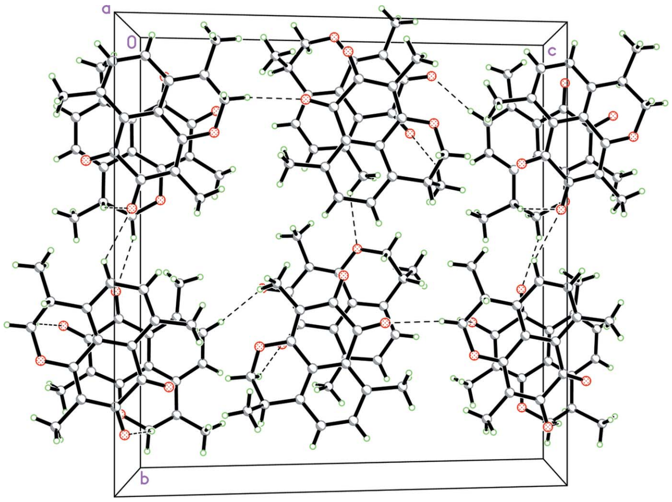


**Figure 2**
The crystal packing of (I), viewed approximately along the $a$ axis. Hydrogen bonds are shown as dashed lines.

of all four molecules are very similar, except for slight deviations in the bond angles subtended at C11 (Table 1), with bond lengths and angles within normal ranges (Allen *et al.*, 1987). The conformations of molecules *A* and *B* are similar, the methylene and methyl groups in the pyran rings being on opposite sides of the mean plane through the molecule, while molecules *C* and *D* have similar conformations with these groups on the same side of the plane. The projections of the groups attached at C11 and C12, viewed along the C12—C11 bond, are shown in (II*a*) for molecules *A* and *B*, and (II*b*) for *C* and *D*.

The naphthoquinone ring system (C1–C10) is essentially planar, with atom C4 deviating by a maximum of 0.090 (4), 0.101 (4), 0.073 (4) and 0.075 (4) Å for molecules *A*, *B*, *C* and *D*, respectively. The pyran ring adopts an envelope conformations in all four molecules, with atom C12 displaced from the C1/C2/C10/C11/O1 plane by 0.640 (5), 0.640 (7) 0.636 (5) and 0.596 (6)Å for molecules *A*, *B*, *C* and *D*, respectively. The puckering parameters (Cremer & Pople, 1975) are $Q$ = 0.468 (4) Å, $\theta$ = 57.2 (5) and $\varphi$ = 293.8 (5)° for molecule *A*, $Q$ = 0.469 (5) Å, $\theta$ = 56.9 (6) and $\varphi$ = 292.7 (6)° for molecule *B*, $Q$ = 0.465 (4) Å, $\theta$ = 123.0 (5) and $\varphi$ = 115.1 (6)° for molecule *C*, and $Q$ = 0.434 (4) Å, $\theta$ = 122.5 (5) and $\varphi$ = 123.1 (6)° for molecule *D*. In molecules *A* and *B*, the methyl group at atom C11 is axially attached, while in *C* and *D* it is equatorially attached. Molecules *A*, *B* and *C*, *D* are related by conformational enantiomerism.

In the crystal structure, the molecules are stacked in layers parallel to the (100) plane. The molecules in a layer and in adjacent layers are linked through C—H···O intermolecular hydrogen bonds (Table 2), forming a three-dimensional network (Fig. 2).

## Experimental

Air-dried heartwood of *T. populnea* (2.1 kg) was extracted with $CH_2Cl_2$ over a period of 5 d at room temperature. The $CH_2Cl_2$ extract (20 ml) was evaporated under reduced pressure, yielding an orange–brown gum (37.5 g), which was subjected to silica-gel column chromatography, affording eight fractions (F1–F8). Fraction F1 was subjected to repeated column chromatography, affording the title compound, (I). Single crystals of (I) were obtained after several days by recrystallization from MeOH–$CH_2Cl_2$ (3:7 *v*/*v*) (m.p. 381–383 K).

### *Crystal data*

$C_{15}H_{14}O_3$
$M_r$ = 242.26
Monoclinic, $P2_1$
$a$ = 7.1615 (1) Å
$b$ = 18.6558 (3) Å
$c$ = 18.0323 (3) Å
$\beta$ = 98.216 (1)°
$V$ = 2384.45 (6) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 100.0 (1) K
0.32 × 0.20 × 0.10 mm

### *Data collection*

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{\min}$ = 0.971, $T_{\max}$ = 0.991
30798 measured reflections
7133 independent reflections
4478 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.080

