



Crystal structure of (*E*)-2-(4-chlorobenzylidene)-3,4-dihydronaphthalen-1(2*H*)-one: a second monoclinic polymorph

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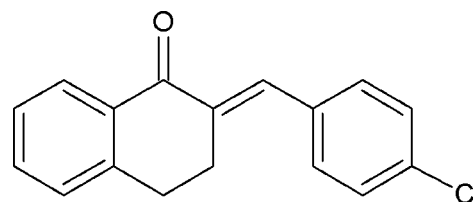
The title compound, C₁₇H₁₃ClO, is the second monoclinic polymorph to crystallize in the space group *P*2₁/*c*. The first polymorph crystallized with two independent molecules in the asymmetric unit [Bolognesi *et al.* (1975). *Acta Cryst.* **A31**, S119; *Z'* = 2; no atomic coordinates available], whereas the title compound has *Z'* = 1. In the title polymorph, the dihedral angle between the plane of the benzene ring of the tetralone moiety and that of the 4-chlorobenzyl ring is 52.21 (11)°. The cyclohex-2-en-1-one ring of the tetralone moiety has a screw-boat conformation. In the crystal, molecules are linked by pairs of C—H···π interactions forming inversion dimers. There are no other significant intermolecular interactions present.

Keywords: crystal structure; α-tetralone; C—H···π interactions.

CCDC reference: 1421217

1. Related literature

For a brief description of the first monoclinic polymorph of the title compound, see: Bolognesi *et al.* (1975). For the crystal structures of related compounds, see: Asiri *et al.* (2012); Dimmock *et al.* (2002); Oloo *et al.* (2002). For the synthesis, see: Kerbal *et al.* (1988).



2. Experimental

2.1. Crystal data

C ₁₇ H ₁₃ ClO	<i>V</i> = 1352.51 (14) Å ³
<i>M_r</i> = 268.72	<i>Z</i> = 4
Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Mo <i>K</i> α radiation
<i>a</i> = 13.3791 (8) Å	<i>μ</i> = 0.27 mm ⁻¹
<i>b</i> = 14.9352 (10) Å	<i>T</i> = 296 K
<i>c</i> = 6.7849 (3) Å	0.38 × 0.30 × 0.26 mm
<i>β</i> = 93.968 (3)°	

2.2. Data collection

Bruker Kappa APEXII CCD diffractometer	11640 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2007)	2954 independent reflections
<i>T</i> _{min} = 0.906, <i>T</i> _{max} = 0.930	1901 reflections with <i>I</i> > 2σ(<i>I</i>)
	<i>R</i> _{int} = 0.029

2.3. Refinement

<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.051	172 parameters
<i>wR</i> (<i>F</i> ²) = 0.170	H-atom parameters constrained
<i>S</i> = 1.05	Δρ _{max} = 0.37 e Å ⁻³
2954 reflections	Δρ _{min} = -0.19 e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C2–C7 ring.

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C13—H13···Cg1 ¹	0.93	2.86	3.552 (2)	132

Symmetry code: (i) $-x, -y, -z$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON*.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5197).

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supporting information

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Crystal structure of (*E*)-2-(4-chlorobenzylidene)-3,4-dihydronaphthalen-1(2*H*)-one: a second monoclinic polymorph

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S1. Comments

The crystal structure of 2-(*p*-chlorobenzylidene)-tetral-1-one (Bolognesi *et al.*, 1975) is the first monoclinic polymorph of the title compound, however no atomic coordinates were reported. The crystal structures of the related structures 2-(2,4-dichlorophenylmethylene)-1-tetralone (Oloo *et al.*, 2002), 2-[(*E*)-4-methoxybenzylidene]-1,2,3,4-tetrahydronaphthalen-1-one (Asiri *et al.*, 2012), and 2-(3,4-dichlorophenylmethylene)-1-tetralone (Dimmock *et al.*, 2002) have been published.

The molecular structure of the title polymorph is illustrated in Fig. 1. The benzene ring (C2–C7) of tetralone (systematic name: 3,4-dihydronaphthalen-1(2*H*)-one) and the mean plane of part of the 4-chlorobenzylidene (C11–C17/C11) moiety are inclined to one another by 52.03 (6)°. The cyclohex-2-en-1-one ring, (C1/C2/C7–C10), has puckering amplitude (Q) = 0.471 (2) Å, and $\theta = 65.6$ (2)° and $\varphi = 210.5$ (3)°, and can be described as having a screw-boat conformation.

