Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(3,4-Dimethoxyphenyl)-3-(3-methoxyphenyl)prop-2-en-1-one

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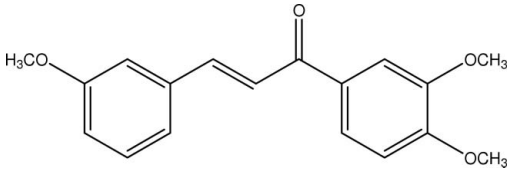
Received 4 June 2007; accepted 5 June 2007

Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.002 Å; R factor = 0.060; wR factor = 0.211; data-to-parameter ratio = 22.4.

The title compound, $C_{18}H_{18}O_4$, was obtained as by-product in a search for nonlinear optical chalcones. In the molecule, the two benzene rings make a dihedral angle of 37.20 (8)°. The crystal packing exhibits weak intermolecular C—H···O interactions.

Related literature

For related crystal structures, see: Patil, Teh, Fun, Babu *et al.* (2007); Patil, Teh, Fun, Razak & Dharmaprakash (2007). For general background, see: Uchida *et al.* (1998); Watson *et al.* (1993). For our previous work on related compounds, see: Patil *et al.* (2006); Patil, Dharmaprakash *et al.* (2007). For related literature, see: Allen *et al.* (1987); Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{18}H_{18}O_4$
M_r = 298.32
Triclinic, $P\overline{1}$
a = 7.4583 (3) Å
b = 10.7134 (4) Å
c = 10.9600 (4) Å
α = 107.062 (2)°
β = 107.744 (2)°
γ = 98.220 (2)°
V = 770.96 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 297 (2) K
0.50 × 0.40 × 0.28 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.891, T_{max} = 0.975
18727 measured reflections
4528 independent reflections
2888 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.211
S = 1.04
4528 reflections
202 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···O2^{i}	0.93	2.57	3.468 (2)	162
C16—H16C···O2^{ii}	0.96	2.50	3.363 (3)	150

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

The authors thank the Malaysian Government and Universiti Sains Malaysia for Fundamental Research Grant Scheme (FRGS) grant No. 203/PFIZIK/671064). PSP thanks the DRDO, Government of India, for a Junior Research Fellowship (JRF).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2259).

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 doi:10.1107/S1600536807027560

supplementary materials

Acta Cryst. (2007). E**63**, o3239 [**doi:10.1107/S1600536807027560**]

1-(3,4-Dimethoxyphenyl)-3-(3-methoxyphenyl)prop-2-en-1-one

M. M. Rosli, P. S. Patil, H.-K. Fun, I. A. Razak, S. M. Dharmaprakash and Y. E. Satheesh

Comment

During our search for non-linear optical chalcones (Patil *et al.*, 2006; Patil, Dharmaprakash *et al.*, 2007; Patil, Teh, Fun, Babu *et al.*, 2007; Patil, Teh, Fun, Razak *et al.* 2007), the title compound, (I) (Fig. 1), was synthesized. We present here a study of the molecular packing in (I), which crystallizes in a centrosymmetric crystal structure and hence don't exhibit second-order non-linear optical properties.

All bond lengths and angles in (I) have normal values (Allen *et al.*, 1987) and are comparable to those in related structures (Patil, Teh, Fun, Babu *et al.*, 2007;; Patil, Teh, Fun, Razak *et al.* 2007). The dihedral angle between the benzene rings is 37.20 (8)°. The least-square plane through the enone group makes dihedral angles of 18.8 (1) and 18.5 (1)° with the C1—C6 and C10—C15 benzene rings, respectively. The three methoxy groups attached at the atoms C4, C12 and C13 are almost coplanar with the attached benzene ring, with C16—O1—C4—C5, C17—O3—C12—C13 and C18—O4—C13—C12 torsion angles of 177.11 (14), −173.07 (14) and 174.17 (14)° respectively.

