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(3*E*,4*Z*)-3-[(1,3-Benzodioxol-5-yl)methylene]-4-(2-pentylidene)tetrahydrofuran-2,5-dione

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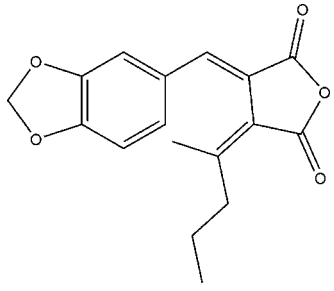
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.056; wR factor = 0.203; data-to-parameter ratio = 18.4.

In the title compound, $C_{17}H_{16}O_5$, the dihydrofuran ring adopts an envelope conformation while the methylenedioxyphenyl ring system is essentially planar. The vinyl group is inclined to the dihydrofuran ring by 31.06 (18)°. The dihedral angle between the atoms defining the planar part of the dihydrofuran ring and the aryl ring is 24.86 (7)°.

Related literature

For related literature, see: Asiri (2003); Heller *et al.* (2000); Uchida *et al.* (1995).



Experimental

Crystal data

$C_{17}H_{16}O_5$
M_r = 300.30
Monoclinic, $P2_1/c$
a = 7.8597 (11) Å
b = 15.383 (3) Å
c = 12.781 (2) Å
β = 105.117 (10)°
V = 1491.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
0.30 × 0.25 × 0.15 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2 Software Suite*; Bruker, 2005)
T_{min} = 0.971, T_{max} = 0.985
14935 measured reflections
3695 independent reflections
2084 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.203
S = 0.94
3695 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *APEX2 Software Suite* (Bruker, 2005); cell refinement: *APEX2 Software Suite*; data reduction: *APEX2 Software Suite*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2014).

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supplementary materials

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(3*E*,4*Z*)-3-[(1,3-Benzodioxol-5-yl)methylene]-4-(2-pentylidene)tetrahydrofuran-2,5-dione

W.-L. Dong, J.-H. Zhang, L.-W. Zheng and B.-X. Zhao

Comment

Organic photochromic compounds, such as fulgides are potential candidates for application in erasable optical information media, attempts have been made to improve their photochromic properties (Uchida *et al.*, 1995; Asiri, 2003). In order to achieve certain desirable properties such as absorption at longer wavelengths and thus higher fatigue resistance to coloration-bleaching cycles, improvements have been made by modifying the fulgide frame (Heller *et al.*, 2000). We report here the crystal structure of the title compound, (I).

Experimental

The 2-(benzo[*d*][1,3]dioxol-5-ylmethylene)-3-(pentan-2-ylidene)succinic acid (0.01 mmol) was dissolved in dichloromethane (10 ml), and to this mixture was added acetyl chloride (5 ml) dropwise with stirring at 273 K, and the mixture was stirred at room temperature for 5 h. After removal of the excess acetyl chloride and dichloromethane, the residue was purified using flash column chromatography on silica gel (petroleum ether/ethyl acetate = 2/1; *v*/*v*) and recrystallized with ethyl acetate to give a solid (yield 4%) as minor product. Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a solution of the solid in ethyl acetate at room temperature for 15 days.

Refinement

H atoms were positioned geometrically (C - H = 0.93 − 0.97 Å) and refined as riding, with U_{iso}(H) = 1.2Ueq(C).

Figures

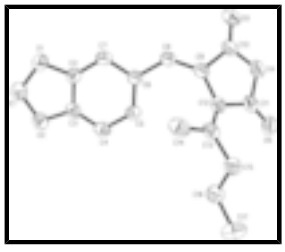


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme with 50% probability displacement ellipsoids; H atoms have been omitted for clarity.

