Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2E,2′*E*)-3,3′-(1,4-Phenylene)bis[1-(3,4-dimethoxyphenyl)prop-2-en-1-one]

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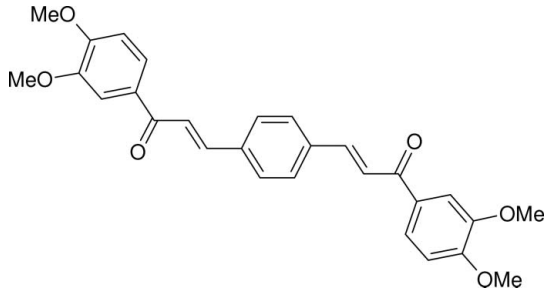
Received 29 May 2007; accepted 29 May 2007

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.117; data-to-parameter ratio = 16.6.

In the centrosymmetric title compound, $C_{28}H_{26}O_6$, the dihedral angle between the central and terminal aromatic rings is 27.72 (9)°.

Related literature

For a related structure, see: Harrison *et al.* (2007*a*). For background, see: Harrison *et al.* (2007*b*).



Experimental

Crystal data

$C_{28}H_{26}O_6$
M_r = 458.48
Monoclinic, $P2_1/n$
a = 10.6974 (9) Å
b = 10.4056 (9) Å
c = 11.0337 (9) Å
β = 111.015 (2)°
V = 1146.50 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 (2) K
0.51 × 0.47 × 0.28 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: none
6150 measured reflections
2593 independent reflections
1845 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.117
S = 1.03
2593 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

HJR and SMD thank DAE-BRNS for financial assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2159).

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  doi:10.1107/S1600536807026281

supplementary materials

Acta Cryst. (2007). E**63**, o3068 [**doi:10.1107/S1600536807026281**]

(2E,2'*E*)-3,3'-(1,4-Phenylene)bis[1-(3,4-dimethoxyphenyl)prop-2-en-1-one]

W. T. A. Harrison, H. J. Ravindra, M. R. Suresh Kumar and S. M. Dharmaprakash

Comment

As part of our ongoing studies of organic nonlinear optical materials derived from chalcone (Harrison *et al.*, 2007*a*,b), we now report the synthesis and structure of the title compound, (I), (Fig. 1).

The molecule of (I) is centrosymmetric and the dihedral angle between the central C1—C3/C1^{i}—C3^{i} (i = −*x*, 1 − *y*, 2 − *z*) and pendant C7—C12 benzene rings is 27.72 (9)°. The dihedral angles for the enone (C4/C5/C6/O1) fragment with respect to C1—C3/C1^{i}—C3^{i} and C7—C12 are 15.54 (12)° and 12.93 (12)°, respectively. The terminal C14 methyl carbon atom is displaced slightly further from the C7—C12 ring than is C13 [deviations = −0.073 (3)Å and 0.151 (3) Å, respectively].

There are no π-π stacking interactions in (I) and the crystal packing (Fig. 2), which bears little if any resemblence to that in the related compound (2E,2'*E*)-3,3'-(1,4-phenylene)bis[1-(4-methoxyphenyl)prop-2-en-1-one] (Harrison *et al.*, 2007*a*), appears to be controlled by van der Waals forces.

Experimental

A solution of ethanol (25 ml) and 10% sodium hydroxide (5 ml) solution were taken in a conical flask. A previously prepared small portion of terephthalaldehyde (0.001 mol) and 1-(3,4-dimethoxyphenyl)ethanone (0.002 mol) dissolved in methanol was added to the conical flask with stirring and the temperature of the solution was maintained between 298–303 K. A precipitate was obtained after stirring the solution for about five minutes. The remaining portion of the aldehyde and ketone mixture was added and the solution was stirred for a further 30 minutes. The separated product was filtered and washed with water and dried. The crude product was recrystallized from DMF solution and single crystals of (I) were grown by slow evaporation of a second DMF solution.

Refinement

The hydrogen atoms were placed in calculated positions (C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate but not to tip to best fit the electron density.

Figures

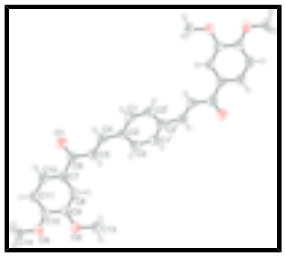

Fig. 1. View of the molecular structure of (I) showing 30% displacement ellipsoids (H atoms are drawn as spheres of arbitrary radius). Symmetry code: (i) −*x*, 1 − *y*, 2 − *z*.