Chains of the molecules of the title compound are stabilized *via* two C—H···O intermolecular interactions which also form cyclic centrosymmetric $R^2_4(14)$ motifs (Bernstein *et al.*, 1995)

Experimental

Compound (I) was prepared by the condensation of 3-Methoxybenzaldehyde (0.01 mol) and 3,4-dimethoxyacetophenone (0.01 mol) in ethanol (60 ml) at room temperature. 10% NaOH aqueous solution (10 g) was added and the mixture was stirred for 4 h. The reaction mixture was poured on to ice cold water and kept aside for 12 h. The precipitate which formed was filtered off and dried. The resulting crude product was recrystallized twice from acetone. Crystals suitable for single-crystal X-ray diffraction experiments were grown by slow evaporation of an acetone solution at room temperature.

Refinement

All H atoms were geometrically positioned (C—H 0.93–0.96 Å) and refined using a riding model, with U_{iso}(H) = 1.2–1.5U_{eq}(C).

Figures

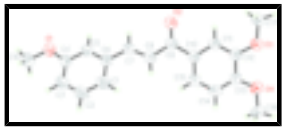

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

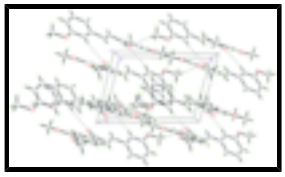

Fig. 2. The crystal packing of (I), viewed down the *c* axis. Hydrogen bonds are shown as dashed lines.

1-(3,4-dimethoxyphenyl)-3-(3-methoxyphenyl)prop-2-en-1-one

Crystal data

$C_{18}H_{18}O_4$	$Z = 2$
$M_r = 298.32$	$F_{000} = 316$
Triclinic, $P\overline{1}$	$D_x = 1.285$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.4583$ (3) Å	Cell parameters from 7560 reflections
$b = 10.7134$ (4) Å	$\theta = 2.1$–30.1°
$c = 10.9600$ (4) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 107.062$ (2)°	$T = 297$ (2) K
$\beta = 107.744$ (2)°	Block, yellow
$\gamma = 98.220$ (2)°	0.50 × 0.40 × 0.28 mm
$V = 770.96$ (5) Å^3	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	4528 independent reflections
Radiation source: fine-focus sealed tube	2888 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.033$
Detector resolution: 8.33 pixels mm^{-1}	$\theta_{max} = 30.1^\circ$
$T = 297(2)$ K	$\theta_{min} = 2.1^\circ$
ω scans	$h = -10 \rightarrow 9$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$k = -15 \rightarrow 13$
$T_{min} = 0.891$, $T_{max} = 0.975$	$l = -15 \rightarrow 15$
18727 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.060$	H-atom parameters constrained
$wR(F^2) = 0.211$	$w = 1/[\sigma^2(F_o^2) + (0.1274P)^2 + 0.0391P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
4528 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}

202 parameters	$\Delta\rho_{min} = -0.21$ 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	0.22866 (19)	0.52126 (11)	0.48587 (12)	0.0732 (4)
O2	0.1287 (2)	−0.09232 (11)	0.65154 (11)	0.0763 (4)
O3	0.09181 (17)	−0.34403 (10)	0.97666 (11)	0.0610 (3)
O4	0.19517 (19)	−0.16003 (11)	1.21401 (11)	0.0680 (4)
C1	0.4955 (2)	0.40910 (16)	0.81197 (17)	0.0623 (4)
H1A	0.5551	0.3822	0.8843	0.075*
C2	0.5629 (3)	0.53736 (17)	0.81696 (19)	0.0706 (5)
H2A	0.6682	0.5967	0.8933	0.085*
C3	0.4782 (2)	0.57993 (15)	0.71157 (18)	0.0632 (4)
H3A	0.5251	0.6674	0.7169	0.076*
C4	0.3224 (2)	0.49149 (14)	0.59727 (16)	0.0533 (4)
C5	0.2528 (2)	0.36155 (14)	0.59124 (15)	0.0518 (4)
H5A	0.1481	0.3023	0.5145	0.062*
C6	0.3372 (2)	0.31899 (14)	0.69796 (15)	0.0495 (3)
C7	0.2570 (2)	0.18182 (14)	0.68832 (15)	0.0521 (4)
H7A	0.1643	0.1241	0.6042	0.063*
C8	0.3036 (2)	0.13212 (15)	0.78734 (16)	0.0561 (4)
H8A	0.3972	0.1867	0.8723	0.067*
C9	0.2112 (2)	−0.00775 (14)	0.76705 (15)	0.0541 (4)
C10	0.2170 (2)	−0.04300 (14)	0.88918 (15)	0.0498 (3)
C11	0.1546 (2)	−0.17913 (14)	0.87190 (14)	0.0489 (3)
H11A	0.1161	−0.2454	0.7855	0.059*
C12	0.1498 (2)	−0.21561 (13)	0.98145 (14)	0.0481 (3)
C13	0.2070 (2)	−0.11488 (15)	1.11254 (15)	0.0520 (4)
C14	0.2677 (3)	0.01868 (16)	1.12890 (16)	0.0618 (4)
H14A	0.3051	0.0854	1.2149	0.074*
C15	0.2735 (2)	0.05406 (15)	1.01826 (16)	0.0595 (4)
H15A	0.3159	0.1445	1.0309	0.071*
C16	0.2872 (3)	0.65426 (17)	0.4888 (2)	0.0767 (5)
H16A	0.2114	0.6609	0.4038	0.115*

