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8,8′-Methylenebis(7,10-dihydroxy-1,3,6,6-tetramethyl-3,4-dihydro-1*H*-benzo[*g*]isochromen-9-one)

Hoong-Kun Fun,[a]* Nawong Boonnak[b] and Suchada Chantrapromma[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

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In the title compound, $C_{23}H_{24}O_8$, the central methylene unit that links the two sets of fused rings lies on a crystallographic twofold rotation axis. The isochromene skeleton is not planar, with the pyran ring in an envelope conformation. Intra- and intermolecular O—H⋯O hydrogen bonds and weak C—H⋯O and C—H⋯π interactions stabilize the crystal structure.

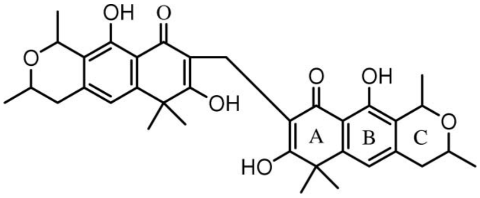
Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.108
Data-to-parameter ratio = 8.2

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

Comment

Many quinonoid compounds such as naphthoquinones, anthraquinones, ventiloquinones and ventilagones have been isolated from the plant species of the *Ventilago* genus (Hanumaiah *et al.*, 1985; Jammula *et al.*, 1991; Ali *et al.*, 1994), which are distributed throughout India and southern Asia. 5,10-Quinones, or ventiloquinones, show antibiotic and antineoplastic activities and their syntheses have been reported previously (Blouin *et al.*, 1990; Cameron *et al.*, 1992). However, the analogous 6,9-quinones are not as widely found in nature. Therefore we have attempted to synthesize the analogous 6,9-quinones to test for their activities. The title compound (I) is the unexpected product of such an attempt and we report its structure here (Fig. 1).



(I)

In (I), the methylene C18 atom that links the two isochromene units lies on a crystallographic twofold rotation axis and the asymmetric unit therefore contains only half of the molecule. In the isochromene skeleton (rings A, B and C), rings A and B are essentially planar with the maximum deviation from the C4—C13 plane of 0.067 (3) Å for atom C10. The dihedral angle between rings A and B is 1.37 (12)°. The pyran ring C adopts an envelope conformation with atom C2 displaced from the C1/O1/C3/C4/C13 plane by 0.381 (3) Å, and with the Cremer & Pople (1975) puckering parameters Q = 0.527 (3) Å, θ = 56.6 (3)° and φ = 302.2 (3)°. Bond lengths and angles observed in the structure are normal (Allen *et al.*, 1987).

Intra- and intermolecular O—H⋯O hydrogen bonds and weak C—H⋯O interactions are observed in the crystal (Table 2). O2—H1O2⋯O3 and weak C14—H14*A*⋯O2 intramolecular hydrogen bonds each generate $S(6)$ ring motifs (Bernstein *et al.*, 1995) (Table 2). Additionally, O4—H1O4···O3^{i} and weak C18—H18···O3^{i} intramolecular hydrogen bonds generate $S(8)$ and $S(5)$ ring motifs, respectively. The molecules are arranged in wave-like chains along the *c* axis and these are stacked along the *b* axis (Fig. 2). These chains are further aggregated by C—H···π interactions; *Cg*1 is the centroid of the C4–C6/C11–C13 benzene ring (Table 1).

Experimental

Compound (I) was obtained as an unexpected product of the reaction between *cis*-2,6-dimethyl-5,6-dihydro-2*H*-pyran-3-carbonyl chloride (170 mg) in dry chloroform (3 ml) and a mixture of 3,6-dimethyl-1,2,4-trimethoxybenzene (300 mg) and anhydrous aluminium trichloride (408 mg) in dry chloroform (7 ml) at 273 K. The mixture was left to stand for 2 h at 273 K, 15 h at 278 K and 4 h at 283 K, then poured into ice–water and extracted with diethyl ether. The ether extract was washed, dried and concentrated, then subjected to column chromatography, eluting with ethyl acetate/hexane (1:3) to give a colourless oil. Nitric acid (0.5 ml, 6*M*) was added to a suspension of the oil (170 mg) and silver(II) oxide (252 mg) in dioxane (5 ml) was added. The mixture was stirred for 15 min, washed with water and extracted with diethyl ether. The ether extract was separated by column chromatography, eluting with hexane to give compound (I), which was recrystallized from $CHCl_3$.

