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(2*E*,6*E*)-2,6-Bis(2,6-dichlorobenzylidene)cyclohexanone

Gholam Hossein Mahdavinia,[a]* Maryam Mirzazadeh,[a] Vahid Amani[b] and Behrouz Notash[c]

[a]Department of Chemistry, Marvdasht Branch, Islamic Azad University, Marvdasht, Iran, [b]Department of Chemistry, Shahre-Rey Branch, Islamic Azad University, Tehran, Iran, and [c]Department of Chemistry, Shahid Beheshti University, G.C., Evin, Tehran 1983963113, Iran
Correspondence e-mail: hmahdavinia@gmail.com

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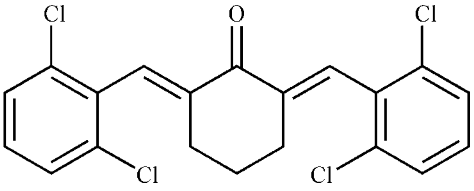
Key indicators: single-crystal X-ray study; *T* = 120 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.031; *wR* factor = 0.071; data-to-parameter ratio = 21.9.

The title compound, $C_{20}H_{14}Cl_4O$, was prepared by the reaction of 2,6-dichlorobenzaldehyde and cyclohexanone. In the molecule, the central cyclohexanone ring adopts an envelope conformation, while the terminal benzene rings make a dihedral angle of 57.87 (9)°.

Related literature

For background and applications of arylidene cycloalkanones, see: Deli *et al.* (1984); Nakano *et al.* (1987); Kawamata *et al.* (1996); Dimmock *et al.* (2003); Raj *et al.* (2003); Gangadhara (1995). For related structures, see: Yu *et al.* (2000); Zhou (2007).



Experimental

Crystal data

$C_{20}H_{14}Cl_4O$
M_r = 412.11
Orthorhombic, $Pna2_1$
a = 17.917 (4) Å
b = 7.3094 (15) Å
c = 14.093 (3) Å
V = 1845.7 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.65 mm^{-1}
T = 120 K
0.6 × 0.35 × 0.33 mm

Data collection

Stoe IPDS 2T diffractometer
13510 measured reflections
4946 independent reflections
4682 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.071
S = 1.04
4946 reflections
226 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.25 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 2369 Friedel pairs
Flack parameter: 0.01 (4)

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5466).

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(2*E*,6*E*)-2,6-Bis(2,6-dichlorobenzylidene)cyclohexanone

Gholam Hossein Mahdavinia, Maryam Mirzazadeh, Vahid Amani and Behrouz Notash

S1. Comment

Cross-aldol condensation of aromatic aldehydes with cyclic ketones is an important protocol for the synthesis of arylidene cycloalkanones, which are very important precursors to potentially bioactive pyrimidine derivates (Deli *et al.*, 1984), intermediates for agrochemical, pharmaceuticals and perfumes (Nakano *et al.*, 1987), new organic material for nonlinear optical applications (Kawamata *et al.*, 1996), cytotoxic analogous (Dimmock *et al.*, 2003), bis-spiropyrrolidines (Raj *et al.*, 2003) and the units of liquid crystalline polymers (Gangadhara, 1995). Usually, this condensation process is catalyzed by strong acid or base.

In the molecule of the title compound, (Fig. 1), the bond lengths and angles are within normal ranges (Yu *et al.*, 2000; Zhou, 2007). A dihedral angle of 57.87 (9) A is found between the mean planes of the two benzene rings.

S2. Experimental

To a 10 ml solution of KOH (0.11 g) in ethanol at 313 K in a round bottom flask, cyclohexanone (5.0 mmol, 0.50 g) and 2,6-dichlorobenzaldehyde (10 mmol, 1.75 g) was added and the mixture was stirred for 2 min. The resulting product was then isolated by simple filtration from the reaction mixture and given washings with water to remove any trace of KOH remaining on the product. Yellow crystals, yield 97%, 1.98 g, m. p. 455–458 K.

