Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*E*,6*E*)-2,6-Bis(4-ethoxybenzylidene)-cyclohexanone

Xiaopeng Shi,* Shuqin Li and Zhenzhen Liu

Department of Chemistry and Biology, Xiangfan University, Xiangfan 441053, People's Republic of China
Correspondence e-mail: chemch@163.com

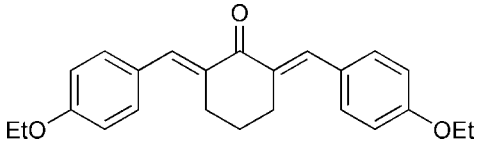
Received 6 September 2008; accepted 20 October 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.048; wR factor = 0.115; data-to-parameter ratio = 8.0.

The title compound, $C_{24}H_{26}O_3$, was prepared by the condensation reaction of 4-ethoxybenzaldehyde with cyclohexanone. The molecule has crystallographic mirror symmetry and exhibits a butterfly-shaped geometry, with a dihedral angle of 5.46 (1)° between the two benzene rings. Weak intermolecular C—H···π interactions help stabilize the crystal structure.

Related literature

For related structures, see: Du *et al.* (2007); Liang *et al.* (2007); Sun *et al.* (2007); Zhou *et al.* (2007). For background information, see: Guilford *et al.* (1999); Ompraba *et al.* (2003); Yu *et al.* (2000).



Experimental

Crystal data

$C_{24}H_{26}O_3$
M_r = 362.45
Orthorhombic, $Cmc2_1$
a = 24.2516 (6) Å
b = 10.8459 (3) Å
c = 7.5270 (2) Å
V = 1979.83 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.974, T_{max} = 0.992
6002 measured reflections
1026 independent reflections
879 reflections with $I > 2\sigma(I)$
R_{int} = 0.121

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.115
S = 1.05
1026 reflections
129 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···$Cg1^{i}$	0.93	2.92	3.601 (2)	132

Symmetry code: (i) $x, -y, z+\frac{1}{2}$. $Cg1$ is the centroid of atoms C6–C11.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors are grateful to Xiangfan University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2205).

References

Bruker (1999). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Du, Z.-Y., Huang, B.-H., Zhang, K. & Fang, Y.-X. (2007). *Acta Cryst.* E**63**, o3216.
Guilford, W. J., Shaw, K. J., Dallas, J. L., Koovakkat, S., Lee, W., Liang, A., Light, D. R., McCarrick, M. A., Whitlow, M., Ye, B. & Morrissey, M. M. (1999). *J. Med. Chem.* **42**, 5415–5425.
Liang, G., Yang, S.-L., Wang, X.-H., Li, Y.-R. & Li, X.-K. (2007). *Acta Cryst.* E**63**, o4118.
Ompraba, G., Rafi, Z. A., Yougave, M., Velmurugan, D., Sekar, K., Karthikeyan, E., Perumal, S., Choudhury, A. R. & Row, T. N. G. (2003). *Cryst. Res. Technol.* **38**, 822–828.
Sheldrick, G. M. (1997). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sun, Y.-F. & Cui, Y.-P. (2007). *Acta Cryst.* E**63**, o1932–o1933.
Yu, R. C., Yakimansky, A. V., Kothe, H., Voigt-Martin, I. G., Schollmeyer, D., Jansen, J., Zandbergen, H. & Tenkovtsev, A. V. (2000). *Acta Cryst.* A**56**, 436–450.
Zhou, L.-Y. (2007). *Acta Cryst.* E**63**, o3113.

supporting information

Acta Cryst. (2008). E**64**, o2199 [doi:10.1107/S1600536808034272]

(2*E*,6*E*)-2,6-Bis(4-ethoxybenzylidene)cyclohexanone

Xiaopeng Shi, Shuqin Li and Zhenzhen Liu

S1. Comment

The development of highly efficient nonlinear optical crystals is extremely important for laser spectroscopy and laser processing. Bis(arylmethylidene) cycloalkanones are reported to exhibit promising nonlinear optical properties (Yu *et al.*, 2000). In addiiton, these compounds are widely used as building blocks for the synthesis of biologically active heterocycles (Guilford *et al.*, 1999) The title compound $C_{24}H_{26}O_3$ (I) was prepared by the condensation reaction of 4-ethoxybenzaldehyde with cyclohexanone. The molecular structure of (I) is shown in Fig. 1. It has crystallographic mirror symmetry and exhibits a butterfly-shaped geometry. Similar structures have been observed in the related substituted cyclohexanone analogues reported by Ompraba *et al.* (2003) and Sun *et al.* (2007). A dihedral angle of 5.46 (1)° is found between the mean planes of the two benzene rings. Molecules are mainly connected by intermolecular weak C—H···π interactions (Table 1).

