Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

2,4-Bis(*o*-tolyl)-3-azabicyclo[3.3.1]-nonan-9-one

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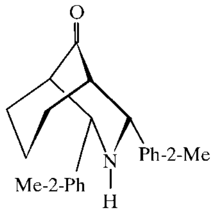
Received 13 March 2000
Accepted 27 June 2000

The crystal structure of the title compound, $C_{22}H_{25}NO$, confirms that the bicyclic ring system adopts the chair–chair conformation. The phenyl rings are equatorially disposed with respect to the bicyclic ring. There is a slight deviation from the chair conformation in the case of the cyclohexane ring.

Comment

The bicyclic [3.3.1]nonane ring can exist in chair–chair, chair–boat and boat–boat conformations. Among these, the chair–chair conformation is the most favourable. In the present study of the title compound, (I), the bicyclic ring system also adopts the chair–chair conformation.



(I)

In the cyclohexane ring of (I), atoms C7 and C9 deviate from the C8/C1/C5/C6 plane by −0.537 (4) and 0.720 (3) Å, respectively, indicating a departure from the ideal chair conformation (Brown *et al.*, 1965; Webb & Becker, 1967). The piperidine ring has near ideal chair conformation. The phenyl rings are equatorially disposed with respect to the piperidine ring, with the torsion angles C9—C5—C4—C16 −177.9 (2) and C10—C2—C1—C9 179.2 (2)°. They are oriented at an angle of 29.4 (1)° to each other. The orientations of the two *o*-tolyl groups are such that their methyl groups point towards the carbonyl group in the fused-ring system.

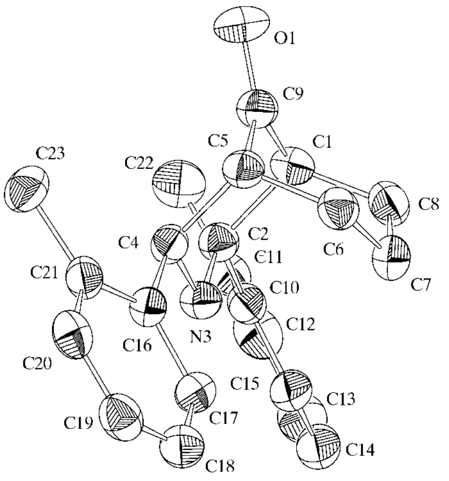


Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

A mixture of cyclohexanone (0.1 mol), *o*-tolualdehyde (0.2 mol) and absolute ethanol (75–100 ml) was added to a warm solution of ammonium acetate (0.15–0.20 mol) in absolute ethanol (200–250 ml). The mixture was gently warmed on a hot plate until the yellow colour changed to orange. Dry ether (100 ml) was added to the reaction mixture after cooling and the solution was kept for 5 d. Pale-yellow crystals of the ketone separated out and were washed with ethanol–ether and then recrystallized from benzene–petroleum ether (b.p. 333–353 K) [yield 35–40%, m.p. 490–491 K].

Crystal data

$C_{22}H_{25}NO$
$M_r = 319.43$
Monoclinic, $P2_1/a$
$a = 7.692$ (3) Å
$b = 16.825$ (3) Å
$c = 13.7247$ (18) Å
$\beta = 99.428$ (18)°
$V = 1752.1$ (8) Å^3
$Z = 4$
$D_x = 1.211$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 2$–$25°$
$\mu = 0.564$ mm^{-1}
$T = 293$ (2) K
Rectangular block, pale yellow
$0.15 \times 0.12 \times 0.10$ mm

Data collection

Rigaku AFC-7*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\rm min} = 0.928$, $T_{\rm max} = 0.942$
3578 measured reflections
3312 independent reflections
2380 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\rm max} = 70.08°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 20$
$l = -16 \rightarrow 16$
3 standard reflections every 100 reflections intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.206$
$S = 1.101$
3312 reflections
222 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1042P)^2 + 0.6146P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.34$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.43$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0067 (10)

Table 1
Selected geometric parameters (Å, °).

N3—C4	1.461 (3)	O1—C9	1.221 (3)
N3—C2	1.465 (3)		
C4—N3—C2	113.1 (2)	N3—C2—C1	110.1 (2)
O1—C9—C5	124.2 (2)	N3—C4—C16	111.5 (2)
N3—C2—C10	111.1 (2)	N3—C4—C5	109.6 (2)
C4—N3—C2—C1	58.3 (2)	N3—C2—C1—C9	−56.9 (2)
C1—C9—C5—C6	63.3 (3)	C10—C2—C1—C8	−60.7 (3)
C1—C9—C5—C4	−61.8 (3)	C9—C1—C8—C7	54.0 (3)
C2—N3—C4—C5	−58.9 (3)	C2—C1—C8—C7	−65.8 (3)
C9—C5—C4—N3	58.4 (2)	C1—C8—C7—C6	−45.0 (3)
C9—C5—C4—C16	−177.9 (2)	C8—C7—C6—C5	44.1 (3)
C6—C5—C4—C16	61.6 (3)	C9—C5—C6—C7	−52.1 (3)
C5—C9—C1—C8	−64.1 (3)	C4—C5—C6—C7	67.9 (3)
C5—C9—C1—C2	60.8 (3)		

All H atoms were located from difference Fourier maps and fixed using the *HFIX* option in *SHELXL*97 (Sheldrick, 1997), with their displacement parameters refined in four groups: N—H, C—H and CH_2 = 0.082 (2) Å^2 and methyl H atoms = 0.129 (6) Å^2. Geometrical calculations were performed using *PARST* (Nardelli, 1996).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL*97.

LV thanks the UGC, India, for the award of an FIP fellowship. Thanks are due to Professor J. Shashidaraprasad and Dr M. A. Sridhar for making the National Data Collection Facility at Mysore University, supported by the Department of Science and Technology, available for data collection.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1106). Services for accessing these data are described at the back of the journal.

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