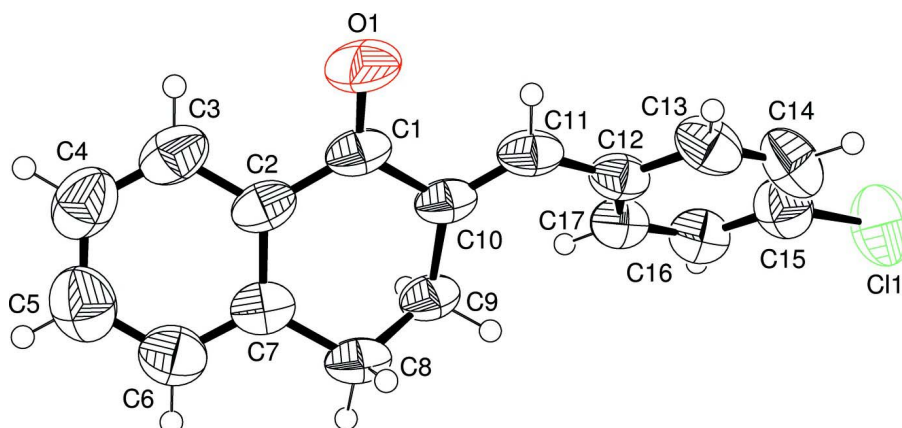
In the crystal, molecules are linked by pairs of C—H \cdots π interactions forming inversion dimers (Table 1). There are no other significant intermolecular interactions present.

S2. Synthesis and crystallization

The synthesis of the title compound was carried out following a published procedure (Kerbal *et al.*, 1988), *viz.* by a condensation of equimolar amounts of 4-chlorobenzaldehyde and α -tetralone using sodium hydroxide in methanol (yield; 87%; m.p.: 426–428 K). The synthesized compound was crystallized in tetrahydrofuran under slow evaporation yielding light-orange prismatic crystals.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H-atoms were positioned geometrically (C–H = 0.93–0.97 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

View of the molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

2-(4-Chlorobenzylidene)-3,4-dihydronaphthalen-1(2H)-one

Crystal data

$C_{17}H_{13}ClO$

$M_r = 268.72$

Monoclinic, $P2_1/c$

$a = 13.3791$ (8) Å

$b = 14.9352$ (10) Å

$c = 6.7849$ (3) Å

$\beta = 93.968$ (3)°

$V = 1352.51$ (14) Å³

$Z = 4$

$F(000) = 560$

$D_x = 1.320$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1901 reflections

$\theta = 2.7$ – 27.0 °

$\mu = 0.27$ mm⁻¹

$T = 296$ K

Prism, light orange

$0.38 \times 0.30 \times 0.26$ mm

Data collection

Bruker Kappa APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 7.70 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2007)

$T_{\min} = 0.906$, $T_{\max} = 0.930$

11640 measured reflections

2954 independent reflections

1901 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 27.0$ °, $\theta_{\min} = 2.7$ °

$h = -17 \rightarrow 17$

$k = -19 \rightarrow 18$

$l = -8 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.170$

$S = 1.05$

2954 reflections

172 parameters

0 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.0931P)^2 + 0.1261P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.37$ e Å⁻³

