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Crystal structure of (*E*)-4-ethyl-2-(4-methoxybenzylidene)-3,4-dihydronaphthalen-1(2*H*)-one

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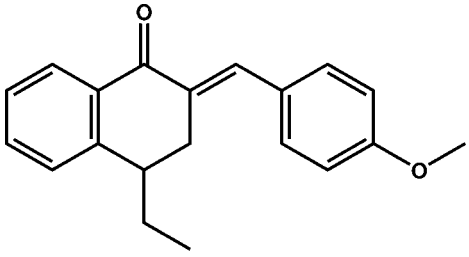
In the title compound, $C_{20}H_{20}O_2$, the exocyclic C=C double bond has an *E* conformation. The ethyl substituent on the cyclohexanone ring is in an axial orientation. The cyclohexanone ring adopts a screw-boat conformation, with the methylene C atom and the C atom bearing the 4-methoxybenzylidene group displaced from the other atoms by 0.812 (1) and 0.334 (1) Å, respectively. The dihedral angle between the planes of the benzene rings is 42.20 (8)°. In the crystal, no directional interactions beyond van der Waals contacts are observed.

Keywords: crystal structure; benzylidene; naphthalenone; dipolar 1,3-cycloaddition reactions.

CCDC reference: 1402624

1. Related literature

For general background to dipolar 1,3-cycloaddition reactions, see: Bennani *et al.* (2007); Kerbal *et al.* (1988); Al Houari *et al.* (2008). For a related structure, see: Akhazzane *et al.* (2010).



2. Experimental

2.1. Crystal data

$C_{20}H_{20}O_2$
M_r = 292.36
Monoclinic, $P2_1/c$
a = 12.0411 (13) Å
b = 8.9698 (9) Å
c = 15.5832 (18) Å
β = 110.721 (3)°
V = 1574.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 K
0.38 × 0.16 × 0.12 mm

2.2. Data collection

Bruker X8 APEX CCD diffractometer
25378 measured reflections
4068 independent reflections
2552 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.129
S = 1.01
4068 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7434).

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supporting information

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Crystal structure of (*E*)-4-ethyl-2-(4-methoxybenzylidene)-3,4-dihydronaphthalen-1(2*H*)-one

Mohamed Akhazzane, Ghali Al Houari, Mohamed El Yazidi, Mohamed Saadi and Lahcen El Ammari

S1. Comment

Knowledge of the configuration and conformation of the title compound is necessary to understand its behaviour in dipolar-1,3 cycloaddition reactions (Bennani *et al.*, 2007; Al Houari *et al.*, 2008). To confirm the (E) conformation of the exocyclic C=C double bond, an X-ray crystal structure determination has been carried out. The present work is a continuation of the investigation of the dihydronaphthalene derivatives published recently by Akhazzane *et al.*, 2010.

The molecule of the title compound is formed by two fused rings linked to an ethyl group and to a 4-methoxybenzylidene moities as shown in Fig.1. The cyclohexanone ring adopts a screw-boat conformation as indicated by the total puckering amplitude QT = 0.477 (2) Å and spherical polar angle θ = 115.9 (2)° with φ = 35.6 (2)°. The benzene rings form a dihedral angle of 42.20 (8)°.

S2. Experimental

The synthesis of the title compound was achieved using the method reported by Kerbal *et al.*, 1988. By a condensation of *para* anisaldehyde with 4-ethyl-3,4-dihydronaphthalen- 1(2*H*)-one in an alkaline medium in ethanol. The resulting residue was recrystallized from ethanol solution by slow evaporation to afford the title compound as colourless needles.

S3. Refinement

H atoms were located in a difference map and treated as riding with C–H = 0.96 Å, C–H = 0.97 Å, and C–H = 0.93 Å for methyl, methylene and aromatic, respectively. All hydrogen with U_{iso}(H) = 1.2 U_{eq} for methylene, aromatic and U_{iso}(H) = 1.5 U_{eq} for methyl.