(3*E*,4*Z*)-3-[1,3-Benzodioxol-5-yl)methylene]-4-(2-\ pentylidene)tetrahydrofuran-2,5-dione

Crystal data

$C_{17}H_{16}O_5$	F_{000} = 632
M_r = 300.30	D_x = 1.337 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å

Hall symbol: -P 2ybc	Cell parameters from 4386 reflections
a = 7.8597 (11) Å	θ = 2.7–23.6º
b = 15.383 (3) Å	μ = 0.10 mm^{-1}
c = 12.781 (2) Å	T = 296 (2) K
β = 105.117 (10)º	Plate, yellow
V = 1491.8 (4) $Å^3$	0.30 × 0.25 × 0.15 mm
Z = 4	

Data collection

Bruker APEX2 CCD area-detector diffractometer	3695 independent reflections
Radiation source: fine-focus sealed tube	2084 reflections with $I > 2.0\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 296(2) K	θ_{max} = 28.4º
φ and ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (APEX2 Software Suite; Bruker, 2005)	$h = -10 \rightarrow 10$
T_{min} = 0.971, T_{max} = 0.985	$k = -13 \rightarrow 20$
14935 measured reflections	$l = -17 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.056	H-atom parameters constrained
$wR(F^2)$ = 0.203	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.6902P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.94	$(\Delta/\sigma)_{max} < 0.001$
3695 reflections	$\Delta\rho_{max}$ = 0.40 e $Å^{-3}$
201 parameters	$\Delta\rho_{min}$ = −0.16 e $Å^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.1718 (3)	0.82579 (16)	1.02559 (15)	0.0841 (6)
O2	−0.3854 (2)	0.80813 (15)	0.86514 (16)	0.0836 (6)
O3	0.5532 (3)	1.07745 (17)	0.87030 (18)	0.0974 (7)
O4	0.4746 (2)	1.07754 (12)	0.68911 (16)	0.0727 (5)
O5	0.3415 (3)	1.05288 (15)	0.51414 (17)	0.0840 (6)
C7	0.0484 (3)	0.89375 (17)	0.94481 (19)	0.0605 (6)
H2	0.1334	0.9009	1.0101	0.073*
C13	0.2597 (3)	0.87611 (18)	0.6060 (2)	0.0622 (6)
C12	0.2982 (3)	0.95405 (16)	0.65540 (19)	0.0572 (6)
C5	−0.0529 (3)	0.90996 (17)	0.75049 (19)	0.0608 (6)
H8	−0.0318	0.9280	0.6856	0.073*
C6	0.0795 (3)	0.91969 (16)	0.84621 (19)	0.0569 (6)
C2	−0.1117 (3)	0.85766 (16)	0.94102 (19)	0.0587 (6)
C3	−0.2401 (3)	0.84713 (16)	0.8457 (2)	0.0592 (6)
C11	0.3643 (3)	1.02957 (18)	0.6057 (2)	0.0655 (7)
C4	−0.2156 (3)	0.87400 (19)	0.7491 (2)	0.0661 (7)
H14	−0.3041	0.8685	0.6850	0.079*
C16	0.0476 (3)	0.87491 (19)	0.4242 (2)	0.0713 (7)
H15A	−0.0283	0.8385	0.4547	0.086*
H15B	0.0143	0.9350	0.4309	0.086*
C9	0.3288 (3)	0.97795 (17)	0.7705 (2)	0.0614 (6)
C15	0.2383 (3)	0.8619 (2)	0.4888 (2)	0.0719 (7)
H17A	0.2752	0.8033	0.4773	0.086*
H17B	0.3133	0.9021	0.4630	0.086*
C8	0.2473 (3)	0.96253 (17)	0.8495 (2)	0.0619 (6)
H18	0.3080	0.9825	0.9176	0.074*
C10	0.4634 (3)	1.04727 (19)	0.7889 (2)	0.0678 (7)
C14	0.2331 (4)	0.79731 (18)	0.6670 (2)	0.0775 (8)
H20A	0.2687	0.8093	0.7434	0.116*
H20B	0.3026	0.7504	0.6507	0.116*
H20C	0.1108	0.7813	0.6464	0.116*
C1	−0.3455 (4)	0.7941 (2)	0.9778 (3)	0.0824 (8)
H21A	−0.3519	0.7326	0.9928	0.099*
H21B	−0.4298	0.8245	1.0081	0.099*
C17	0.0189 (6)	0.8531 (2)	0.3058 (2)	0.1014 (11)
H22A	0.0941	0.8887	0.2753	0.152*
H22B	−0.1020	0.8637	0.2682	0.152*
H22C	0.0462	0.7929	0.2985	0.152*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0716 (11)	0.1183 (17)	0.0632 (11)	−0.0185 (11)	0.0189 (9)	0.0098 (11)
O2	0.0510 (9)	0.1139 (17)	0.0833 (13)	−0.0151 (10)	0.0127 (8)	0.0099 (11)