H16B	0.4226	0.6749	0.5013	0.115*
H16C	0.2672	0.7171	0.5632	0.115*
C17	0.0536 (3)	−0.45077 (15)	0.85133 (17)	0.0675 (5)
H17A	0.0160	−0.5359	0.8609	0.101*
H17B	0.1689	−0.4466	0.8292	0.101*
H17C	−0.0498	−0.4419	0.7793	0.101*
C18	0.2687 (3)	−0.0633 (2)	1.34896 (16)	0.0791 (6)
H18A	0.2534	−0.1068	1.4116	0.119*
H18B	0.1984	0.0053	1.3528	0.119*
H18C	0.4043	−0.0227	1.3740	0.119*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1054 (10)	0.0505 (6)	0.0593 (7)	0.0082 (6)	0.0249 (6)	0.0244 (5)
O2	0.1247 (11)	0.0433 (6)	0.0503 (6)	0.0126 (6)	0.0249 (7)	0.0136 (5)
O3	0.0870 (8)	0.0438 (5)	0.0490 (6)	0.0099 (5)	0.0237 (5)	0.0163 (5)
O4	0.0932 (9)	0.0601 (7)	0.0456 (6)	0.0103 (6)	0.0241 (6)	0.0175 (5)
C1	0.0617 (9)	0.0548 (9)	0.0584 (9)	0.0077 (7)	0.0108 (7)	0.0184 (7)
C2	0.0626 (10)	0.0527 (9)	0.0699 (11)	−0.0042 (7)	0.0074 (8)	0.0112 (8)
C3	0.0703 (10)	0.0426 (8)	0.0694 (10)	0.0012 (7)	0.0272 (8)	0.0142 (7)
C4	0.0661 (9)	0.0441 (7)	0.0526 (8)	0.0107 (6)	0.0282 (7)	0.0161 (6)
C5	0.0592 (8)	0.0423 (7)	0.0479 (7)	0.0053 (6)	0.0203 (6)	0.0108 (6)
C6	0.0558 (8)	0.0413 (7)	0.0493 (7)	0.0099 (6)	0.0212 (6)	0.0126 (6)
C7	0.0623 (8)	0.0410 (7)	0.0484 (8)	0.0101 (6)	0.0193 (6)	0.0120 (6)
C8	0.0663 (9)	0.0463 (7)	0.0510 (8)	0.0109 (6)	0.0183 (7)	0.0158 (6)
C9	0.0709 (9)	0.0419 (7)	0.0483 (8)	0.0164 (6)	0.0205 (7)	0.0147 (6)
C10	0.0576 (8)	0.0433 (7)	0.0472 (7)	0.0149 (6)	0.0173 (6)	0.0156 (6)
C11	0.0591 (8)	0.0413 (7)	0.0425 (7)	0.0135 (6)	0.0161 (6)	0.0119 (5)
C12	0.0529 (7)	0.0429 (7)	0.0463 (7)	0.0127 (5)	0.0158 (6)	0.0153 (6)
C13	0.0571 (8)	0.0533 (8)	0.0436 (7)	0.0141 (6)	0.0163 (6)	0.0170 (6)
C14	0.0808 (11)	0.0481 (8)	0.0435 (7)	0.0099 (7)	0.0177 (7)	0.0065 (6)
C15	0.0761 (10)	0.0410 (7)	0.0521 (8)	0.0089 (6)	0.0190 (7)	0.0110 (6)
C16	0.1139 (15)	0.0487 (9)	0.0759 (12)	0.0227 (9)	0.0402 (11)	0.0273 (8)
C17	0.0930 (12)	0.0451 (8)	0.0586 (9)	0.0092 (8)	0.0286 (9)	0.0137 (7)
C18	0.1081 (15)	0.0776 (12)	0.0417 (8)	0.0153 (10)	0.0212 (9)	0.0179 (8)