S2. Experimental

The title compound was synthesized as previously described (Sun *et al.*, 2007). 4-Ethoxybenzaldehyde (15.0 g, 0.1 mol) and cyclohexanone (4.9 g, 0.05 mol) were dissolved in 80 ml of ethanol. To this solution, a 10% NaOH aqueous solution (20 ml) was added dropwise with stirring at room temperature. The reaction mixture was stirred for a further 8 h and then poured into a mixture of 100 ml water and diluted hydrochloric acid. The precipitate was filtered and washed thoroughly with water and finally with ethanol. The product was dried at room temperature and crystallized from ethanol to give the title compound as pale yellow solid (12.7 g, yield 70%). Crystals of (I) suitable for X-ray data collection were obtained by slow evaporation of a CH_2Cl_2 and MeOH solution in a ratio of 3:2 at 293 K.

S3. Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C) (1.5 U_{eq}(C) for methyl) of the parent atoms. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

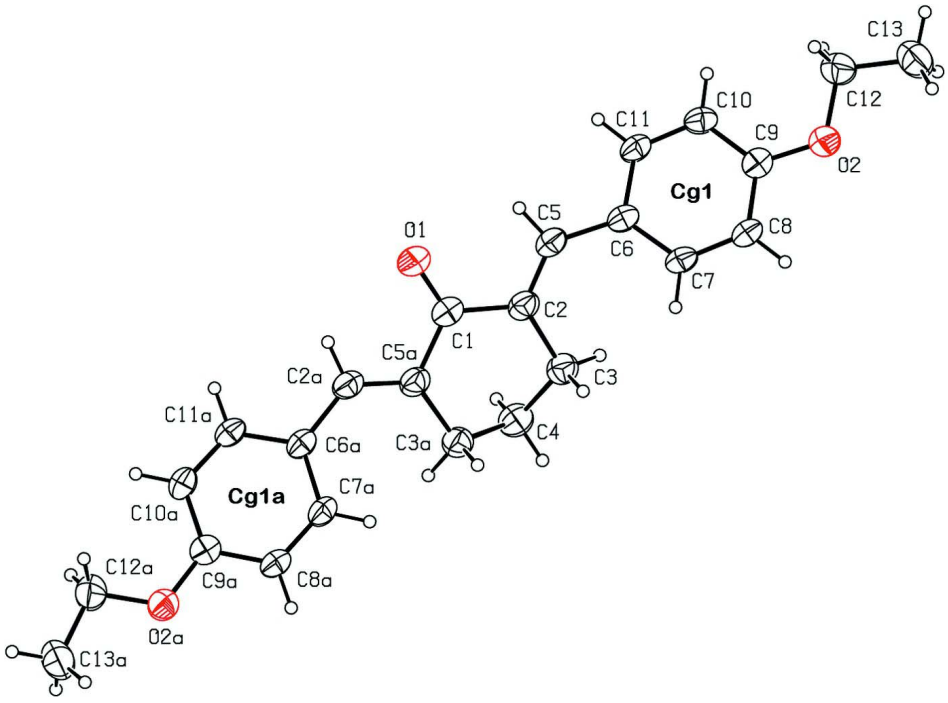


Figure 1

View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Symmetry code "a" represents the operation 1-x, y, z

(2*E*,6*E*)-2,6-Bis(4-ethoxybenzylidene)cyclohexanone

Crystal data

$C_{24}H_{26}O_3$
$M_r = 362.45$
Orthorhombic, $Cmc2_1$
Hall symbol: C2c-2
$a = 24.2516\ (6)$ Å
$b = 10.8459\ (3)$ Å
$c = 7.5270\ (2)$ Å
$V = 1979.83\ (9)$ Å^3
$Z = 4$

$F(000) = 776$
$D_x = 1.216$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1880 reflections
$\theta = 2.7–21.4°$
$\mu = 0.08$ mm^{-1}
$T = 298$ K
Block, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min} = 0.974$, $T_{max} = 0.992$