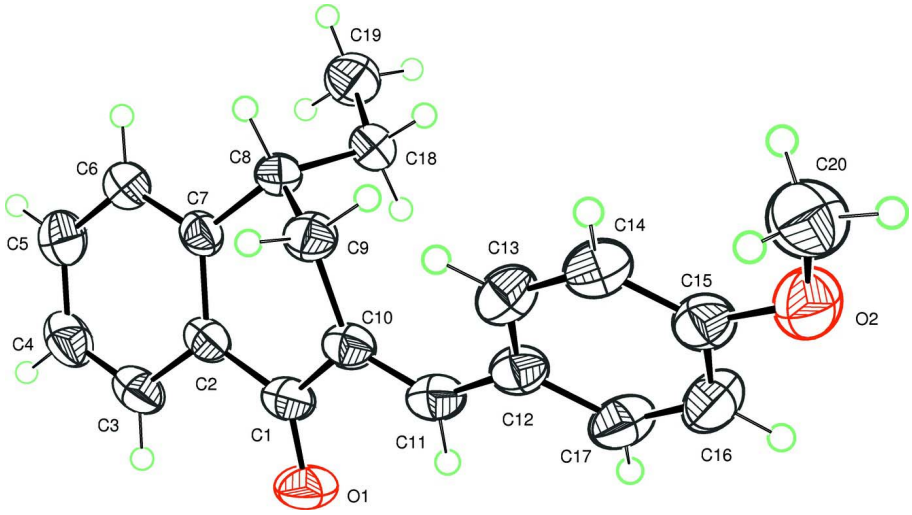

Figure 1

Plot of the molecule of the title compound with displacement ellipsoids drawn at the 50% probability level.

(*E*)-4-Ethyl-2-(4-methoxybenzylidene)-3,4-dihydronaphthalen-1(2*H*)-\ one

Crystal data

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β = 110.721 (3)°
V = 1574.2 (3) Å^3
Z = 4
$F(000)$ = 624
D_x = 1.234 Mg m^{-3}
Melting point: 383 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4068 reflections
θ = 2.7–28.7°
μ = 0.08 mm^{-1}
T = 296 K
Needles, colourless
0.38 × 0.16 × 0.12 mm

Data collection

Bruker X8 APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
25378 measured reflections
4068 independent reflections
2552 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 28.7°, θ_{min} = 2.7°
h = −14→16
k = −12→12
l = −20→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.129
S = 1.01
4068 reflections
200 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.2497P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL2013* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0088 (16)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.64653 (13)	0.60708 (16)	0.83826 (9)	0.0452 (3)
C2	0.52292 (13)	0.64122 (14)	0.83030 (9)	0.0415 (3)
C3	0.49026 (15)	0.62928 (17)	0.90761 (10)	0.0520 (4)
H3	0.5468	0.6017	0.9636	0.062*
C4	0.37607 (17)	0.65769 (19)	0.90212 (11)	0.0614 (4)
H4	0.3552	0.6486	0.9539	0.074*
C5	0.29205 (16)	0.6999 (2)	0.81910 (12)	0.0621 (4)
H5	0.2144	0.7191	0.8150	0.075*
C6	0.32321 (14)	0.71366 (17)	0.74230 (10)	0.0514 (4)
H6	0.2662	0.7429	0.6870	0.062*
C7	0.43793 (12)	0.68471 (14)	0.74609 (9)	0.0405 (3)
C8	0.47260 (12)	0.69755 (15)	0.66233 (9)	0.0415 (3)
H8	0.4205	0.7717	0.6213	0.050*
C9	0.60029 (13)	0.75335 (16)	0.68939 (10)	0.0466 (4)
H9A	0.6231	0.7542	0.6356	0.056*
H9B	0.6047	0.8549	0.7118	0.056*
C10	0.68618 (12)	0.65723 (15)	0.76252 (9)	0.0432 (3)
C11	0.79269 (12)	0.60980 (17)	0.76459 (10)	0.0485 (4)
H11	0.8315	0.5479	0.8140	0.058*
C12	0.85911 (12)	0.63703 (16)	0.70394 (10)	0.0464 (3)
C13	0.84423 (13)	0.75855 (17)	0.64532 (11)	0.0521 (4)
H13	0.7884	0.8310	0.6440	0.063*
C14	0.91024 (13)	0.77445 (17)	0.58897 (11)	0.0530 (4)
H14	0.8978	0.8561	0.5499	0.064*
C15	0.99443 (13)	0.66918 (18)	0.59086 (11)	0.0525 (4)
C16	1.01364 (15)	0.5491 (2)	0.65050 (12)	0.0643 (5)
H16	1.0715	0.4788	0.6531	0.077*
C17	0.94721 (14)	0.53437 (19)	0.70550 (12)	0.0602 (4)
H17	0.9612	0.4534	0.7452	0.072*
C18	0.45730 (13)	0.55031 (17)	0.60935 (10)	0.0509 (4)
H18A	0.4894	0.5627	0.5608	0.061*
H18B	0.5042	0.4744	0.6508	0.061*
C19	0.33144 (15)	0.4944 (2)	0.56723 (12)	0.0692 (5)
H19A	0.3303	0.4052	0.5329	0.104*
H19C	0.2833	0.5692	0.5271	0.104*
H19B	0.3006	0.4732	0.6149	0.104*
O2	1.06414 (11)	0.67254 (15)	0.53835 (9)	0.0708 (4)
C20	1.05297 (19)	0.7950 (2)	0.47844 (14)	0.0811 (6)
H20A	1.1071	0.7828	0.4462	0.122*

