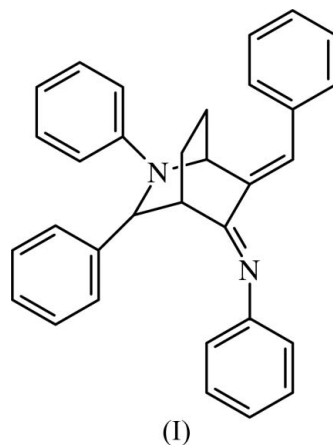


N*-{6-[(*Z*)-Benzylidene]-2,3-diphenyl-2-azabicyclo[2.2.2]octan-5-ylidene}aniline*K. Ravikumar,^{a*} B. Sridhar,^a
M. Mahesh^b and V. V.
Narayana Reddy^b**^aLaboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and ^bOrganic Chemistry Division-II, Indian Institute of Chemical Technology, Hyderabad 500 007, IndiaCorrespondence e-mail:
ravikumar_iict@yahoo.co.in**Key indicators**Single-crystal X-ray study
T = 273 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.049
wR factor = 0.134
Data-to-parameter ratio = 18.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{32}\text{H}_{28}\text{N}_2$, was obtained from the Diels–Alder reaction of *N*-benzylideneaniline and cyclohexen-2-one catalysed by zirconium tetrachloride. All three rings in the azabicyclo[2.2.2] ring system adopt boat conformations. The crystal packing is stabilized by van der Waals forces.

Comment

The presence of an isoquinuclidine ring system in a molecule is frequently associated with significant pharmacological properties (Iriepa *et al.*, 2002). In addition, it also acts as a precursor for the synthesis of naturally occurring piperidine alkaloids of the prosopis family (Birkinshaw *et al.*, 1988). The structure of the title compound, (I), is reported here as a part of our ongoing structural study of this series of compounds (Ravikumar *et al.*, 2005).



The molecule of (I) consists of four benzene rings, two of which are directly connected to the azabicyclic system while the other two are connected through a double-bond linkage as an extended ‘arm’ (Fig. 1). All the bond lengths and angles (Table 1) are in a good agreement with those found in related structures (Sonar *et al.*, 2003, 2004).

Geometrical isomerism around the double bonds $\text{C}_6=\text{C}_{27}$ and $\text{C}_5=\text{N}_3$ affords the possibility of *E* and *Z* isomers. The $\text{C}_{27}-\text{C}_{28}$ bond is in a *trans* configuration with respect to the C_5-C_6 bond [$\text{C}_5-\text{C}_6-\text{C}_{27}-\text{C}_{28} = -172.43 (14)^\circ$]. Similarly, the N_3-C_{21} bond is also in a *trans* disposition with respect to the C_5-C_6 bond [$\text{C}_6-\text{C}_5-\text{N}_3-\text{C}_{21} = 177.92 (13)^\circ$]. The bond angles around atoms C_6 , C_{21} and C_{28} are close to the ideal value of 120° , while the $\text{C}_1-\text{C}_6-\text{C}_{27}$ [$128.2 (1)^\circ$] and $\text{N}_3-\text{C}_5-\text{C}_4$ [$127.4 (1)^\circ$] angles are more distorted, as a consequence of the strain induced by the $\text{C}_6=\text{C}_{27}$ and $\text{C}_5=\text{N}_3$ double-bond linkages. Atom N_2 is in

Received 27 July 2005
Accepted 12 September 2005
Online 17 September 2005Imino Diels–Alder adducts.
VIII.

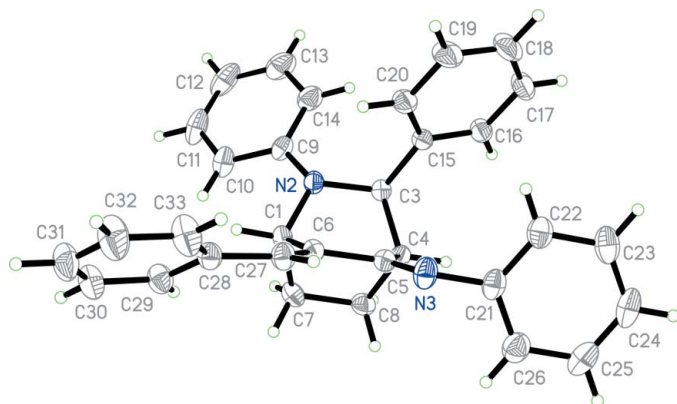


Figure 1
View of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as small spheres of arbitrary radii.

