Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,5-Bis(3,4-dimethoxybenzylidene)cyclopentanone

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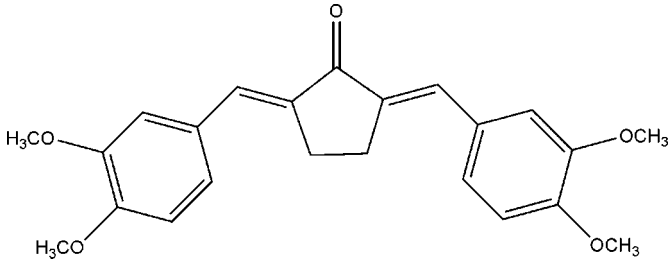
Received 18 May 2007; accepted 15 June 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.128; data-to-parameter ratio = 21.1.

The title compound, $C_{23}H_{24}O_5$, a bis(3,4-dimethoxyphenyl)-cyclopentanone derivative related to curcumin, shows noncrystallographic C_{2v} symmetry with two noncrystallographic mirror planes intersecting the cyclopentanone ring, resulting in a noncrystallographic twofold axis along the C=O double bond. The molecule is basically planar, the angles between the planes of the cyclopentanone ring and those of the two benzene rings being 2.5 (3) and 1.2 (7)°. The dihedral angles of both methoxy groups with the benzene rings are nearly identical [179.05 (11), 177.88 (11), −172.51 (12) and 176.11 (13)°]. The two 3,4-dimethoxyphenyl groups also lie within the plane of the molecule and the angle between them is 1.4 (1)°. The crystal packing is stabilized by intermolecular C—H···O hydrogen bonds.

Related literature

For the synthesis of the title compound, see: Furniss *et al.* (1989). For biological activity of the title compound and curcumin, see: Du *et al.* (2006) and Reksohadiprodjo *et al.* (2004), respectively. For related structures, see: Girija *et al.* (2004); Theocharis *et al.* (1981, 1982); Butcher *et al.* (2006).



Experimental

Crystal data

$C_{23}H_{24}O_5$
M_r = 380.42
Monoclinic, $P2_1/c$
a = 7.9492 (9) Å
b = 8.8428 (10) Å
c = 27.603 (3) Å
β = 92.421 (2)°
V = 1938.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 K
0.50 × 0.48 × 0.27 mm

Data collection

Bruker APEX II CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.861, T_{max} = 1.000
21225 measured reflections
5434 independent reflections
4383 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.128
S = 1.02
5434 reflections
257 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4B—H4BA···O1A^{i}	0.95	2.42	3.2556 (15)	146

Symmetry code: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS90* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 2000).

BKS thanks AICTE, Government of India, for financial assistance through a Career Award for Young Teachers Scheme and SB thanks the University of Mysore for research facilities. RJB acknowledges the Laboratory for the Structure of Matter at the Naval Research Laboratory for access to their diffractometers.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2029).

References

Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2006). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Butcher, R. J., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Vijaya Raj, K. K. (2006). *Acta Cryst.* E**62**, o1973–o1975.
Du, Z. Y., Bao, Y. D., Liu, Z., Qiao, W., Ma, L., Huang, Z. S., Gu, L. Q. & Chan, A. S. C. (2006). *Arch. Pharm.* **339**, 123–128.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Furniss, B. S., Hannaford, A. J., Smith, P. W. G. & Tatchell, A. R. (1989). Editors. *Vogel's Textbook of Practical Organic Chemistry*, 5th ed., p. 1033. London: Longman Group UK Ltd.
Girija, C. R., Begum, N. S., Syed, A. A. & Thiruvenkatam, V. (2004). *Acta Cryst.* C**60**, o611–o613.
Reksohadiprodjo, M. S., Timmerman, H., Sardjiman, M. S. A., Martono, S., Sugiyanto, H. L. R., Nurlaila, H. A. R., Puspitasari, I., Nurrochmad, A., Purwantiningsih, O. & Yuwono, T. (2004). US Patent 6 777 447 B2.

