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3-[(1,3-Benzodioxol-6-yl)methylene]-4-(pentan-3-ylidene)tetrahydrofuran-2,5-dione

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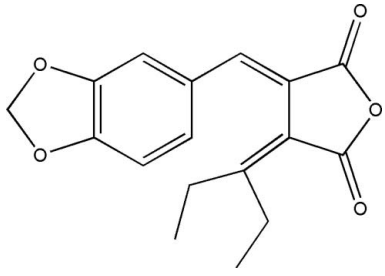
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.150; data-to-parameter ratio = 16.9.

In the structure of 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 30.22 (12)°. The dihedral angle between the atoms defining the planar part of the dihydrofuran ring and the aryl ring is 19.12 (8)°

Related literature

For related literature, see: Heller *et al.* (2000); Liang *et al.* (2001); Walz *et al.* (1993).



Experimental

Crystal data

$C_{17}H_{17}O_5$
M_r = 301.31
Monoclinic, $C2/c$
a = 21.0914 (3) Å
b = 10.4909 (2) Å
c = 14.2684 (2) Å
β = 110.6560 (10)°
V = 2954.18 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
0.34 × 0.31 × 0.31 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
T_{min} = 0.804, T_{max} = 1.000
(expected range 0.7798–0.9696)
9328 measured reflections
3397 independent reflections
2265 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.150
S = 1.03
3397 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; 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: BV2051).

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supplementary materials

Acta Cryst. (2007). E**63**, o3066 [**doi:10.1107/S1600536807025470**]

3-[(1,3-Benzodioxol-6-yl)methylene]-4-(pentan-3-ylidene)tetrahydrofuran-2,5-dione

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

Comment

Photochromic fulgides with aromatic heterocycles such as furan, thiophene, pyrrole, indole and thiazole rings have been synthesized and their spectroscopic and photochromic properties have been previously described (Liang *et al.*, 2001; Walz *et al.*, 1993). In order to achieve certain properties, such as absorption of the colored form at longer wavelengths and 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-6-yl)methylene)-3-(pentan-3-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 0°C, 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 76%). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a solution of the solid in dichloromethane at room temperature for 10 d.

Refinement

(type here to add refinement details)

Figures

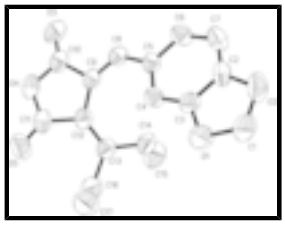


Fig. 1. (type here to add)

3-[(1,3-Benzodioxol-6-yl)methylene]-4-(pentan-3-ylidene)tetrahydrofuran- 2,5-dione

Crystal data

$C_{17}H_{17}O_5$	F_{000} = 1272
M_r = 301.31	D_x = 1.355 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
a = 21.0914 (3) Å	Cell parameters from 2665 reflections

b = 10.4909 (2) Å	θ = 2.2–24.8º
c = 14.2684 (2) Å	μ = 0.10 mm^{-1}
β = 110.6560 (10)º	T = 296 (2) K
V = 2954.18 (8) Å^3	Block, orange-yellow
Z = 8	0.34 × 0.31 × 0.31 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	3397 independent reflections
Radiation source: fine-focus sealed tube	2265 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.019
T = 296(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (APEX2; Bruker, 2005)	h = −27→26
T_{min} = 0.804, T_{max} = 1.000	k = −9→13
9328 measured reflections	l = −17→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.049	H-atom parameters constrained
$wR(F^2)$ = 0.150	$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 1.5071P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.013
3397 reflections	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
201 parameters	$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. All H atoms were placed in geometrically calculated positions and refined using a riding model with C—H = 0.97%A (for CH_2 groups) and 0.96%A (for CH_3 groups), their isotropic displacement parameters were set to 1.2 times (1.5 times for CH_3 groups) the equivalent displacement parameter of their parent atoms.