$\Delta\rho_{\min} = -0.18$ 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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	−0.45111 (6)	0.12595 (6)	0.64763 (13)	0.1115 (4)
O1	0.01032 (15)	0.14379 (12)	−0.1910 (2)	0.0905 (6)
C1	0.04921 (18)	0.13078 (12)	−0.0242 (3)	0.0592 (5)
C2	0.15886 (18)	0.13063 (12)	0.0132 (3)	0.0594 (5)
C3	0.2190 (2)	0.16509 (15)	−0.1286 (4)	0.0758 (7)
H3	0.1893	0.1867	−0.2471	0.091*
C4	0.3206 (2)	0.16757 (17)	−0.0960 (5)	0.0903 (8)
H4	0.3597	0.1910	−0.1918	0.108*
C5	0.3657 (2)	0.13533 (18)	0.0791 (5)	0.0947 (9)
H5	0.4350	0.1373	0.1021	0.114*
C6	0.3064 (2)	0.09969 (16)	0.2216 (4)	0.0767 (7)
H6	0.3369	0.0774	0.3389	0.092*
C7	0.20322 (18)	0.09703 (13)	0.1913 (3)	0.0597 (5)
C8	0.13788 (16)	0.05948 (14)	0.3412 (3)	0.0638 (6)
H8A	0.1739	0.0605	0.4703	0.077*
H8B	0.1220	−0.0024	0.3086	0.077*
C9	0.04115 (17)	0.11253 (13)	0.3489 (3)	0.0588 (5)
H9A	−0.0018	0.0835	0.4390	0.071*
H9B	0.0564	0.1721	0.3992	0.071*
C10	−0.01328 (17)	0.11966 (12)	0.1487 (3)	0.0550 (5)
C11	−0.11281 (18)	0.11818 (12)	0.1126 (3)	0.0599 (6)
H11	−0.1350	0.1175	−0.0203	0.072*
C12	−0.19169 (17)	0.11744 (12)	0.2505 (3)	0.0572 (5)
C13	−0.28306 (18)	0.07683 (15)	0.1952 (3)	0.0736 (6)
H13	−0.2913	0.0487	0.0729	0.088*
C14	−0.36166 (18)	0.07709 (18)	0.3162 (4)	0.0859 (8)
H14	−0.4212	0.0477	0.2782	0.103*
C15	−0.3506 (2)	0.12143 (16)	0.4938 (4)	0.0779 (7)
C16	−0.26219 (17)	0.16368 (14)	0.5526 (3)	0.0689 (6)
H16	−0.2559	0.1942	0.6723	0.083*
C17	−0.18321 (17)	0.16064 (13)	0.4338 (3)	0.0614 (5)
H17	−0.1229	0.1878	0.4760	0.074*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0757 (5)	0.1284 (7)	0.1334 (8)	0.0051 (4)	0.0296 (5)	-0.0236 (5)
O1	0.1112 (14)	0.1190 (15)	0.0393 (8)	-0.0036 (11)	-0.0092 (8)	0.0101 (8)
C1	0.0875 (16)	0.0509 (11)	0.0383 (10)	-0.0005 (10)	-0.0030 (9)	0.0017 (8)
C2	0.0872 (15)	0.0442 (10)	0.0469 (10)	0.0026 (9)	0.0066 (10)	0.0004 (8)
C3	0.103 (2)	0.0587 (13)	0.0674 (14)	0.0112 (12)	0.0194 (13)	0.0091 (10)
C4	0.103 (2)	0.0666 (15)	0.105 (2)	0.0074 (14)	0.0359 (16)	0.0120 (14)
C5	0.0834 (19)	0.0756 (17)	0.126 (3)	0.0051 (14)	0.0139 (18)	-0.0056 (16)
C6	0.0839 (18)	0.0650 (14)	0.0801 (16)	0.0072 (12)	-0.0013 (13)	-0.0022 (12)
C7	0.0798 (15)	0.0456 (10)	0.0529 (11)	0.0036 (9)	-0.0008 (10)	-0.0052 (8)
C8	0.0849 (15)	0.0601 (12)	0.0446 (10)	0.0039 (10)	-0.0093 (9)	0.0068 (9)
C9	0.0785 (14)	0.0581 (11)	0.0387 (9)	-0.0024 (10)	-0.0037 (9)	0.0037 (8)
C10	0.0790 (15)	0.0441 (10)	0.0404 (10)	-0.0013 (9)	-0.0070 (9)	0.0016 (7)
C11	0.0843 (16)	0.0474 (11)	0.0453 (10)	0.0016 (9)	-0.0137 (10)	-0.0023 (8)
C12	0.0691 (13)	0.0440 (10)	0.0563 (11)	0.0052 (8)	-0.0109 (9)	-0.0017 (8)
C13	0.0747 (15)	0.0690 (14)	0.0740 (14)	0.0063 (12)	-0.0172 (12)	-0.0173 (11)
C14	0.0631 (15)	0.0827 (17)	0.109 (2)	0.0007 (12)	-0.0133 (14)	-0.0218 (15)
C15	0.0698 (16)	0.0701 (15)	0.0940 (18)	0.0099 (11)	0.0064 (13)	-0.0040 (13)
C16	0.0787 (16)	0.0575 (12)	0.0696 (13)	0.0069 (11)	-0.0009 (11)	-0.0084 (10)
C17	0.0744 (14)	0.0477 (11)	0.0607 (12)	-0.0011 (9)	-0.0068 (10)	-0.0051 (9)

Geometric parameters (\AA , $^\circ$)

C11—C15	1.759 (3)	C8—H8B	0.9700
O1—C1	1.227 (2)	C9—C10	1.500 (3)
C1—C2	1.471 (3)	C9—H9A	0.9700
C1—C10	1.496 (3)	C9—H9B	0.9700
C2—C3	1.394 (3)	C10—C11	1.337 (3)
C2—C7	1.402 (3)	C11—C12	1.458 (3)
C3—C4	1.363 (4)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.393 (3)
C4—C5	1.382 (4)	C12—C17	1.399 (3)
C4—H4	0.9300	C13—C14	1.378 (3)
C5—C6	1.398 (4)	C13—H13	0.9300
C5—H5	0.9300	C14—C15	1.374 (4)
C6—C7	1.383 (3)	C14—H14	0.9300
C6—H6	0.9300	C15—C16	1.376 (3)
C7—C8	1.495 (3)	C16—C17	1.373 (3)
C8—C9	1.521 (3)	C16—H16	0.9300
C8—H8A	0.9700	C17—H17	0.9300
O1—C1—C2	120.87 (19)	C8—C9—H9A	109.3
O1—C1—C10	121.1 (2)	C10—C9—H9B	109.3
C2—C1—C10	117.97 (16)	C8—C9—H9B	109.3
C3—C2—C7	119.8 (2)	H9A—C9—H9B	108.0
C3—C2—C1	119.8 (2)	C11—C10—C1	117.49 (17)