Geometric parameters (Å, °)

O1—C4	1.3613 (19)	C8—H8A	0.9300
O1—C16	1.4174 (19)	C9—C10	1.484 (2)
O2—C9	1.2227 (18)	C10—C15	1.381 (2)
O3—C12	1.3624 (16)	C10—C11	1.4034 (19)
O3—C17	1.4221 (19)	C11—C12	1.3760 (19)
O4—C13	1.3571 (18)	C11—H11A	0.9300
O4—C18	1.416 (2)	C12—C13	1.413 (2)
C1—C2	1.373 (2)	C13—C14	1.378 (2)
C1—C6	1.398 (2)	C14—C15	1.383 (2)
C1—H1A	0.9300	C14—H14A	0.9300

C2—C3	1.374 (2)	C15—H15A	0.9300
C2—H2A	0.9300	C16—H16A	0.9600
C3—C4	1.385 (2)	C16—H16B	0.9600
C3—H3A	0.9300	C16—H16C	0.9600
C4—C5	1.3893 (19)	C17—H17A	0.9600
C5—C6	1.385 (2)	C17—H17B	0.9600
C5—H5A	0.9300	C17—H17C	0.9600
C6—C7	1.465 (2)	C18—H18A	0.9600
C7—C8	1.321 (2)	C18—H18B	0.9600
C7—H7A	0.9300	C18—H18C	0.9600
C8—C9	1.478 (2)		
C4—O1—C16	118.13 (14)	C12—C11—C10	120.71 (13)
C12—O3—C17	117.15 (11)	C12—C11—H11A	119.6
C13—O4—C18	116.96 (13)	C10—C11—H11A	119.6
C2—C1—C6	119.89 (15)	O3—C12—C11	125.75 (12)
C2—C1—H1A	120.1	O3—C12—C13	114.61 (12)
C6—C1—H1A	120.1	C11—C12—C13	119.64 (12)
C1—C2—C3	121.40 (15)	O4—C13—C14	125.03 (14)
C1—C2—H2A	119.3	O4—C13—C12	115.55 (13)
C3—C2—H2A	119.3	C14—C13—C12	119.42 (13)
C2—C3—C4	119.44 (14)	C13—C14—C15	120.43 (14)
C2—C3—H3A	120.3	C13—C14—H14A	119.8
C4—C3—H3A	120.3	C15—C14—H14A	119.8
O1—C4—C3	124.81 (13)	C10—C15—C14	120.96 (14)
O1—C4—C5	115.55 (14)	C10—C15—H15A	119.5
C3—C4—C5	119.64 (14)	C14—C15—H15A	119.5
C6—C5—C4	120.92 (14)	O1—C16—H16A	109.5
C6—C5—H5A	119.5	O1—C16—H16B	109.5
C4—C5—H5A	119.5	H16A—C16—H16B	109.5
C5—C6—C1	118.70 (13)	O1—C16—H16C	109.5
C5—C6—C7	119.09 (13)	H16A—C16—H16C	109.5
C1—C6—C7	122.20 (14)	H16B—C16—H16C	109.5
C8—C7—C6	126.38 (14)	O3—C17—H17A	109.5
C8—C7—H7A	116.8	O3—C17—H17B	109.5
C6—C7—H7A	116.8	H17A—C17—H17B	109.5
C7—C8—C9	121.95 (14)	O3—C17—H17C	109.5
C7—C8—H8A	119.0	H17A—C17—H17C	109.5
C9—C8—H8A	119.0	H17B—C17—H17C	109.5
O2—C9—C8	120.54 (14)	O4—C18—H18A	109.5
O2—C9—C10	120.60 (13)	O4—C18—H18B	109.5
C8—C9—C10	118.84 (13)	H18A—C18—H18B	109.5
C15—C10—C11	118.84 (13)	O4—C18—H18C	109.5
C15—C10—C9	122.24 (13)	H18A—C18—H18C	109.5
C11—C10—C9	118.86 (13)	H18B—C18—H18C	109.5
C6—C1—C2—C3	−0.1 (3)	O2—C9—C10—C11	10.2 (2)
C1—C2—C3—C4	−0.5 (3)	C8—C9—C10—C11	−170.99 (14)
C16—O1—C4—C3	−3.3 (3)	C15—C10—C11—C12	−0.1 (2)
C16—O1—C4—C5	177.11 (14)	C9—C10—C11—C12	−177.19 (12)