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 $F^2 > 2$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}
C1	0.50597 (11)	0.1433 (2)	0.09603 (19)	0.0667 (6)
H1A	0.5511	0.1470	0.1464	0.080*
H1B	0.5102	0.1303	0.0312	0.080*
C2	0.41037 (10)	0.09418 (19)	0.12185 (15)	0.0510 (5)
C3	0.41168 (9)	0.22456 (18)	0.10944 (13)	0.0464 (4)
C4	0.36026 (9)	0.30202 (17)	0.11198 (14)	0.0443 (4)
H4	0.3619	0.3897	0.1033	0.053*
C5	0.30475 (9)	0.24430 (17)	0.12824 (13)	0.0431 (4)
C6	0.30334 (10)	0.11233 (19)	0.13665 (15)	0.0529 (5)
H6	0.2656	0.0744	0.1444	0.063*
C7	0.35620 (11)	0.03484 (19)	0.13395 (17)	0.0610 (6)
H7	0.3547	−0.0533	0.1401	0.073*
C8	0.24709 (9)	0.31722 (18)	0.13190 (13)	0.0458 (4)
H8	0.2082	0.2691	0.1218	0.055*
C9	0.24012 (9)	0.44169 (18)	0.14725 (14)	0.0466 (4)
C10	0.17018 (10)	0.4924 (2)	0.11986 (15)	0.0543 (5)
C11	0.23940 (11)	0.6642 (2)	0.12756 (16)	0.0579 (5)
C12	0.28464 (9)	0.55359 (18)	0.17105 (15)	0.0492 (5)
H12	0.2982	0.5431	0.1125	0.059*
C13	0.34872 (9)	0.56812 (18)	0.23532 (14)	0.0504 (5)
C14	0.38614 (11)	0.4624 (2)	0.30374 (16)	0.0605 (6)
H14A	0.4288	0.4474	0.2940	0.073*
H14B	0.3596	0.3847	0.2860	0.073*
C15	0.40001 (14)	0.4921 (3)	0.41240 (17)	0.0838 (8)
H15A	0.4255	0.5698	0.4300	0.126*
H15B	0.4255	0.4237	0.4532	0.126*
H15C	0.3579	0.5016	0.4233	0.126*
C16	0.38779 (11)	0.6895 (2)	0.24180 (18)	0.0658 (6)
H16A	0.4253	0.6924	0.3052	0.079*
H16B	0.3586	0.7618	0.2392	0.079*
C17	0.41472 (13)	0.6982 (3)	0.1561 (2)	0.0820 (8)
H17A	0.4412	0.6238	0.1562	0.123*
H17B	0.4425	0.7729	0.1645	0.123*
H17C	0.3774	0.7034	0.0937	0.123*
O1	0.47048 (7)	0.25917 (14)	0.09591 (13)	0.0676 (4)
O2	0.46851 (8)	0.04115 (14)	0.11749 (13)	0.0715 (5)
O3	0.11796 (7)	0.44018 (16)	0.10856 (12)	0.0705 (5)
O4	0.17249 (7)	0.62320 (14)	0.10505 (11)	0.0634 (4)
O5	0.25015 (9)	0.77286 (16)	0.11528 (14)	0.0801 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0493 (12)	0.0696 (15)	0.0853 (16)	0.0149 (10)	0.0290 (11)	−0.0041 (12)