  doi:10.1107/S1600536807029339

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. University of Göttingen, Germany.
Theocharis, C. R., Jones, W., Motevalli, M. & Hursthouse, M. B. (1982). *J. Chem. Crystallogr.* **12**, 377–389.
Theocharis, C. R., Nakanishi, H. & Jones, W. (1981). *Acta Cryst.* B**37**, 756–758.

supplementary materials

Acta Cryst. (2007). E**63**, o3270-o3271 [**doi:10.1107/S1600536807029339**]

2,5-Bis(3,4-dimethoxybenzylidene)cyclopentanone

R. J. Butcher, J. P. Jasinski, B. Narayana, B. K. Sarojini, S. Bindya and H. S. Yathirajan

Comment

The title compound, (I), 2,5-bis(3,4-dimethoxyphenyl)cyclopentanone, (synthesis; Fig. 3) is a biologically active compound. Derivatives of dibenzylidene acetone, cyclopentanone and cyclohexanone exhibit potent anti-inflammatory, antibacterial and antioxidation activity. Closely realted to the title compound is curcumin, (1,7-bis(4-hydroxy-3-methoxy-phenyl)hepta-1,6-diene-3,5-dione), which is the main constituent of turmeric, the commonly used spice. It has been widely used as an anti-inflammatory, antibacterial, antioxidant, antihepatotoxic, hypocholesterolanemia, anti-cyclooxygenase, anti-cancer and radical scavenger agent. It is reported that curcumin is nontoxic in high doses and substitution on the aromatic rings with electron donating and withdrawing groups increases anti-inflammatory activity (Reksohadiprodjo *et al.*, 2004). Certain curcumin analogs are described to be potent aldose reductase inhibitors (Du *et al.*, 2006).

Hydrogen-bonding interactions in 1,7-bis(4-hydroxy-3-methoxyphenyl)heptane-3,5-dione have been reported (Girija *et al.*, 2004), and related crystal and molecular structural studies of 2-(*p*-methylbenzyl)cyclopentanone and 2-(*p*-chlorobenzyl)-5-(*p*-bromobenzylidene)cyclopentanone (Theocharis *et al.*, 1982) as well as one of the photodimerizable compound 5-benzylidene-2-(*p*-chlorobenzyl)cyclopentanone (Theocharis *et al.*, 1981) and 1,5-bis(4-chlorophenyl)penta-1,4-dien-3-one (Butcher *et al.*, 2006) have been reported. In view of the importance of these dibenzylidene derivatives, the present paper reports the crystal structure of the title compound, (I), $C_{23}H_{24}O_5$.

Experimental

The title compound was synthesized according to the method reported in the literature (Furniss *et al.*, 1989). A solution of 25 g of NaOH in 250 ml of water and 200 ml of ethanol was placed in a 500 ml bolt head flask provided with a mechanical stirrer. The flask was immersed in a water bath and the temperature of the solution was maintained at 393–398 K. The solution was stirred vigorously and added to one half of a previously prepared mixture of 3,4-dimethoxybenzaldehyde (41.5 g, 0.25 mol) and cyclopentanone (10.5 g, 0.125 mol). A flocculent precipitate is formed within 5–10 minutes. After 25 minutes, the remainder of the aldehyde-cyclopentanone mixture was added and the stirring was continued for additional 45 minutes. The crude product obtained was filtered and washed with cold water to eliminate the NaOH as completely as possible. The compound was purified by crystallization from ethanol (yield: 65%). Single crystal were grown in a acetone-toluene mixture (2:8) by slow evaporation (m.p.: 398–403 K). Analysis for $C_{23}H_{24}O_5$: Found (Calculated): C:72.55 (72.61); H: 6.29% (6.36%).

Refinement

The H atoms were included in the riding model approximation with C—H = 0.95–0.99 Å, and with U_{iso}(H) = 1.18–1.52U_{eq}(C).

