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

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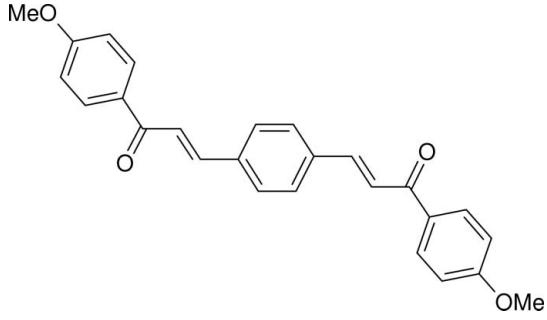
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.050; wR factor = 0.150; data-to-parameter ratio = 14.3.

In the centrosymmetric title compound, $C_{26}H_{22}O_4$, the dihedral angle between the central and terminal aromatic rings is 13.78 (12)°. A weak C—H$\cdots\pi$ interaction may help to consolidate the crystal packing.

Related literature

For background, see: Harrison *et al.* (2007). For commentary on the use of an *I*-centred monoclinic cell, see: Mighell (2003).



Experimental

Crystal data

$C_{26}H_{22}O_4$
M_r = 398.44
Monoclinic, *I*2/*a*
a = 13.0715 (12) Å
b = 5.8535 (6) Å
c = 26.377 (6) Å
β = 90.339 (2)°
V = 2018.2 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 (2) K
0.51 × 0.50 × 0.17 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: none
5231 measured reflections
1956 independent reflections
1449 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.150
S = 1.02
1956 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 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) and *Mercury* (Macrae *et al.*, 2006); 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: BT2384).

References

Bruker (1999). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Harrison, W. T. A., Kumari, V., Ravindra, H. J. & Dharmaprakash, S. M. (2007). *Acta Cryst.* E**63**, o2928.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Mighell, A. D. (2003). *Acta Cryst.* B**59**, 300–302.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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

Comment

As part of our ongoing studies of organic nonlinear optical materials derived from substituted chalcones (Harrison *et al.*, 2007), 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*, −*y*, 1 − *z*; centroid = *Cg*1) and pendant C7—C12 benzene rings is 13.78 (12)°. The dihedral angles for the enone (C4/C5/C6/O1) fragment with respect to C1—C3/C1^{i}—C3^{i} and C7—C12 are 12.05 (12)° and 17.65 (12)°, respectively. The terminal C13 methyl carbon atom is almost co-planar with the C7—C12 ring [deviation = 0.049 (4) Å].

The only possible directional interaction in (I) is a weak C8^{ii}—H8^{ii}···*Cg*1 (ii = 1/2 − *x*, *y*, 1 − *z*) [H^{ii}···*Cg*1 = 2.96 Å, C^{ii}—H^{ii}···*Cg*1 = 120°] bond, which leads to [100] chains in the crystal (Fig. 2). Overall, pseudo (001) sheets of molecules are seen (Fig. 3) in the unit-cell packing of (I).

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-(4-methoxyphenyl)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 30 minutes. The separated product was filtered and washed with water and dried and purified by recrystallization from DMF solution. The single crystals of (I) required for X-ray diffraction analysis was grown by slow evaporation of a DMF solutions.

Refinement

The I-centred setting of the cell was chosen to avoid an obtuse β angle for the conventional C-centred setting (Mighell, 2003).

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 group was allowed to rotate but not to tip to best fit the electron density.

Figures

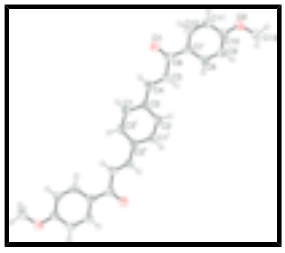
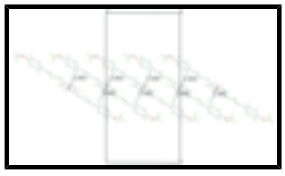


Fig. 1. View of the molecular structure of (I) showing 30% displacement ellipsoids (H atoms are drawn as spheres of arbitrary radius). Symmetry code: (i) −*x*, −*y*, 1 − *z*.



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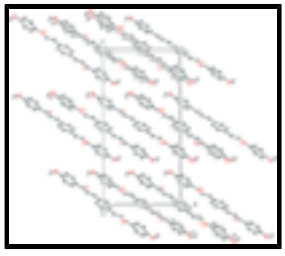

Fig. 3. Unit cell packing for (I) with hydrogen atoms omitted for clarity.

