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(*Z*)-2-(4-Methoxybenzylidene)-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) = 0.003 Å
R factor = 0.034
wR factor = 0.077
Data-to-parameter ratio = 10.1

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

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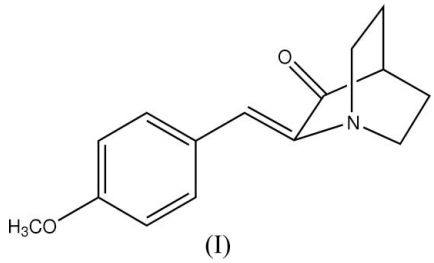
The title compound, $C_{15}H_{17}NO_2$, was prepared by the base-catalyzed reaction of 4-methoxybenzaldehyde with 1-azabicyclo[2.2.2]octan-3-one. The configuration about the olefinic bond connecting the methoxyphenyl and 1-azabicylo[2.2.2]octan-3-one moieties is *Z*.

Comment

The title compound, (I), was prepared by the base-catalyzed condensation of 4-methoxybenzaldehyde with 1-azabicyclo[2.2.2]octan-3-one, to afford (I) as a single geometrical isomer. In order to confirm the double-bond geometry, and to determine how the molecular conformation in the crystal structure is affected by the position of the *para*-methoxy group, the X-ray analysis of this positional isomer has been carried out and the results are presented here. This is a companion study together with the previous communication on the isomeric 2-methoxy analogue (Sonar *et al.*, 2006).



(I)

Fig. 1 illustrates an ellipsoid plot of (I), with the atom-numbering scheme; selected geometric parameters are listed in Table 1. The configuration about the olefinic bond connecting the 4-methoxyphenyl and 1-azabicylo[2.2.2]octan-3-one moieties is *Z*. The double bond has a nearly planar atomic arrangement, since the r.m.s. deviation from the mean plane passing through atoms N1, C8, C9, C7 and C1 for (I) is 0.0197 (11) Å.

There are no significant differences in the geometric parameters of (*Z*)-2-(2-methoxy-benzylidene)-1-azabicyclo[2.2.2]-octan-3-one and (*Z*)-2-(4-methoxy-benzylidene)-1-azabicyclo[2.2.2]octan-3-one. This suggests that the position of the methoxy group does not have much influence on the overall molecular conformation in the 2- and 4-positional isomers.

Experimental

Compound (I) was prepared following the method described previously for the 2-methoxy analogue (Sonar *et al.*, 2006), but utilizing 4-methoxybenzaldehyde in place of 2-methoxy-

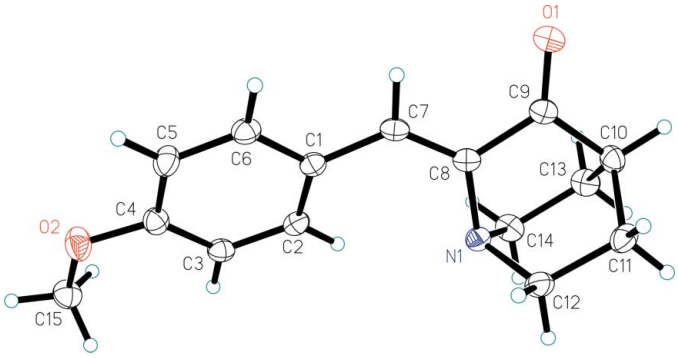


Figure 1
A view of the molecule of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

benzaldehyde. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 1.99–2.04 (*td*, 4H), 2.59–2.62 (*p*, 1H), 2.93–3.03 (*m*, 2H), 3.09–3.19 (*m*, 2H), 3.83 (*s*, 3H), 6.89 (*dd*, 2H),6.98 (*s*, 1H), 8.02 (*dd*, 2H); ^{13}C NMR ($CDCl_3$, δ, p.p.m.): 26.4, 40.6, 47.8, 55.5, 114.1, 125.1, 127.0, 134.1, 143.0, 160.8, 206.4.

Crystal data

$C_{15}H_{17}NO_2$
M_r = 243.30
Orthorhombic, $P2_12_12_1$
a = 5.8425 (2) Å
b = 9.9252 (3) Å
c = 21.3739 (7) Å
V = 1239.43 (7) Å^3
Z = 4
D_x = 1.304 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1641 reflections
θ = 1.0–27.5°
μ = 0.09 mm^{-1}
T = 90.0 (2) K
Block, colourless
0.30 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.975, T_{max} = 0.987
10079 measured reflections
1664 independent reflections
1323 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.5°
$h = -7 \rightarrow 7$
$k = -12 \rightarrow 12$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.077
S = 1.04
1664 reflections
165 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 0.1346P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.013 (2)

Table 1
Selected geometric parameters (Å, °).

C1—C7	1.463 (2)	O2—C15	1.429 (2)
N1—C8	1.447 (2)	C7—C8	1.336 (2)
O1—C9	1.227 (2)	C8—C9	1.485 (2)
O2—C4	1.369 (2)	C9—C10	1.508 (3)
C2—C1—C7	123.56 (17)	C7—C8—C9	121.39 (17)
C6—C1—C7	118.35 (17)	N1—C8—C9	113.57 (15)
C4—O2—C15	117.91 (16)	O1—C9—C8	124.48 (17)
C8—C7—C1	130.35 (17)	C8—C9—C10	110.75 (15)
C15—O2—C4—C3	−5.4 (3)	C6—C1—C7—C8	160.91 (19)
C2—C1—C7—C8	−21.9 (3)	C7—C8—C9—O1	0.0 (3)

In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. H atoms were positioned geometrically and treated as riding, with C—H distances in the range 0.95–0.99 Å and with U_{iso}(H) = 1.2–1.5U_{eq}(C).

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/PC* (Sheldrick, 1995); software used to prepare material for publication: *SHELX97-2* (Sheldrick, 1997) and local procedures.

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