Figures

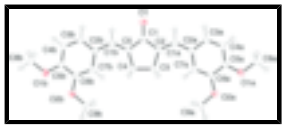


Fig. 1. Molecular structure of $C_{23}H_{24}O_5$, (I), showing the atom labeling and 50% probability displacement ellipsoids.

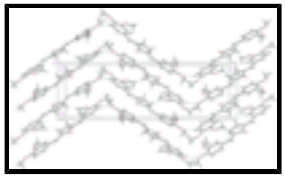


Fig. 2. Packing diagram of $C_{23}H_{24}O_5$ viewed down the *a* axis. Dashed lines indicate C—H···O hydrogen bonds.

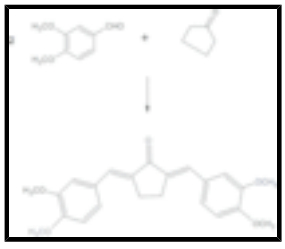


Fig. 3. Synthesis scheme of $C_{23}H_{24}O_5$.

2,5-Bis(3,4-dimethoxybenzylidene)cyclopentanone

Crystal data

$C_{23}H_{24}O_5$	F_{000} = 808
M_r = 380.42	D_x = 1.303 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.9492 (9) Å	Cell parameters from 6322 reflections
b = 8.8428 (10) Å	θ = 2.7–29.5º
c = 27.603 (3) Å	μ = 0.09 mm^{-1}
β = 92.421 (2)º	T = 173 K
V = 1938.6 (4) Å^3	Plate, colorless
Z = 4	0.50 × 0.48 × 0.27 mm

Data collection

Bruker APEX II CCD area-detector diffractometer	5434 independent reflections
Radiation source: fine-focus sealed tube	4383 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.025
T = 173 K	θ_{max} = 30.1º
φ and ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = −10→10
T_{min} = 0.861, T_{max} = 1.000	k = −9→12
21225 measured reflections	l = −37→37

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.128$	$w = 1/[\sigma^2(F_o^2) + (0.0653P)^2 + 0.4601P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
5434 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
257 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 $F^2 > 2$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	0.81476 (12)	0.25378 (12)	0.56791 (4)	0.0441 (3)
O1A	0.63787 (11)	−0.20756 (11)	0.30071 (3)	0.0363 (2)
O2A	0.38946 (10)	−0.05138 (11)	0.32971 (3)	0.0360 (2)
O1B	0.11569 (11)	0.80398 (12)	0.71096 (3)	0.0394 (2)
O2B	0.00659 (11)	0.69043 (12)	0.62888 (4)	0.0416 (2)
C1	0.67617 (15)	0.27066 (14)	0.54724 (4)	0.0292 (2)
C2	0.62053 (14)	0.20262 (13)	0.50019 (4)	0.0264 (2)
C3	0.44328 (14)	0.25268 (13)	0.48725 (4)	0.0263 (2)
H3A	0.3677	0.1640	0.4836	0.032*
H3B	0.4394	0.3096	0.4563	0.032*
C4	0.38814 (14)	0.35517 (14)	0.52927 (4)	0.0264 (2)
H4A	0.3577	0.4572	0.5170	0.032*
H4B	0.2894	0.3112	0.5448	0.032*
C5	0.53647 (14)	0.36409 (13)	0.56496 (4)	0.0264 (2)
C1A	0.72554 (14)	0.11349 (14)	0.47582 (4)	0.0289 (2)
H1AA	0.8338	0.1006	0.4912	0.035*
C2A	0.70094 (14)	0.03375 (13)	0.42997 (4)	0.0261 (2)
C3A	0.83432 (14)	−0.05166 (14)	0.41327 (4)	0.0300 (2)