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Crystal data

$C_{26}H_{22}O_4$	F_{000} = 840
M_r = 398.44	D_x = 1.311 Mg m^{-3}
Monoclinic, *I*2/*a*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -I 2ya	Cell parameters from 1969 reflections
a = 13.0715 (12) Å	θ = 4.4–26.0º
b = 5.8535 (6) Å	μ = 0.09 mm^{-1}
c = 26.377 (6) Å	*T* = 295 (2) K
β = 90.339 (2)º	Chunky plate, pale yellow
V = 2018.2 (5) Å^3	0.51 × 0.50 × 0.17 mm
Z = 4	

Data collection

Bruker SMART 1000 CCD diffractometer	1449 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.046
Monochromator: graphite	θ_{max} = 26.0º
T = 295(2) K	θ_{min} = 4.4º
ω scans	*h* = −16→12
Absorption correction: none	*k* = −7→6

5231 measured reflections
1956 independent reflections

$l = -32 \rightarrow 32$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.150$

$S = 1.02$

1956 reflections

137 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0954P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.19$ e Å^{-3}

$\Delta\rho_{min} = -0.25$ e Å^{-3}

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 $F^2 > 2$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	−0.03417 (12)	0.2125 (3)	0.48510 (6)	0.0440 (4)
H1	−0.0578	0.3553	0.4750	0.053*
C2	0.06485 (11)	0.1447 (3)	0.47189 (6)	0.0402 (4)
C3	0.09755 (12)	−0.0719 (3)	0.48714 (6)	0.0440 (4)
H3	0.1627	−0.1220	0.4785	0.053*
C4	0.12999 (13)	0.3030 (3)	0.44335 (6)	0.0452 (4)
H4	0.1023	0.4459	0.4363	0.054*
C5	0.22381 (13)	0.2650 (3)	0.42646 (6)	0.0473 (4)
H5	0.2539	0.1231	0.4319	0.057*
C6	0.28212 (12)	0.4445 (3)	0.39897 (6)	0.0457 (4)
C7	0.37501 (12)	0.3779 (3)	0.37068 (6)	0.0426 (4)
C8	0.42572 (13)	0.1724 (3)	0.37741 (6)	0.0528 (5)
H8	0.3996	0.0665	0.4002	0.063*
C9	0.51388 (13)	0.1198 (3)	0.35134 (7)	0.0552 (5)
H9	0.5471	−0.0183	0.3572	0.066*
C10	0.55274 (13)	0.2734 (3)	0.31642 (6)	0.0468 (5)

C11	0.50199 (15)	0.4779 (3)	0.30838 (7)	0.0606 (5)
H11	0.5267	0.5807	0.2845	0.073*
C12	0.41560 (14)	0.5308 (3)	0.33523 (7)	0.0585 (5)
H12	0.3834	0.6704	0.3298	0.070*
C13	0.69434 (15)	0.0334 (4)	0.29605 (9)	0.0701 (6)
H13A	0.7540	0.0341	0.2750	0.105*
H13B	0.6523	−0.0956	0.2875	0.105*
H13C	0.7147	0.0232	0.3310	0.105*
O1	0.25322 (10)	0.6437 (2)	0.39963 (5)	0.0668 (4)
O2	0.63797 (9)	0.2384 (2)	0.28815 (5)	0.0599 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0395 (9)	0.0379 (10)	0.0548 (9)	0.0016 (7)	0.0072 (7)	−0.0034 (7)
C2	0.0356 (8)	0.0382 (9)	0.0469 (8)	−0.0051 (7)	0.0062 (6)	−0.0072 (7)
C3	0.0325 (8)	0.0440 (11)	0.0555 (9)	0.0003 (7)	0.0092 (6)	−0.0074 (7)
C4	0.0429 (9)	0.0391 (10)	0.0536 (9)	−0.0030 (7)	0.0087 (7)	−0.0040 (7)
C5	0.0423 (9)	0.0445 (10)	0.0554 (10)	−0.0011 (8)	0.0111 (7)	0.0046 (7)
C6	0.0430 (9)	0.0411 (11)	0.0530 (9)	−0.0026 (8)	0.0045 (7)	0.0016 (7)
C7	0.0401 (9)	0.0412 (10)	0.0466 (8)	−0.0050 (7)	0.0042 (7)	0.0045 (7)
C8	0.0489 (10)	0.0519 (12)	0.0577 (10)	0.0018 (9)	0.0159 (8)	0.0185 (8)
C9	0.0495 (10)	0.0481 (11)	0.0683 (11)	0.0072 (9)	0.0158 (8)	0.0162 (9)
C10	0.0422 (9)	0.0504 (11)	0.0479 (9)	−0.0065 (8)	0.0089 (7)	0.0033 (7)
C11	0.0624 (11)	0.0511 (12)	0.0685 (12)	−0.0036 (10)	0.0225 (9)	0.0187 (9)
C12	0.0567 (11)	0.0424 (11)	0.0765 (12)	0.0019 (9)	0.0177 (9)	0.0155 (9)
C13	0.0554 (11)	0.0684 (15)	0.0866 (14)	0.0066 (10)	0.0264 (10)	0.0066 (11)
O1	0.0639 (9)	0.0440 (9)	0.0927 (10)	0.0028 (7)	0.0279 (7)	0.0038 (7)
O2	0.0519 (8)	0.0609 (9)	0.0671 (8)	0.0011 (6)	0.0244 (6)	0.0091 (6)

