Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Acenapthene-2-spiro-5′-perhydrodipyrrolo[1,2-*a*;-2′,1′-*c*]pyrazine-6′-spiro-2″-acenapthene-1,1″-dione

S. Sugaleshini,[a] D. Gayathri,[b] D. Velmurugan,[b]* K. Ravikumar[c] and A. R. Sureshbabu[d]

[a]Bioinformatics Research Institute, Ashok Nagar, Chennai 600 083, India, [b]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [c]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [d]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

Correspondence e-mail: d_velu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.128
Data-to-parameter ratio = 16.8

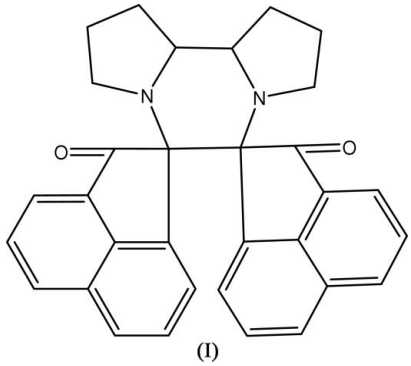
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 11 May 2006
Accepted 12 May 2006

The five-membered ring in the acenapthene ring system of the title compound, $C_{32}H_{26}N_2O_2$, slightly deviates from planarity. The two pyrrolidine rings adopt half-chair conformations and the piperazine ring adopts a chair conformation. The molecular structure is stabilized by weak C—H···O intramolecular interactions and the crystal packing is stabilized by C—H···O intermolecular interactions forming a chain running along (011).

Comment

Acenaphthene, a polycyclic aromatic hydrocarbon consisting of naphthalene with an olefin bridge, is used to make dyes, plastics and pesticides. Derivatives of acenaphthene are used as conformationally restricted ligands for melatonin receptors (Jellimann *et al.*, 2000), liver regeneration (Gershbein, 1975) and antitumoral agents (Boido *et al.*, 1994). Pyrrolidine has gained much attention in the pharmacological industry for its medicinal value. Pyrrolidine compounds have antifungal and antimicrobial activity (Amal Raj *et al.*, 2003). Owing to the high medicinal importance of pyrrolidine and acenaphthene derivatives we have undertaken the X-ray structure determination of the title compound, (I), containing both acenaphthene and pyrrolidine.



(I)

Fig. 1 shows the molecular structure of the title compound. The sums of the bond angles at N1 and N2 of the pyrrolidine rings are 338.9 and 338.3°, respectively, indicating sp^3-hybridization. The bond lengths and bond angles of the acenaphthene ring system are comparable to reported values (Edwards *et al.*, 1980; Selvanayagam *et al.*, 2004).

The two pyrrolidine rings adopt half-chair conformations; the piperazine ring adopts a chair conformation. The puckering parameters (q_2 and φ; Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Δ; Nardelli, 1983) are, for the pyrrolidine ring (N1/C25–C28), q_2 = 0.418 (2) Å, φ = 346.1 (3)° and Δ_2(C26) = 6.0 (2); for the pyrrolidine

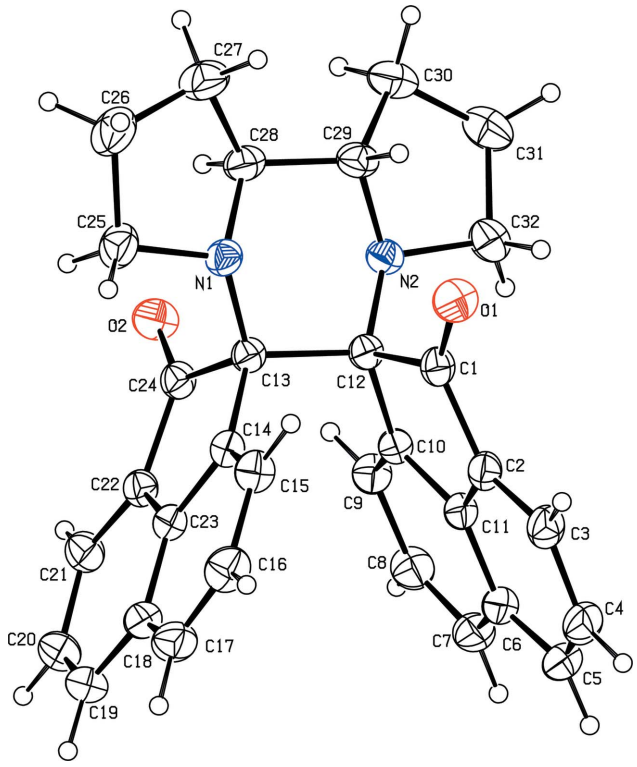


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids.