S3. Refinement

All H atoms were positioned geometrically with C–H = 0.93–0.97 Å and constrained to ride on their parent atoms, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

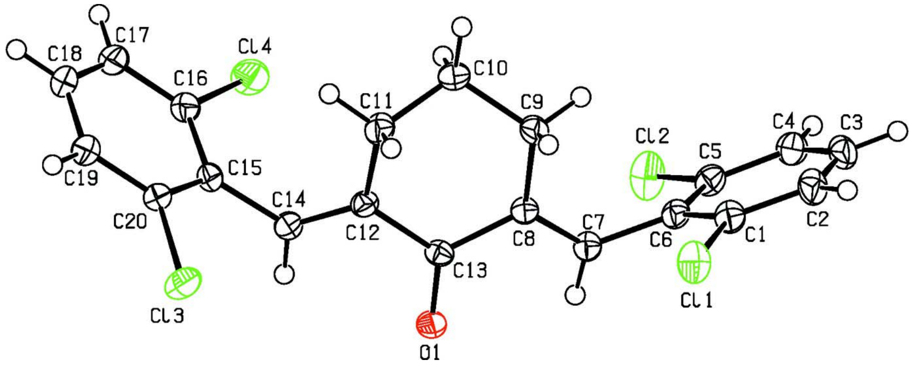

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

(2*E*,6*E*)-2,6-Bis(2,6-dichlorobenzylidene)cyclohexanone

Crystal data

$C_{20}H_{14}Cl_4O$	$F(000) = 840$
$M_r = 412.11$	$D_x = 1.483$ Mg m^{-3}
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2c -2n	Cell parameters from 4949 reflections
$a = 17.917$ (4) Å	$\theta = 2.3$–29.2°
$b = 7.3094$ (15) Å	$\mu = 0.65$ mm^{-1}
$c = 14.093$ (3) Å	$T = 120$ K
$V = 1845.7$ (7) Å^3	Needle, yellow
$Z = 4$	0.6 × 0.35 × 0.33 mm

Data collection

Stoe IPDS 2T diffractometer	4682 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.043$
Graphite monochromator	$\theta_{\max} = 29.2°$, $\theta_{\min} = 2.3°$
rotation method scans	$h = -24 \rightarrow 24$
13510 measured reflections	$k = -8 \rightarrow 10$
4946 independent reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.031$	$w = 1/[\sigma^2(F_o^2) + (0.0318P)^2 + 0.5994P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.071$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.04$	$\Delta\rho_{\max} = 0.25$ e Å^{-3}
4946 reflections	$\Delta\rho_{\min} = -0.20$ e Å^{-3}
226 parameters	Absolute structure: Flack (1983), 2369 Friedel pairs
1 restraint	Absolute structure parameter: 0.01 (4)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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.

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

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.23181 (3)	1.40628 (7)	0.99333 (3)	0.03179 (11)
Cl3	−0.14648 (3)	0.88978 (7)	0.82912 (3)	0.03028 (10)
Cl4	0.05216 (3)	0.35099 (7)	0.89917 (4)	0.03460 (11)
Cl2	0.30403 (3)	0.77001 (7)	1.16748 (4)	0.03902 (13)

