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3-[(*E*)-2,4-Dichlorobenzylidene]-1-methylpiperidin-4-one

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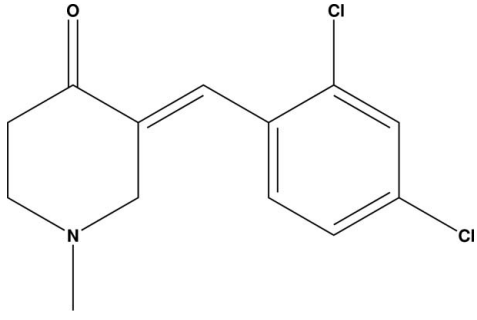
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.121; data-to-parameter ratio = 19.8.

The piperidine ring of the title compound, $C_{13}H_{13}Cl_2NO$, adopts an envelope conformation. Intermolecular C—H···O interactions link the molecules into a $C(7)$ chain running along the b axis.

Related literature

For biological activities of 4-piperidones, see: Badorrey *et al.* (1999); Grishina *et al.* (1994); Nalanishi *et al.* (1974*a*,*b*). For ring conformations, see: Cremer & Pople (1975); Nardelli (1983).



Experimental

Crystal data

$C_{13}H_{13}Cl_2NO$
M_r = 270.14
Monoclinic, $P2_1/n$
a = 12.2013 (9) Å
b = 8.5901 (6) Å
c = 12.6391 (9) Å
β = 92.997 (1)°
V = 1322.90 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 293 (2) K
0.24 × 0.23 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
14377 measured reflections
3071 independent reflections
2654 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.120
S = 0.97
3071 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O1^{i}	0.93	2.46	3.366 (2)	163

Symmetry code: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{5}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2557).

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supporting information

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3-[(*E*)-2,4-Dichlorobenzylidene]-1-methylpiperidin-4-one

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S1. Comment

Synthesis of 4-piperidones is of current interest due to their potential medical applications (Grishina *et al.*, 1994). 4-Piperidones have been found to exhibit blood cholesterol-lowering activities (Nalanishi *et al.*, 1974*a*,*b*). Various piperidones and piperidine derivatives are present in numerous alkaloids (Badorrey *et al.*, 1999). As the title compound is of biological significance, the crystal structure of the title compound has been determined by X-ray diffraction.

The sum of the bond angles around atom N1 (330 °) indicates sp^3– hybridization. Atoms Cl1 and Cl2 deviate from the plane of the attached benzene ring by 0.075 (1) and -0.094 (1) Å, respectively. The piperidine ring adopts an envelope conformation, with puckering parameters (Cremer & Pople, 1975) and smallest displacement asymmetry parameters (Nardelli, 1983) of Q = 0.504 (2) Å, θ = 141.1 (2)°, φ = 193.7 (4)° and ΔC_s[N1] = 8.7 (2)°.

In the crystal structure, the C—H···O intermolecular interactions generate a C(7) chain running along the *b* axis.

S2. Experimental

A mixture of 1-methyl-4-piperidone (1 mmol) and pyrrolidine (1.2 mmol) was taken in a glass tube, mixed well and kept aside for 5 min at ambient temperature. To this mixture, 2,4-dichlorobenzaldehyde (1 mmol) was added, mixed thoroughly and the tube containing the mixture was partially immersed in a silica bath placed in a microwave oven and irradiated at 4 power level for 7 minutes. The progress of the reaction was monitored after every 1 min of irradiation by TLC with petroleum ether:ethyl acetate (1:2 *v*/*v* mixture) as eluent. fter each irradiation, the reaction mixture was cooled to room temperature and mixed well. The maximum temperature of the silica bath, measured immediately after each irradiation was over by stirring the silica bath with the thermometer, was found to be 338 K. After completion of the reaction as evident from the TLC, the product was purified by column chromatography using petroleum ether:ethyl acetate (7:2 *v*/*v*) mixture and crystallized from ethyl acetate.