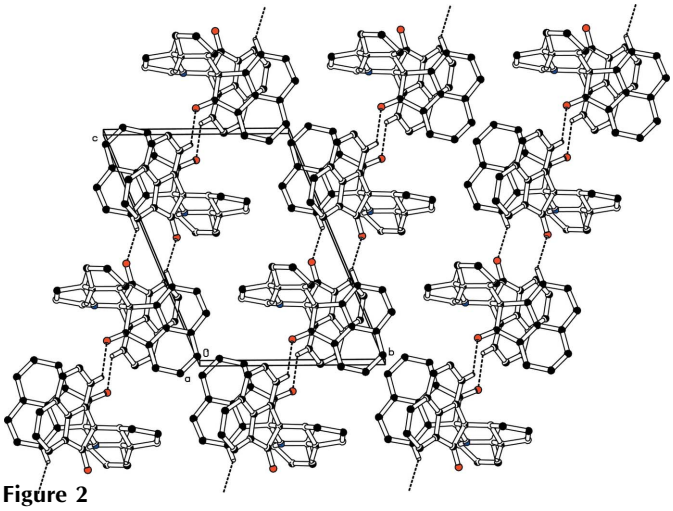


Figure 2
The crystal packing of (I), viewed down the *a* axis. For clarity, H atoms not involved in intermolecular hydrogen bonds have been omitted. Hydrogen bonds are shown as dashed lines.

ring (N2/C29—C32), q_2 = 0.427 (2) Å, φ = 193.6 (3)° and Δ_2(C31) = 6.8 (2); and for the piperazine ring, q_2 = 0.024 (1) Å, q_3 = −0.552 (1) Å, Q_T = 0.552 (1) Å and θ = 177.5 (2)°.

The molecular structure is stabilized by a weak C—H···O intramolecular interaction and the crystal packing is stabilized by two C—H···O intermolecular interactions (Table 2). The interactions involving C9 and C15 generate centrosymmetric $R_2^2(14)$ rings. As a result, a chain is formed running along [011].

Experimental

A mixture of acenapthenequinone (1 mmol) and L-proline (1 mmol) in methanol (20 ml) was refluxed on a water bath until the disappearance of the starting materials. The excess solvent was removed in vacuum and the crude product was recrystallized from methanol.

Crystal data

$C_{32}H_{26}N_2O_2$
M_r = 470.55
Triclinic, $P\overline{1}$
a = 8.8721 (7) Å
b = 10.7042 (8) Å
c = 14.2846 (11) Å
α = 109.418 (1)°
β = 100.584 (1)°
γ = 102.447 (1)°
V = 1200.37 (16) Å^3
Z = 2
D_x = 1.302 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, colorless
0.24 × 0.23 × 0.21 mm

Data collection

Bruker SMART APEX CCD area detector diffractometer
ω scans
Absorption correction: none
13844 measured reflections
5468 independent reflections
4621 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.128
S = 1.02
5468 reflections
325 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.068P)^2 + 0.217P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C13	1.454 (1)	O2—C24	1.206 (2)
N1—C25	1.464 (2)	C1—C12	1.586 (2)
N1—C28	1.471 (2)	C10—C12	1.522 (2)
N2—C12	1.453 (2)	C12—C13	1.578 (2)
N2—C32	1.461 (2)	C13—C14	1.526 (2)
N2—C29	1.470 (2)	C13—C24	1.586 (2)
O1—C1	1.207 (2)	C28—C29	1.5019 (19)
C13—N1—C25	117.0 (1)	C12—N2—C32	116.8 (1)
C13—N1—C28	116.0 (1)	C12—N2—C29	116.3 (1)
C25—N1—C28	105.9 (1)	C32—N2—C29	105.2 (1)
C29—N2—C12—C13	55.2 (1)	C13—N1—C28—C29	−59.1 (1)
C28—N1—C13—C12	55.7 (1)	C12—N2—C29—C28	−58.3 (1)
N2—C12—C13—N1	−50.3 (1)	N1—C28—C29—N2	55.1 (1)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C28—H28···O2	0.98	2.41	3.058 (2)	124
C29—H29···O1	0.98	2.43	3.075 (2)	123
C9—H9···O2^{i}	0.93	2.48	3.217 (2)	136
C15—H15···O1^{ii}	0.93	2.51	3.261 (2)	138

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

All H atoms were positioned geometrically and allowed to ride on their parent C atoms, with C—H distances in the range 0.93–0.98 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

DG and DV thank the University Grants Commission, India, for funding. Financial support from the University Grants Commission (UGC-SAP) and Department of Science and Technology (DST-FIST), Government of India, is acknowledged for providing facilities to the department.

References

Amal Raj, A., Raghunathan, R., Sridevi Kumari, M. R. & Raman, N. (2003). *Bioorg. Med. Chem.* **11**, 407–409.
Boido, A., Vazzana, I. & Sparatore, F. (1994). *Farmaco*, **49**, 97–104.
Bruker (2001). *SMART* (Version 5.625/NT/2000) and *SAINT* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Edwards, J. M., Mangion, M., Anderson, J. B., Rapposch, M., Moews, P. & Hite, G. (1980). *Acta Cryst.* B**36**, 1241–1244.
Gershbein, L. L. (1975). *Res. Commun. Chem. Pathol. Pharm.* **11**, 445–466.
Jellimann, C., Mathe-Allainmat, M., Andrieux, J., Kloubert, S., Boutin, J. A., Nicolas, J. P., Bennejean, C., Delagrange, P. & Langlois, M. (2000). *J. Med. Chem.* **43**, 4051–4062.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Selvanayagam, S., Velmurugan, D., Ravikumar, K., Jayashankaran, J., Durga, R. R. & Raghunathan, R. (2004). *Acta Cryst.* E**60**, o2216–o2218.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.