Geometric parameters (Å, °)

C1—C3^{i}	1.381 (2)	C7—C12	1.401 (2)
C1—C2	1.400 (2)	C8—C9	1.380 (2)
C1—H1	0.9300	C8—H8	0.9300
C2—C3	1.396 (2)	C9—C10	1.386 (2)
C2—C4	1.469 (2)	C9—H9	0.9300
C3—C1^{i}	1.381 (2)	C10—O2	1.360 (2)
C3—H3	0.9300	C10—C11	1.385 (3)
C4—C5	1.326 (2)	C11—C12	1.372 (2)
C4—H4	0.9300	C11—H11	0.9300
C5—C6	1.489 (2)	C12—H12	0.9300
C5—H5	0.9300	C13—O2	1.423 (2)
C6—O1	1.226 (2)	C13—H13A	0.9600
C6—C7	1.481 (2)	C13—H13B	0.9600
C7—C8	1.384 (3)	C13—H13C	0.9600
C3^{i}—C1—C2	121.45 (16)	C9—C8—H8	119.0
C3^{i}—C1—H1	119.3	C7—C8—H8	119.0

C2—C1—H1	119.3	C8—C9—C10	119.80 (17)
C3—C2—C1	117.87 (15)	C8—C9—H9	120.1
C3—C2—C4	122.88 (14)	C10—C9—H9	120.1
C1—C2—C4	119.24 (16)	O2—C10—C11	116.06 (14)
C1^{i}—C3—C2	120.67 (15)	O2—C10—C9	124.88 (17)
C1^{i}—C3—H3	119.7	C11—C10—C9	119.06 (16)
C2—C3—H3	119.7	C12—C11—C10	120.72 (16)
C5—C4—C2	127.35 (17)	C12—C11—H11	119.6
C5—C4—H4	116.3	C10—C11—H11	119.6
C2—C4—H4	116.3	C11—C12—C7	121.12 (18)
C4—C5—C6	121.50 (17)	C11—C12—H12	119.4
C4—C5—H5	119.2	C7—C12—H12	119.4
C6—C5—H5	119.2	O2—C13—H13A	109.5
O1—C6—C7	120.75 (15)	O2—C13—H13B	109.5
O1—C6—C5	120.37 (15)	H13A—C13—H13B	109.5
C7—C6—C5	118.88 (15)	O2—C13—H13C	109.5
C8—C7—C12	117.24 (15)	H13A—C13—H13C	109.5
C8—C7—C6	123.88 (14)	H13B—C13—H13C	109.5
C12—C7—C6	118.87 (16)	C10—O2—C13	118.14 (13)
C9—C8—C7	122.03 (15)		
C3^{i}—C1—C2—C3	0.8 (2)	C12—C7—C8—C9	1.3 (3)
C3^{i}—C1—C2—C4	−178.57 (15)	C6—C7—C8—C9	−178.01 (17)
C1—C2—C3—C1^{i}	−0.8 (2)	C7—C8—C9—C10	−1.4 (3)
C4—C2—C3—C1^{i}	178.55 (15)	C8—C9—C10—O2	−179.07 (16)
C3—C2—C4—C5	2.9 (3)	C8—C9—C10—C11	0.0 (3)
C1—C2—C4—C5	−177.79 (16)	O2—C10—C11—C12	−179.50 (17)
C2—C4—C5—C6	−178.47 (15)	C9—C10—C11—C12	1.3 (3)
C4—C5—C6—O1	13.9 (3)	C10—C11—C12—C7	−1.4 (3)
C4—C5—C6—C7	−165.72 (15)	C8—C7—C12—C11	0.1 (3)
O1—C6—C7—C8	164.35 (18)	C6—C7—C12—C11	179.43 (17)
C5—C6—C7—C8	−16.0 (2)	C11—C10—O2—C13	178.32 (18)
O1—C6—C7—C12	−15.0 (3)	C9—C10—O2—C13	−2.5 (3)
C5—C6—C7—C12	164.68 (16)		