S3. Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.5U_{eq}(methyl C) or 1.2U_{eq}(C). A rotating group model was used for the methyl groups.

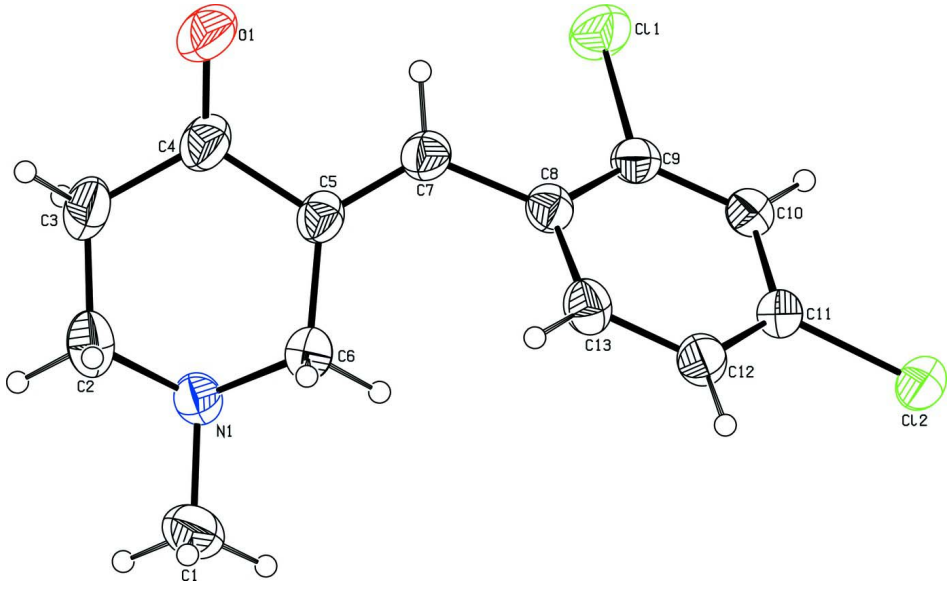

Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

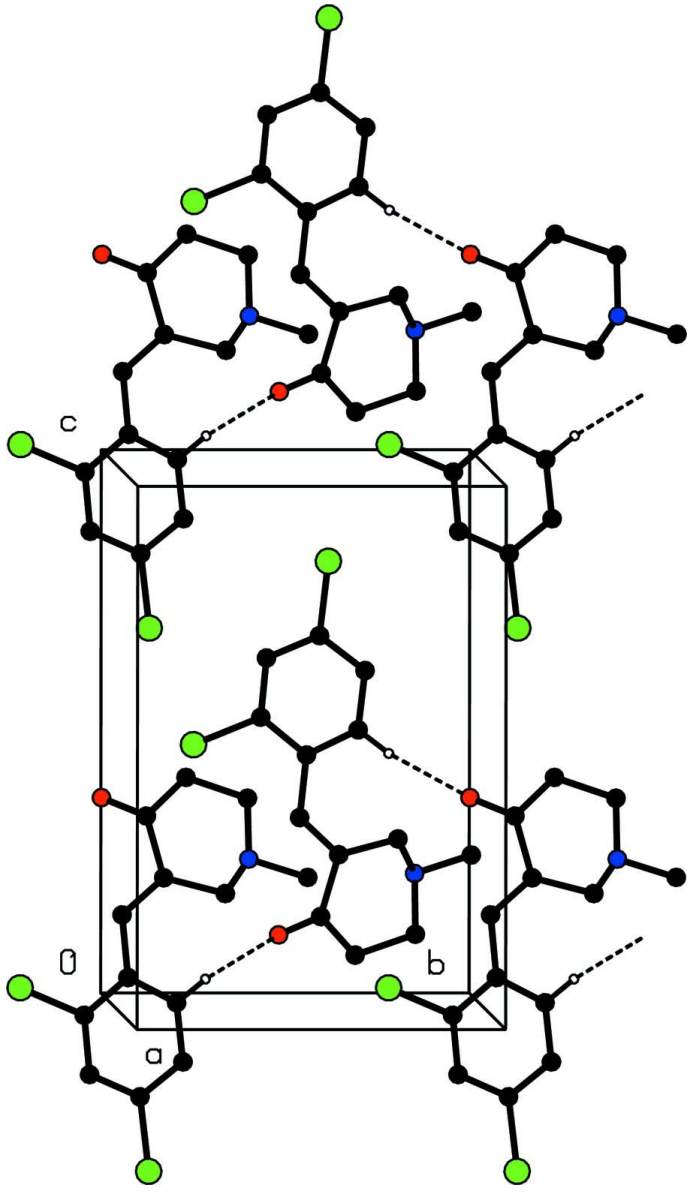

Figure 2

The molecular packing in the title compound, viewed approximately down the *a* axis.

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Crystal data

$C_{13}H_{13}Cl_2NO$
$M_r = 270.14$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 12.2013\ (9)$ Å
$b = 8.5901\ (6)$ Å
$c = 12.6391\ (9)$ Å
$\beta = 92.997\ (1)°$
$V = 1322.90\ (16)$ Å^3
$Z = 4$

$F(000) = 560$
$D_x = 1.356$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1509 reflections
$\theta = 2.3–25.0°$
$\mu = 0.47$ mm^{-1}
$T = 293$ K
Block, pale yellow
0.24 × 0.23 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
14377 measured reflections
3071 independent reflections