H3AA	0.9381	−0.0537	0.4316	0.036*
C4A	0.81865 (14)	−0.13364 (15)	0.37044 (4)	0.0305 (3)
H4AA	0.9110	−0.1913	0.3599	0.037*
C5A	0.66858 (14)	−0.13152 (14)	0.34312 (4)	0.0282 (2)
C6A	0.53173 (13)	−0.04561 (14)	0.35913 (4)	0.0270 (2)
C7A	0.54844 (14)	0.03504 (13)	0.40180 (4)	0.0269 (2)
H7AA	0.4559	0.0924	0.4124	0.032*
C8A	0.77269 (18)	−0.29440 (18)	0.28243 (5)	0.0421 (3)
H8AA	0.7333	−0.3476	0.2529	0.063*
H8AB	0.8114	−0.3682	0.3069	0.063*
H8AC	0.8659	−0.2270	0.2749	0.063*
C9A	0.24621 (15)	0.02963 (17)	0.34540 (5)	0.0369 (3)
H9AA	0.1519	0.0158	0.3218	0.055*
H9AB	0.2736	0.1374	0.3481	0.055*
H9AC	0.2148	−0.0088	0.3771	0.055*
C1B	0.55581 (14)	0.43827 (14)	0.60739 (4)	0.0287 (2)
H1BA	0.6626	0.4251	0.6236	0.034*
C2B	0.44034 (14)	0.53533 (14)	0.63258 (4)	0.0270 (2)
C3B	0.49604 (15)	0.59992 (15)	0.67642 (4)	0.0321 (3)
H3BA	0.6086	0.5825	0.6880	0.039*
C4B	0.39165 (16)	0.68903 (15)	0.70360 (4)	0.0332 (3)
H4BA	0.4324	0.7299	0.7337	0.040*
C5B	0.22851 (15)	0.71848 (14)	0.68699 (4)	0.0301 (2)
C6B	0.16958 (14)	0.65609 (14)	0.64237 (4)	0.0291 (2)
C7B	0.27334 (14)	0.56639 (14)	0.61594 (4)	0.0278 (2)
H7BA	0.2321	0.5247	0.5860	0.033*
C8B	0.18210 (19)	0.88207 (18)	0.75296 (5)	0.0444 (3)
H8BA	0.0956	0.9491	0.7653	0.067*
H8BB	0.2800	0.9421	0.7443	0.067*
H8BC	0.2162	0.8085	0.7780	0.067*
C9B	−0.05314 (17)	0.6372 (2)	0.58261 (6)	0.0497 (4)
H9BA	−0.1684	0.6733	0.5760	0.075*
H9BB	−0.0519	0.5264	0.5824	0.075*
H9BC	0.0198	0.6754	0.5576	0.075*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0382 (5)	0.0514 (6)	0.0414 (5)	0.0209 (4)	−0.0143 (4)	−0.0106 (5)
O1A	0.0337 (4)	0.0433 (6)	0.0320 (4)	0.0100 (4)	0.0021 (3)	−0.0113 (4)
O2A	0.0249 (4)	0.0460 (6)	0.0366 (5)	0.0072 (4)	−0.0038 (3)	−0.0109 (4)
O1B	0.0330 (4)	0.0439 (6)	0.0415 (5)	−0.0023 (4)	0.0055 (4)	−0.0180 (4)
O2B	0.0237 (4)	0.0534 (6)	0.0471 (5)	0.0050 (4)	−0.0058 (4)	−0.0182 (5)
C1	0.0311 (5)	0.0274 (6)	0.0287 (5)	0.0075 (4)	−0.0035 (4)	0.0006 (4)
C2	0.0289 (5)	0.0232 (5)	0.0268 (5)	0.0038 (4)	−0.0018 (4)	0.0024 (4)
C3	0.0252 (5)	0.0264 (6)	0.0272 (5)	0.0014 (4)	−0.0003 (4)	−0.0008 (4)
C4	0.0244 (5)	0.0271 (6)	0.0277 (5)	0.0012 (4)	0.0002 (4)	−0.0009 (4)
C5	0.0275 (5)	0.0246 (6)	0.0269 (5)	0.0039 (4)	−0.0015 (4)	0.0034 (4)