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

Fig. 1

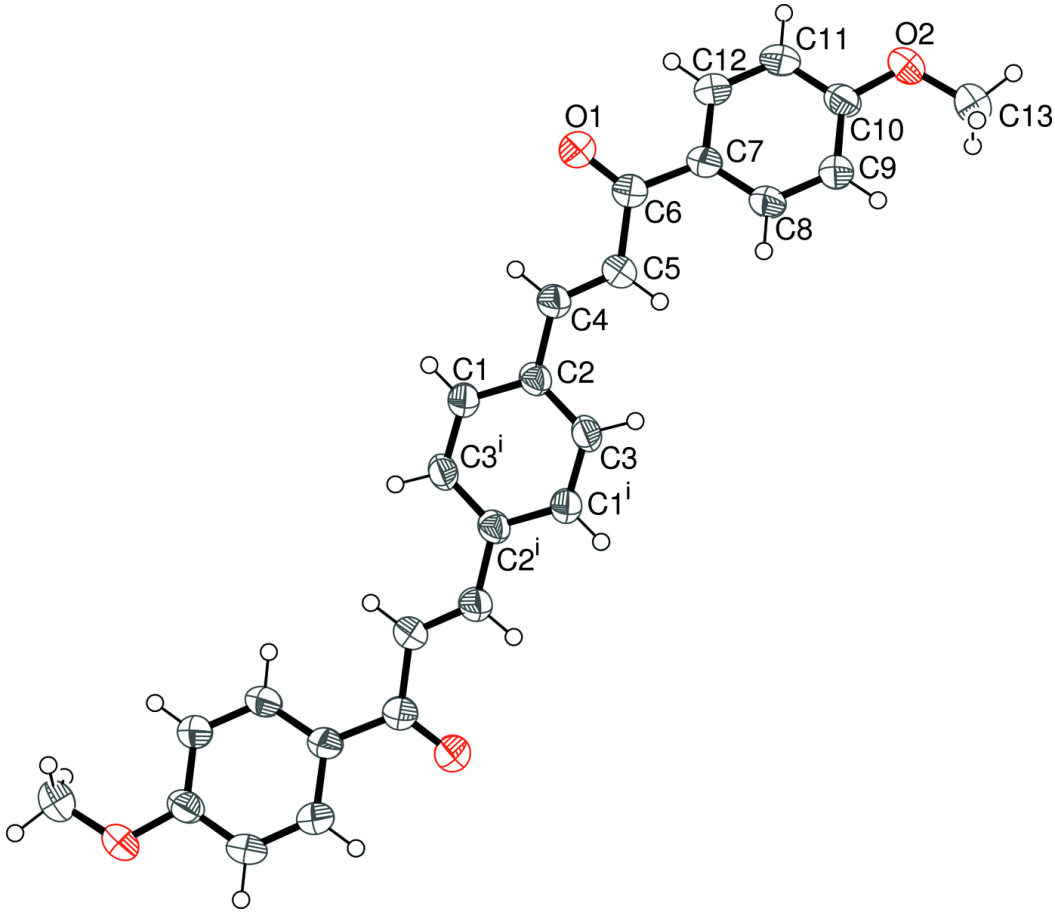


Fig. 2

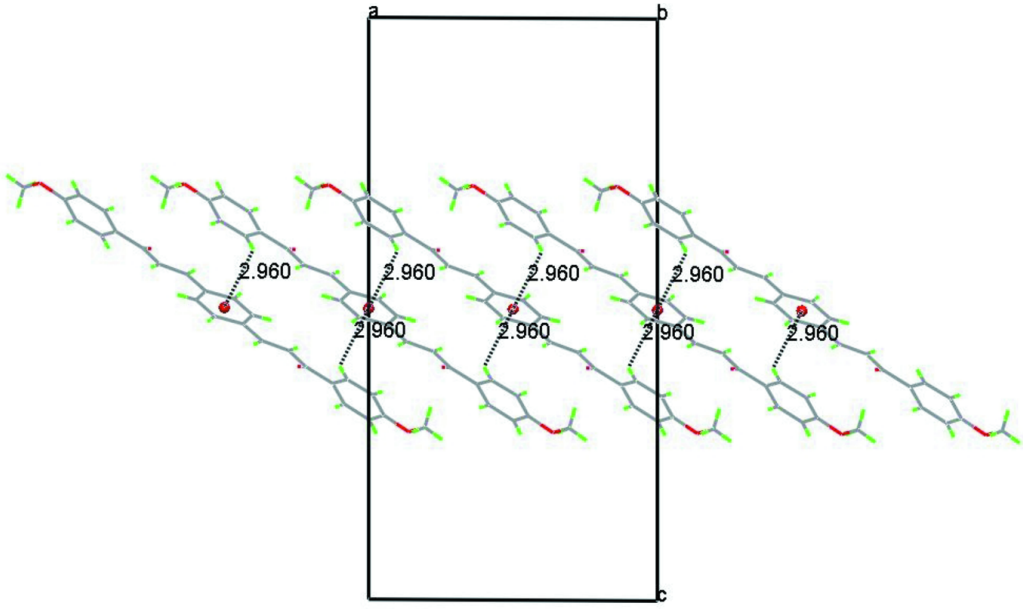
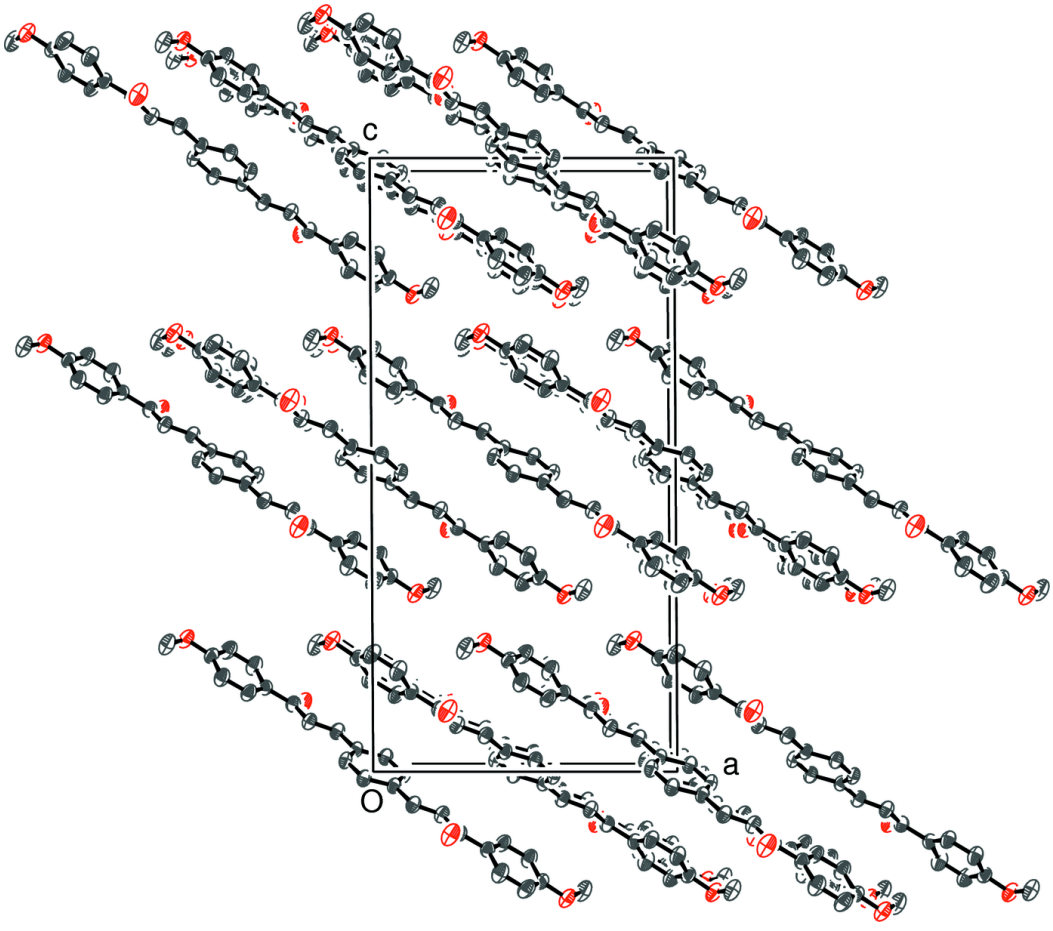


Fig. 3



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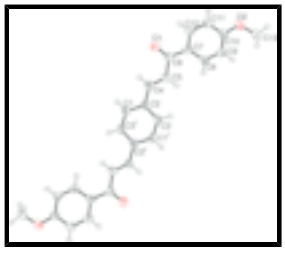

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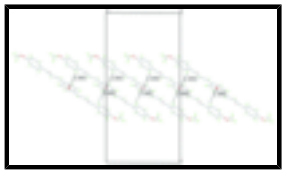

Fig. 2. Fragment of a [100] chain of molecules in (I) linked by weak C—H···π interactions (dashed lines).

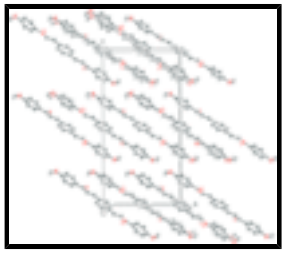

Fig. 3. Unit cell packing for (I) with hydrogen atoms omitted for clarity.