2654 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 28.1°$, $\theta_{min} = 2.3°$
$h = -15 \rightarrow 15$
$k = -10 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.120$
$S = 0.97$
3071 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 0.3356P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.53390 (18)	0.5088 (2)	1.24853 (18)	0.0781 (5)
H1A	0.5518	0.5079	1.1755	0.117*
H1B	0.5966	0.5421	1.2917	0.117*
H1C	0.4740	0.5792	1.2576	0.117*
C2	0.47091 (17)	0.3512 (2)	1.38999 (14)	0.0700 (5)
H2A	0.4038	0.4104	1.3959	0.084*
H2B	0.5280	0.4004	1.4346	0.084*
C3	0.4536 (2)	0.1860 (3)	1.42729 (13)	0.0791 (6)
H3A	0.5248	0.1374	1.4403	0.095*
H3B	0.4182	0.1896	1.4942	0.095*
C4	0.38604 (15)	0.0855 (2)	1.35183 (13)	0.0660 (5)
C5	0.37681 (12)	0.1336 (2)	1.23733 (11)	0.0524 (3)
C6	0.41053 (13)	0.29745 (19)	1.21048 (12)	0.0532 (3)
H6A	0.4317	0.3008	1.1376	0.064*
H6B	0.3484	0.3667	1.2168	0.064*
C7	0.34326 (12)	0.0240 (2)	1.16663 (12)	0.0545 (4)
H7	0.3257	−0.0728	1.1939	0.065*

