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Methyl 2-(1*H*-indol-3-ylmethyl)-1,2,3,3a,4,11c-hexahydronaphtho[2′,1′:2,3]pyrano[4,5-*b*]pyrrole-2-carboxylate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.002 Å
R factor = 0.050
wR factor = 0.162
Data-to-parameter ratio = 17.3

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

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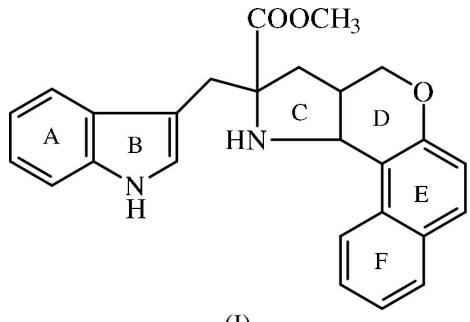
In the title compound, $C_{26}H_{24}N_2O_3$, the hydrogenated pyrrole ring adopts a twist conformation and the pyran ring adopts a twist–boat conformation. The molecular packing is stabilized by N—H···O and C—H···O intermolecular hydrogen bonds and also by weak intermolecular π–π interactions.

Comment

Pyrrole derivatives possess antimycobacterial (Biava *et al.*, 2005), anti-inflammatory (Fernandes *et al.*, 2004) and antiviral (Borthwick *et al.*, 2003) activities. These derivatives decrease afferent pelvic nerve activity (Tanaka *et al.*, 2003). Indole derivatives have been proven to display high aldose reductase inhibitory activity (Rajeswaran *et al.*, 1999). In view of its medicinal importance, the crystal structure determination of the title compound, (I), was carried out by X-ray diffraction.



(I)

A displacement ellipsoid plot of (I) is shown in Fig. 1. It consists of an indole group (*A* and *B*), four fused rings (*C*, *D*, *E* and *F*) and a methoxycarbonyl group. The geometry of the indole group is comparable with that in a related reported structure (Chandrakantha *et al.*, 1990). The C═O distances are normal. All the C—C and C—N bond lengths in ring *C* are comparable with literature values (Ueda *et al.*, 1991).

The indole group is planar. The methoxycarbonyl group is oriented at a dihedral angle of 72.6 (1)° with respect to the indole group. The naphthyl fragment is oriented at an angle of 67.6 (1)° with respect to the indole group. The methoxycarbonyl group (C25/O2/O3/C26) is planar, with a maximum deviation of 0.007 (2) Å for atom C25. The C—O bond of the ester group is in a *syn* orientation. The torsion angle C26—O3—C25—O2 is 1.6 (2)°.

Ring *C* adopts a twist conformation, with puckering parameters q_2 = 0.326 (1) Å and φ = −97.9 (3)° (Cremer & Pople, 1975), the displacement asymmetry parameters being Δ_S(C11) = 0.043 (1) and Δ_2(C10) = 0.028 (1) (Nardelli, 1983). The pyran ring *D* adopts a twist–boat conformation, with puck-

