

5-Benzoyl-1-cyclohexyl-3-(*m*-nitrophenyl)-2-phenylpyrrolidine-spiro-4,2'-(6'-methoxy-1'-tetralone)

A. Jeyabharathi,^a M. N. Ponnuswamy,^{a*} A. Amal Raj,^b R. Raghunathan^b and Hoong-Kun Fun^c

^aDepartment of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, ^bDepartment of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India, and ^cX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: mnpsy@hotmail.com

Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$

R factor = 0.068

wR factor = 0.182

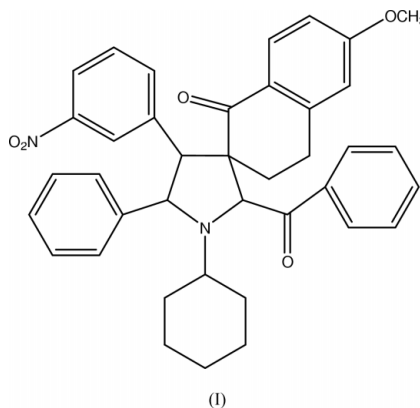
Data-to-parameter ratio = 13.6

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

In the title molecule, $\text{C}_{39}\text{H}_{38}\text{N}_2\text{O}_5$, the pyrrolidine ring is in an envelope conformation and the cyclohexyl ring adopts a chair conformation. The cyclohexanone ring in the tetralone moiety adopts a sofa conformation. The molecular packing in the crystal is stabilized by $\text{C}-\text{H}\cdots\pi$ interactions in addition to van der Waals forces.

Comment

Substituted pyrrolidine compounds have gained much importance in the past years, since they are the basic structural elements of many alkaloids and pharmacologically active compounds. For example, several amino acids which contain the pyrrolidine motif have been investigated (Galeazzi *et al.*, 1999). The title compound, (I), a pyrrolidine derivative was chosen for crystallographic study to determine its structure and conformation.



A displacement ellipsoid plot of (I) is shown in Fig. 1. The pyrrolidine ring is in an envelope conformation with the puckering parameters (Cremer & Pople, 1975) $q_2 = 0.364(3)\text{ \AA}$ and $\varphi_2 = 179.0(6)^\circ$. The cyclohexanone ring in the tetralone moiety adopts a sofa conformation. The cyclohexyl ring substituted at N1 is equatorially oriented with respect to the pyrrolidine ring and adopts a chair conformation with torsion angle values ranging from $53.7(4)$ to $57.3(5)^\circ$. The mean plane through the tetralone moiety makes a dihedral angle of $86.6(2)^\circ$ with the mean plane through the pyrrolidine ring. The displacement parameters of the O atoms of the nitro group are high, as a result of the greater thermal motion of these terminal atoms; similar effects have been reported by Ravikumar & Mehdi (1993). The nitro group is oriented at an angle of $10.7(2)^\circ$ with respect to the attached phenyl ring.

Carbonyl atom O2 is involved in weak intramolecular $\text{C}-\text{H}\cdots\text{O}$ (Desiraju, 1996) interactions with H13B and H16. The crystal structure is stabilized by $\text{C}-\text{H}\cdots\pi$ interactions (see

Received 19 April 2002

Accepted 29 April 2002

Online 24 May 2002

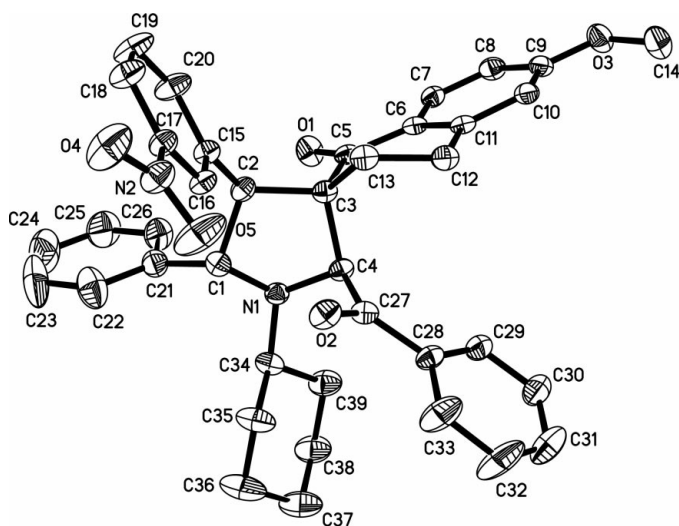


Figure 1
Displacement ellipsoid (30% probability) plot of (I), showing the atom-numbering scheme. H atoms have been omitted for clarity.