6002 measured reflections
1026 independent reflections
879 reflections with $I > 2\sigma(I)$
$R_{int} = 0.121$
$\theta_{max} = 25.5°$, $\theta_{min} = 1.7°$
$h = -24\rightarrow29$
$k = -12\rightarrow13$
$l = -9\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.115$
$S = 1.05$
1026 reflections
129 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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. The authors have merged Friedel pairs before the final refinement. In the absence of anomalous scatterers, no attempt was made to establish the absolute configuration of the title compound.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5000	0.2069 (4)	0.1013 (7)	0.0752 (13)
C2	0.44694 (11)	0.1650 (2)	0.1831 (4)	0.0599 (7)
C3	0.44890 (11)	0.0930 (3)	0.3532 (4)	0.0641 (8)
H3A	0.4486	0.0056	0.3262	0.077*
H3B	0.4163	0.1114	0.4231	0.077*
C4	0.5000	0.1232 (4)	0.4615 (5)	0.0713 (11)
H4A	0.5000	0.2100	0.4926	0.086*
H4B	0.5000	0.0755	0.5705	0.086*
C5	0.40084 (12)	0.1962 (2)	0.0943 (4)	0.0604 (8)
H5	0.4067	0.2472	−0.0033	0.072*
C6	0.34301 (12)	0.1637 (2)	0.1252 (3)	0.0534 (7)
C7	0.32483 (12)	0.0581 (2)	0.2149 (4)	0.0568 (8)
H7	0.3507	0.0059	0.2663	0.068*
C8	0.26987 (12)	0.0298 (2)	0.2288 (3)	0.0561 (7)
H8	0.2590	−0.0412	0.2885	0.067*
C9	0.23052 (12)	0.1066 (2)	0.1543 (4)	0.0535 (7)
C10	0.24723 (12)	0.2127 (2)	0.0674 (4)	0.0585 (7)
H10	0.2212	0.2658	0.0188	0.070*
C11	0.30222 (11)	0.2389 (2)	0.0536 (4)	0.0572 (7)
H11	0.3128	0.3101	−0.0062	0.069*
C12	0.13522 (12)	0.1324 (3)	0.0784 (6)	0.0798 (10)
H12A	0.1433	0.1310	−0.0477	0.096*
H12B	0.1331	0.2177	0.1168	0.096*

C13	0.08165 (14)	0.0681 (4)	0.1142 (7)	0.1005 (14)
H13A	0.0865	−0.0193	0.1008	0.151*
H13B	0.0543	0.0965	0.0316	0.151*
H13C	0.0698	0.0858	0.2332	0.151*
O1	0.5000	0.2707 (5)	−0.0304 (6)	0.1303 (19)
O2	0.17725 (8)	0.06933 (16)	0.1745 (3)	0.0673 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.074 (3)	0.070 (2)	0.082 (3)	0.000	0.000	0.028 (2)
C2	0.0684 (18)	0.0474 (13)	0.0640 (18)	0.0046 (12)	0.0043 (15)	0.0077 (13)
C3	0.0661 (18)	0.0647 (19)	0.0614 (17)	0.0063 (13)	0.0094 (15)	0.0044 (15)
C4	0.085 (3)	0.077 (3)	0.052 (2)	0.000	0.000	0.000 (2)
C5	0.073 (2)	0.0483 (14)	0.0598 (17)	0.0014 (11)	0.0048 (14)	0.0111 (12)
C6	0.0702 (17)	0.0420 (13)	0.0481 (14)	0.0040 (11)	−0.0010 (13)	0.0023 (11)
C7	0.071 (2)	0.0422 (13)	0.0567 (17)	0.0091 (12)	0.0011 (14)	0.0086 (12)
C8	0.0723 (18)	0.0427 (14)	0.0532 (15)	0.0006 (12)	0.0008 (14)	0.0082 (12)
C9	0.0663 (17)	0.0460 (14)	0.0482 (14)	−0.0013 (11)	0.0004 (13)	−0.0063 (12)
C10	0.0701 (19)	0.0449 (14)	0.0605 (16)	0.0083 (12)	−0.0071 (14)	0.0016 (13)
C11	0.0731 (17)	0.0403 (14)	0.0583 (15)	−0.0021 (11)	−0.0034 (15)	0.0083 (11)
C12	0.072 (2)	0.075 (2)	0.092 (3)	0.0082 (16)	−0.008 (2)	0.0095 (19)
C13	0.065 (2)	0.099 (2)	0.138 (4)	0.0051 (17)	−0.004 (2)	0.008 (3)
O1	0.076 (2)	0.170 (4)	0.145 (4)	0.000	0.000	0.110 (4)
O2	0.0661 (13)	0.0624 (10)	0.0733 (14)	−0.0012 (9)	−0.0051 (12)	0.0077 (11)

Geometric parameters (Å, º)