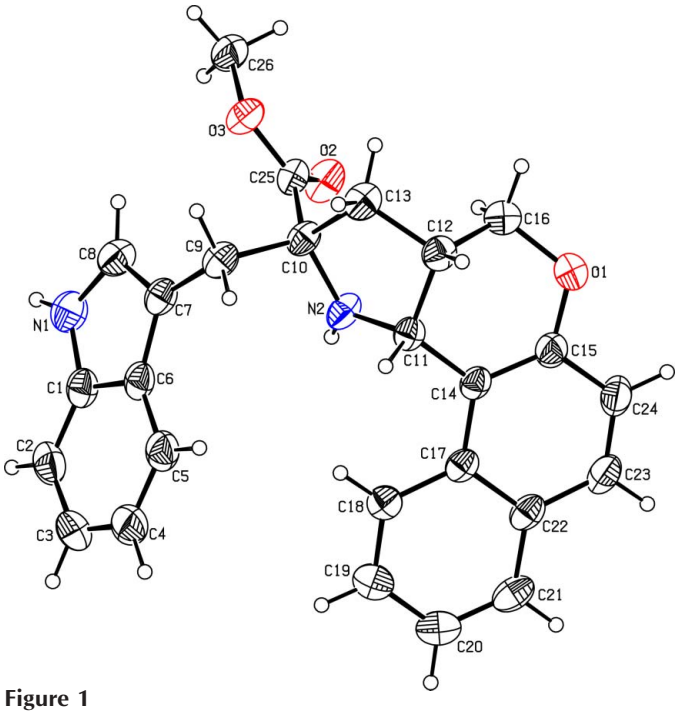


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

ering parameters of q_2 = 0.381 (2), q_3 = −0.286 (1), Q_T = 0.477 (2) Å, respectively and φ = −80.5 (2)° (Cremer & Pople, 1975).

In addition to van der Waals interactions, the molecular packing is stabilized by intermolecular N—H···O and C—H···O hydrogen bonds (Table 2 and Fig. 2) and also by intermolecular π–π interactions between the benzene rings *F* (C17–C22) at (*x*, *y*, *z*) and (1 − *x*, 1 − *y*, −*z*) (Fig. 3), with a centroid–centroid separation of 3.530 (1) Å.

Experimental

A solution of tryptophan methyl ester hydrochloride (1 mmol), *o*-allylnapthaldehyde (1 mmol) in dry dichloromethane (20 ml) and triethylamine (1.1 mmol) was stirred for 16 h, then poured into water and extracted with dichloromethane. The organic layer was dried with anhydrous magnesium sulfate and concentrated *in vacuo*. The resulting imine was then refluxed in dry toluene for 12 h to obtain the title compound. The compound was recrystallized by slow evaporation of a methanol solution to obtain good diffraction quality crystals.

Crystal data

$C_{26}H_{24}N_2O_3$
M_r = 412.47
Monoclinic, $P2_1/c$
a = 8.7481 (8) Å
b = 19.0068 (18) Å
c = 12.4940 (12) Å
β = 95.891 (2)°
V = 2066.4 (3) Å^3
Z = 4
D_x = 1.326 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 7163 reflections
θ = 2.3–27.6°
μ = 0.09 mm^{-1}
T = 273 (2) K
Block, colourless
0.26 × 0.22 × 0.20 mm

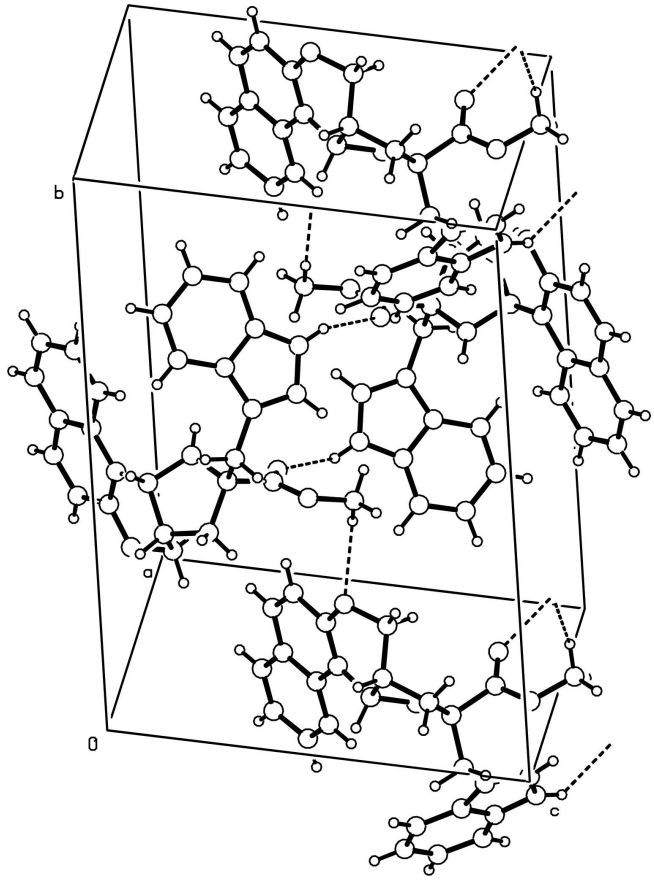


Figure 2
The molecular packing of (I), viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

