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(*Z*)-2-(4-Methylbenzylidene)-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 = 0.047
wR factor = 0.128
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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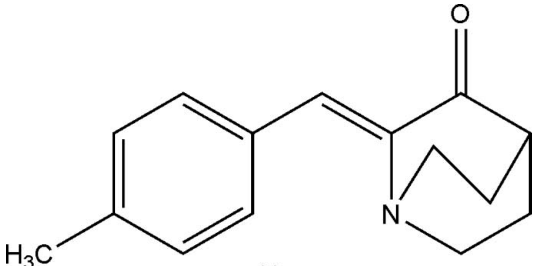
The title compound, $C_{15}H_{17}NO$, was synthesized by base-catalyzed condensation of 4-methylbenzaldehyde with 1-azabicyclo[2.2.2]octan-3-one and crystallization of the product from ethyl acetate. The geometry of the C═C bond is *Z*.

Comment

The title compound, (I), was prepared by base-catalyzed condensation of 4-methylbenzaldehyde with 1-azabicyclo[2.2.2]octan-3-one and the resultant product was crystallized from ethyl acetate to afford a single geometric isomer. The present X-ray crystallographic determination was carried out in order to obtain more detailed information on the conformation of the molecule and to confirm the geometry of the double bond.



(I)

Fig. 1 shows a view of (I), and selected geometric parameters are presented in Table 1. In the title compound, the C1—C7 bond is in a *trans* disposition with respect to the C8—C13 bond. Deviations from ideal bond-angle geometry around the Csp^2 atoms of the double bonds are observed. The bond angles N9—C8—C13, C7═C8—N9 and C8═C7–C1 (Table 1)

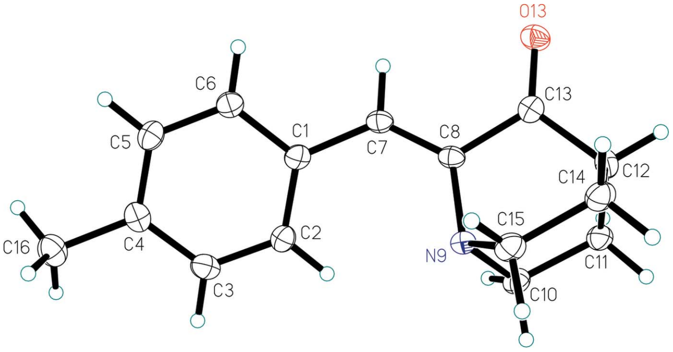


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

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are distorted because of the steric hindrance of the double bond linking the 4-methylphenyl ring with the azabicyclic moiety. These deviations contribute significantly to the relief of the intramolecular non-bonded interactions present in this portion of the molecule. The C2—C1—C7═C8 torsion angle indicates the deviation of the double bond from the plane of the benzene ring. However, the C1—C7 bond length suggests conjugation of the C7═C8 bond π electrons with those of the 4-methylphenyl ring (Wilson, 1992).

Experimental

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

Crystal data

$C_{15}H_{17}NO$
$M_r = 227.30$
Orthorhombic, $P2_12_12_1$
$a = 5.8527$ (2) Å
$b = 9.9840$ (3) Å
$c = 20.3309$ (6) Å
$V = 1188.00$ (6) Å^3

$Z = 4$
$D_x = 1.271$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 90.0$ (2) K
Block, yellow
0.25 × 0.20 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min} = 0.980$, $T_{\max} = 0.982$

2724 measured reflections
1602 independent reflections
1262 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.128$
$S = 1.07$
1602 reflections
155 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0819P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C7	1.468 (3)	C8—C13	1.498 (3)
C4—C16	1.510 (3)	N9—C10	1.484 (3)
C7—C8	1.332 (3)	O13—C13	1.221 (3)
C8—N9	1.438 (3)		
C2—C1—C7	123.5 (2)	C8—N9—C10	108.6 (2)
C8—C7—C1	129.2 (2)	O13—C13—C8	125.1 (2)
C7—C8—N9	125.5 (2)	O13—C13—C12	124.5 (2)
C7—C8—C13	120.8 (2)	C8—C13—C12	110.4 (2)
N9—C8—C13	113.62 (19)		
C2—C1—C7—C8	−27.5 (4)	C7—C8—C13—O13	0.0 (4)
C1—C7—C8—C13	179.0 (2)		

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$_2$), 0.98 (RCH$_3$) and 0.95 Å (Csp^2). $U_{\rm iso}$(H) values were set to either $1.5U_{\rm eq}$ of the attached C atom (CH$_3$) or $1.2U_{\rm eq}$ for all other H atoms. In the absence of significant anomalous scattering effects, Friedel pairs have been 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: *SHELXL97* and local procedures.

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