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3-Acetyl-4-furyl-1-methylspiro[pyrrolidine-2,2′-indol]-2′(3′*H*)-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.130
Data-to-parameter ratio = 10.2

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

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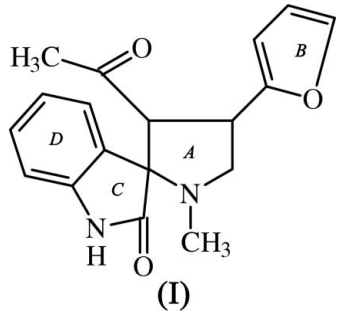
In the title compound, $C_{18}H_{18}N_2O_3$, the central pyrrolidine ring adopts a half-chair conformation. The furan ring is almost perpendicular to the oxindole system. Intermolecular C—H···O and N—H···N hydrogen bonds link the molecules into ribbons running along the *b*-axis direction.

Comment

The pyrrolidine skeleton occurs in many families of biologically important compounds. The resulting functionality, due to ease of substitution and therefore of modification at several positions (Baldwin *et al.*, 1994*a*,*b*), has been utilized to synthesize compounds with varying properties. These substituted pyrrolidine compounds have been found to have antimicrobial and antifungal activities against various pathogens (Amalraj *et al.*, 2003). The spiro-indole-pyrrolidine ring system is encountered in many pharmacologically important alkaloids, as typified by Vincrinstine, Vinblastine and spirotypostatins (Cordel, 1981; Bindra, 1973). Oxindole derivatives are found to be potent aldose reductase inhibitors (ARIs), which help to treat and prevent diabetic complications arising from elevated levels of sorbitol (Rajeswaran *et al.*, 1999). In view of this medicinal importance, and as a continuation of our studies, an X-ray crystallographic analysis of the title compound, (I), was carried out and the results are presented here.



(I)

Fig. 1 shows a displacement ellipsoid plot of (I) with the atomic numbering scheme. In the benzene ring of the oxindole system, the endocyclic angles at C15 and C18 are narrowed to 118.6 (3) and 118.0 (3)°, respectively, while those at C16 and C19 are widened to 121.0 (2) and 121.8 (2)°, respectively. The deviations of these angles from the normal value of 120° may be due to fusion of the smaller pyrrole ring to the six-membered benzene ring, the strain being taken up by angular distortion rather than by bond-length distortions (Sethu Sankar *et al.*, 2002; Govind *et al.*, 2004).

The orientation of the furan ring with respect to the pyrrolidine ring is given by the torsion angles N1—C5—C4—

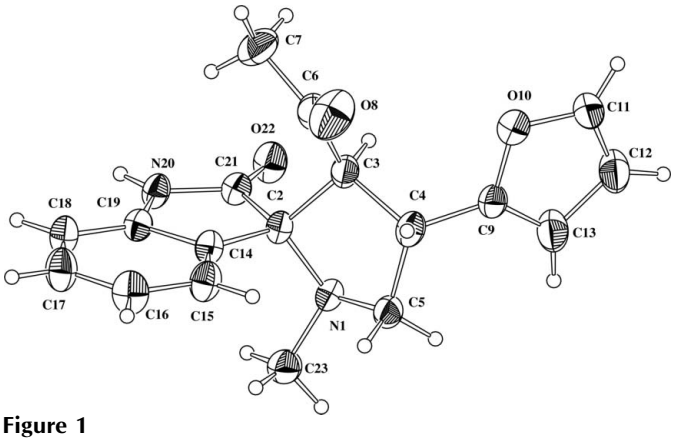


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

C9 = −106.8 (2)° and C3—C4—C9—O10 = 134.5 (2)°. The acetyl group is twisted with respect to the pyrrolidine ring, as evidenced by the torsion angles C7—C6—C3—C2 = −67.1 (3)° and C7—C6—C3—C4 = 171.8 (3)°. The keto atom O22 is displaced by 0.043 (2) Å from the indole plane (C2/C14–C19/N20/C21). The furan ring is almost perpendicular to the oxindole ring system, forming a dihedral angle of 88.8 (1)°.

Ring *A* adopts a half-chair conformation, with puckering parameters (q_2 and φ; Cremer & Pople, 1975) and asymmetry parameters (Δ; Nardelli, 1983) as follows: q_2 = 0.405 (2) Å, φ = 164.0 (3)° and ΔC_2(C4) = 0.011 (1). Rings *B*, *C* and *D* are each planar.

The crystal structure of (I) is stabilized by intermolecular C—H···O and N—H···N hydrogen bonds (Table 1), which link the molecules into ribbons running along the *b*-axis direction.

Experimental

A solution of furyl acetone (1 mmol), sarcosine (1 mmol) and isatin (1 mmol) was refluxed in aqueous methanol (20 ml) for 4 h. After completion of the reaction, as evidenced by thin-layer chromatography, the residue was chromatographed with petroleum ether–ethyl acetate (4:1) to obtain the title compound. Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{18}H_{18}N_2O_3$
M_r = 310.34
Orthorhombic, $P2_12_12_1$
a = 7.7404 (6) Å
b = 10.0227 (7) Å
c = 20.5622 (16) Å
V = 1595.2 (2) Å^3
Z = 4
D_x = 1.292 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.30 × 0.21 × 0.13 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
9691 measured reflections
2125 independent reflections
1796 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.130
S = 1.09
2125 reflections
209 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0753P)^2 + 0.1621P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O8	0.98	2.39	2.822 (4)	106
N20—H20···N1^{i}	0.86	2.24	3.058 (3)	158
C18—H18···O22^{i}	0.93	2.37	3.226 (3)	153

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

All H atoms were positioned geometrically and were treated as riding on their parent atoms, with N—H = 0.86 Å and C—H = 0.93–0.98 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C,N) for other H atoms. Owing to the absence of any significant anomalous scatterers in the molecule, Friedel pairs were merged before the final refinement.

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: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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