C1A	0.0271 (5)	0.0287 (6)	0.0304 (6)	0.0039 (4)	−0.0031 (4)	0.0012 (5)
C2A	0.0251 (5)	0.0243 (6)	0.0290 (5)	0.0032 (4)	0.0014 (4)	0.0018 (4)
C3A	0.0240 (5)	0.0324 (6)	0.0336 (6)	0.0043 (4)	−0.0004 (4)	0.0009 (5)
C4A	0.0253 (5)	0.0336 (6)	0.0330 (6)	0.0063 (4)	0.0055 (4)	−0.0005 (5)
C5A	0.0284 (5)	0.0291 (6)	0.0276 (5)	0.0017 (4)	0.0043 (4)	−0.0020 (4)
C6A	0.0221 (5)	0.0288 (6)	0.0300 (5)	0.0015 (4)	0.0003 (4)	0.0002 (4)
C7A	0.0234 (5)	0.0274 (6)	0.0301 (5)	0.0042 (4)	0.0035 (4)	−0.0013 (4)
C8A	0.0437 (7)	0.0458 (8)	0.0373 (7)	0.0151 (6)	0.0081 (6)	−0.0077 (6)
C9A	0.0234 (5)	0.0486 (8)	0.0386 (7)	0.0070 (5)	0.0001 (5)	−0.0015 (6)
C1B	0.0287 (5)	0.0302 (6)	0.0267 (5)	0.0056 (4)	−0.0050 (4)	0.0023 (4)
C2B	0.0278 (5)	0.0287 (6)	0.0242 (5)	0.0025 (4)	−0.0028 (4)	0.0012 (4)
C3B	0.0305 (6)	0.0374 (7)	0.0276 (5)	0.0040 (5)	−0.0078 (4)	−0.0016 (5)
C4B	0.0364 (6)	0.0373 (7)	0.0255 (5)	−0.0013 (5)	−0.0052 (5)	−0.0060 (5)
C5B	0.0303 (5)	0.0296 (6)	0.0305 (6)	−0.0031 (5)	0.0027 (4)	−0.0054 (5)
C6B	0.0231 (5)	0.0319 (6)	0.0322 (6)	−0.0016 (4)	−0.0019 (4)	−0.0034 (5)
C7B	0.0271 (5)	0.0303 (6)	0.0257 (5)	−0.0008 (4)	−0.0027 (4)	−0.0036 (4)
C8B	0.0472 (8)	0.0455 (8)	0.0413 (7)	−0.0106 (6)	0.0103 (6)	−0.0193 (6)
C9B	0.0262 (6)	0.0704 (11)	0.0513 (8)	0.0032 (6)	−0.0120 (6)	−0.0175 (8)

Geometric parameters (Å, °)

O1—C1	1.2282 (14)	C4A—H4AA	0.9500
O1A—C5A	1.3631 (14)	C5A—C6A	1.4129 (15)
O1A—C8A	1.4278 (15)	C6A—C7A	1.3787 (16)
O2A—C6A	1.3652 (14)	C7A—H7AA	0.9500
O2A—C9A	1.4280 (14)	C8A—H8AA	0.9800
O1B—C5B	1.3652 (15)	C8A—H8AB	0.9800
O1B—C8B	1.4310 (16)	C8A—H8AC	0.9800
O2B—C6B	1.3671 (14)	C9A—H9AA	0.9800
O2B—C9B	1.4236 (17)	C9A—H9AB	0.9800
C1—C2	1.4819 (16)	C9A—H9AC	0.9800
C1—C5	1.4836 (16)	C1B—C2B	1.4548 (16)
C2—C1A	1.3483 (16)	C1B—H1BA	0.9500
C2—C3	1.5054 (15)	C2B—C3B	1.3933 (16)
C3—C4	1.5498 (16)	C2B—C7B	1.4133 (15)
C3—H3A	0.9900	C3B—C4B	1.3876 (18)
C3—H3B	0.9900	C3B—H3BA	0.9500
C4—C5	1.5064 (15)	C4B—C5B	1.3819 (17)
C4—H4A	0.9900	C4B—H4BA	0.9500
C4—H4B	0.9900	C5B—C6B	1.4114 (16)
C5—C1B	1.3454 (16)	C6B—C7B	1.3758 (17)
C1A—C2A	1.4546 (16)	C7B—H7BA	0.9500
C1A—H1AA	0.9500	C8B—H8BA	0.9800
C2A—C3A	1.3959 (16)	C8B—H8BB	0.9800
C2A—C7A	1.4123 (15)	C8B—H8BC	0.9800
C3A—C4A	1.3881 (17)	C9B—H9BA	0.9800
C3A—H3AA	0.9500	C9B—H9BB	0.9800
C4A—C5A	1.3842 (16)	C9B—H9BC	0.9800
C5A—O1A—C8A	117.32 (10)	O1A—C8A—H8AA	109.5