C8	0.33134 (11)	0.04126 (18)	1.05058 (11)	0.0486 (3)
C9	0.35991 (12)	−0.08015 (17)	0.98306 (12)	0.0489 (3)
C10	0.35760 (12)	−0.06504 (17)	0.87383 (12)	0.0496 (3)
H10	0.3791	−0.1465	0.8311	0.059*
C11	0.32225 (13)	0.07535 (18)	0.83036 (11)	0.0500 (3)
C12	0.28628 (14)	0.19534 (19)	0.89226 (13)	0.0565 (4)
H12	0.2592	0.2867	0.8613	0.068*
C13	0.29116 (13)	0.17732 (19)	1.00152 (13)	0.0557 (4)
H13	0.2670	0.2581	1.0434	0.067*
N1	0.50222 (11)	0.35202 (16)	1.28025 (10)	0.0539 (3)
O1	0.34393 (15)	−0.0325 (2)	1.38258 (12)	0.1014 (6)
Cl1	0.40318 (5)	−0.25836 (5)	1.03652 (4)	0.07721 (18)
Cl2	0.32208 (5)	0.09909 (5)	0.69335 (3)	0.07158 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0843 (13)	0.0546 (10)	0.0934 (14)	−0.0057 (9)	−0.0158 (11)	0.0027 (10)
C2	0.0752 (11)	0.0799 (12)	0.0544 (9)	0.0079 (9)	−0.0035 (8)	−0.0182 (9)
C3	0.0949 (14)	0.1009 (15)	0.0412 (8)	−0.0155 (12)	0.0012 (8)	0.0010 (9)
C4	0.0617 (9)	0.0896 (13)	0.0472 (8)	−0.0133 (9)	0.0065 (7)	0.0089 (8)
C5	0.0465 (7)	0.0672 (9)	0.0437 (7)	−0.0049 (6)	0.0037 (6)	0.0033 (6)
C6	0.0523 (8)	0.0595 (9)	0.0475 (7)	0.0023 (7)	−0.0001 (6)	−0.0007 (7)
C7	0.0509 (8)	0.0625 (9)	0.0501 (8)	−0.0084 (7)	0.0038 (6)	0.0046 (7)
C8	0.0424 (7)	0.0541 (8)	0.0493 (7)	−0.0043 (6)	0.0011 (5)	−0.0007 (6)
C9	0.0473 (7)	0.0448 (7)	0.0543 (8)	−0.0020 (6)	−0.0011 (6)	0.0045 (6)
C10	0.0512 (7)	0.0460 (7)	0.0513 (8)	−0.0012 (6)	0.0016 (6)	−0.0035 (6)
C11	0.0535 (8)	0.0504 (7)	0.0455 (7)	−0.0036 (6)	−0.0018 (6)	0.0003 (6)
C12	0.0639 (9)	0.0483 (8)	0.0563 (8)	0.0069 (7)	−0.0084 (7)	−0.0006 (7)
C13	0.0561 (8)	0.0555 (8)	0.0549 (8)	0.0071 (7)	−0.0032 (6)	−0.0090 (7)
N1	0.0546 (7)	0.0524 (7)	0.0540 (7)	0.0017 (5)	−0.0029 (5)	−0.0036 (5)
O1	0.1116 (12)	0.1262 (13)	0.0654 (8)	−0.0543 (11)	−0.0048 (8)	0.0325 (9)
Cl1	0.1059 (4)	0.0545 (3)	0.0708 (3)	0.0144 (2)	0.0005 (3)	0.01340 (19)
Cl2	0.1041 (4)	0.0622 (3)	0.0482 (2)	−0.0037 (2)	0.0024 (2)	0.00406 (17)

Geometric parameters (Å, º)

C1—N1	1.463 (2)	C6—H6A	0.97
C1—H1A	0.96	C6—H6B	0.97
C1—H1B	0.96	C7—C8	1.474 (2)
C1—H1C	0.96	C7—H7	0.93
C2—N1	1.458 (2)	C8—C13	1.400 (2)
C2—C3	1.514 (3)	C8—C9	1.403 (2)
C2—H2A	0.97	C9—C10	1.385 (2)
C2—H2B	0.97	C9—Cl1	1.7440 (15)
C3—C4	1.501 (3)	C10—C11	1.385 (2)
C3—H3A	0.97	C10—H10	0.93
C3—H3B	0.97	C11—C12	1.380 (2)

C4—O1	1.209 (2)	C11—Cl2	1.7435 (15)
C4—C5	1.504 (2)	C12—C13	1.388 (2)
C5—C7	1.346 (2)	C12—H12	0.93
C5—C6	1.510 (2)	C13—H13	0.93
C6—N1	1.465 (2)		