C2—C3—C4—O1	−178.98 (16)	C17—O3—C12—C11	7.4 (2)
C2—C3—C4—C5	0.6 (2)	C17—O3—C12—C13	−173.07 (14)
O1—C4—C5—C6	179.54 (13)	C10—C11—C12—O3	179.89 (13)
C3—C4—C5—C6	−0.1 (2)	C10—C11—C12—C13	0.4 (2)
C4—C5—C6—C1	−0.5 (2)	C18—O4—C13—C14	−6.4 (3)
C4—C5—C6—C7	178.97 (13)	C18—O4—C13—C12	174.17 (14)
C2—C1—C6—C5	0.6 (2)	O3—C12—C13—O4	−0.3 (2)
C2—C1—C6—C7	−178.86 (16)	C11—C12—C13—O4	179.21 (13)
C5—C6—C7—C8	−170.61 (14)	O3—C12—C13—C14	−179.81 (14)
C1—C6—C7—C8	8.9 (3)	C11—C12—C13—C14	−0.3 (2)
C6—C7—C8—C9	179.07 (14)	O4—C13—C14—C15	−179.67 (15)
C7—C8—C9—O2	19.4 (3)	C12—C13—C14—C15	−0.2 (3)
C7—C8—C9—C10	−159.47 (14)	C11—C10—C15—C14	−0.5 (3)
O2—C9—C10—C15	−166.86 (15)	C9—C10—C15—C14	176.57 (14)
C8—C9—C10—C15	12.0 (2)	C13—C14—C15—C10	0.6 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5A···O2^{i}	0.93	2.57	3.468 (2)	162
C16—H16C···O2^{ii}	0.96	2.50	3.363 (3)	150

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

Fig. 1

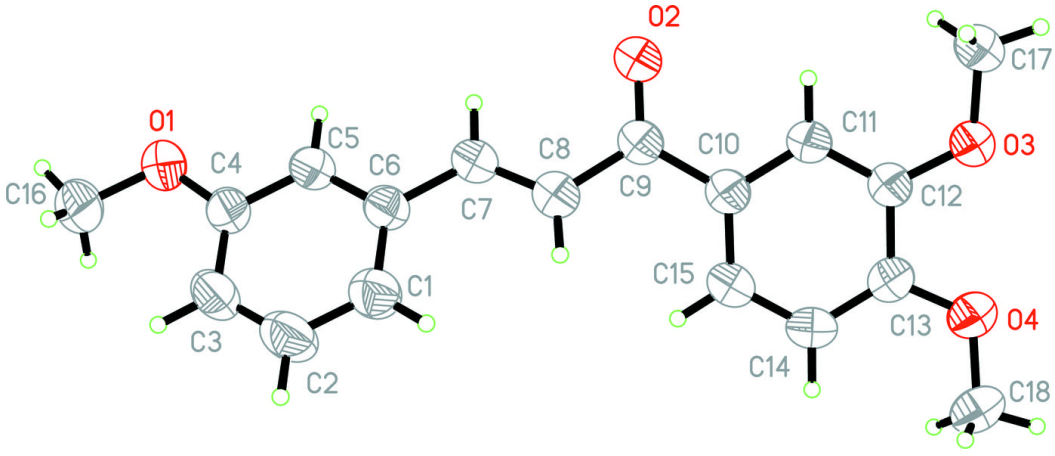
O2
C17
O1
C5
C11
O3
C4
C6
C8
C10
C16
C7
C9
C12
C15
C1
C13
O4
C3
C14
C2
C18

Fig. 2