Crystal data

$C_{35}H_{40}O_8$
$M_r = 588.67$
Tetragonal, $P4_32_12$
$a = 8.4385\ (11)$ Å
$c = 42.901\ (6)$ Å
$V = 3054.9\ (7)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 297\ (2)$ K
0.52 × 0.43 × 0.31 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.955$, $T_{\max} = 0.973$
18799 measured reflections
2260 independent reflections
2063 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.109$
$S = 1.15$
2260 reflections
275 parameters
All H-atom parameter refined
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C4–C6/C11–C13 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1O2···O3	0.94 (3)	1.70 (3)	2.549 (2)	149 (3)
O4—H1O4···O3^{i}	0.94 (4)	1.77 (4)	2.690 (2)	168 (3)
C14—H14*A*···O2	0.95 (4)	2.49 (3)	3.038 (4)	117 (3)
C18—H18···O3^{i}	1.04 (2)	2.37 (2)	2.7938 (19)	102.8 (16)
C1—H1···*Cg*1^{ii}	0.99 (3)	2.83 (3)	3.780 (3)	159 (2)
C14—H14*B*···*Cg*1^{iii}	0.99 (4)	2.87 (4)	3.729 (4)	146 (3)

Symmetry codes: (i) $y, x, -z$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+\frac{1}{4}$; (iii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+\frac{1}{4}$.

All H atoms were located in difference maps and refined isotropically [C—H = 0.94 (3)–1.05 (4) Å]. In the absence of significant anomalous scattering effects, 1418 Friedel pairs were merged.

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* and *PLATON* (Spek, 2003).

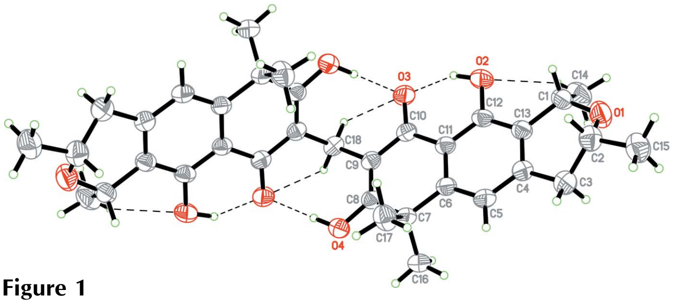


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Intramolecular hydrogen bonds are drawn as dashed lines. Unlabelled atoms are related to labelled atoms by (y, x, $-z$).

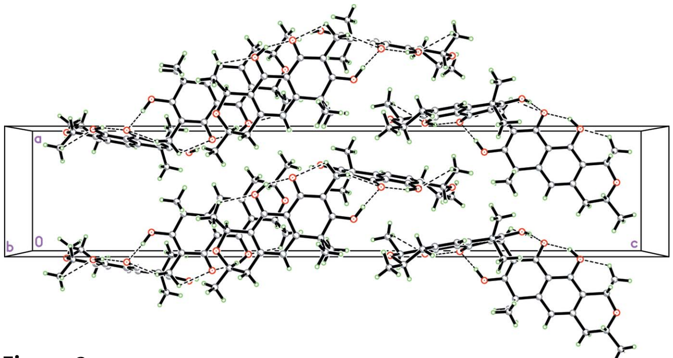


Figure 2
The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are drawn as dashed lines.

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o2318 Fun *et al.* • $C_{35}H_{40}O_8$ *Acta Cryst.* (2007). E**63**, o2317–o2318