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4,5-Dimethoxy-2-(1-methoxy-3-phenylallylidene)-cyclopent-4-ene-1,3-dione (methyl linderone)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.054
wR factor = 0.149
Data-to-parameter ratio = 15.5

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

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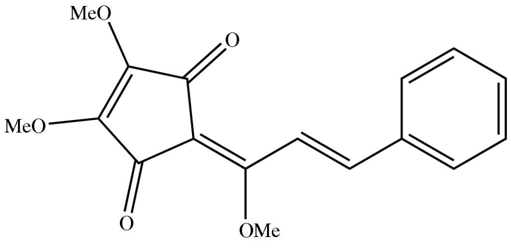
The title compound, $C_{17}H_{16}O_5$, was isolated from the plant *Lindera poliantha*. The cyclopentenedione and phenyl rings are connected by the central C_3O fragment, which makes dihedral angles with these rings of 7.10 (6) and 16.24 (10)°, respectively.

Comment

Methyl linderone, (I), is a cyclopent-4-ene-1,3-dione derivative isolated from several plants such as *Lindera lucida* (Leong *et al.*, 1998). The biological importance and structural diversity of the cyclopentanoid natural products have made these compounds valuable synthetic targets (Liebeskind & Bombrun, 1994). In the present study, the title compound was obtained from the plant *Lindera poliantha*, collected from the Cibodas Botanical Garden, Indonesia. The molecule is discrete (Fig. 1), with normal bond lengths and angles (Allen *et al.*, 1987). The endocylic C4═C5 double bond is slightly shorter than the exocyclic C2═C6 double bond (Table 1). The substituted cyclopentenedione ring, the phenyl ring and the central connecting O5/C6/C7/C8 fragment are essentially coplanar. The maximum deviation from the plane is 0.054 (2) Å, found for C7. The dihedral angles between the central O5/C6/C7/C8 fragment and the cyclopentenedione and phenyl rings are 7.10 (6) and 16.24 (10)°, respectively. The O5—C15 and O3—C17 bonds lie markedly out of the plane of the molecule, with torsion angles C15—O5—C6—C2 and C17—O3—C4—C3 of −66.7 (3) and 52.9 (2)°, respectively. The presence of a number of intramolecular C—H⋯O hydrogen bonds (Table 2) may contribute to the stability and planarity of the molecule.



(I)

Experimental

Lindera polyantha was collected from the Cibodas Botanical Garden, West Java, Indonesia, in July 2004. The powdered dried wood of the plant (1 kg) was macerated with MeOH at room temperature to yield a dark-brown extract (104 g). A portion (50 g) of the MeOH extract was partitioned into EtOAc to give an EtOAc fraction (17.5 g). Fractionation of the EtOAc fraction using chromatography (silica gel) eluted with *n*-hexane–EtOAc of increasing polarity gave three

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major fractions *A*–*C* (670, 120 and 600 mg, respectively). Fraction *A* was then purified using radial chromatography (silica gel), eluted with *n*-hexane–EtOAC (9:1) to afford methyl linderone (70 mg) as pale-yellow crystals; m.p. 348–349 K (literature 348 K; Leong *et al.*, 1998). ^{1}H NMR (400 MHz): δ 7.93 (1H, *d*, J = 16.0 Hz, H-8), 7.60 (2H, *dd*, J = 8.0 and 2.0 Hz, H-10/14), 7.51 (1H, *d*, J = 16.0 Hz, H-7), 7.36 (3H, *m*), 4.19, 4.18 (3H, 4,5-OMe), 4.09 (6-OMe); ^{13}C NMR: δ 60.0, 60.1 (5,6-OMe), 64.5 (6-OMe), 109.6 (C-2), 121.4 (C-7), 128.5 (C-11/C13), 129.1 (C-10/14), 130.2 (C-12), 135.8 (C-9), 141.4 (C-8), 148.0, 149.1 (C-4,5), 165.6 (C-6), 184.9, 187.4 (C-1,-3), in agreement with the literature (Leong *et al.*, 1998).

Crystal data

$C_{17}H_{16}O_5$
M_r = 300.30
Triclinic, $P\overline{1}$
a = 7.380 (3) Å
b = 8.139 (3) Å
c = 13.781 (5) Å
α = 78.224 (5)°
β = 87.913 (6)°
γ = 68.280 (5)°
V = 752.1 (5) Å^3
Z = 2
D_x = 1.326 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 866 reflections
θ = 1.5–26.4°
μ = 0.10 mm^{-1}
T = 298 (2) K
Block, pale yellow
0.48 × 0.32 × 0.31 mm

Data collection

Bruker SMART APEX area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.954, $T_{\max}$ = 0.970
8035 measured reflections
3079 independent reflections
2588 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.018
$\theta_{\max}$ = 26.4°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.149
S = 1.06
3079 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0726P)^2 + 0.1615P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ <0.001
$\Delta\rho_{\max}$ = 0.25 e Å^{-3}
$\Delta\rho_{\min}$ = −0.18 e Å^{-3}

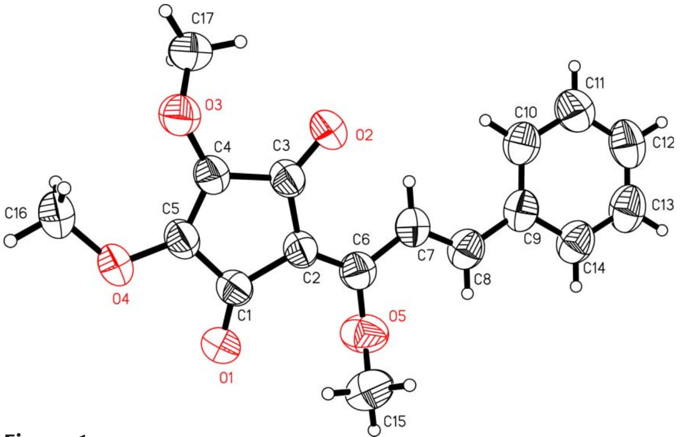

Figure 1
Molecular structure of the title compound, (I), with 50% probability displacement ellipsoids for the non-H atoms. H atoms are included as spheres of arbitrary radii.

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.210 (2)	C6—C2	1.357 (3)
O2—C3	1.219 (2)	O5—C6	1.347 (2)
C4—C5	1.347 (2)	O5—C15	1.412 (2)
C10—C9—C8—C7	−10.1 (3)	C16—O4—C5—C4	11.0 (3)
O5—C6—C2—C1	−7.1 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*A*···O2	0.93	2.35	2.976 (3)	124
C8—H8*A*···O5	0.93	2.36	2.730 (3)	103
C15—H15*B*···O1	0.96	2.34	3.022 (3)	128
C16—H16*C*···O3	0.96	2.52	2.929 (3)	106
C17—H17*C*···O2	0.96	2.36	2.992 (3)	123

After the location of H atoms in a difference map, their geometrical parameters were idealized. The H atoms were allowed to ride on the parent C with C—H = 0.93–0.96 Å and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for CH_3 and 1.2$U_{\rm eq}$(C) for CH groups.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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