C2	0.0536 (11)	0.0480 (11)	0.0537 (12)	0.0170 (8)	0.0218 (9)	0.0052 (8)
C3	0.0421 (10)	0.0482 (11)	0.0507 (11)	0.0013 (8)	0.0187 (8)	−0.0029 (8)
C4	0.0471 (10)	0.0379 (9)	0.0511 (10)	0.0024 (7)	0.0211 (8)	−0.0017 (8)
C5	0.0428 (9)	0.0453 (10)	0.0415 (9)	0.0049 (7)	0.0154 (7)	−0.0010 (7)
C6	0.0573 (12)	0.0476 (11)	0.0610 (12)	−0.0037 (9)	0.0299 (10)	0.0008 (9)
C7	0.0740 (14)	0.0411 (11)	0.0748 (15)	0.0077 (10)	0.0349 (12)	0.0074 (9)
C8	0.0406 (9)	0.0532 (11)	0.0458 (10)	0.0015 (8)	0.0179 (8)	−0.0013 (8)
C9	0.0384 (9)	0.0549 (11)	0.0479 (11)	0.0094 (8)	0.0167 (8)	0.0004 (8)
C10	0.0458 (11)	0.0692 (14)	0.0491 (11)	0.0145 (9)	0.0183 (9)	−0.0020 (9)
C11	0.0581 (12)	0.0594 (14)	0.0594 (13)	0.0171 (10)	0.0246 (10)	0.0021 (10)
C12	0.0488 (11)	0.0499 (11)	0.0541 (11)	0.0133 (8)	0.0247 (9)	0.0003 (8)
C13	0.0469 (10)	0.0512 (11)	0.0536 (11)	0.0072 (8)	0.0186 (9)	−0.0081 (9)
C14	0.0531 (12)	0.0655 (14)	0.0554 (12)	0.0083 (10)	0.0098 (9)	−0.0047 (10)
C15	0.0821 (17)	0.102 (2)	0.0558 (14)	0.0153 (15)	0.0102 (12)	−0.0097 (13)
C16	0.0587 (12)	0.0553 (13)	0.0827 (16)	0.0017 (10)	0.0240 (11)	−0.0093 (11)
C17	0.0752 (16)	0.0712 (16)	0.111 (2)	0.0034 (13)	0.0475 (15)	0.0022 (14)
O1	0.0496 (8)	0.0570 (9)	0.1072 (13)	0.0041 (7)	0.0412 (8)	−0.0038 (8)
O2	0.0639 (9)	0.0612 (10)	0.0965 (12)	0.0269 (7)	0.0372 (9)	0.0110 (8)
O3	0.0409 (8)	0.0901 (12)	0.0817 (11)	0.0092 (7)	0.0231 (7)	−0.0024 (8)
O4	0.0519 (8)	0.0672 (10)	0.0702 (10)	0.0235 (7)	0.0204 (7)	0.0069 (7)
O5	0.0864 (12)	0.0522 (10)	0.1033 (13)	0.0185 (8)	0.0353 (10)	0.0146 (9)

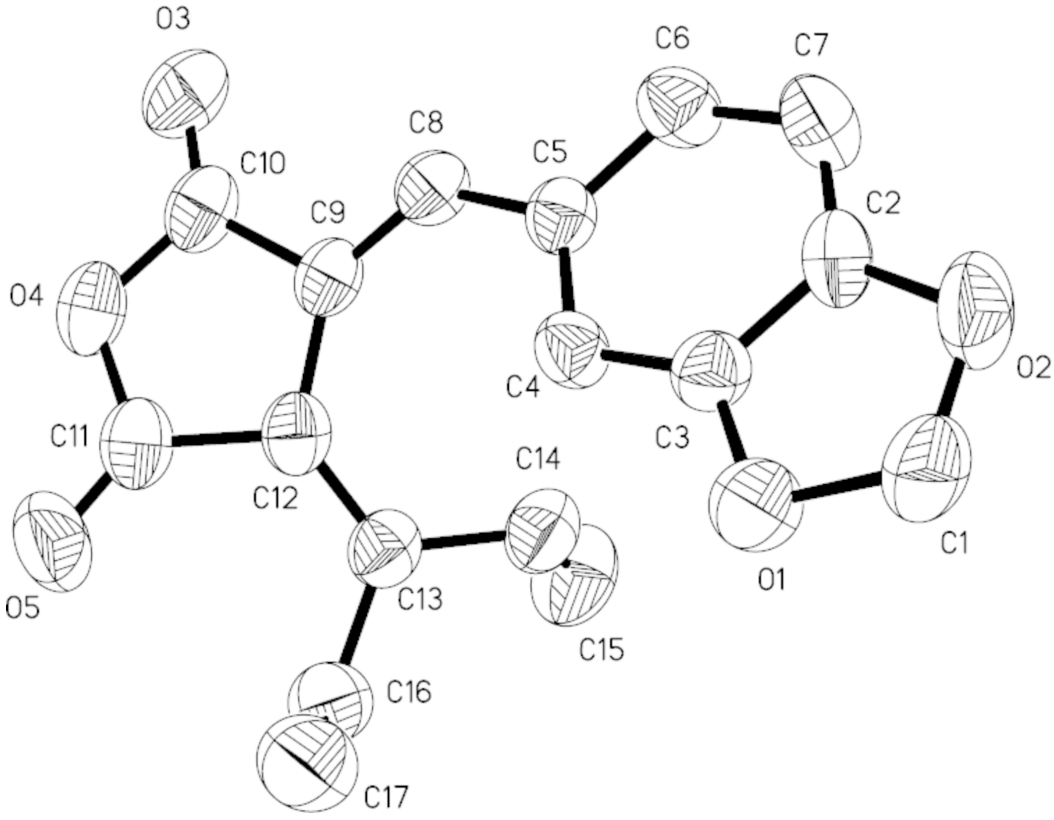
Geometric parameters (Å, °)

