Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*E*,5*E*)-2,5-Bis(2-bromobenzylidene)-cyclopentanone

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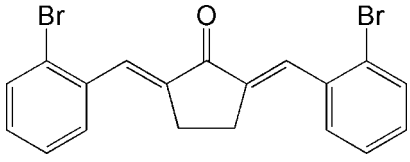
Received 13 July 2007; accepted 24 July 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.095; data-to-parameter ratio = 14.4.

In the biologically active title compound, $C_{19}H_{14}Br_2O$, a derivative of curcumin, the dihedral angle between the aromatic ring planes is 59.78 (19)°.

Related literature

For related literature, see: Artico *et al.* (1998); Began *et al.* (1999); Butcher, Jasinski, Narayana *et al.* (2007); Butcher, Jasinski, Sarojini *et al.* (2007); Kawamon *et al.* (1999); Poorichaya *et al.* (2007); Subbagh *et al.* (2000).



Experimental

Crystal data

$C_{19}H_{14}Br_2O$
M_r = 418.12
Monoclinic, $P2_1/c$
a = 8.6504 (8) Å
b = 8.4789 (8) Å
c = 22.126 (2) Å
β = 93.119 (2)°
V = 1620.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.00 mm^{-1}
T = 298 (2) K
0.33 × 0.20 × 0.19 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.309, T_{max} = 0.391
8202 measured reflections
2857 independent reflections
2292 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.095
S = 1.04
2857 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.96 e Å^{-3}

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

This work was supported by a Key Grant (No. 2005C13019) from the Science and Technology Department of Zhejiang Province, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2485).

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  doi:10.1107/S1600536807036185

supplementary materials

Acta Cryst. (2007). E**63**, o3630 [**doi:10.1107/S1600536807036185**]

(2*E*,5*E*)-2,5-Bis(2-bromobenzylidene)cyclopentanone

G. Liang, J.-L. Tian, C.-G. Zhao and X.-K. Li

Comment

The title compound, (2E, 5E)-2,5-bis(2-bromobenzylidene) cyclopentanone, (I), derived from curcumin, is a biologically active compound. Curcumin has been found to possess a variety of pharmaceutical applications, for example, inhibiting carcinogen-induced mutations and the formation of tumour, antioxidation, anti-inflammation, anti-virus, decreasing total cholesterol and LDL cholesterol level (Began *et al.*, 1999; Kawamon *et al.*, 1999; Poorichaya *et al.*, 2007). However, curcumin is unstable at a pH over 6.5, due to the presence of the methylene group. Omitting the methylene group and one carbonyl group, therefore, some mono-carbonyl curcumin analogues without the central methylene functional groups, were synthesized and their biological activity *in vitro* were evaluated. Part of bisbenzylidene cyclopentanone derivatives showed stronger bio-activity than curcumin (Artico *et al.*, 1998; Butcher *et al.*, 2007; Subbagh *et al.*, 2000). The crystal structure of 2,5-bis(3,4-dimethoxybenzylidene) cyclopentanone (Butcher *et al.*, 2007) has been reported. As part of our research in this area, we synthesized the title compound $C_{19}H_{14}Br_2O$ and describe its structure here. Its geometrical parameters are normal; the dihedral angle between the aromatic ring planes is 59.78 (19)°.

Experimental

To a solution of 15 mmol 2-bromobenzaldehyde in MeOH (10 ml) was added 7.5 mmol cyclopentanone. The solution was stirred at room temperature for 20 min, followed by added dropwise 20% (*w*/*v*) NaOH (1.5 ml, 7.5 mmol). The mixture was stirred at RT and monitored with TLC. When the reaction finished, the residue was poured into saturated NH_4Cl solution and filtered. The precipitate was washed and purified by chromatography over silica gel using CH_2Cl_2/CH_3OH as the eluent to afford the pure product (yield: 65%). Single crystals of (I) were grown in a CH_2Cl_2–CH_3OH mixture (8:2 *v*/*v*) by slow evaporation (mp 436–437 K). 1*H*-NMR ($CDCl_3$): 1.93 (4*H*, s, CH2—CH2), 7.23 (2*H*, m, Ar—H6), 7.37 (2*H*, m, Ar—H4), 7.52 (2*H*, m, Ar—H5), 7.66 (2*H*, m, Ar—H3), 7.86 (2*H*, s, Ar—CH=C2). ESI-MS m/*z*: 418.94 (*M*+1)+, calcd for $C_{19}H_{14}Br_2O$: 418.12.