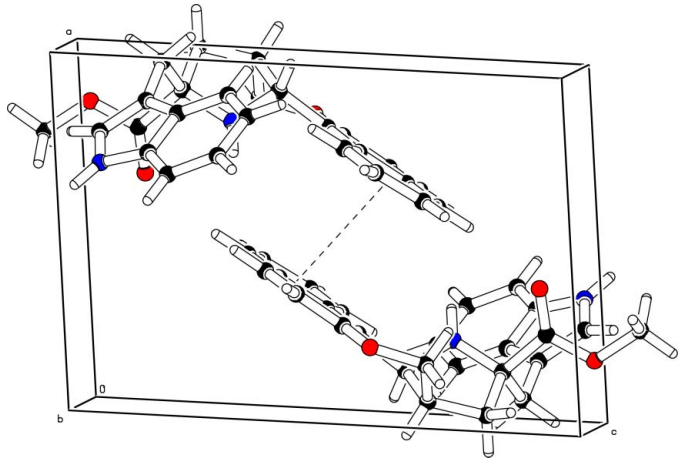


Figure 3
The molecular packing of (I), viewed down the *b* axis, showing the π–π interaction as a dashed line.

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
17 613 measured reflections
4839 independent reflections
3776 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 28.0°
h = −11 → 11
k = −24 → 24
l = −16 → 16

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1029P)^2 + 0.2052P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.050$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.162$	$\Delta\rho_{max} = 0.43$ e Å^{-3}
$S = 1.01$	$\Delta\rho_{min} = -0.34$ e Å^{-3}
4839 reflections	
280 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—C8	1.368 (2)	O1—C16	1.435 (2)
N1—C1	1.363 (2)	O2—C25	1.201 (2)
N2—C10	1.458 (2)	O3—C25	1.325 (2)
N2—C11	1.467 (2)	O3—C26	1.437 (2)
O1—C15	1.371 (2)		
C26—O3—C25—O2	1.6 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	2.38	3.075 (2)	139
C26—H26*C*···O1^{ii}	0.96	2.42	3.209 (2)	139

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

The H atoms were positioned geometrically and were treated as riding on their parent atoms, with an N—H distance of 0.86 Å and C—H distances of 0.93–0.98 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H and 1.2U_{eq}(C,N) for other H atoms.

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: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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References

Biava, M., Porretta, G. C., Poce, G., Deidda, D., Pompei, R., Tafi, A. & Manetti, F. (2005). *Bioorg. Med. Chem.* **13**, 1221–1230.
Borthwick, A. D., Davies, D. E., Erti, P. F., Exall, A. M., Haley, T. M., Hart, G. J., Jackson, D. L., Parry, N. R., Patikis, A., Trivedi, N., Weingarten, C. G. & Woolven, J. M. (2003). *J. Med. Chem.* **46**, 4428–4449.
Bruker (2001). *SAINT* (Version 6.28*a*) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
Chandrakantha, T. N., Puttaraja & Vasantha Pattabhi (1990). *Acta Cryst.* C**46**, 1697–1700.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Fernandes, E., Costa, D., Toste, S. A., Lima, J. L. & Reis, S. (2004). *Free Radic. Biol. Med.* **37**, 1895–1905.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Rajeswaran, W. G., Labroo, R. B. & Cohen, L. A. (1999). *J. Org. Chem.* **64**, 1369–1371.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tanaka, M., Sasaki, Y., Kimura, Y., Fukui, T., Hamada, K. & Ukai, Y. (2003). *BJU Int.* **92**, 1031–1036.
Ueda, I., Marubayashi, N., Ueno, K. & Tuge, O. (1991). *Acta Cryst.* C**47**, 842–845.