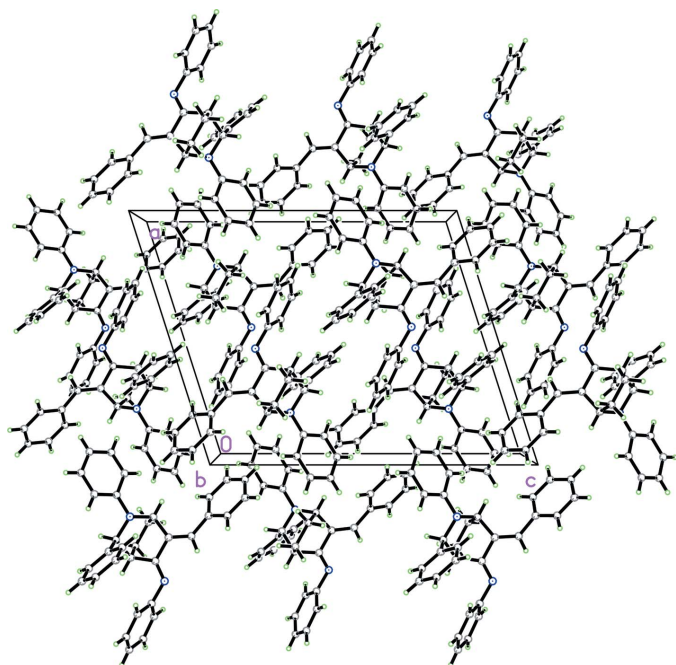


Figure 2
A packing diagram of (I), viewed down the *b* axis.

pyramidal configuration and the sum of the angles around the atom is 350.6°.

All three six-membered rings of the azabicyclo[2.2.2] system adopt the expected boat conformation, with asymmetry parameters (Nardelli, 1983) $\Delta C_s(C1)$ of 0.015 (1), 0.289 (1) and 0.002 (1) for the rings C3/N2/C1/C7/C8/C4, C1/C4–C8 and C1/N2/C3–C6, respectively. In the absence of H-atom-donating groups, the crystal packing (Fig. 2) is stabilized by van der Waals forces.

Experimental

To a solution of *N*-benzylideneaniline (5.5 mmol) in CH_2Cl_2 (5 ml) at room temperature were sequentially added ZrCl_4 (10 mol%) and cyclohexen-2-one (5.5 mmol), and the mixture was stirred for 6 h. After completion of the reaction, as indicated by thin-layer chro-

matography, the reaction was quenched with water, diluted with CH_2Cl_2 (20 ml) and washed with water (10 ml). The aqueous layer was extracted with CH_2Cl_2 (2×10 ml). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo* to yield a dark-brown residue, which was purified by column chromatography using 2–10% ethyl acetate in hexane as eluant to obtain the pure product.

Crystal data

$\text{C}_{32}\text{H}_{28}\text{N}_2$
 $M_r = 440.56$
Monoclinic, $P2_1/c$
 $a = 14.6209$ (13) Å
 $b = 9.5899$ (9) Å
 $c = 17.9541$ (16) Å
 $\beta = 107.701$ (2)°
 $V = 2398.2$ (4) Å³
 $Z = 4$

$D_x = 1.220$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 4667 reflections
 $\theta = 2.4$ – 27.4 °
 $\mu = 0.07$ mm⁻¹
 $T = 273$ (2) K
Block, colourless
 $0.19 \times 0.11 \times 0.09$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
14781 measured reflections
5570 independent reflections
4256 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$
 $\theta_{\text{max}} = 28.0$ °
 $h = -18 \rightarrow 19$
 $k = -7 \rightarrow 12$
 $l = -23 \rightarrow 23$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.134$
 $S = 1.05$
5570 reflections
307 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 0.3708P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

N2–C1	1.4735 (17)	C5–C6	1.4819 (17)
N3–C5	1.2717 (17)	C6–C27	1.3343 (18)
C1–C6	1.5119 (18)		
N3–C5–C6	121.80 (12)	C26–C21–N3	120.59 (15)
C27–C6–C5	121.47 (12)	C29–C28–C33	116.96 (13)
C22–C21–N3	119.98 (14)	C29–C28–C27	124.03 (13)
C1–N2–C9–C10	−0.10 (19)	C5–N3–C21–C22	96.93 (18)
C4–C3–C15–C16	79.58 (15)	C6–C27–C28–C29	30.1 (3)

H atoms were included in calculated positions and refined as riding, with C–H distances in the range 0.93–0.98 Å and with $U_{\text{iso}}(\text{H}) = 1.2$ – $1.5U_{\text{eq}}$ of the parent atom.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

MM thanks the Council of Scientific and Industrial Research (CSIR), India, for the award of a Senior Research Fellowship.

References

Birkinshaw, T. N., Tabor, A. B., Holmes, A. B., Kaye, P., Mayne, P. M. & Raithby, P. R. (1988). *Chem. Commun.* **24**, 1599–1600.

- Bruker (2001). *SMART* (Version 5.625) and *SAINTE* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.
- Iriepa, I., Villasante, F. J., Galvez, E., Labeaga, L., Innerarity, A. & Orjales, A. (2002). *Bioorg. Med. Chem. Lett.* **12**, 189–192.
- Nardelli, M. (1983). *Acta Cryst.* **C39**, 1141–1142.
- Ravikumar, K., Sridhar, B., Mahesh, M. & Narayana Reddy, V. V. (2005). *Acta Cryst.* **E61**, o2277–o2279.
- Sheldrick, G. M. (1990). *SHELXTL/PC User Manual*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sonar, V. N., Parkin, S. & Crooks, P. A. (2003). *Acta Cryst.* **E59**, o2010–o2012.
- Sonar, V. N., Parkin, S. & Crooks, P. A. (2004). *Acta Cryst.* **C60**, o659–o661.