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.166
$S$ = 1.01
7133 reflections
661 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{\min}$ = −0.23 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1*A*—C2*A* | 1.354 (5) | O1*C*—C2*C* | 1.351 (5) |
| O1*A*—C12*A* | 1.450 (5) | O1*C*—C12*C* | 1.448 (5) |
| O2*A*—C4*A* | 1.221 (5) | O2*C*—C4*C* | 1.227 (5) |
| O3*A*—C5*A* | 1.204 (5) | O3*C*—C5*C* | 1.214 (5) |
| O1*B*—C2*B* | 1.347 (5) | O1*D*—C2*D* | 1.359 (5) |
| O1*B*—C12*B* | 1.452 (6) | O1*D*—C12*D* | 1.448 (5) |
| O2*B*—C4*B* | 1.226 (5) | O2*D*—C4*D* | 1.227 (5) |
| O3*B*—C5*B* | 1.207 (5) | O3*D*—C5*D* | 1.211 (5) |
| | | | |
| C12*A*—C11*A*—C15*A* | 112.7 (4) | C12*C*—C11*C*—C15*C* | 108.9 (3) |
| C12*B*—C11*B*—C15*B* | 113.6 (4) | C12*D*—C11*D*—C15*D* | 108.0 (4) |

o2128 Fun et al. • $C_{15}H_{14}O_3$ Acta Cryst. (2007). E63, o2127–o2129

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C9*C*—H9*CA*···O2*C*$^{i}$ | 0.93 | 2.45 | 3.185 (5) | 136 |
| C8*D*—H8*DA*···O2*B*$^{ii}$ | 0.93 | 2.43 | 3.352 (5) | 169 |
| C11*D*—H11*D*···O2*C*$^{ii}$ | 0.98 | 2.55 | 3.449 (5) | 152 |
| C12*A*—H12*A*···O3*B*$^{ii}$ | 0.97 | 2.43 | 3.248 (5) | 142 |
| C12*C*—H12*E*···O3*A*$^{iii}$ | 0.97 | 2.43 | 3.368 (5) | 162 |
| C12*C*—H12*F*···O2*D* | 0.97 | 2.43 | 3.349 (5) | 158 |
| C12*D*—H12*G*···O3*D*$^{iv}$ | 0.97 | 2.46 | 3.343 (5) | 151 |
| C12*D*—H12*H*···O2*C* | 0.97 | 2.51 | 3.358 (6) | 146 |
| C14*B*—H14*F*···O1*B*$^{v}$ | 0.96 | 2.56 | 3.273 (5) | 131 |
| C15*A*—H15*B*···O3*B* | 0.96 | 2.52 | 3.410 (5) | 154 |

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+2$; (ii) $x+1, y, z$; (iii) $x, y, z+1$; (iv) $-x+2, y+\frac{1}{2}, -z+2$; (v) $-x, y+\frac{1}{2}, -z+1$.

H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å. The $U_{\rm iso}$ values were set equal to $1.5U_{\rm eq}$ of the carrier atom for methyl H atoms and $1.2U_{\rm eq}$ for the remaining H atoms. A rotating-group model was used for the methyl groups. In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

The authors thank the Directed Basic Research in Medicinal Chemistry (Thailand Research Fund), Prince of Songkla University, the Malaysian Government and Universiti Sains Malaysia for the Scientific Advancement Grant Allocation (SAGA) grant No. 304/PFIZIK/635003/A118. SB thanks the Thailand Research Fund through the Royal Golden Jubilee PhD Program (grant No. PHD/0136/2548) and the Center for Innovation in Chemistry: Postgraduate Education and Research Program in Chemistry (PERCH–CIC), Commission on Higher Education, Ministry of Education, Thailand.

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Boonnak, N., Chantrapromma, S., Fun, H.-K. & Karalai, C. (2005). *Acta Cryst.* E**61**, o4376–o4378.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chantrapromma, S., Boonsri, S., Fun, H.-K., Anjum, S. & Kanjana-opas, A. (2006). *Acta Cryst.* E**62**, o1254–o1256.

Chantrapromma, S., Boonnak, N., Fun, H.-K. & Karalai, C. (2006). *Acta Cryst.* E**62**, o360–o362.

Chantrapromma, S., Boonnak, N., Fun, H.-K., Anjum, S. & Atta-ur-Rahman (2005). *Acta Cryst.* E**61**, o2136–o2138.

Chantrapromma, S., Fun, H.-K., Koysomboon, S. & Chantrapromma, K. (2006). *Acta Cryst.* E**62**, o1984–o1986.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Fun, H.-K., Razak, I. A., Boonnak, N., Laphookhieo, S. & Chantrapromma, S. (2005). *Acta Cryst.* E**61**, o3086–o3088.

Kim, J.-H., Kim, W.-G., Koshino, H., Jung, J. & Yoo, I.-D. (1996). *Phytochemistry*, **43**, 425–430.

Marini Bettolo, G. B., Casinovi, C. G. & Galeffi, C. (1965). *Tetrahedron Lett.* **52**, 4875–4864.

Milbrodt, M., Konig, W. A. & Hausen, B. M. (1997). *Phytochemistry*, **45**, 1523–1525.

Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Tanaka, N., Yasue, M. & Imamura, H. (1966). *Tetrahedron Lett.* **24**, 2767–2773.

*Acta Cryst.* (2007). E**63**, o2127–o2129 Fun *et al.* • $C_{15}H_{14}O_3$ **o2129**