C12	0.04419 (9)	0.8310 (2)	0.91901 (11)	0.0196 (3)
O1	0.04467 (7)	0.94480 (19)	1.07669 (9)	0.0237 (3)
C6	0.27374 (10)	1.0891 (2)	1.07795 (12)	0.0211 (3)
C13	0.07918 (9)	0.9234 (2)	1.00226 (12)	0.0185 (3)
C7	0.19499 (9)	1.0268 (2)	1.07289 (12)	0.0199 (3)
H7	0.1686	1.0178	1.1295	0.024*
C9	0.19554 (9)	0.9844 (3)	0.89597 (12)	0.0253 (3)
H9A	0.2490	0.9679	0.9032	0.030*
H9B	0.1872	1.1026	0.8666	0.030*
C15	−0.05043 (9)	0.6110 (2)	0.85940 (12)	0.0209 (3)
C5	0.32884 (10)	0.9818 (3)	1.12039 (12)	0.0252 (4)
C18	−0.11451 (11)	0.3989 (3)	0.71361 (15)	0.0317 (4)
H18	−0.1354	0.3295	0.6651	0.038*
C8	0.15938 (9)	0.9827 (2)	0.99274 (12)	0.0186 (3)
C1	0.29745 (11)	1.2584 (3)	1.04280 (12)	0.0253 (4)
C16	−0.02454 (10)	0.4355 (3)	0.83722 (14)	0.0246 (3)
C10	0.16500 (10)	0.8350 (3)	0.83127 (13)	0.0278 (4)
H10A	0.1873	0.8457	0.7688	0.033*
H10B	0.1777	0.7157	0.8568	0.033*
C19	−0.14239 (10)	0.5721 (3)	0.73292 (14)	0.0281 (4)
H19	−0.1820	0.6191	0.6979	0.034*
C11	0.08055 (10)	0.8530 (3)	0.82365 (12)	0.0260 (4)
H11A	0.0681	0.9721	0.7978	0.031*
H11B	0.0615	0.7605	0.7806	0.031*
C3	0.42366 (10)	1.2027 (3)	1.08876 (13)	0.0318 (4)
H3	0.4733	1.2393	1.0916	0.038*
C14	−0.01489 (9)	0.7245 (2)	0.93404 (12)	0.0211 (3)
H14	−0.0350	0.7210	0.9948	0.025*
C20	−0.11038 (10)	0.6741 (3)	0.80531 (12)	0.0222 (3)
C17	−0.05563 (11)	0.3289 (3)	0.76625 (15)	0.0295 (4)
H17	−0.0373	0.2122	0.7541	0.035*
C4	0.40302 (11)	1.0347 (3)	1.12538 (14)	0.0296 (4)
H4	0.4384	0.9584	1.1530	0.036*
C2	0.37123 (11)	1.3166 (3)	1.04810 (13)	0.0298 (4)
H2	0.3850	1.4306	1.0246	0.036*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0347 (2)	0.0280 (2)	0.0326 (2)	−0.00606 (18)	−0.00506 (19)	0.0103 (2)
Cl3	0.0346 (2)	0.0290 (2)	0.0272 (2)	0.00852 (18)	−0.00735 (18)	−0.00764 (19)
Cl4	0.0329 (2)	0.0303 (2)	0.0406 (3)	0.00904 (18)	−0.0048 (2)	−0.0030 (2)
Cl2	0.0393 (2)	0.0295 (2)	0.0483 (3)	−0.0027 (2)	−0.0170 (2)	0.0103 (2)
C12	0.0205 (7)	0.0212 (8)	0.0172 (7)	0.0006 (6)	−0.0003 (6)	−0.0023 (6)
O1	0.0229 (6)	0.0316 (7)	0.0167 (5)	−0.0022 (5)	0.0025 (4)	−0.0034 (5)
C6	0.0233 (7)	0.0252 (8)	0.0147 (6)	−0.0025 (6)	−0.0014 (6)	−0.0006 (6)
C13	0.0185 (7)	0.0214 (7)	0.0157 (7)	0.0020 (6)	0.0008 (6)	0.0017 (6)
C7	0.0214 (7)	0.0217 (7)	0.0166 (7)	−0.0015 (6)	−0.0011 (6)	0.0020 (6)

