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(2*E*)-2-[4-(Dimethylamino)benzylidene]-5-methylcyclohexanone

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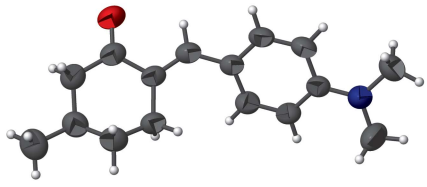
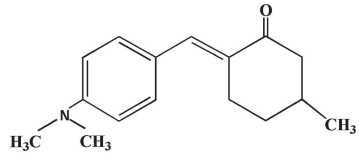
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{16}H_{21}NO$, the cyclohexanone ring adopts a half-chair conformation; the dihedral angle between this ring (all atoms) and the benzene ring is 41.74 (16)°. No directional interactions could be identified in the crystal.

3D view



Chemical scheme



Structure description

Cyclohexanone derivatives have various applications as drugs (*e.g.* Chen *et al.*, 2010). As part our studies in this area, we now describe the synthesis *via* a Claissen–Schmidt condensation and crystal structure of the title compound (Fig. 1). In the arbitrarily chosen asymmetric molecule, C13 has an *R* configuration, but symmetry generates a racemic mixture in the crystal.

The geometric parameters for the title compound are comparable with the corresponding values for similar reported structures (*e.g.* Shalini *et al.*, 2013). The cyclohexanone ring adopts a half-chair conformation, with C10/C11/C14/C15 roughly coplanar (r.m.s. deviation = 0.075 Å) and C12 and C13 deviating by 0.465 (5) and −0.234 (4) Å, respectively, from the other atoms. The dihedral angle between the cyclohexanone ring (all atoms) and the benzene ring is 41.74 (15)°. The N1/C1/C2 dimethylamino group is almost coplanar with its attached benzene ring [dihedral angle = 2.6 (4)°] and the bond-angle sum at the nitrogen atom of 359.8° clearly indicates sp^2 hybridization.

No directional interactions could be identified in the crystal (Fig. 2) and van der Waals forces must be responsible for crystal cohesion.

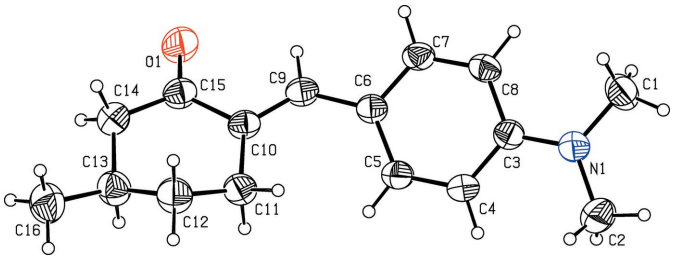


Figure 1
The molecular structure, with displacement ellipsoids drawn at the 30% probability level.

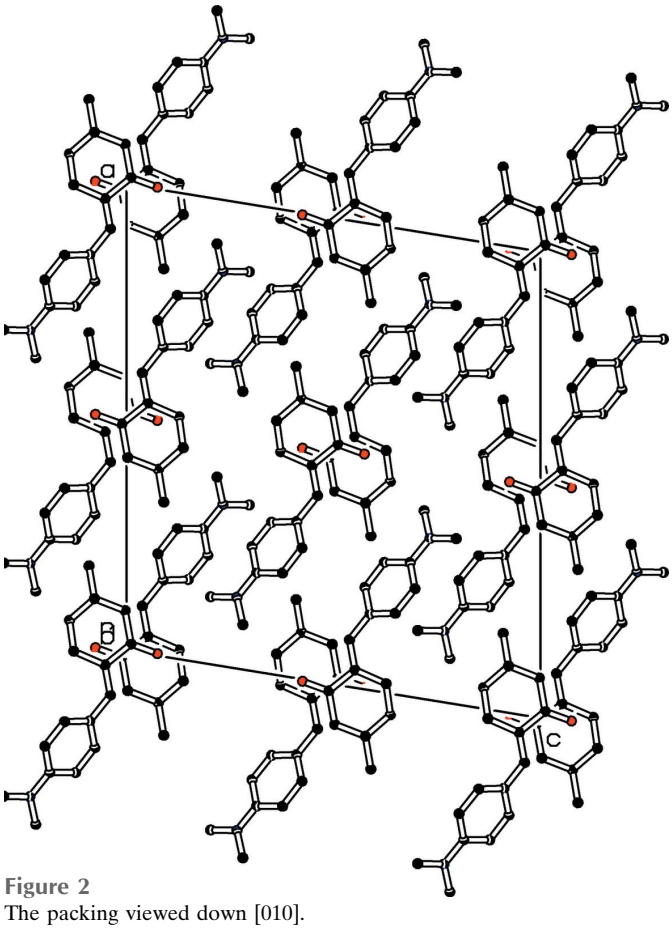


Figure 2
The packing viewed down [010].