H20B	1.0711	0.8857	0.5134	0.122*
H20C	0.9731	0.7995	0.4352	0.122*
O1	0.71294 (10)	0.54013 (13)	0.90568 (7)	0.0630 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0520 (8)	0.0409 (7)	0.0352 (7)	−0.0027 (6)	0.0061 (6)	0.0005 (6)
C2	0.0554 (8)	0.0349 (7)	0.0328 (7)	−0.0029 (6)	0.0138 (6)	−0.0014 (5)
C3	0.0705 (11)	0.0487 (8)	0.0354 (7)	−0.0050 (7)	0.0167 (7)	−0.0013 (6)
C4	0.0812 (12)	0.0662 (11)	0.0471 (9)	−0.0006 (9)	0.0356 (9)	−0.0030 (8)
C5	0.0641 (10)	0.0721 (11)	0.0578 (10)	0.0081 (8)	0.0311 (9)	−0.0022 (9)
C6	0.0540 (9)	0.0578 (9)	0.0428 (8)	0.0075 (7)	0.0179 (7)	0.0000 (7)
C7	0.0511 (8)	0.0347 (7)	0.0354 (7)	0.0002 (6)	0.0150 (6)	−0.0028 (6)
C8	0.0475 (8)	0.0427 (7)	0.0324 (7)	0.0081 (6)	0.0119 (6)	0.0052 (6)
C9	0.0515 (8)	0.0464 (8)	0.0421 (8)	0.0026 (6)	0.0170 (7)	0.0080 (6)
C10	0.0461 (8)	0.0407 (7)	0.0377 (7)	−0.0043 (6)	0.0086 (6)	−0.0005 (6)
C11	0.0450 (8)	0.0477 (8)	0.0435 (8)	−0.0032 (6)	0.0043 (6)	0.0020 (6)
C12	0.0367 (7)	0.0495 (8)	0.0461 (8)	−0.0051 (6)	0.0060 (6)	−0.0018 (7)
C13	0.0423 (8)	0.0444 (8)	0.0663 (10)	−0.0017 (6)	0.0151 (7)	0.0018 (7)
C14	0.0443 (8)	0.0502 (9)	0.0592 (9)	−0.0053 (7)	0.0119 (7)	0.0068 (7)
C15	0.0413 (8)	0.0611 (9)	0.0507 (9)	−0.0044 (7)	0.0110 (7)	−0.0033 (7)
C16	0.0520 (10)	0.0674 (11)	0.0740 (11)	0.0157 (8)	0.0227 (9)	0.0117 (9)
C17	0.0490 (9)	0.0637 (10)	0.0617 (10)	0.0096 (7)	0.0120 (8)	0.0167 (8)
C18	0.0550 (9)	0.0558 (9)	0.0396 (8)	0.0073 (7)	0.0138 (7)	−0.0057 (7)
C19	0.0635 (11)	0.0683 (11)	0.0614 (10)	0.0017 (9)	0.0042 (9)	−0.0173 (9)
O2	0.0642 (7)	0.0829 (9)	0.0717 (8)	0.0064 (6)	0.0320 (6)	0.0094 (7)
C20	0.0813 (14)	0.0945 (15)	0.0748 (13)	−0.0010 (11)	0.0367 (11)	0.0165 (11)
O1	0.0627 (7)	0.0745 (8)	0.0423 (6)	0.0079 (6)	0.0068 (5)	0.0166 (5)