C6A—O2A—C9A	116.87 (9)	O1A—C8A—H8AB	109.5
C5B—O1B—C8B	115.62 (10)	H8AA—C8A—H8AB	109.5
C6B—O2B—C9B	116.58 (10)	O1A—C8A—H8AC	109.5
O1—C1—C2	125.91 (11)	H8AA—C8A—H8AC	109.5
O1—C1—C5	125.71 (11)	H8AB—C8A—H8AC	109.5
C2—C1—C5	108.38 (9)	O2A—C9A—H9AA	109.5
C1A—C2—C1	120.43 (10)	O2A—C9A—H9AB	109.5
C1A—C2—C3	130.28 (11)	H9AA—C9A—H9AB	109.5
C1—C2—C3	109.29 (9)	O2A—C9A—H9AC	109.5
C2—C3—C4	106.61 (9)	H9AA—C9A—H9AC	109.5
C2—C3—H3A	110.4	H9AB—C9A—H9AC	109.5
C4—C3—H3A	110.4	C5—C1B—C2B	130.72 (10)
C2—C3—H3B	110.4	C5—C1B—H1BA	114.6
C4—C3—H3B	110.4	C2B—C1B—H1BA	114.6
H3A—C3—H3B	108.6	C3B—C2B—C7B	117.53 (10)
C5—C4—C3	106.37 (9)	C3B—C2B—C1B	118.24 (10)
C5—C4—H4A	110.5	C7B—C2B—C1B	124.22 (10)
C3—C4—H4A	110.5	C4B—C3B—C2B	121.82 (11)
C5—C4—H4B	110.5	C4B—C3B—H3BA	119.1
C3—C4—H4B	110.5	C2B—C3B—H3BA	119.1
H4A—C4—H4B	108.6	C5B—C4B—C3B	120.12 (11)
C1B—C5—C1	119.93 (10)	C5B—C4B—H4BA	119.9
C1B—C5—C4	130.72 (10)	C3B—C4B—H4BA	119.9
C1—C5—C4	109.34 (10)	O1B—C5B—C4B	124.75 (11)
C2—C1A—C2A	130.85 (11)	O1B—C5B—C6B	116.03 (11)
C2—C1A—H1AA	114.6	C4B—C5B—C6B	119.22 (11)
C2A—C1A—H1AA	114.6	O2B—C6B—C7B	124.47 (11)
C3A—C2A—C7A	117.96 (10)	O2B—C6B—C5B	115.27 (10)
C3A—C2A—C1A	118.26 (10)	C7B—C6B—C5B	120.26 (10)
C7A—C2A—C1A	123.76 (10)	C6B—C7B—C2B	121.04 (10)
C4A—C3A—C2A	121.45 (11)	C6B—C7B—H7BA	119.5
C4A—C3A—H3AA	119.3	C2B—C7B—H7BA	119.5
C2A—C3A—H3AA	119.3	O1B—C8B—H8BA	109.5
C5A—C4A—C3A	120.08 (10)	O1B—C8B—H8BB	109.5
C5A—C4A—H4AA	120.0	H8BA—C8B—H8BB	109.5
C3A—C4A—H4AA	120.0	O1B—C8B—H8BC	109.5
O1A—C5A—C4A	125.46 (10)	H8BA—C8B—H8BC	109.5
O1A—C5A—C6A	114.93 (10)	H8BB—C8B—H8BC	109.5
C4A—C5A—C6A	119.61 (11)	O2B—C9B—H9BA	109.5
O2A—C6A—C7A	125.11 (10)	O2B—C9B—H9BB	109.5
O2A—C6A—C5A	115.04 (10)	H9BA—C9B—H9BB	109.5
C7A—C6A—C5A	119.85 (10)	O2B—C9B—H9BC	109.5
C6A—C7A—C2A	121.04 (10)	H9BA—C9B—H9BC	109.5
C6A—C7A—H7AA	119.5	H9BB—C9B—H9BC	109.5
C2A—C7A—H7AA	119.5		
O1—C1—C2—C1A	−0.3 (2)	O1A—C5A—C6A—C7A	179.55 (11)
C5—C1—C2—C1A	179.10 (11)	C4A—C5A—C6A—C7A	−0.16 (18)
O1—C1—C2—C3	−179.31 (13)	O2A—C6A—C7A—C2A	179.80 (11)
C5—C1—C2—C3	0.12 (13)	C5A—C6A—C7A—C2A	0.17 (18)

