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1,13-Dimethyl-6*H*,7*H*,8*H*-chromeno-[3′,4′:5,6]pyrano[3,2-*c*]chromene-6,8-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.136
Data-to-parameter ratio = 11.4

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

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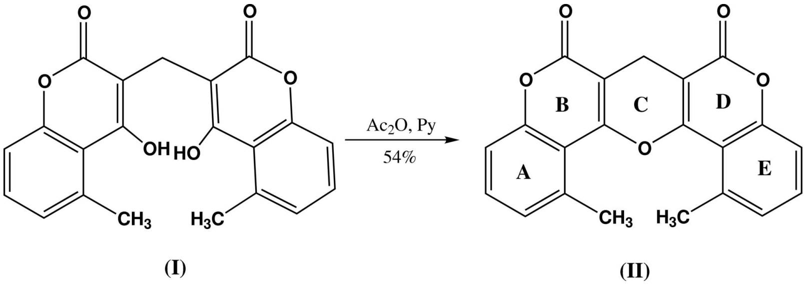
The title compound, $C_{21}H_{14}O_5$, is a pentacyclic chromene derivative. The dihedral angle between the two outer benzene rings is 30.3 (1)°. The pyran and one of the pyranone rings adopt twist-boat conformations. The crystal packing is stabilized by C—H···O, C—H···π and π–π interactions

Comment

Diospyros canaliculata De Wildeman (Ebenaceae) is a tree up to 15–20 m tall (Letouzey & White, 1970) found throughout the forest zone of West and Central Africa (Jeffreys *et al.*, 1983). The bark extract is poisonous and causes blistering and gangrene around wounds, and is a common ingredient of the Guere (Ivory Coast) arrow poisons (Irvine, 1961). Previous investigation of the stem bark of this plant led to the isolation of gerberinol, (I) (Sengupta *et al.*, 1985; Paknikar *et al.*, 1996; Tangmouo *et al.*, 2005). In the present work, we have undertaken the X-ray structure determination of the title compound, (II) (Sengupta *et al.*, 1985), derived from (I).



The bond lengths in compound (II) show normal values (Allen *et al.*, 1987). The molecule is non-planar, with the outer benzene rings *A* and *E* forming a dihedral angle of 30.3 (1)°. The pyranone ring *B* is planar, with a weighted average absolute torsion angle (τ) of 4.1°. The pyran ring *C* and pyranone ring *D* are slightly puckered and adopt twist–boat conformations; the Cremer and Pople (1975) puckering parameters *Q*, θ and φ are 0.185 (2) Å, 82.6 (6)° and 157.0 (8)° for ring *C*, and 0.182 (2) Å, 89.6 (9)° and 30.9 (8)° for ring *D*, respectively. The C1—C14*B*—C14*A* [128.3 (2)°] and C13—C13*A*—C13*B* [128.30 (2)°] angles are larger than 120° as a result of steric interactions between the methyl groups at the C1 and C13 positions.

As shown in Fig. 1, the C16—H16*C*···O3 and C15—H15*C*···O3 hydrogen bonds generate rings of graph-set motif *S*(6) (Bernstein *et al.*, 1995). The crystal packing is stabilized by C—H···O, C—H···π (Table 1) and π···π interactions. The π···π interactions involve rings *A*, *B* and *E* of inversion-related molecules with *Cg*2···*Cg*2^{ii} = 3.593 (3) Å, *Cg*1···*Cg*1^{iii} = 3.544 (3) Å and *Cg*3···*Cg*3^{iii} = 3.790 (4) Å [symmetry codes:

(ii) $1-x, 1-y, 1-z$; (iii) $1-x, 2-y, 1-z$; (iii) $1-x, 1-y, -z$], where *Cg*1, *Cg*2 and *Cg*3 are the centroids of rings *A*, *B* and *E*, respectively. The C—H$\cdots\pi$ interaction involves ring *A*. A view of the molecular packing is shown in Fig. 2.

Experimental

Compound (II) was prepared from gerberinol (I) using the literature method of Sengupta *et al.* (1985). A solution of (I) (15 mg) in pyridine (0.5 ml) and acetic anhydride (0.5 ml) was stirred for 2 d at room temperature. The solvent was evaporated and the resulting solid pink residue was crystallized from $CHCl_3$–CH_3OH (1:1 *v*/*v*) to afford compound (II) as shiny pink crystals (yield 54%, 8 mg; m.p. 523 K).