Refinement

The H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

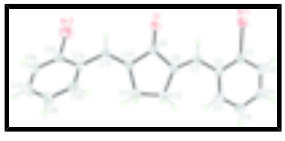


Fig. 1. The molecular structure of (I) shoiwng 30% displacement ellipsoids for the non-hydrogen atoms.

(2E,5E)-2,5-Bis(2-bromobenzylidene)cyclopentanone

Crystal data

$C_{19}H_{14}Br_2O$	F_{000} = 824
M_r = 418.12	D_x = 1.714 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2746 reflections
a = 8.6504 (8) Å	θ = 2.5–24.8º
b = 8.4789 (8) Å	μ = 5.00 mm^{-1}
c = 22.126 (2) Å	T = 298 (2) K
β = 93.119 (2)º	Block, colourless
V = 1620.5 (3) Å^3	0.33 × 0.20 × 0.19 mm
Z = 4	

Data collection

Bruker APEX CCD area-detector diffractometer	2857 independent reflections
Radiation source: fine-focus sealed tube	2292 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	h = −10→10
T_{min} = 0.309, T_{max} = 0.391	k = −10→9
8202 measured reflections	l = −26→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H-atom parameters constrained
$wR(F^2)$ = 0.095	$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 1.7145P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
2857 reflections	$\Delta\rho_{max}$ = 0.69 e Å^{-3}
199 parameters	$\Delta\rho_{min}$ = −0.96 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}
Br1	0.33176 (5)	0.34999 (5)	1.049559 (18)	0.05462 (16)
Br2	1.11065 (5)	0.18597 (7)	0.77243 (3)	0.0767 (2)
O1	0.6144 (3)	0.3500 (3)	0.86877 (12)	0.0550 (7)
C1	0.2692 (4)	0.1396 (4)	1.02839 (17)	0.0432 (8)
C2	0.3168 (4)	0.0684 (4)	0.97524 (16)	0.0410 (8)
C3	0.2662 (4)	−0.0868 (5)	0.96568 (17)	0.0505 (9)
H3	0.2942	−0.1396	0.9311	0.061*
C4	0.1760 (5)	−0.1643 (5)	1.00582 (19)	0.0575 (11)
H4	0.1448	−0.2677	0.9982	0.069*
C5	0.1322 (5)	−0.0883 (5)	1.05707 (19)	0.0565 (11)
H5	0.0708	−0.1403	1.0840	0.068*
C6	0.1786 (4)	0.0636 (5)	1.06871 (17)	0.0501 (10)
H6	0.1494	0.1150	1.1034	0.060*
C7	0.4145 (4)	0.1517 (4)	0.93415 (16)	0.0421 (8)
H7	0.4464	0.2509	0.9477	0.050*
C8	0.4664 (4)	0.1114 (4)	0.88048 (16)	0.0389 (8)
C9	0.5734 (4)	0.2199 (4)	0.85095 (15)	0.0395 (8)
C10	0.6236 (4)	0.1421 (4)	0.79538 (15)	0.0370 (8)
C11	0.5245 (5)	−0.0008 (5)	0.78286 (17)	0.0502 (9)
H11A	0.5873	−0.0898	0.7717	0.060*
H11B	0.4477	0.0199	0.7502	0.060*
C12	0.4453 (4)	−0.0347 (4)	0.84245 (16)	0.0431 (8)
H12A	0.3362	−0.0569	0.8343	0.052*
H12B	0.4931	−0.1248	0.8630	0.052*
C13	0.7469 (4)	0.1931 (4)	0.76776 (16)	0.0405 (8)
H13	0.7969	0.2809	0.7847	0.049*
C14	0.8131 (4)	0.1269 (4)	0.71370 (16)	0.0398 (8)
C15	0.9727 (4)	0.1184 (4)	0.70760 (18)	0.0479 (9)
C16	1.0353 (5)	0.0583 (5)	0.6568 (2)	0.0603 (11)
H16	1.1422	0.0531	0.6542	0.072*
C17	0.9385 (6)	0.0059 (5)	0.6099 (2)	0.0696 (14)
H17	0.9801	−0.0346	0.5752	0.083*
C18	0.7796 (6)	0.0129 (5)	0.61369 (19)	0.0634 (12)