C7—C2—C1	120.46 (18)	C11—C10—C9	125.41 (19)
C4—C3—C2	121.0 (2)	C1—C10—C9	117.09 (19)
C4—C3—H3	119.5	C10—C11—C12	129.69 (18)
C2—C3—H3	119.5	C10—C11—H11	115.2
C3—C4—C5	120.1 (3)	C12—C11—H11	115.2
C3—C4—H4	120.0	C13—C12—C17	117.1 (2)
C5—C4—H4	120.0	C13—C12—C11	119.44 (18)
C4—C5—C6	119.5 (3)	C17—C12—C11	123.31 (19)
C4—C5—H5	120.2	C14—C13—C12	122.0 (2)
C6—C5—H5	120.2	C14—C13—H13	119.0
C7—C6—C5	121.1 (2)	C12—C13—H13	119.0
C7—C6—H6	119.4	C15—C14—C13	119.0 (2)
C5—C6—H6	119.4	C15—C14—H14	120.5
C6—C7—C2	118.5 (2)	C13—C14—H14	120.5
C6—C7—C8	122.29 (19)	C14—C15—C16	120.9 (2)
C2—C7—C8	119.2 (2)	C14—C15—C11	119.9 (2)
C7—C8—C9	111.51 (16)	C16—C15—C11	119.2 (2)
C7—C8—H8A	109.3	C17—C16—C15	119.8 (2)
C9—C8—H8A	109.3	C17—C16—H16	120.1
C7—C8—H8B	109.3	C15—C16—H16	120.1
C9—C8—H8B	109.3	C16—C17—C12	121.2 (2)
H8A—C8—H8B	108.0	C16—C17—H17	119.4
C10—C9—C8	111.52 (17)	C12—C17—H17	119.4
C10—C9—H9A	109.3		
O1—C1—C2—C3	14.6 (3)	C2—C1—C10—C11	-178.76 (17)
C10—C1—C2—C3	-162.05 (18)	O1—C1—C10—C9	-174.51 (19)
O1—C1—C2—C7	-166.10 (19)	C2—C1—C10—C9	2.2 (2)
C10—C1—C2—C7	17.2 (3)	C8—C9—C10—C11	143.7 (2)
C7—C2—C3—C4	-0.7 (3)	C8—C9—C10—C1	-37.3 (2)
C1—C2—C3—C4	178.6 (2)	C1—C10—C11—C12	-173.08 (18)
C2—C3—C4—C5	0.3 (4)	C9—C10—C11—C12	5.9 (3)
C3—C4—C5—C6	0.4 (4)	C10—C11—C12—C13	-151.1 (2)
C4—C5—C6—C7	-0.7 (4)	C10—C11—C12—C17	33.0 (3)
C5—C6—C7—C2	0.3 (3)	C17—C12—C13—C14	-1.3 (3)
C5—C6—C7—C8	-179.9 (2)	C11—C12—C13—C14	-177.4 (2)
C3—C2—C7—C6	0.4 (3)	C12—C13—C14—C15	2.3 (4)
C1—C2—C7—C6	-178.84 (19)	C13—C14—C15—C16	-1.1 (4)
C3—C2—C7—C8	-179.42 (18)	C13—C14—C15—C11	177.9 (2)
C1—C2—C7—C8	1.3 (3)	C14—C15—C16—C17	-0.9 (4)
C6—C7—C8—C9	142.9 (2)	C11—C15—C16—C17	180.00 (17)
C2—C7—C8—C9	-37.3 (3)	C15—C16—C17—C12	1.9 (3)
C7—C8—C9—C10	54.1 (2)	C13—C12—C17—C16	-0.8 (3)
O1—C1—C10—C11	4.6 (3)	C11—C12—C17—C16	175.17 (19)

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 is the centroid of the C2–C7 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C13—H13 \cdots Cg1 ⁱ	0.93	2.86	3.552 (2)	132

Symmetry code: (i) $-x, -y, -z$.