C9	0.0218 (8)	0.0344 (10)	0.0197 (7)	−0.0056 (7)	0.0038 (7)	−0.0032 (8)
C15	0.0188 (7)	0.0219 (8)	0.0219 (7)	−0.0041 (6)	0.0023 (6)	−0.0011 (6)
C5	0.0273 (9)	0.0279 (9)	0.0204 (8)	−0.0009 (7)	−0.0027 (7)	−0.0023 (7)
C18	0.0259 (9)	0.0345 (10)	0.0348 (10)	−0.0081 (8)	0.0025 (7)	−0.0151 (9)
C8	0.0196 (7)	0.0193 (7)	0.0169 (6)	−0.0001 (6)	0.0001 (6)	0.0010 (6)
C1	0.0267 (8)	0.0313 (9)	0.0179 (7)	−0.0039 (7)	−0.0008 (6)	0.0017 (7)
C16	0.0228 (7)	0.0228 (8)	0.0283 (9)	−0.0013 (6)	0.0026 (7)	−0.0026 (7)
C10	0.0276 (8)	0.0360 (10)	0.0197 (7)	−0.0038 (7)	0.0050 (7)	−0.0068 (8)
C19	0.0233 (8)	0.0340 (10)	0.0270 (9)	−0.0018 (7)	−0.0015 (7)	−0.0090 (8)
C11	0.0289 (8)	0.0331 (10)	0.0160 (7)	−0.0077 (7)	0.0011 (7)	−0.0018 (7)
C3	0.0220 (8)	0.0518 (12)	0.0216 (8)	−0.0093 (8)	0.0008 (7)	−0.0069 (8)
C14	0.0212 (7)	0.0233 (8)	0.0187 (7)	−0.0006 (7)	−0.0001 (6)	−0.0014 (6)
C20	0.0217 (8)	0.0232 (8)	0.0216 (8)	−0.0014 (7)	0.0017 (6)	−0.0040 (6)
C17	0.0281 (9)	0.0241 (9)	0.0362 (10)	−0.0048 (7)	0.0066 (8)	−0.0085 (8)
C4	0.0232 (8)	0.0412 (11)	0.0243 (8)	0.0021 (8)	−0.0063 (7)	−0.0062 (8)
C2	0.0299 (9)	0.0386 (10)	0.0210 (8)	−0.0137 (8)	0.0032 (7)	0.0017 (8)

Geometric parameters (Å, º)

Cl1—C1	1.743 (2)	C5—C4	1.386 (3)
Cl3—C20	1.7370 (19)	C18—C17	1.388 (3)
Cl4—C16	1.7414 (19)	C18—C19	1.388 (3)
Cl2—C5	1.742 (2)	C18—H18	0.9300
C12—C14	1.331 (2)	C1—C2	1.391 (3)
C12—C13	1.492 (2)	C16—C17	1.385 (3)
C12—C11	1.502 (2)	C10—C11	1.523 (3)
O1—C13	1.228 (2)	C10—H10A	0.9700
C6—C5	1.396 (3)	C10—H10B	0.9700
C6—C1	1.399 (3)	C19—C20	1.388 (2)
C6—C7	1.484 (2)	C19—H19	0.9300
C13—C8	1.507 (2)	C11—H11A	0.9700
C7—C8	1.337 (2)	C11—H11B	0.9700
C7—H7	0.9300	C3—C2	1.380 (3)
C9—C8	1.510 (2)	C3—C4	1.382 (3)
C9—C10	1.524 (3)	C3—H3	0.9300
C9—H9A	0.9700	C14—H14	0.9300
C9—H9B	0.9700	C17—H17	0.9300
C15—C20	1.396 (2)	C4—H4	0.9300
C15—C16	1.399 (3)	C2—H2	0.9300
C15—C14	1.483 (2)		
C14—C12—C13	118.29 (15)	C15—C16—Cl4	118.34 (14)
C14—C12—C11	123.35 (15)	C11—C10—C9	109.69 (15)
C13—C12—C11	118.23 (14)	C11—C10—H10A	109.7
C5—C6—C1	115.73 (17)	C9—C10—H10A	109.7
C5—C6—C7	121.37 (17)	C11—C10—H10B	109.7
C1—C6—C7	122.89 (16)	C9—C10—H10B	109.7
O1—C13—C12	121.19 (15)	H10A—C10—H10B	108.2