H18	0.7143	−0.0213	0.5815	0.076*
C19	0.7185 (5)	0.0710 (5)	0.66541 (16)	0.0504 (10)
H19	0.6116	0.0730	0.6682	0.060*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0688 (3)	0.0473 (2)	0.0492 (2)	0.00208 (19)	0.01663 (19)	−0.00735 (18)
Br2	0.0447 (3)	0.0869 (4)	0.0983 (4)	0.0006 (2)	0.0019 (2)	−0.0170 (3)
O1	0.0654 (18)	0.0410 (16)	0.0605 (17)	−0.0107 (13)	0.0218 (14)	−0.0135 (13)
C1	0.0397 (19)	0.044 (2)	0.046 (2)	0.0049 (17)	0.0023 (16)	−0.0012 (16)
C2	0.0358 (19)	0.045 (2)	0.0428 (19)	0.0014 (16)	0.0020 (15)	0.0018 (16)
C3	0.052 (2)	0.052 (2)	0.048 (2)	−0.005 (2)	0.0111 (18)	−0.0045 (18)
C4	0.060 (3)	0.055 (3)	0.059 (3)	−0.012 (2)	0.008 (2)	−0.002 (2)
C5	0.047 (2)	0.064 (3)	0.060 (3)	−0.009 (2)	0.0128 (19)	0.014 (2)
C6	0.044 (2)	0.061 (3)	0.046 (2)	0.0028 (19)	0.0086 (17)	0.0020 (19)
C7	0.0402 (19)	0.0360 (19)	0.050 (2)	0.0002 (16)	0.0062 (16)	−0.0028 (16)
C8	0.0357 (18)	0.0379 (19)	0.043 (2)	0.0024 (15)	0.0042 (15)	−0.0032 (15)
C9	0.0398 (19)	0.039 (2)	0.0397 (19)	0.0021 (16)	0.0039 (15)	−0.0032 (16)
C10	0.0344 (18)	0.0375 (19)	0.0394 (18)	0.0031 (15)	0.0039 (14)	−0.0026 (15)
C11	0.050 (2)	0.050 (2)	0.051 (2)	−0.0075 (19)	0.0114 (17)	−0.0139 (18)
C12	0.043 (2)	0.042 (2)	0.045 (2)	−0.0052 (16)	0.0040 (16)	−0.0075 (16)
C13	0.043 (2)	0.0348 (19)	0.0439 (19)	0.0020 (16)	0.0032 (16)	−0.0042 (15)
C14	0.044 (2)	0.0315 (18)	0.045 (2)	0.0043 (16)	0.0082 (16)	0.0014 (15)
C15	0.048 (2)	0.037 (2)	0.060 (2)	0.0021 (17)	0.0161 (18)	0.0045 (17)
C16	0.068 (3)	0.048 (3)	0.068 (3)	0.007 (2)	0.033 (2)	0.008 (2)
C17	0.104 (4)	0.052 (3)	0.056 (3)	0.009 (3)	0.042 (3)	0.001 (2)
C18	0.098 (4)	0.048 (3)	0.045 (2)	−0.006 (2)	0.012 (2)	−0.0036 (19)
C19	0.057 (2)	0.046 (2)	0.049 (2)	0.0000 (19)	0.0094 (19)	0.0020 (18)