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

Crystal data

$C_{26}H_{22}O_4$	F_{000} = 840
M_r = 398.44	D_x = 1.311 Mg m^{-3}
Monoclinic, *I*2/*a*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -I 2ya	Cell parameters from 1969 reflections
a = 13.0715 (12) Å	θ = 4.4–26.0º
b = 5.8535 (6) Å	μ = 0.09 mm^{-1}
c = 26.377 (6) Å	*T* = 295 (2) K
β = 90.339 (2)º	Chunky plate, pale yellow
V = 2018.2 (5) Å^3	0.51 × 0.50 × 0.17 mm
Z = 4	

Data collection

Bruker SMART 1000 CCD diffractometer	1449 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.046
Monochromator: graphite	θ_{max} = 26.0º
T = 295(2) K	θ_{min} = 4.4º
ω scans	*h* = −16→12
Absorption correction: none	*k* = −7→6

5231 measured reflections
1956 independent reflections

$l = -32 \rightarrow 32$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.150$

$S = 1.02$

1956 reflections

137 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0954P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.19$ e Å^{-3}

$\Delta\rho_{min} = -0.25$ e Å^{-3}

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 $F^2 > 2$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	−0.03417 (12)	0.2125 (3)	0.48510 (6)	0.0440 (4)
H1	−0.0578	0.3553	0.4750	0.053*
C2	0.06485 (11)	0.1447 (3)	0.47189 (6)	0.0402 (4)
C3	0.09755 (12)	−0.0719 (3)	0.48714 (6)	0.0440 (4)
H3	0.1627	−0.1220	0.4785	0.053*
C4	0.12999 (13)	0.3030 (3)	0.44335 (6)	0.0452 (4)
H4	0.1023	0.4459	0.4363	0.054*
C5	0.22381 (13)	0.2650 (3)	0.42646 (6)	0.0473 (4)
H5	0.2539	0.1231	0.4319	0.057*
C6	0.28212 (12)	0.4445 (3)	0.39897 (6)	0.0457 (4)
C7	0.37501 (12)	0.3779 (3)	0.37068 (6)	0.0426 (4)
C8	0.42572 (13)	0.1724 (3)	0.37741 (6)	0.0528 (5)
H8	0.3996	0.0665	0.4002	0.063*
C9	0.51388 (13)	0.1198 (3)	0.35134 (7)	0.0552 (5)
H9	0.5471	−0.0183	0.3572	0.066*
C10	0.55274 (13)	0.2734 (3)	0.31642 (6)	0.0468 (5)

C11	0.50199 (15)	0.4779 (3)	0.30838 (7)	0.0606 (5)
H11	0.5267	0.5807	0.2845	0.073*
C12	0.41560 (14)	0.5308 (3)	0.33523 (7)	0.0585 (5)
H12	0.3834	0.6704	0.3298	0.070*
C13	0.69434 (15)	0.0334 (4)	0.29605 (9)	0.0701 (6)
H13A	0.7540	0.0341	0.2750	0.105*
H13B	0.6523	−0.0956	0.2875	0.105*
H13C	0.7147	0.0232	0.3310	0.105*
O1	0.25322 (10)	0.6437 (2)	0.39963 (5)	0.0668 (4)
O2	0.63797 (9)	0.2384 (2)	0.28815 (5)	0.0599 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0395 (9)	0.0379 (10)	0.0548 (9)	0.0016 (7)	0.0072 (7)	−0.0034 (7)
C2	0.0356 (8)	0.0382 (9)	0.0469 (8)	−0.0051 (7)	0.0062 (6)	−0.0072 (7)
C3	0.0325 (8)	0.0440 (11)	0.0555 (9)	0.0003 (7)	0.0092 (6)	−0.0074 (7)
C4	0.0429 (9)	0.0391 (10)	0.0536 (9)	−0.0030 (7)	0.0087 (7)	−0.0040 (7)
C5	0.0423 (9)	0.0445 (10)	0.0554 (10)	−0.0011 (8)	0.0111 (7)	0.0046 (7)
C6	0.0430 (9)	0.0411 (11)	0.0530 (9)	−0.0026 (8)	0.0045 (7)	0.0016 (7)
C7	0.0401 (9)	0.0412 (10)	0.0466 (8)	−0.0050 (7)	0.0042 (7)	0.0045 (7)
C8	0.0489 (10)	0.0519 (12)	0.0577 (10)	0.0018 (9)	0.0159 (8)	0.0185 (8)
C9	0.0495 (10)	0.0481 (11)	0.0683 (11)	0.0072 (9)	0.0158 (8)	0.0162 (9)
C10	0.0422 (9)	0.0504 (11)	0.0479 (9)	−0.0065 (8)	0.0089 (7)	0.0033 (7)
C11	0.0624 (11)	0.0511 (12)	0.0685 (12)	−0.0036 (10)	0.0225 (9)	0.0187 (9)
C12	0.0567 (11)	0.0424 (11)	0.0765 (12)	0.0019 (9)	0.0177 (9)	0.0155 (9)
C13	0.0554 (11)	0.0684 (15)	0.0866 (14)	0.0066 (10)	0.0264 (10)	0.0066 (11)
O1	0.0639 (9)	0.0440 (9)	0.0927 (10)	0.0028 (7)	0.0279 (7)	0.0038 (7)
O2	0.0519 (8)	0.0609 (9)	0.0671 (8)	0.0011 (6)	0.0244 (6)	0.0091 (6)