Geometric parameters (Å, º)

C1—O1	1.2285 (16)	C11—H11	0.9300
C1—C2	1.481 (2)	C12—C13	1.392 (2)
C1—C10	1.491 (2)	C12—C17	1.398 (2)
C2—C3	1.3975 (19)	C13—C14	1.385 (2)
C2—C7	1.4040 (19)	C13—H13	0.9300
C3—C4	1.371 (2)	C14—C15	1.378 (2)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.384 (2)	C15—O2	1.3644 (19)
C4—H4	0.9300	C15—C16	1.387 (2)
C5—C6	1.381 (2)	C16—C17	1.370 (2)
C5—H5	0.9300	C16—H16	0.9300
C6—C7	1.386 (2)	C17—H17	0.9300
C6—H6	0.9300	C18—C19	1.508 (2)
C7—C8	1.5089 (18)	C18—H18A	0.9700
C8—C9	1.528 (2)	C18—H18B	0.9700
C8—C18	1.5337 (19)	C19—H19A	0.9600

C8—H8	0.9800	C19—H19C	0.9600
C9—C10	1.5092 (19)	C19—H19B	0.9600
C9—H9A	0.9700	O2—C20	1.417 (2)
C9—H9B	0.9700	C20—H20A	0.9600
C10—C11	1.341 (2)	C20—H20B	0.9600
C11—C12	1.459 (2)	C20—H20C	0.9600
O1—C1—C2	120.23 (13)	C12—C11—H11	114.0
O1—C1—C10	122.05 (14)	C13—C12—C17	116.60 (14)
C2—C1—C10	117.72 (12)	C13—C12—C11	125.69 (14)
C3—C2—C7	119.51 (14)	C17—C12—C11	117.70 (14)
C3—C2—C1	119.53 (13)	C14—C13—C12	121.82 (15)
C7—C2—C1	120.95 (12)	C14—C13—H13	119.1
C4—C3—C2	120.93 (14)	C12—C13—H13	119.1
C4—C3—H3	119.5	C15—C14—C13	119.92 (15)
C2—C3—H3	119.5	C15—C14—H14	120.0
C3—C4—C5	119.65 (15)	C13—C14—H14	120.0
C3—C4—H4	120.2	O2—C15—C14	125.17 (15)
C5—C4—H4	120.2	O2—C15—C16	115.27 (15)
C6—C5—C4	120.12 (16)	C14—C15—C16	119.56 (15)
C6—C5—H5	119.9	C17—C16—C15	119.89 (15)
C4—C5—H5	119.9	C17—C16—H16	120.1
C5—C6—C7	121.25 (15)	C15—C16—H16	120.1
C5—C6—H6	119.4	C16—C17—C12	122.17 (15)
C7—C6—H6	119.4	C16—C17—H17	118.9
C6—C7—C2	118.54 (13)	C12—C17—H17	118.9
C6—C7—C8	121.70 (12)	C19—C18—C8	115.56 (13)
C2—C7—C8	119.76 (13)	C19—C18—H18A	108.4
C7—C8—C9	110.23 (11)	C8—C18—H18A	108.4
C7—C8—C18	112.49 (12)	C19—C18—H18B	108.4
C9—C8—C18	110.38 (11)	C8—C18—H18B	108.4
C7—C8—H8	107.9	H18A—C18—H18B	107.5
C9—C8—H8	107.9	C18—C19—H19A	109.5
C18—C8—H8	107.9	C18—C19—H19C	109.5
C10—C9—C8	112.04 (11)	H19A—C19—H19C	109.5
C10—C9—H9A	109.2	C18—C19—H19B	109.5
C8—C9—H9A	109.2	H19A—C19—H19B	109.5
C10—C9—H9B	109.2	H19C—C19—H19B	109.5
C8—C9—H9B	109.2	C15—O2—C20	118.51 (14)
H9A—C9—H9B	107.9	O2—C20—H20A	109.5
C11—C10—C1	117.18 (13)	O2—C20—H20B	109.5
C11—C10—C9	126.41 (13)	H20A—C20—H20B	109.5
C1—C10—C9	116.38 (12)	O2—C20—H20C	109.5
C10—C11—C12	132.10 (14)	H20A—C20—H20C	109.5
C10—C11—H11	114.0	H20B—C20—H20C	109.5