O1—C13—C8	121.32 (16)
C12—C13—C8	117.45 (14)
C8—C7—C6	124.63 (15)
C8—C7—H7	117.7
C6—C7—H7	117.7
C8—C9—C10	112.35 (15)
C8—C9—H9A	109.1
C10—C9—H9A	109.1
C8—C9—H9B	109.1
C10—C9—H9B	109.1
H9A—C9—H9B	107.9
C20—C15—C16	115.85 (16)
C20—C15—C14	122.20 (16)
C16—C15—C14	121.93 (16)
C4—C5—C6	122.88 (19)
C4—C5—Cl2	118.28 (15)
C6—C5—Cl2	118.83 (14)
C17—C18—C19	120.36 (18)
C17—C18—H18	119.8
C19—C18—H18	119.8
C7—C8—C13	116.69 (15)
C7—C8—C9	123.82 (15)
C13—C8—C9	119.47 (14)
C2—C1—C6	122.69 (18)
C2—C1—Cl1	118.25 (16)
C6—C1—Cl1	119.03 (14)
C17—C16—C15	122.94 (18)
C17—C16—Cl4	118.69 (15)
C18—C19—C20	119.04 (18)
C18—C19—H19	120.5
C20—C19—H19	120.5
C12—C11—C10	111.02 (15)
C12—C11—H11A	109.4
C10—C11—H11A	109.4
C12—C11—H11B	109.4
C10—C11—H11B	109.4
H11A—C11—H11B	108.0
C2—C3—C4	120.58 (18)
C2—C3—H3	119.7
C4—C3—H3	119.7
C12—C14—C15	123.77 (15)
C12—C14—H14	118.1
C15—C14—H14	118.1
C19—C20—C15	122.83 (18)
C19—C20—Cl3	118.41 (14)
C15—C20—Cl3	118.76 (13)
C16—C17—C18	118.97 (18)
C16—C17—H17	120.5
C18—C17—H17	120.5
C3—C4—C5	119.07 (19)
C3—C4—H4	120.5
C5—C4—H4	120.5
C3—C2—C1	119.02 (19)
C3—C2—H2	120.5
C1—C2—H2	120.5

C14—C12—C13—O1	−20.1 (3)
C11—C12—C13—O1	163.96 (17)
C14—C12—C13—C8	157.57 (16)
C11—C12—C13—C8	−18.4 (2)
C5—C6—C7—C8	−112.5 (2)
C1—C6—C7—C8	68.8 (3)
C1—C6—C5—C4	−2.0 (3)
C7—C6—C5—C4	179.24 (17)
C1—C6—C5—Cl2	179.18 (13)
C7—C6—C5—Cl2	0.4 (2)
C6—C7—C8—C13	−179.65 (16)
C6—C7—C8—C9	2.1 (3)
O1—C13—C8—C7	12.3 (2)
C12—C13—C8—C7	−165.39 (16)
O1—C13—C8—C9	−169.41 (17)
C12—C13—C8—C9	12.9 (2)
C10—C9—C8—C7	146.11 (18)
C10—C9—C8—C13	−32.1 (2)
C5—C6—C1—C2	0.9 (3)
C14—C15—C16—Cl4	−0.5 (2)
C8—C9—C10—C11	55.7 (2)
C17—C18—C19—C20	0.2 (3)
C14—C12—C11—C10	−132.96 (18)
C13—C12—C11—C10	42.8 (2)
C9—C10—C11—C12	−61.2 (2)
C13—C12—C14—C15	−173.89 (16)
C11—C12—C14—C15	1.8 (3)
C20—C15—C14—C12	−92.9 (2)
C16—C15—C14—C12	85.4 (2)
C18—C19—C20—C15	0.7 (3)
C18—C19—C20—Cl3	−179.49 (15)
C16—C15—C20—C19	−0.8 (3)
C14—C15—C20—C19	177.55 (17)
C16—C15—C20—Cl3	179.37 (13)
C14—C15—C20—Cl3	−2.3 (2)
C15—C16—C17—C18	0.8 (3)
Cl4—C16—C17—C18	−176.99 (15)
C19—C18—C17—C16	−0.9 (3)

C7—C6—C1—C2	179.69 (17)	C2—C3—C4—C5	0.2 (3)
C5—C6—C1—Cl1	−177.17 (13)	C6—C5—C4—C3	1.4 (3)
C7—C6—C1—Cl1	1.6 (2)	Cl2—C5—C4—C3	−179.70 (14)
C20—C15—C16—C17	0.1 (3)	C4—C3—C2—C1	−1.2 (3)
C14—C15—C16—C17	−178.32 (17)	C6—C1—C2—C3	0.6 (3)
C20—C15—C16—Cl4	177.86 (13)	Cl1—C1—C2—C3	178.73 (14)