Synthesis and crystallization

An aqueous solution of NaOH (10%, 10 ml) was added to a solution of 3-methylcyclohexanone (0.02 mol) and 4-*N*,*N*-methylaminobenzaldehyde (0.02 mol) in absolute ethanol (40 ml). The reaction mixture was stirred for 2 h and was left overnight. On addition of ice-cold water, a dark-yellow solid was obtained, which was filtered, washed with ice-cold water and dried. The product was recrystallized from ethyl acetate solution to yield yellow blocks after seven days (yield: 87%, m.p. 70°C).

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{21}NO$
M_r	243.34
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	296
a, *b*, *c* (Å)	20.3655 (16), 7.6148 (7), 18.2999 (16)
β (°)	99.254 (3)
V (Å^3)	2801.0 (4)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.07
Crystal size (mm)	0.30 × 0.25 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.979, 0.986
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19385, 2462, 1528
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.073, 0.218, 1.07
No. of reflections	2458
No. of parameters	167
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.19

Computer programs: *APEX2* (Bruker, 2004), *APEX2* and *SAINT* (Bruker, 2004), *SAINT* and *XPREP* (Bruker, 2004), *SIR92* (Altomare *et al.*, 1993), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x171706 [https://doi.org/10.1107/S2414314617017060]

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Crystal data

$C_{16}H_{21}NO$
M_r = 243.34
Monoclinic, *C*2/*c*
a = 20.3655 (16) Å
b = 7.6148 (7) Å
c = 18.2999 (16) Å
β = 99.254 (3)°
V = 2801.0 (4) Å^3
Z = 8
F(000) = 1056
D_x = 1.154 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 6070 reflections
θ = 2.8–25.5°
μ = 0.07 mm^{-1}
T = 296 K
Block, yellow
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.979, T_{max} = 0.986
19385 measured reflections
2462 independent reflections
1528 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.0°, θ_{min} = 2.3°
h = −24→22
k = −9→9
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.218
S = 1.07
2458 reflections
167 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0719P)^2 + 5.4525P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0022 (6)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Hydrogen atoms were positioned geometrically and treated as riding on their parent atoms and refined with C—H distances of 0.93–0.97 Å, with U_{iso}(H)= 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(methyl C).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.40379 (18)	0.7797 (6)	0.2188 (2)	0.0897 (12)
H1A	0.4390	0.7688	0.2603	0.135*
H1B	0.3919	0.9012	0.2115	0.135*
H1C	0.4185	0.7348	0.1752	0.135*
C2	0.3551 (2)	0.5670 (6)	0.2965 (2)	0.0978 (13)
H2A	0.3988	0.5828	0.3246	0.147*
H2B	0.3498	0.4472	0.2803	0.147*
H2C	0.3223	0.5946	0.3269	0.147*
C3	0.28784 (15)	0.6857 (4)	0.18527 (16)	0.0596 (8)
C4	0.23324 (16)	0.5821 (4)	0.19661 (18)	0.0659 (9)
H4	0.2375	0.5059	0.2368	0.079*
C5	0.17401 (16)	0.5914 (4)	0.14940 (17)	0.0658 (9)
H5	0.1392	0.5197	0.1582	0.079*
C6	0.16364 (15)	0.7044 (4)	0.08843 (16)	0.0578 (8)
C7	0.21890 (16)	0.8012 (4)	0.07624 (17)	0.0619 (8)
H7	0.2150	0.8736	0.0349	0.074*
C8	0.27912 (16)	0.7939 (4)	0.12295 (17)	0.0637 (9)
H8	0.3144	0.8622	0.1128	0.076*
C9	0.10106 (16)	0.7265 (4)	0.03870 (17)	0.0614 (8)
H9	0.1051	0.7536	−0.0099	0.074*
C10	0.03833 (16)	0.7142 (4)	0.05177 (16)	0.0593 (8)
C11	0.01972 (17)	0.6781 (5)	0.12709 (17)	0.0714 (10)
H11A	0.0166	0.5522	0.1338	0.086*
H11B	0.0547	0.7220	0.1650	0.086*
C12	−0.04529 (19)	0.7616 (6)	0.1373 (2)	0.0830 (11)
H12A	−0.0566	0.7253	0.1845	0.100*
H12B	−0.0403	0.8883	0.1382	0.100*
C13	−0.10030 (18)	0.7122 (5)	0.0772 (2)	0.0778 (10)
H13	−0.1018	0.5836	0.0755	0.093*
C14	−0.08539 (17)	0.7743 (5)	0.00311 (19)	0.0738 (10)
H14A	−0.1154	0.7144	−0.0355	0.089*
H14B	−0.0954	0.8988	−0.0014	0.089*
C15	−0.01578 (17)	0.7473 (4)	−0.01116 (18)	0.0676 (9)
C16	−0.1685 (2)	0.7745 (6)	0.0903 (3)	0.1008 (14)
H16A	−0.2018	0.7330	0.0509	0.151*
H16B	−0.1693	0.9005	0.0914	0.151*
H16C	−0.1774	0.7292	0.1366	0.151*
N1	0.34694 (14)	0.6815 (4)	0.23302 (15)	0.0731 (8)
O1	−0.00436 (13)	0.7606 (4)	−0.07460 (13)	0.0917 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.068 (2)	0.119 (3)	0.084 (3)	−0.014 (2)	0.0187 (19)	0.003 (2)
C2	0.091 (3)	0.114 (3)	0.080 (3)	−0.011 (2)	−0.011 (2)	0.023 (2)