C1—O1	1.427 (2)	C10—O4	1.392 (3)
C1—O2	1.427 (3)	C11—O5	1.187 (3)
C1—H1A	0.9700	C11—O4	1.400 (3)
C1—H1B	0.9700	C11—C12	1.492 (3)
C2—C7	1.364 (3)	C12—C13	1.348 (3)
C2—O2	1.368 (2)	C12—H12	0.9800
C2—C3	1.381 (3)	C13—C16	1.502 (3)
C3—C4	1.366 (2)	C13—C14	1.504 (3)
C3—O1	1.369 (2)	C14—C15	1.505 (3)
C4—C5	1.408 (2)	C14—H14A	0.9700
C4—H4	0.9300	C14—H14B	0.9700
C5—C6	1.391 (3)	C15—H15A	0.9600
C5—C8	1.453 (2)	C15—H15B	0.9600
C6—C7	1.391 (3)	C15—H15C	0.9600
C6—H6	0.9300	C16—C17	1.521 (3)
C7—H7	0.9300	C16—H16A	0.9700
C8—C9	1.341 (3)	C16—H16B	0.9700
C8—H8	0.9300	C17—H17A	0.9600
C9—C12	1.466 (3)	C17—H17B	0.9600
C9—C10	1.485 (2)	C17—H17C	0.9600
C10—O3	1.189 (2)		
O1—C1—O2	108.05 (15)	C13—C12—C9	130.89 (18)
O1—C1—H1A	110.1	C13—C12—C11	122.39 (18)
O2—C1—H1A	110.1	C9—C12—C11	105.27 (16)
O1—C1—H1B	110.1	C13—C12—H12	93.9