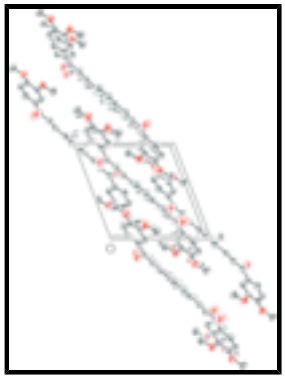

Fig. 2. Unit cell packing for (I) with hydrogen atoms omitted for clarity.

(2E,2'*E*)-3,3'-(1,4-Phenylene)bis[1-(3,4-methoxyphenyl)prop-2-en-1-one]

Crystal data

$C_{28}H_{26}O_6$	F_{000} = 484
M_r = 458.48	D_x = 1.328 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2493 reflections
a = 10.6974 (9) Å	θ = 4.4–27.3º
b = 10.4056 (9) Å	μ = 0.09 mm^{-1}
c = 11.0337 (9) Å	T = 295 (2) K
β = 111.015 (2)º	Slab, pale yellow
V = 1146.50 (17) Å^3	0.51 × 0.47 × 0.28 mm
Z = 2	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	1845 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.037
Monochromator: graphite	θ_{max} = 27.6º
T = 295(2) K	θ_{min} = 4.5º
ω scans	h = −12→13
Absorption correction: none	k = −13→7
6150 measured reflections	l = −14→13
2593 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.042	H-atom parameters constrained
$wR(F^2)$ = 0.117	$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.102P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
2593 reflections	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
156 parameters	$\Delta\rho_{min}$ = −0.23 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}
C1	0.02570 (14)	0.62724 (13)	0.98401 (14)	0.0531 (4)
H1	0.0426	0.7134	0.9735	0.064*
C2	0.06430 (13)	0.53394 (12)	0.91311 (12)	0.0458 (3)
C3	0.03669 (14)	0.40569 (13)	0.93106 (14)	0.0543 (4)
H3	0.0608	0.3416	0.8849	0.065*
C4	0.13242 (13)	0.57377 (13)	0.82596 (13)	0.0492 (3)
H4	0.1527	0.6608	0.8269	0.059*
C5	0.16902 (14)	0.50108 (13)	0.74548 (14)	0.0520 (3)
H5	0.1510	0.4134	0.7409	0.062*
C6	0.23769 (14)	0.55662 (13)	0.66305 (14)	0.0522 (3)
C7	0.24029 (13)	0.48429 (12)	0.54784 (13)	0.0480 (3)
C8	0.16721 (13)	0.37084 (12)	0.50367 (13)	0.0488 (3)
H8	0.1149	0.3380	0.5479	0.059*
C9	0.17175 (13)	0.30731 (13)	0.39578 (13)	0.0480 (3)
C10	0.25223 (13)	0.35637 (14)	0.32880 (13)	0.0497 (3)
C11	0.32134 (15)	0.46974 (14)	0.37027 (15)	0.0567 (4)
H11	0.3720	0.5041	0.3250	0.068*
C12	0.31592 (15)	0.53259 (14)	0.47846 (15)	0.0561 (4)
H12	0.3636	0.6085	0.5055	0.067*
C13	0.01953 (17)	0.14495 (16)	0.40800 (18)	0.0678 (4)
H13A	−0.0219	0.0676	0.3645	0.102*
H13B	−0.0484	0.2067	0.4043	0.102*
H13C	0.0716	0.1260	0.4970	0.102*
C14	0.34061 (18)	0.32855 (19)	0.16052 (17)	0.0758 (5)
H14A	0.3366	0.2690	0.0927	0.114*
H14B	0.4312	0.3346	0.2207	0.114*
H14C	0.3109	0.4116	0.1235	0.114*