O3	0.0794 (13)	0.1191 (19)	0.0881 (14)	−0.0453 (13)	0.0117 (11)	−0.0243 (13)
O4	0.0639 (10)	0.0713 (12)	0.0861 (13)	−0.0182 (9)	0.0252 (9)	−0.0034 (10)
O5	0.0748 (12)	0.1033 (16)	0.0788 (13)	−0.0082 (11)	0.0288 (10)	0.0186 (11)
C7	0.0569 (12)	0.0686 (16)	0.0497 (12)	−0.0075 (11)	0.0028 (10)	0.0000 (11)
C13	0.0421 (10)	0.0699 (17)	0.0715 (15)	−0.0003 (10)	0.0090 (10)	−0.0049 (12)
C12	0.0437 (10)	0.0632 (15)	0.0637 (14)	−0.0021 (10)	0.0120 (9)	0.0017 (11)
C5	0.0562 (12)	0.0693 (16)	0.0525 (13)	−0.0033 (11)	0.0067 (10)	0.0061 (11)
C6	0.0544 (12)	0.0571 (14)	0.0549 (13)	−0.0060 (10)	0.0065 (10)	0.0023 (10)
C2	0.0573 (12)	0.0636 (15)	0.0547 (13)	−0.0010 (11)	0.0137 (10)	0.0038 (11)
C3	0.0455 (11)	0.0631 (15)	0.0667 (14)	−0.0002 (10)	0.0102 (10)	0.0014 (11)
C11	0.0480 (11)	0.0743 (18)	0.0766 (17)	−0.0036 (11)	0.0207 (11)	−0.0002 (14)
C4	0.0494 (11)	0.0827 (19)	0.0587 (14)	0.0002 (12)	0.0009 (10)	0.0032 (12)
C16	0.0633 (14)	0.0708 (18)	0.0796 (18)	0.0047 (12)	0.0182 (13)	−0.0089 (14)
C9	0.0520 (11)	0.0640 (15)	0.0628 (14)	−0.0082 (11)	0.0053 (10)	0.0014 (11)
C15	0.0547 (13)	0.085 (2)	0.0785 (17)	−0.0023 (12)	0.0225 (12)	−0.0165 (14)
C8	0.0558 (12)	0.0678 (16)	0.0552 (13)	−0.0112 (11)	0.0019 (10)	0.0020 (11)
C10	0.0529 (12)	0.0734 (18)	0.0745 (17)	−0.0109 (12)	0.0122 (12)	−0.0091 (14)
C14	0.0718 (16)	0.0621 (17)	0.089 (2)	−0.0019 (13)	0.0040 (14)	0.0004 (14)
C1	0.0666 (16)	0.103 (2)	0.0833 (19)	−0.0119 (15)	0.0289 (14)	−0.0008 (17)
C17	0.130 (3)	0.090 (2)	0.0701 (19)	0.016 (2)	0.0009 (18)	−0.0112 (16)