O2—C1—H1B	110.1	C9—C12—H12	93.9
H1A—C1—H1B	108.4	C11—C12—H12	93.9
C7—C2—O2	128.63 (18)	C12—C13—C16	122.31 (18)
C7—C2—C3	121.59 (17)	C12—C13—C14	121.92 (18)
O2—C2—C3	109.76 (17)	C16—C13—C14	115.75 (17)
C4—C3—O1	127.80 (17)	C13—C14—C15	112.49 (19)
C4—C3—C2	122.26 (17)	C13—C14—H14A	109.1
O1—C3—C2	109.93 (16)	C15—C14—H14A	109.1
C3—C4—C5	117.56 (17)	C13—C14—H14B	109.1
C3—C4—H4	121.2	C15—C14—H14B	109.1
C5—C4—H4	121.2	H14A—C14—H14B	107.8
C6—C5—C4	119.11 (16)	C14—C15—H15A	109.5
C6—C5—C8	118.51 (16)	C14—C15—H15B	109.5
C4—C5—C8	122.32 (16)	H15A—C15—H15B	109.5
C5—C6—C7	122.52 (18)	C14—C15—H15C	109.5
C5—C6—H6	118.7	H15A—C15—H15C	109.5
C7—C6—H6	118.7	H15B—C15—H15C	109.5
C2—C7—C6	116.86 (18)	C13—C16—C17	110.73 (18)
C2—C7—H7	121.6	C13—C16—H16A	109.5
C6—C7—H7	121.6	C17—C16—H16A	109.5
C9—C8—C5	131.29 (18)	C13—C16—H16B	109.5
C9—C8—H8	114.4	C17—C16—H16B	109.5
C5—C8—H8	114.4	H16A—C16—H16B	108.1
C8—C9—C12	136.01 (17)	C16—C17—H17A	109.5
C8—C9—C10	117.51 (18)	C16—C17—H17B	109.5
C12—C9—C10	105.64 (16)	H17A—C17—H17B	109.5
O3—C10—O4	120.93 (18)	C16—C17—H17C	109.5
O3—C10—C9	131.1 (2)	H17A—C17—H17C	109.5
O4—C10—C9	107.97 (17)	H17B—C17—H17C	109.5
O5—C11—O4	119.41 (19)	C3—O1—C1	105.95 (15)
O5—C11—C12	132.9 (2)	C2—O2—C1	106.10 (15)
O4—C11—C12	107.52 (18)	C10—O4—C11	110.78 (15)
C7—C2—C3—C4	2.3 (3)	C10—C9—C12—C11	−16.76 (19)
O2—C2—C3—C4	−179.03 (17)	O5—C11—C12—C13	22.6 (4)
C7—C2—C3—O1	−178.17 (19)	O4—C11—C12—C13	−152.96 (18)
O2—C2—C3—O1	0.5 (2)	O5—C11—C12—C9	−169.8 (2)
O1—C3—C4—C5	−179.23 (18)	O4—C11—C12—C9	14.7 (2)
C2—C3—C4—C5	0.2 (3)	C9—C12—C13—C16	174.36 (18)
C3—C4—C5—C6	−2.7 (3)	C11—C12—C13—C16	−21.5 (3)
C3—C4—C5—C8	−179.70 (17)	C9—C12—C13—C14	−4.3 (3)
C4—C5—C6—C7	2.8 (3)	C11—C12—C13—C14	159.83 (18)
C8—C5—C6—C7	179.99 (19)	C12—C13—C14—C15	−113.0 (2)
O2—C2—C7—C6	179.5 (2)	C16—C13—C14—C15	68.3 (2)
C3—C2—C7—C6	−2.2 (3)	C12—C13—C16—C17	−78.3 (2)
C5—C6—C7—C2	−0.4 (3)	C14—C13—C16—C17	100.4 (2)
C6—C5—C8—C9	163.7 (2)	C4—C3—O1—C1	−178.1 (2)
C4—C5—C8—C9	−19.3 (3)	C2—C3—O1—C1	2.4 (2)
C5—C8—C9—C12	−3.8 (4)	O2—C1—O1—C3	−4.3 (2)
C5—C8—C9—C10	164.00 (18)	C7—C2—O2—C1	175.4 (2)

C8—C9—C10—O3	20.8 (3)	C3—C2—O2—C1	−3.1 (2)
C12—C9—C10—O3	−168.0 (2)	O1—C1—O2—C2	4.6 (2)
C8—C9—C10—O4	−157.82 (17)	O3—C10—O4—C11	176.97 (18)
C12—C9—C10—O4	13.4 (2)	C9—C10—O4—C11	−4.2 (2)
C8—C9—C12—C13	−41.9 (4)	O5—C11—O4—C10	177.19 (19)
C10—C9—C12—C13	149.4 (2)	C12—C11—O4—C10	−6.6 (2)
C8—C9—C12—C11	152.0 (2)		

Fig. 1


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