Table 1) in addition to van der Waals forces. In Table 1, $Cg(A)$ and $Cg(B)$ denote the centroids of the phenyl rings A (C21–C26) and B (C6–C11), respectively. The geometry of these interactions is comparable with those reported in the literature (Abdul Ajees *et al.*, 2001; Gallagher *et al.*, 2000; Kooijman *et al.*, 2000).

Experimental

A mixture of (*E*)-2-(*m*-nitrobenzylidene)-6-methoxy-1-tetralone and *cis*-1-cyclohexyl-2-phenyl-3-benzoylaziridine was refluxed in xylene under a nitrogen atmosphere for 36 h. On completion of the reaction, the solvent was evaporated *in vacuo* and the resulting crude product was purified by column chromatography using hexane–benzene mixture (7:3) as eluent. The title compound was recrystallized from ethanol.

Crystal data

$C_{39}H_{38}N_2O_5$	$Z = 2$
$M_r = 614.71$	$D_x = 1.239 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 11.2284 (1) \text{ \AA}$	Cell parameters from 3239 reflections
$b = 12.6346 (4) \text{ \AA}$	$\theta = 2.7\text{--}28.4^\circ$
$c = 12.9056 (3) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$\alpha = 102.453 (2)^\circ$	$T = 293 (2) \text{ K}$
$\beta = 96.412 (2)^\circ$	Slab, yellow
$\gamma = 109.775 (1)^\circ$	$0.40 \times 0.20 \times 0.14 \text{ mm}$
$V = 1648.05 (7) \text{ \AA}^3$	

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
Absorption correction: none
9073 measured reflections
5638 independent reflections

2873 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.059$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -13 \rightarrow 12$
 $k = -14 \rightarrow 15$
 $l = -15 \rightarrow 14$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.182$
 $S = 0.91$
5638 reflections
416 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.018 (2)

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
C13–H13B \cdots O2	0.97	2.43	3.063 (4)	122
C16–H16 \cdots O2	0.93	2.49	3.303 (4)	146
C14–H14C \cdots $Cg(A^i)$	0.96	2.83	3.653 (5)	144
C19–H19 \cdots $Cg(B^{ii})$	0.93	2.65	3.488 (4)	151

Symmetry codes: (i) $x, y, 1 + z$; (ii) $2 - x, 1 - y, 1 - z$.

The H atoms were fixed geometrically and treated as riding atoms on their parent C atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2000).

AJ thanks the University Grants Commission for the award of a teacher fellowship under FIP.

References

- Abdul Ajees, A., Parthasarathy, S., Manikandan, S. & Raghunathan, R. (2001). *Acta Cryst.* **C57**, 473–475.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Desiraju, G. R. (1996). *Acc. Chem. Res.* **29**, 441–449.
Gallagher, J. F., Brady, F. & Murphy, C. (2000). *Acta Cryst.* **C56**, 365–368.
Galeazzi, R., Geremia, S., Mobbli, G. & Orina, M. (1999). *Tetrahedron Asymmetry*, **10**, 587–605.
Kooijman, H., Spek, A. L., Kleijn, H., van Maanen, H. L., Jastrzebski, J. T. B. H. & van Koten, G. (2000). *Acta Cryst.* **C56**, 481–483.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Ravikumar, K. & Mehdi, S. (1993). *Acta Cryst.* **C49**, 2027–2030.
Siemens (1996). *SMART*, *SAINT* and *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2000). *PLATON*. University of Utrecht, The Netherlands.