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(*Z*)-2-(Cyclohexylidene)-1-azabicyclo[2.2.2]-octan-3-one

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Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.077
Data-to-parameter ratio = 10.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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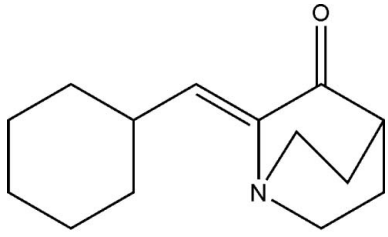
The reaction of cyclohexanecarboxaldehyde with 1-azabicylo[2.2.2]octan-3-one in methanolic KOH afforded the title compound, $C_{14}H_{21}NO$. The cyclohexane ring adopts a chair conformation and the olefinic double bond has *Z* geometry.

Comment

The title compound, (I), is a synthetic intermediate in our ongoing synthesis of 2-(substituted benzylidene/heteroaryl-3-ylmethylene)-1-azabicylo[2.2.2]octan-3-ones (Sonar *et al.*, 2003). The title compound was obtained from the reaction of cyclohexanecarboxaldehyde with 1-azabicylo[2.2.2]octan-3-one in methanolic KOH under reflux to afford a single geometrical isomer. In order to confirm the geometry of the product, and to obtain detailed information on the structural conformation of the molecule, its crystal structure determination has been carried out.



(I)

The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters are presented in Table 1. The cyclohexane ring adopts a chair conformation and the double bond has *Z* geometry. The C1—C7═C8, C7═C8—C12,

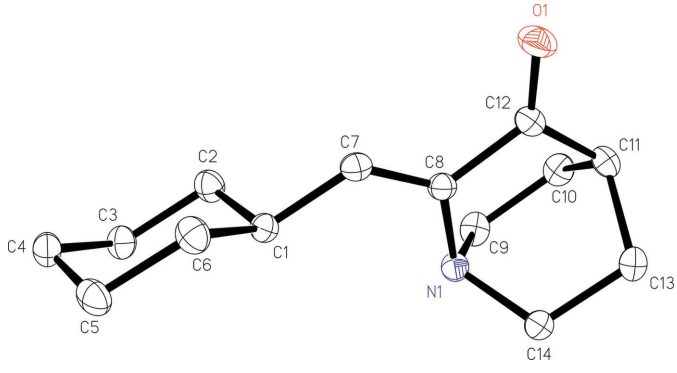


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted.

C7═C8—N1 and C8—C12—C11 bond angles deviate from the ideal bond angle of 120°. These deviations result from the relief of strain induced by the double-bond linkage to atom C8 of the azabicyclic unit.

Experimental

The title compound was prepared according to the previously reported procedure of Sonar *et al.* (2003). Crystallization from ethyl acetate afforded colourless crystals.

Crystal data

$C_{14}H_{21}NO$
M_r = 219.32
Monoclinic, $P2_1$
a = 9.2795 (2) Å
b = 6.7778 (1) Å
c = 9.8401 (2) Å
β = 101.5236 (9)°
V = 606.41 (2) Å^3
Z = 2
D_x = 1.201 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 90.0 (2) K
Cut block, colourless
0.40 × 0.30 × 0.25 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.971, T_{max} = 0.982
2756 measured reflections
1498 independent reflections
1437 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.077
S = 1.03
1498 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 0.135P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C8	1.4492 (18)	C7—C8	1.328 (2)
O1—C12	1.2200 (19)	C8—C12	1.494 (2)
C1—C6	1.532 (2)		
C8—N1—C9	107.57 (12)	O1—C12—C11	125.01 (15)
C7—C1—C6	111.94 (12)	C1—C7—C8	125.57 (13)
C7—C1—C2	109.89 (13)	C7—C8—C12	123.52 (13)
N1—C8—C12	113.46 (12)	C7—C8—N1	122.95 (13)
O1—C12—C8	124.32 (14)	C8—C12—C11	110.66 (12)
C1—C7—C8—N1	−0.1 (2)	N1—C8—C12—O1	179.55 (16)
C1—C7—C8—C12	176.60 (14)		

H atoms were found in difference Fourier maps and subsequently placed in idealized positions, with constrained C—H distances of 1.00 (R_3CH), 0.99 (R_2CH_2) and 0.95 Å (Csp^2). U_{iso}(H) values were set at 1.2U_{eq} of the attached C atom. In the absence of significant anomalous dispersion effects, Friedel pairs were merged.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*–SMN (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1995); software used to prepare material for publication: *SHELX97* and local procedures.

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