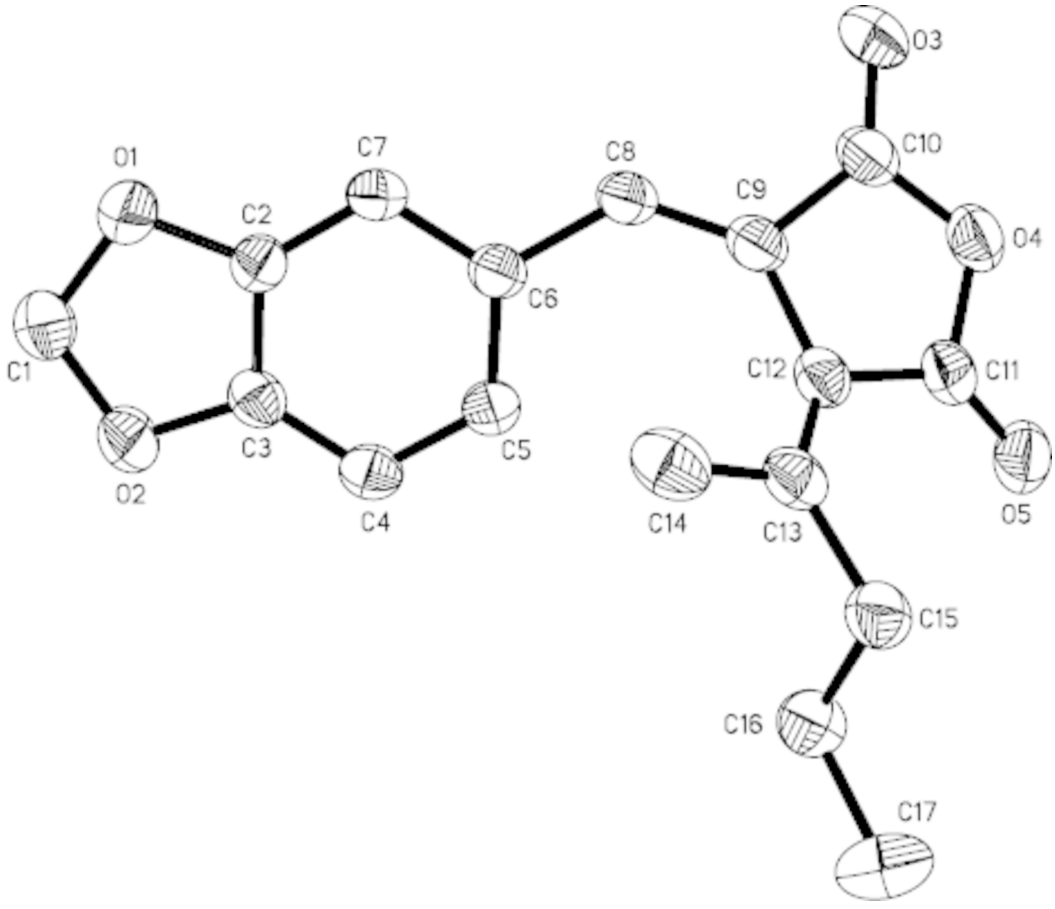
Geometric parameters (Å, °)

O1—C2	1.377 (3)	C2—C3	1.374 (3)
O1—C1	1.428 (3)	C3—C4	1.362 (3)
O2—C3	1.369 (3)	C4—H14	0.9300
O2—C1	1.408 (4)	C16—C17	1.509 (4)
O3—C10	1.189 (3)	C16—C15	1.524 (4)
O4—C10	1.382 (3)	C16—H15A	0.9700
O4—C11	1.396 (3)	C16—H15B	0.9700
O5—C11	1.192 (3)	C9—C8	1.351 (3)
C7—C2	1.365 (3)	C9—C10	1.477 (3)
C7—C6	1.403 (3)	C15—H17A	0.9700
C7—H2	0.9300	C15—H17B	0.9700
C13—C12	1.352 (3)	C8—H18	0.9300
C13—C15	1.480 (4)	C14—H20A	0.9600
C13—C14	1.485 (4)	C14—H20B	0.9600
C12—C9	1.473 (3)	C14—H20C	0.9600
C12—C11	1.481 (4)	C1—H21A	0.9700
C5—C4	1.389 (3)	C1—H21B	0.9700
C5—C6	1.392 (3)	C17—H22A	0.9600
C5—H8	0.9300	C17—H22B	0.9600
C6—C8	1.465 (3)	C17—H22C	0.9600
C2—O1—C1	105.7 (2)	H15A—C16—H15B	107.8
C3—O2—C1	106.2 (2)	C8—C9—C12	135.8 (2)
C10—O4—C11	110.5 (2)	C8—C9—C10	117.9 (2)
C2—C7—C6	117.3 (2)	C12—C9—C10	105.3 (2)
C2—C7—H2	121.3	C13—C15—C16	111.4 (2)
C6—C7—H2	121.3	C13—C15—H17A	109.3