C1—O1	1.209 (6)	C7—H7	0.9300
C1—C2^{i}	1.497 (4)	C8—C9	1.385 (4)
C1—C2	1.497 (4)	C8—H8	0.9300
C2—C5	1.346 (4)	C9—O2	1.362 (3)
C2—C3	1.500 (4)	C9—C10	1.384 (4)
C3—C4	1.519 (4)	C10—C11	1.367 (4)
C3—H3A	0.9700	C10—H10	0.9300
C3—H3B	0.9700	C11—H11	0.9300
C4—C3^{i}	1.519 (4)	C12—O2	1.425 (4)
C4—H4A	0.9700	C12—C13	1.499 (5)
C4—H4B	0.9700	C12—H12A	0.9700
C5—C6	1.465 (4)	C12—H12B	0.9700
C5—H5	0.9300	C13—H13A	0.9600
C6—C11	1.391 (4)	C13—H13B	0.9600
C6—C7	1.400 (4)	C13—H13C	0.9600
C7—C8	1.372 (4)		
O1—C1—C2^{i}	120.73 (18)	C6—C7—H7	119.1
O1—C1—C2	120.73 (18)	C7—C8—C9	120.3 (2)
C2^{i}—C1—C2	118.5 (4)	C7—C8—H8	119.9

C5—C2—C1	115.7 (3)	C9—C8—H8	119.9
C5—C2—C3	125.5 (3)	O2—C9—C10	125.3 (2)
C1—C2—C3	118.8 (3)	O2—C9—C8	115.4 (2)
C2—C3—C4	111.8 (3)	C10—C9—C8	119.3 (3)
C2—C3—H3A	109.3	C11—C10—C9	119.6 (3)
C4—C3—H3A	109.3	C11—C10—H10	120.2
C2—C3—H3B	109.3	C9—C10—H10	120.2
C4—C3—H3B	109.3	C10—C11—C6	122.8 (3)
H3A—C3—H3B	107.9	C10—C11—H11	118.6
C3^{i}—C4—C3	109.3 (3)	C6—C11—H11	118.6
C3^{i}—C4—H4A	109.8	O2—C12—C13	107.8 (3)
C3—C4—H4A	109.8	O2—C12—H12A	110.2
C3^{i}—C4—H4B	109.8	C13—C12—H12A	110.2
C3—C4—H4B	109.8	O2—C12—H12B	110.2
H4A—C4—H4B	108.3	C13—C12—H12B	110.2
C2—C5—C6	131.0 (3)	H12A—C12—H12B	108.5
C2—C5—H5	114.5	C12—C13—H13A	109.5
C6—C5—H5	114.5	C12—C13—H13B	109.5
C11—C6—C7	116.3 (3)	H13A—C13—H13B	109.5
C11—C6—C5	118.6 (2)	C12—C13—H13C	109.5
C7—C6—C5	125.1 (3)	H13A—C13—H13C	109.5
C8—C7—C6	121.7 (2)	H13B—C13—H13C	109.5
C8—C7—H7	119.1	C9—O2—C12	118.6 (2)

O1—C1—C2—C5	4.1 (7)	C5—C6—C7—C8	−175.8 (3)
C2^{i}—C1—C2—C5	−174.1 (3)	C6—C7—C8—C9	−0.4 (4)
O1—C1—C2—C3	−176.0 (5)	C7—C8—C9—O2	179.5 (3)
C2^{i}—C1—C2—C3	5.7 (6)	C7—C8—C9—C10	−0.7 (4)
C5—C2—C3—C4	−153.2 (3)	O2—C9—C10—C11	−179.0 (3)
C1—C2—C3—C4	27.0 (4)	C8—C9—C10—C11	1.2 (4)
C2—C3—C4—C3^{i}	−59.6 (4)	C9—C10—C11—C6	−0.7 (5)
C1—C2—C5—C6	174.4 (3)	C7—C6—C11—C10	−0.4 (4)
C3—C2—C5—C6	−5.4 (5)	C5—C6—C11—C10	176.5 (3)
C2—C5—C6—C11	158.6 (3)	C10—C9—O2—C12	9.9 (4)
C2—C5—C6—C7	−24.8 (5)	C8—C9—O2—C12	−170.3 (3)
C11—C6—C7—C8	1.0 (4)	C13—C12—O2—C9	176.1 (3)

Symmetry code: (i) −*x*+1, *y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···*Cg*1^{ii}	0.93	2.92	3.601 (2)	132

Symmetry code: (ii) *x*, −*y*, *z*+1/2.