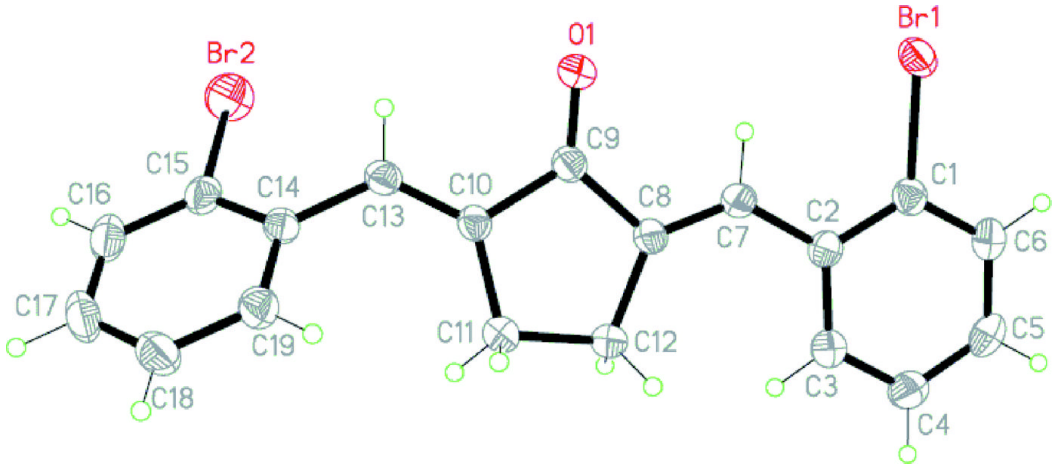
Geometric parameters (Å, °)

Br1—C1	1.915 (4)	C10—C13	1.330 (5)
Br2—C15	1.904 (4)	C10—C11	1.501 (5)
O1—C9	1.217 (4)	C11—C12	1.545 (5)
C1—C6	1.379 (5)	C11—H11A	0.9700
C1—C2	1.403 (5)	C11—H11B	0.9700
C2—C3	1.400 (5)	C12—H12A	0.9700
C2—C7	1.457 (5)	C12—H12B	0.9700
C3—C4	1.380 (6)	C13—C14	1.466 (5)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.375 (6)	C14—C19	1.393 (5)
C4—H4	0.9300	C14—C15	1.396 (5)
C5—C6	1.369 (6)	C15—C16	1.372 (5)
C5—H5	0.9300	C16—C17	1.372 (7)
C6—H6	0.9300	C16—H16	0.9300
C7—C8	1.337 (5)	C17—C18	1.384 (7)
C7—H7	0.9300	C17—H17	0.9300
C8—C9	1.482 (5)	C18—C19	1.377 (5)