O1	0.29246 (12)	0.66240 (10)	0.68998 (12)	0.0766 (4)
O2	0.10457 (11)	0.19627 (10)	0.34583 (11)	0.0651 (3)
O3	0.25563 (10)	0.28434 (11)	0.22740 (10)	0.0629 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0634 (9)	0.0385 (7)	0.0622 (8)	−0.0027 (6)	0.0285 (7)	−0.0062 (6)
C2	0.0445 (7)	0.0438 (7)	0.0477 (7)	0.0000 (5)	0.0148 (6)	−0.0045 (5)
C3	0.0655 (9)	0.0433 (7)	0.0609 (8)	−0.0002 (6)	0.0311 (7)	−0.0108 (6)
C4	0.0506 (7)	0.0428 (7)	0.0547 (8)	−0.0004 (6)	0.0196 (6)	−0.0022 (6)
C5	0.0585 (8)	0.0417 (7)	0.0610 (8)	−0.0018 (6)	0.0278 (7)	−0.0023 (6)
C6	0.0536 (8)	0.0422 (7)	0.0668 (9)	0.0002 (6)	0.0287 (7)	−0.0003 (6)
C7	0.0497 (7)	0.0412 (7)	0.0576 (8)	0.0022 (6)	0.0249 (6)	0.0044 (6)
C8	0.0500 (7)	0.0453 (7)	0.0586 (8)	−0.0011 (6)	0.0286 (6)	0.0045 (6)
C9	0.0450 (7)	0.0449 (7)	0.0568 (8)	−0.0017 (5)	0.0216 (6)	0.0023 (6)
C10	0.0490 (7)	0.0548 (8)	0.0480 (7)	0.0026 (6)	0.0205 (6)	0.0052 (6)
C11	0.0615 (8)	0.0566 (8)	0.0624 (9)	−0.0058 (7)	0.0347 (7)	0.0075 (7)
C12	0.0610 (8)	0.0455 (7)	0.0683 (9)	−0.0090 (6)	0.0312 (7)	0.0016 (7)
C13	0.0689 (10)	0.0598 (9)	0.0853 (11)	−0.0209 (8)	0.0404 (9)	−0.0075 (8)
C14	0.0750 (11)	0.1011 (14)	0.0658 (10)	−0.0157 (10)	0.0426 (9)	−0.0087 (9)
O1	0.0974 (9)	0.0538 (7)	0.1000 (9)	−0.0231 (6)	0.0612 (7)	−0.0188 (6)
O2	0.0719 (7)	0.0612 (6)	0.0760 (7)	−0.0203 (5)	0.0433 (6)	−0.0156 (5)
O3	0.0667 (6)	0.0735 (7)	0.0580 (6)	−0.0107 (5)	0.0339 (5)	−0.0067 (5)

Geometric parameters (Å, °)

C1—C3^{i}	1.3746 (19)	C8—H8	0.9300
C1—C2	1.3988 (18)	C9—O2	1.3679 (16)
C1—H1	0.9300	C9—C10	1.4157 (18)
C2—C3	1.3962 (19)	C10—O3	1.3580 (16)
C2—C4	1.4594 (18)	C10—C11	1.380 (2)
C3—C1^{i}	1.3746 (19)	C11—C12	1.380 (2)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.3279 (18)	C12—H12	0.9300
C4—H4	0.9300	C13—O2	1.4250 (17)
C5—C6	1.4765 (18)	C13—H13A	0.9600
C5—H5	0.9300	C13—H13B	0.9600
C6—O1	1.2322 (17)	C13—H13C	0.9600
C6—C7	1.4861 (19)	C14—O3	1.4366 (18)
C7—C12	1.3918 (18)	C14—H14A	0.9600
C7—C8	1.4033 (18)	C14—H14B	0.9600
C8—C9	1.3777 (19)	C14—H14C	0.9600
C3^{i}—C1—C2	121.41 (13)	O2—C9—C10	115.10 (12)
C3^{i}—C1—H1	119.3	C8—C9—C10	119.64 (12)
C2—C1—H1	119.3	O3—C10—C11	125.13 (12)
C3—C2—C1	117.65 (12)	O3—C10—C9	115.60 (12)
C3—C2—C4	123.09 (12)	C11—C10—C9	119.26 (13)

C1—C2—C4	119.25 (12)	C10—C11—C12	120.53 (12)
C1^{i}—C3—C2	120.94 (12)	C10—C11—H11	119.7
C1^{i}—C3—H3	119.5	C12—C11—H11	119.7
C2—C3—H3	119.5	C11—C12—C7	121.16 (13)
C5—C4—C2	127.99 (13)	C11—C12—H12	119.4
C5—C4—H4	116.0	C7—C12—H12	119.4
C2—C4—H4	116.0	O2—C13—H13A	109.5
C4—C5—C6	121.34 (12)	O2—C13—H13B	109.5
C4—C5—H5	119.3	H13A—C13—H13B	109.5
C6—C5—H5	119.3	O2—C13—H13C	109.5
O1—C6—C5	119.98 (13)	H13A—C13—H13C	109.5
O1—C6—C7	120.36 (12)	H13B—C13—H13C	109.5
C5—C6—C7	119.65 (12)	O3—C14—H14A	109.5
C12—C7—C8	118.30 (13)	O3—C14—H14B	109.5
C12—C7—C6	118.72 (12)	H14A—C14—H14B	109.5
C8—C7—C6	122.96 (12)	O3—C14—H14C	109.5
C9—C8—C7	121.06 (12)	H14A—C14—H14C	109.5
C9—C8—H8	119.5	H14B—C14—H14C	109.5
C7—C8—H8	119.5	C9—O2—C13	117.16 (11)
O2—C9—C8	125.26 (12)	C10—O3—C14	117.09 (12)
C3^{i}—C1—C2—C3	0.3 (2)	C7—C8—C9—O2	179.78 (12)
C3^{i}—C1—C2—C4	−178.89 (12)	C7—C8—C9—C10	0.5 (2)
C1—C2—C3—C1^{i}	−0.3 (2)	O2—C9—C10—O3	−2.31 (18)
C4—C2—C3—C1^{i}	178.86 (13)	C8—C9—C10—O3	177.00 (12)
C3—C2—C4—C5	5.4 (2)	O2—C9—C10—C11	178.49 (12)
C1—C2—C4—C5	−175.49 (14)	C8—C9—C10—C11	−2.2 (2)
C2—C4—C5—C6	179.98 (13)	O3—C10—C11—C12	−176.94 (13)
C4—C5—C6—O1	18.6 (2)	C9—C10—C11—C12	2.2 (2)
C4—C5—C6—C7	−161.43 (13)	C10—C11—C12—C7	−0.5 (2)
O1—C6—C7—C12	5.9 (2)	C8—C7—C12—C11	−1.2 (2)
C5—C6—C7—C12	−174.11 (13)	C6—C7—C12—C11	−179.64 (13)
O1—C6—C7—C8	−172.50 (14)	C8—C9—O2—C13	2.3 (2)
C5—C6—C7—C8	7.5 (2)	C10—C9—O2—C13	−178.43 (13)
C12—C7—C8—C9	1.1 (2)	C11—C10—O3—C14	1.6 (2)
C6—C7—C8—C9	179.53 (12)	C9—C10—O3—C14	−177.53 (13)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+2.