C1A—C2—C3—C4	−179.60 (12)	C3A—C2A—C7A—C6A	0.02 (17)
C1—C2—C3—C4	−0.75 (13)	C1A—C2A—C7A—C6A	−178.56 (11)
C2—C3—C4—C5	1.09 (12)	C1—C5—C1B—C2B	−179.78 (12)
O1—C1—C5—C1B	−0.6 (2)	C4—C5—C1B—C2B	−0.6 (2)
C2—C1—C5—C1B	179.94 (11)	C5—C1B—C2B—C3B	−179.06 (13)
O1—C1—C5—C4	−179.98 (13)	C5—C1B—C2B—C7B	2.2 (2)
C2—C1—C5—C4	0.59 (13)	C7B—C2B—C3B—C4B	1.34 (19)
C3—C4—C5—C1B	179.71 (12)	C1B—C2B—C3B—C4B	−177.47 (12)
C3—C4—C5—C1	−1.04 (13)	C2B—C3B—C4B—C5B	−1.3 (2)
C1—C2—C1A—C2A	179.44 (12)	C8B—O1B—C5B—C4B	8.17 (19)
C3—C2—C1A—C2A	−1.8 (2)	C8B—O1B—C5B—C6B	−172.51 (12)
C2—C1A—C2A—C3A	−179.99 (13)	C3B—C4B—C5B—O1B	179.72 (12)
C2—C1A—C2A—C7A	−1.4 (2)	C3B—C4B—C5B—C6B	0.4 (2)
C7A—C2A—C3A—C4A	−0.23 (18)	C9B—O2B—C6B—C7B	−4.6 (2)
C1A—C2A—C3A—C4A	178.44 (11)	C9B—O2B—C6B—C5B	176.11 (13)
C2A—C3A—C4A—C5A	0.24 (19)	O1B—C5B—C6B—O2B	0.33 (17)
C8A—O1A—C5A—C4A	−1.26 (19)	C4B—C5B—C6B—O2B	179.69 (12)
C8A—O1A—C5A—C6A	179.05 (11)	O1B—C5B—C6B—C7B	−179.01 (11)
C3A—C4A—C5A—O1A	−179.72 (12)	C4B—C5B—C6B—C7B	0.35 (19)
C3A—C4A—C5A—C6A	−0.04 (18)	O2B—C6B—C7B—C2B	−179.56 (12)
C9A—O2A—C6A—C7A	−1.76 (18)	C5B—C6B—C7B—C2B	−0.28 (19)
C9A—O2A—C6A—C5A	177.88 (11)	C3B—C2B—C7B—C6B	−0.54 (18)
O1A—C5A—C6A—O2A	−0.11 (16)	C1B—C2B—C7B—C6B	178.18 (12)
C4A—C5A—C6A—O2A	−179.82 (11)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4B—H4BA···O1A^{i}	0.95	2.42	3.2556 (15)	146