N1—C1—H1A	109.5	N1—C6—H6B	109.3
N1—C1—H1B	109.5	C5—C6—H6B	109.3
H1A—C1—H1B	109.5	H6A—C6—H6B	107.9
N1—C1—H1C	109.5	C5—C7—C8	126.95 (15)
H1A—C1—H1C	109.5	C5—C7—H7	116.5
H1B—C1—H1C	109.5	C8—C7—H7	116.5
N1—C2—C3	110.39 (15)	C13—C8—C9	116.34 (13)
N1—C2—H2A	109.6	C13—C8—C7	122.59 (14)
C3—C2—H2A	109.6	C9—C8—C7	121.07 (14)
N1—C2—H2B	109.6	C10—C9—C8	122.94 (13)
C3—C2—H2B	109.6	C10—C9—Cl1	117.27 (11)
H2A—C2—H2B	108.1	C8—C9—Cl1	119.78 (12)
C4—C3—C2	114.99 (16)	C11—C10—C9	117.82 (13)
C4—C3—H3A	108.5	C11—C10—H10	121.1
C2—C3—H3A	108.5	C9—C10—H10	121.1
C4—C3—H3B	108.5	C12—C11—C10	121.83 (14)
C2—C3—H3B	108.5	C12—C11—Cl2	119.47 (12)
H3A—C3—H3B	107.5	C10—C11—Cl2	118.69 (12)
O1—C4—C5	121.85 (17)	C11—C12—C13	118.85 (15)
O1—C4—C3	120.41 (16)	C11—C12—H12	120.6
C5—C4—C3	117.69 (16)	C13—C12—H12	120.6
C7—C5—C4	116.86 (16)	C12—C13—C8	121.99 (14)
C7—C5—C6	125.39 (14)	C12—C13—H13	119.0
C4—C5—C6	117.70 (14)	C8—C13—H13	119.0
N1—C6—C5	111.81 (13)	C1—N1—C2	110.56 (15)
N1—C6—H6A	109.3	C1—N1—C6	109.51 (14)
C5—C6—H6A	109.3	C2—N1—C6	109.96 (13)

N1—C2—C3—C4	−46.3 (2)	C13—C8—C9—Cl1	−176.27 (11)
C2—C3—C4—O1	−161.3 (2)	C7—C8—C9—Cl1	3.54 (19)
C2—C3—C4—C5	21.1 (3)	C8—C9—C10—C11	−2.0 (2)
O1—C4—C5—C7	−15.5 (3)	Cl1—C9—C10—C11	179.32 (11)
C3—C4—C5—C7	162.09 (18)	C9—C10—C11—C12	−2.4 (2)
O1—C4—C5—C6	167.14 (19)	C9—C10—C11—Cl2	178.30 (11)
C3—C4—C5—C6	−15.2 (2)	C10—C11—C12—C13	3.4 (2)
C7—C5—C6—N1	−142.95 (16)	Cl2—C11—C12—C13	−177.33 (13)
C4—C5—C6—N1	34.12 (19)	C11—C12—C13—C8	0.0 (2)
C4—C5—C7—C8	−177.87 (14)	C9—C8—C13—C12	−4.0 (2)
C6—C5—C7—C8	−0.8 (3)	C7—C8—C13—C12	176.22 (14)
C5—C7—C8—C13	−38.6 (2)	C3—C2—N1—C1	−172.10 (17)
C5—C7—C8—C9	141.58 (17)	C3—C2—N1—C6	66.9 (2)
C13—C8—C9—C10	5.0 (2)	C5—C6—N1—C1	178.07 (14)

C7—C8—C9—C10	−175.16 (13)	C5—C6—N1—C2	−60.26 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O1^{i}	0.93	2.46	3.366 (2)	163

Symmetry code: (i) −*x*+1/2, *y*+1/2, −*z*+5/2.