The title compound, C₁₅H₁₇NO, 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.

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 Cₛ₃ atoms of the double bonds are observed. The bond angles N₉—C₈—C₁₃, C₇—C₈—N₉ and C₈—C₇—C₁ (Table 1)
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 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

\[ \text{C}_{15}\text{H}_{17}\text{NO} \]

\[ M_r = 227.30 \]

Orthorhombic, \( P2_12_12_1 \)

\( a = 5.8527 (2) \) Å

\( b = 9.9840 (3) \) Å

\( c = 20.3309 (6) \) Å

\( V = 1188.00 (6) \) Å³

\( Z = 4 \)

\( D_x = 1.271 \text{ Mg m}^{-3} \)

\( \mu = 0.08 \text{ mm}^{-1} \)

\( T = 90.0 (2) \) K

Block, yellow

0.25 \times 0.20 \times 0.20 \text{ mm}

Data collection

Nonius KappaCCD area-detector

diffractometer

\( \omega \) scans

Absorption correction: multi-scan

(SCALEPACK; Otwinowski & Minor, 1997)

\( T_{\text{max}} = 0.980, T_{\text{min}} = 0.982 \)

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

\[ w = 1/[\sigma^2(F^2) + (0.0819P)^2] \]

where \( P = (F^2 + 2F_c^2)/3 \)

\( \Delta \rho_{\text{max}} = 0.26 \text{ e Å}^{-3} \)

\( \Delta \rho_{\text{min}} = -0.26 \text{ e Å}^{-3} \)

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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References