Fig. 1

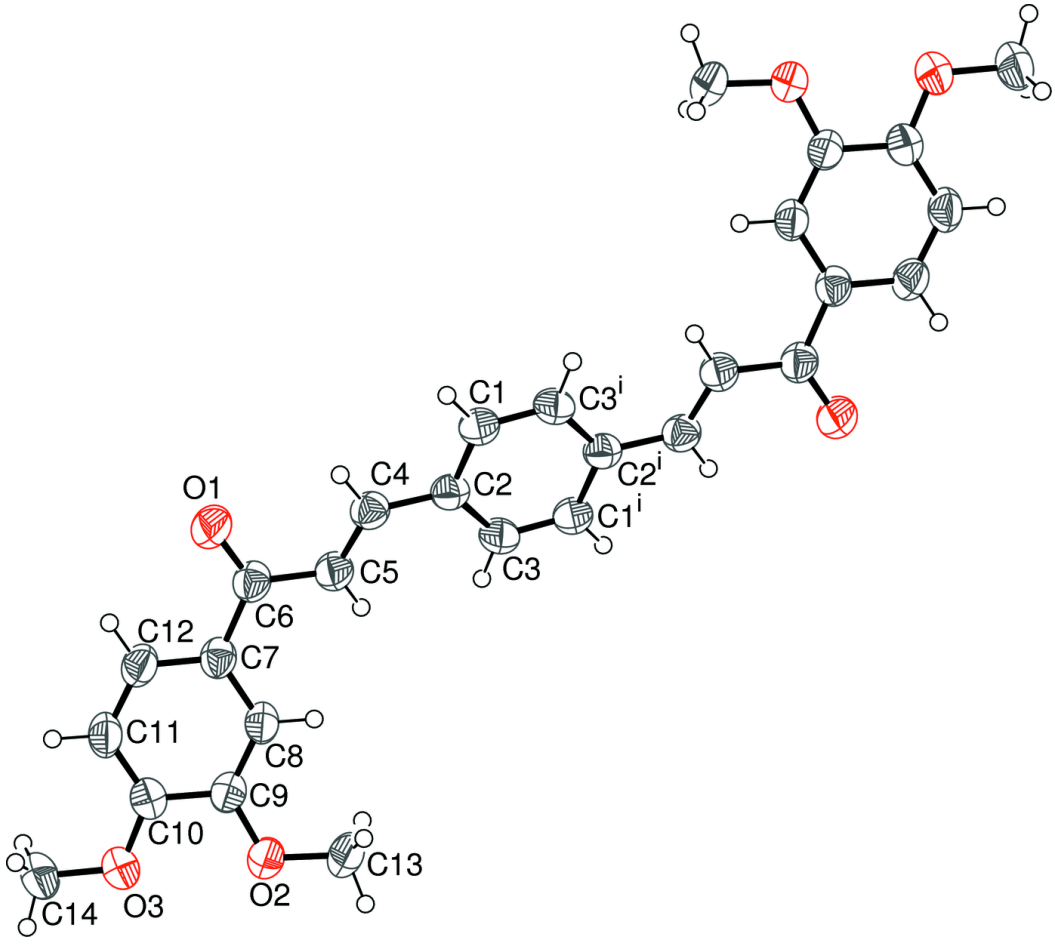

Fig. 2