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

Fig. 1

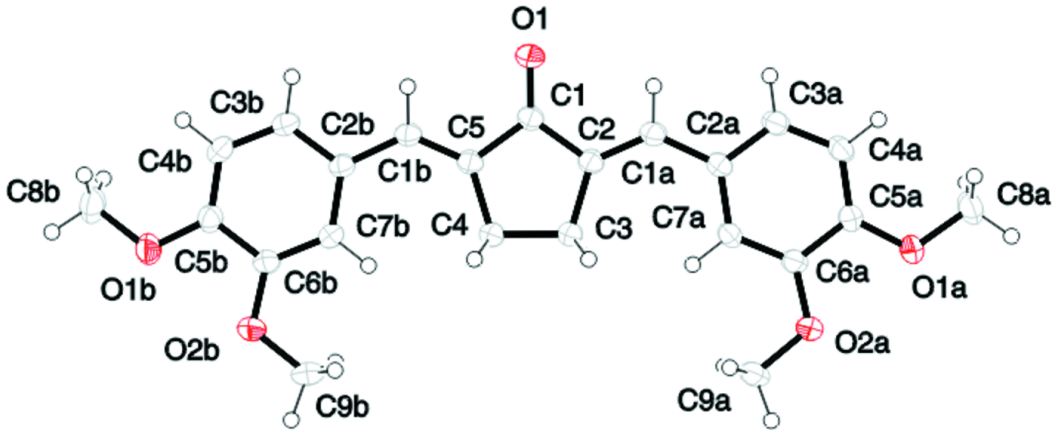

O1
C1
C2
C3
C4
C5
C1a
C2a
C3a
C4a
C5a
C6a
C7a
C8a
O1a
O2a
C9a
C1b
C2b
C3b
C4b
C5b
C6b
C7b
C8b
O1b
O2b
C9b

Fig. 2

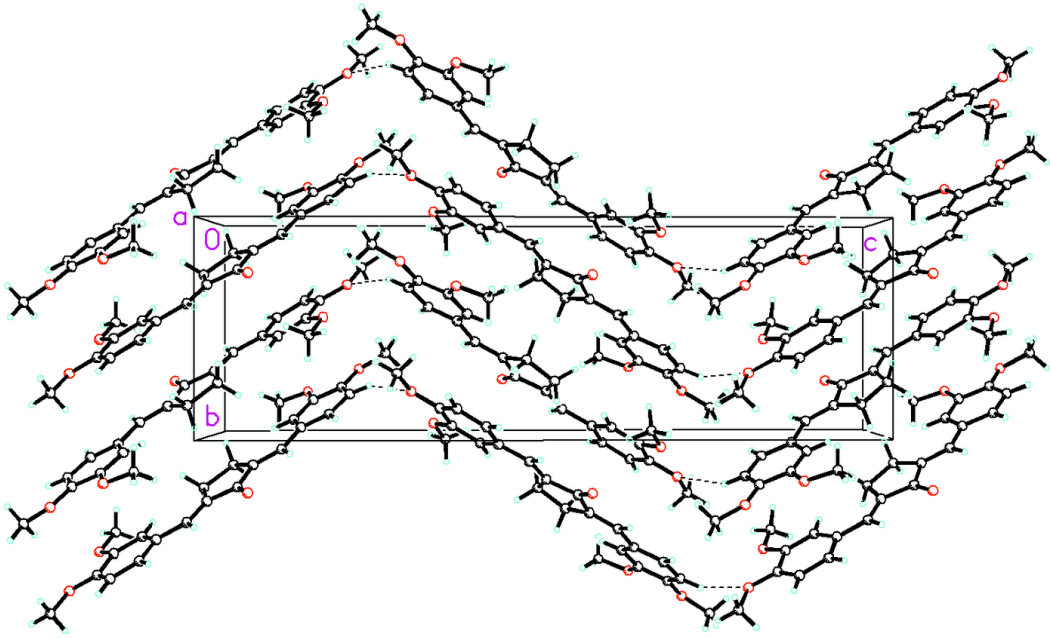


Fig. 3