Crystal data

$C_{21}H_{14}O_5$
$M_r = 346.32$
Triclinic, $P\bar{1}$
$a = 8.052\ (6)$ Å
$b = 8.492\ (7)$ Å
$c = 12.544\ (10)$ Å
$\alpha = 91.441\ (13)^\circ$
$\beta = 106.324\ (13)^\circ$
$\gamma = 109.489\ (13)^\circ$
$V = 769.2\ (11)$ Å^3
$Z = 2$
$D_x = 1.495$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3408 reflections
$\theta = 1.7$–25.0°
$\mu = 0.11$ mm^{-1}
$T = 293\ (2)$ K
Plate, pink
$0.24 \times 0.12 \times 0.06$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.975$, $T_{max} = 0.994$
6917 measured reflections
2695 independent reflections
1842 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 25.0^\circ$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.136$
$S = 1.05$
2695 reflections
237 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2 + 0.1079P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

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

*Cg*1, *Cg*2 and *Cg*3 are the centroids of rings *A*, *B* and *E*, respectively.

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C2—H2*A*$\cdots$O1^{i}	0.93	2.57	3.442 (4)	155
C15—H15*C*$\cdots$O3	0.96	2.43	2.848 (4)	106
C16—H16*C*$\cdots$O3	0.96	2.37	2.937 (4)	117
C7—H7*A*$\cdots$*Cg*1^{ii}	0.97	2.84	3.583 (4)	134

Symmetry codes: (i) $x+1, y+1, z$; (ii) $-x+1, -y+1, -z+1$.

All H atoms were positioned geometrically and allowed to ride on the parent carbon atoms, with C—H distances in the range 0.93–0.97 Å. and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C)$ for other H atoms.

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).

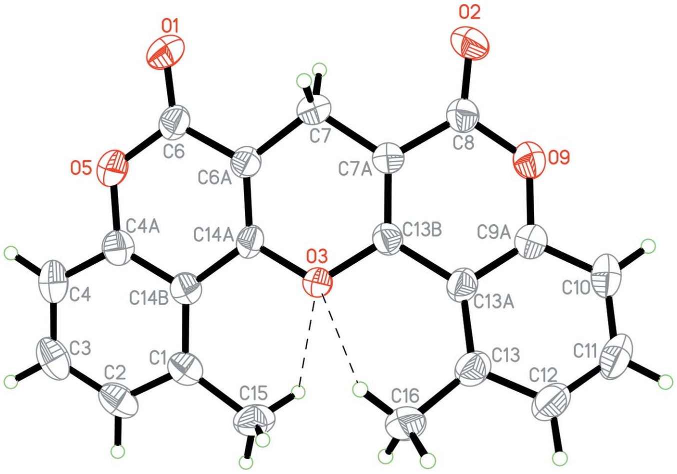


Figure 1
The molecular structure of (II), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate hydrogen bonds.

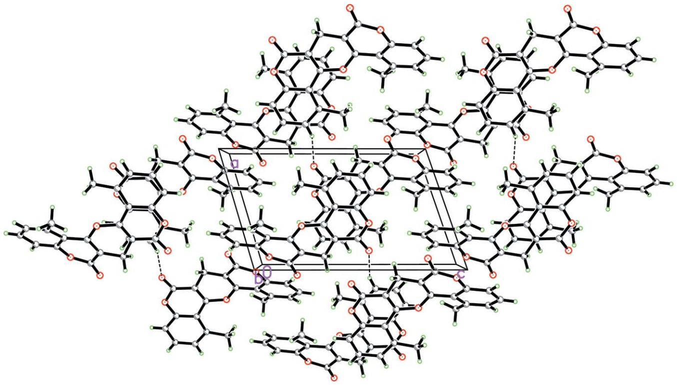


Figure 2
The crystal packing of (II), viewed approximately along the *b* axis. Dashed lines indicate hydrogen bonds.

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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–S19.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N. -L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Irvine, F. R. (1961). *Woody Plants of Ghana*, pp. 576–578. London: Oxford University Press.
Jeffreys, J. A. D., Muhamad, B. Z., Waterman, P. G. & Zhong, S. M. (1983). *Tetrahedron Lett.* **24**, 1085–1088.
Letouzey, R. & White, F. (1970). *Flore du Cameroun: Ebénacées*, vol. 11, p. 43. Muséum National d'Histoire Naturelle, 16 rue Buffon, Paris 5, France.
Paknikar, S. K., Pai, F. K. P., Kirtany, J. K. & Natori, S. (1996). *Phytochemistry*, **41**, 931–933.
Sengupta, P., Sen, M., Karuri, P., Wenkert, E. & Halls, T. D. J. (1985). *J. Indian Chem. Soc.* **62**, 916–919.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

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Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-Ray Instruments Inc., Madison, Wisconsin, USA.

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

Tangmouo, J. G., Lontsi, D., Ngounou, F. N., Kuete, V., Meli, A. L., Manfouo, R. N., Kamdem, H . W., Tane, P., Penlap, B. V., Sondengam, B. L. & Connolly, J. D. (2005). *Bull. Chem. Soc. Ethiop.* **19**, 81–88.

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