C3	0.0627 (19)	0.068 (2)	0.0508 (17)	0.0034 (15)	0.0157 (15)	−0.0007 (15)
C4	0.071 (2)	0.068 (2)	0.0604 (19)	−0.0013 (17)	0.0127 (16)	0.0149 (16)
C5	0.069 (2)	0.063 (2)	0.066 (2)	−0.0061 (16)	0.0114 (16)	0.0093 (16)
C6	0.0641 (19)	0.0634 (19)	0.0476 (16)	0.0003 (15)	0.0144 (14)	−0.0014 (14)
C7	0.070 (2)	0.068 (2)	0.0523 (17)	0.0066 (16)	0.0229 (15)	0.0073 (15)
C8	0.066 (2)	0.070 (2)	0.0610 (19)	−0.0038 (16)	0.0251 (16)	0.0043 (16)
C9	0.074 (2)	0.063 (2)	0.0482 (17)	0.0002 (16)	0.0143 (15)	0.0002 (14)
C10	0.071 (2)	0.0618 (19)	0.0446 (16)	−0.0026 (15)	0.0094 (14)	−0.0016 (14)
C11	0.074 (2)	0.092 (3)	0.0482 (17)	−0.0057 (19)	0.0105 (15)	0.0011 (17)
C12	0.084 (3)	0.106 (3)	0.062 (2)	0.003 (2)	0.0206 (19)	−0.004 (2)
C13	0.074 (2)	0.088 (3)	0.074 (2)	0.0013 (19)	0.0185 (18)	−0.003 (2)
C14	0.074 (2)	0.077 (2)	0.069 (2)	0.0023 (18)	0.0063 (17)	0.0050 (18)
C15	0.079 (2)	0.070 (2)	0.0534 (19)	−0.0015 (17)	0.0084 (16)	0.0005 (16)
C16	0.082 (3)	0.114 (4)	0.109 (3)	0.013 (2)	0.022 (2)	0.003 (3)
N1	0.0655 (17)	0.089 (2)	0.0648 (17)	−0.0025 (15)	0.0109 (14)	0.0141 (15)
O1	0.0925 (19)	0.129 (2)	0.0523 (14)	0.0035 (16)	0.0089 (12)	0.0090 (14)

Geometric parameters (Å, º)

C1—N1	1.437 (4)	C9—C10	1.340 (4)
C1—H1A	0.9600	C9—H9	0.9300
C1—H1B	0.9600	C10—C15	1.482 (5)
C1—H1C	0.9600	C10—C11	1.512 (4)
C2—N1	1.440 (4)	C11—C12	1.507 (5)
C2—H2A	0.9600	C11—H11A	0.9700
C2—H2B	0.9600	C11—H11B	0.9700
C2—H2C	0.9600	C12—C13	1.486 (5)
C3—N1	1.369 (4)	C12—H12A	0.9700
C3—C8	1.395 (4)	C12—H12B	0.9700
C3—C4	1.406 (4)	C13—C14	1.513 (5)
C4—C5	1.368 (4)	C13—C16	1.522 (5)
C4—H4	0.9300	C13—H13	0.9800
C5—C6	1.398 (4)	C14—C15	1.497 (5)
C5—H5	0.9300	C14—H14A	0.9700
C6—C7	1.393 (4)	C14—H14B	0.9700
C6—C9	1.452 (4)	C15—O1	1.224 (4)
C7—C8	1.379 (4)	C16—H16A	0.9600
C7—H7	0.9300	C16—H16B	0.9600
C8—H8	0.9300	C16—H16C	0.9600
N1—C1—H1A	109.5	C12—C11—C10	113.0 (3)
N1—C1—H1B	109.5	C12—C11—H11A	109.0
H1A—C1—H1B	109.5	C10—C11—H11A	109.0
N1—C1—H1C	109.5	C12—C11—H11B	109.0
H1A—C1—H1C	109.5	C10—C11—H11B	109.0
H1B—C1—H1C	109.5	H11A—C11—H11B	107.8
N1—C2—H2A	109.5	C13—C12—C11	112.0 (3)
N1—C2—H2B	109.5	C13—C12—H12A	109.2