Geometric parameters (Å, °)

C1—C3^{i}	1.381 (2)	C7—C12	1.401 (2)
C1—C2	1.400 (2)	C8—C9	1.380 (2)
C1—H1	0.9300	C8—H8	0.9300
C2—C3	1.396 (2)	C9—C10	1.386 (2)
C2—C4	1.469 (2)	C9—H9	0.9300
C3—C1^{i}	1.381 (2)	C10—O2	1.360 (2)
C3—H3	0.9300	C10—C11	1.385 (3)
C4—C5	1.326 (2)	C11—C12	1.372 (2)
C4—H4	0.9300	C11—H11	0.9300
C5—C6	1.489 (2)	C12—H12	0.9300
C5—H5	0.9300	C13—O2	1.423 (2)
C6—O1	1.226 (2)	C13—H13A	0.9600
C6—C7	1.481 (2)	C13—H13B	0.9600
C7—C8	1.384 (3)	C13—H13C	0.9600
C3^{i}—C1—C2	121.45 (16)	C9—C8—H8	119.0
C3^{i}—C1—H1	119.3	C7—C8—H8	119.0

C2—C1—H1	119.3	C8—C9—C10	119.80 (17)
C3—C2—C1	117.87 (15)	C8—C9—H9	120.1
C3—C2—C4	122.88 (14)	C10—C9—H9	120.1
C1—C2—C4	119.24 (16)	O2—C10—C11	116.06 (14)
C1^{i}—C3—C2	120.67 (15)	O2—C10—C9	124.88 (17)
C1^{i}—C3—H3	119.7	C11—C10—C9	119.06 (16)
C2—C3—H3	119.7	C12—C11—C10	120.72 (16)
C5—C4—C2	127.35 (17)	C12—C11—H11	119.6
C5—C4—H4	116.3	C10—C11—H11	119.6
C2—C4—H4	116.3	C11—C12—C7	121.12 (18)
C4—C5—C6	121.50 (17)	C11—C12—H12	119.4
C4—C5—H5	119.2	C7—C12—H12	119.4
C6—C5—H5	119.2	O2—C13—H13A	109.5
O1—C6—C7	120.75 (15)	O2—C13—H13B	109.5
O1—C6—C5	120.37 (15)	H13A—C13—H13B	109.5
C7—C6—C5	118.88 (15)	O2—C13—H13C	109.5
C8—C7—C12	117.24 (15)	H13A—C13—H13C	109.5
C8—C7—C6	123.88 (14)	H13B—C13—H13C	109.5
C12—C7—C6	118.87 (16)	C10—O2—C13	118.14 (13)
C9—C8—C7	122.03 (15)		
C3^{i}—C1—C2—C3	0.8 (2)	C12—C7—C8—C9	1.3 (3)
C3^{i}—C1—C2—C4	−178.57 (15)	C6—C7—C8—C9	−178.01 (17)
C1—C2—C3—C1^{i}	−0.8 (2)	C7—C8—C9—C10	−1.4 (3)
C4—C2—C3—C1^{i}	178.55 (15)	C8—C9—C10—O2	−179.07 (16)
C3—C2—C4—C5	2.9 (3)	C8—C9—C10—C11	0.0 (3)
C1—C2—C4—C5	−177.79 (16)	O2—C10—C11—C12	−179.50 (17)
C2—C4—C5—C6	−178.47 (15)	C9—C10—C11—C12	1.3 (3)
C4—C5—C6—O1	13.9 (3)	C10—C11—C12—C7	−1.4 (3)
C4—C5—C6—C7	−165.72 (15)	C8—C7—C12—C11	0.1 (3)
O1—C6—C7—C8	164.35 (18)	C6—C7—C12—C11	179.43 (17)
C5—C6—C7—C8	−16.0 (2)	C11—C10—O2—C13	178.32 (18)
O1—C6—C7—C12	−15.0 (3)	C9—C10—O2—C13	−2.5 (3)
C5—C6—C7—C12	164.68 (16)		