C8—C12	1.503 (5)	C18—H18	0.9300
C9—C10	1.481 (5)	C19—H19	0.9300
C6—C1—C2	123.0 (4)	C12—C11—H11A	110.6
C6—C1—Br1	116.1 (3)	C10—C11—H11B	110.6
C2—C1—Br1	120.9 (3)	C12—C11—H11B	110.6
C3—C2—C1	115.3 (3)	H11A—C11—H11B	108.8
C3—C2—C7	123.3 (3)	C8—C12—C11	106.1 (3)
C1—C2—C7	121.4 (3)	C8—C12—H12A	110.5
C4—C3—C2	122.3 (4)	C11—C12—H12A	110.5
C4—C3—H3	118.9	C8—C12—H12B	110.5
C2—C3—H3	118.9	C11—C12—H12B	110.5
C5—C4—C3	119.9 (4)	H12A—C12—H12B	108.7
C5—C4—H4	120.1	C10—C13—C14	127.5 (3)
C3—C4—H4	120.1	C10—C13—H13	116.2
C6—C5—C4	120.3 (4)	C14—C13—H13	116.2
C6—C5—H5	119.8	C19—C14—C15	116.8 (3)
C4—C5—H5	119.8	C19—C14—C13	121.1 (3)
C5—C6—C1	119.3 (4)	C15—C14—C13	122.1 (3)
C5—C6—H6	120.4	C16—C15—C14	122.4 (4)
C1—C6—H6	120.4	C16—C15—Br2	118.0 (3)
C8—C7—C2	131.7 (3)	C14—C15—Br2	119.6 (3)
C8—C7—H7	114.2	C17—C16—C15	119.2 (4)
C2—C7—H7	114.2	C17—C16—H16	120.4
C7—C8—C9	118.8 (3)	C15—C16—H16	120.4
C7—C8—C12	132.2 (3)	C16—C17—C18	120.5 (4)
C9—C8—C12	108.9 (3)	C16—C17—H17	119.8
O1—C9—C10	125.4 (3)	C18—C17—H17	119.8
O1—C9—C8	126.7 (3)	C19—C18—C17	119.6 (4)
C10—C9—C8	107.9 (3)	C19—C18—H18	120.2
C13—C10—C9	121.0 (3)	C17—C18—H18	120.2
C13—C10—C11	129.9 (3)	C18—C19—C14	121.5 (4)
C9—C10—C11	108.8 (3)	C18—C19—H19	119.2
C10—C11—C12	105.5 (3)	C14—C19—H19	119.2
C10—C11—H11A	110.6		
C6—C1—C2—C3	−0.1 (5)	C8—C9—C10—C11	11.3 (4)
Br1—C1—C2—C3	178.6 (3)	C13—C10—C11—C12	157.8 (4)
C6—C1—C2—C7	−179.1 (3)	C9—C10—C11—C12	−16.4 (4)
Br1—C1—C2—C7	−0.4 (5)	C7—C8—C12—C11	175.4 (4)
C1—C2—C3—C4	−0.1 (6)	C9—C8—C12—C11	−8.5 (4)
C7—C2—C3—C4	178.8 (4)	C10—C11—C12—C8	15.1 (4)
C2—C3—C4—C5	0.4 (6)	C9—C10—C13—C14	178.3 (3)
C3—C4—C5—C6	−0.5 (6)	C11—C10—C13—C14	4.8 (7)
C4—C5—C6—C1	0.2 (6)	C10—C13—C14—C19	39.3 (6)
C2—C1—C6—C5	0.1 (6)	C10—C13—C14—C15	−141.5 (4)
Br1—C1—C6—C5	−178.7 (3)	C19—C14—C15—C16	0.0 (5)
C3—C2—C7—C8	5.1 (6)	C13—C14—C15—C16	−179.2 (4)
C1—C2—C7—C8	−176.1 (4)	C19—C14—C15—Br2	−178.0 (3)
C2—C7—C8—C9	−176.3 (3)	C13—C14—C15—Br2	2.7 (5)

C2—C7—C8—C12	−0.6 (7)	C14—C15—C16—C17	0.7 (6)
C7—C8—C9—O1	−4.5 (6)	Br2—C15—C16—C17	178.8 (3)
C12—C8—C9—O1	178.8 (4)	C15—C16—C17—C18	−0.3 (7)
C7—C8—C9—C10	175.2 (3)	C16—C17—C18—C19	−0.9 (7)
C12—C8—C9—C10	−1.5 (4)	C17—C18—C19—C14	1.6 (6)
O1—C9—C10—C13	16.3 (6)	C15—C14—C19—C18	−1.2 (6)
C8—C9—C10—C13	−163.4 (3)	C13—C14—C19—C18	178.0 (4)
O1—C9—C10—C11	−168.9 (4)		

Fig. 1


Br2
O1
Br1
C15
C9
C16
C14
C10
C8
C1
C13
C7
C2
C6
C17
C19
C11
C12
C5
C18
C3
C4