H2A—C2—H2B	109.5	C11—C12—H12A	109.2
N1—C2—H2C	109.5	C13—C12—H12B	109.2
H2A—C2—H2C	109.5	C11—C12—H12B	109.2
H2B—C2—H2C	109.5	H12A—C12—H12B	107.9
N1—C3—C8	121.2 (3)	C12—C13—C14	110.4 (3)
N1—C3—C4	121.8 (3)	C12—C13—C16	113.8 (3)
C8—C3—C4	116.9 (3)	C14—C13—C16	111.3 (3)
C5—C4—C3	121.0 (3)	C12—C13—H13	107.0
C5—C4—H4	119.5	C14—C13—H13	107.0
C3—C4—H4	119.5	C16—C13—H13	107.0
C4—C5—C6	122.7 (3)	C15—C14—C13	116.5 (3)
C4—C5—H5	118.6	C15—C14—H14A	108.2
C6—C5—H5	118.6	C13—C14—H14A	108.2
C7—C6—C5	115.5 (3)	C15—C14—H14B	108.2
C7—C6—C9	119.2 (3)	C13—C14—H14B	108.2
C5—C6—C9	125.2 (3)	H14A—C14—H14B	107.3
C8—C7—C6	122.7 (3)	O1—C15—C10	121.3 (3)
C8—C7—H7	118.6	O1—C15—C14	118.9 (3)
C6—C7—H7	118.6	C10—C15—C14	119.7 (3)
C7—C8—C3	120.9 (3)	C13—C16—H16A	109.5
C7—C8—H8	119.5	C13—C16—H16B	109.5
C3—C8—H8	119.5	H16A—C16—H16B	109.5
C10—C9—C6	130.2 (3)	C13—C16—H16C	109.5
C10—C9—H9	114.9	H16A—C16—H16C	109.5
C6—C9—H9	114.9	H16B—C16—H16C	109.5
C9—C10—C15	117.4 (3)	C3—N1—C1	121.6 (3)
C9—C10—C11	124.1 (3)	C3—N1—C2	120.3 (3)
C15—C10—C11	118.4 (3)	C1—N1—C2	117.9 (3)

N1—C3—C4—C5	178.2 (3)	C10—C11—C12—C13	53.8 (4)
C8—C3—C4—C5	−1.6 (5)	C11—C12—C13—C14	−61.4 (4)
C3—C4—C5—C6	−0.9 (5)	C11—C12—C13—C16	172.7 (3)
C4—C5—C6—C7	3.3 (5)	C12—C13—C14—C15	43.0 (5)
C4—C5—C6—C9	−176.7 (3)	C16—C13—C14—C15	170.3 (3)
C5—C6—C7—C8	−3.2 (5)	C9—C10—C15—O1	10.1 (5)
C9—C6—C7—C8	176.8 (3)	C11—C10—C15—O1	−172.9 (3)
C6—C7—C8—C3	0.8 (5)	C9—C10—C15—C14	−166.7 (3)
N1—C3—C8—C7	−178.2 (3)	C11—C10—C15—C14	10.3 (5)
C4—C3—C8—C7	1.7 (5)	C13—C14—C15—O1	165.0 (3)
C7—C6—C9—C10	−148.5 (3)	C13—C14—C15—C10	−18.1 (5)
C5—C6—C9—C10	31.5 (5)	C8—C3—N1—C1	−3.9 (5)
C6—C9—C10—C15	179.0 (3)	C4—C3—N1—C1	176.2 (3)
C6—C9—C10—C11	2.3 (6)	C8—C3—N1—C2	−179.2 (3)
C9—C10—C11—C12	149.3 (3)	C4—C3—N1—C2	1.0 (5)
C15—C10—C11—C12	−27.4 (5)		