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

Fig. 1

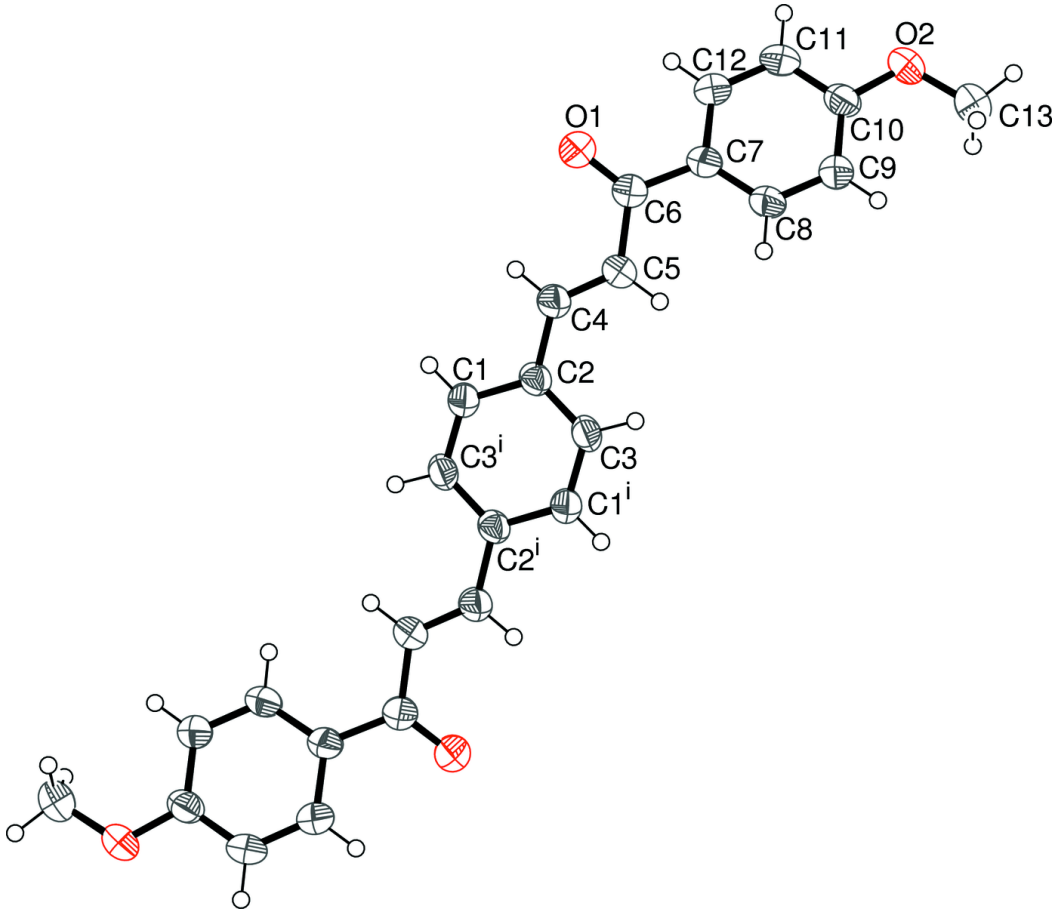

Fig. 2

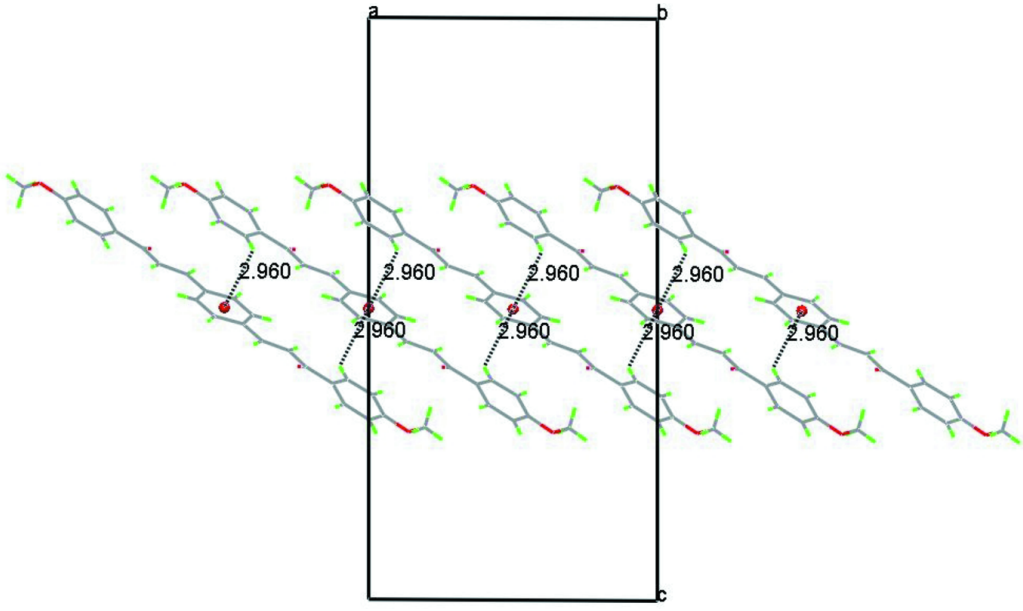

Fig. 3