C12—C13—C15	123.8 (3)	C16—C15—H17A	109.3
C12—C13—C14	121.6 (2)	C13—C15—H17B	109.3
C15—C13—C14	114.6 (2)	C16—C15—H17B	109.3
C13—C12—C9	130.2 (2)	H17A—C15—H17B	108.0
C13—C12—C11	123.8 (2)	C9—C8—C6	131.1 (2)
C9—C12—C11	104.3 (2)	C9—C8—H18	114.5
C4—C5—C6	121.9 (2)	C6—C8—H18	114.5
C4—C5—H8	119.1	O3—C10—O4	120.7 (2)
C6—C5—H8	119.1	O3—C10—C9	131.2 (3)
C5—C6—C7	119.6 (2)	O4—C10—C9	108.1 (2)
C5—C6—C8	122.3 (2)	C13—C14—H20A	109.5
C7—C6—C8	118.0 (2)	C13—C14—H20B	109.5
C7—C2—C3	122.5 (2)	H20A—C14—H20B	109.5
C7—C2—O1	128.2 (2)	C13—C14—H20C	109.5
C3—C2—O1	109.3 (2)	H20A—C14—H20C	109.5
C4—C3—O2	128.2 (2)	H20B—C14—H20C	109.5
C4—C3—C2	121.5 (2)	O2—C1—O1	108.6 (2)
O2—C3—C2	110.2 (2)	O2—C1—H21A	110.0
O5—C11—O4	119.7 (2)	O1—C1—H21A	110.0
O5—C11—C12	132.6 (3)	O2—C1—H21B	110.0
O4—C11—C12	107.7 (2)	O1—C1—H21B	110.0
C3—C4—C5	117.2 (2)	H21A—C1—H21B	108.4
C3—C4—H14	121.4	C16—C17—H22A	109.5
C5—C4—H14	121.4	C16—C17—H22B	109.5
C17—C16—C15	112.5 (2)	H22A—C17—H22B	109.5
C17—C16—H15A	109.1	C16—C17—H22C	109.5
C15—C16—H15A	109.1	H22A—C17—H22C	109.5
C17—C16—H15B	109.1	H22B—C17—H22C	109.5
C15—C16—H15B	109.1		
C15—C13—C12—C9	−177.9 (2)	C9—C12—C11—O4	18.3 (2)
C14—C13—C12—C9	2.9 (4)	O2—C3—C4—C5	−178.3 (3)
C15—C13—C12—C11	−14.8 (4)	C2—C3—C4—C5	2.1 (4)
C14—C13—C12—C11	166.0 (2)	C6—C5—C4—C3	−1.0 (4)
C4—C5—C6—C7	−0.9 (4)	C13—C12—C9—C8	−47.3 (5)
C4—C5—C6—C8	−176.0 (2)	C11—C12—C9—C8	147.2 (3)
C2—C7—C6—C5	1.6 (4)	C13—C12—C9—C10	145.7 (2)
C2—C7—C6—C8	177.0 (2)	C11—C12—C9—C10	−19.8 (2)
C6—C7—C2—C3	−0.6 (4)	C12—C13—C15—C16	−90.1 (3)
C6—C7—C2—O1	178.6 (2)	C14—C13—C15—C16	89.1 (3)
C1—O1—C2—C7	−179.0 (3)	C17—C16—C15—C13	−174.9 (3)
C1—O1—C2—C3	0.2 (3)	C12—C9—C8—C6	−6.4 (5)
C1—O2—C3—C4	−179.4 (3)	C10—C9—C8—C6	159.5 (3)
C1—O2—C3—C2	0.2 (3)	C5—C6—C8—C9	−20.4 (5)
C7—C2—C3—C4	−1.4 (4)	C7—C6—C8—C9	164.4 (3)
O1—C2—C3—C4	179.4 (2)	C11—O4—C10—O3	176.9 (3)
C7—C2—C3—O2	179.0 (2)	C11—O4—C10—C9	−3.5 (3)
O1—C2—C3—O2	−0.3 (3)	C8—C9—C10—O3	24.7 (5)
C10—O4—C11—O5	171.4 (2)	C12—C9—C10—O3	−165.5 (3)
C10—O4—C11—C12	−9.4 (3)	C8—C9—C10—O4	−154.8 (2)

C13—C12—C11—O5	30.6 (4)	C12—C9—C10—O4	15.0 (3)
C9—C12—C11—O5	−162.7 (3)	C3—O2—C1—O1	−0.1 (3)
C13—C12—C11—O4	−148.4 (2)	C2—O1—C1—O2	−0.1 (3)

Fig. 1


O3
C10
O1
C7
C8
C9
O4
C2
C6
C1
C12
C11
C3
C5
O2
C4
C14
C13
O5
C15
C16
C17