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1-(2-Bromo-1-methyl-1*H*-indol-3-ylcarbonyl)-pyrrolidine

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.016 Å
R factor = 0.074
wR factor = 0.277
Data-to-parameter ratio = 17.4

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

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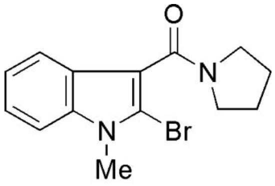
In the title compound, $C_{14}H_{15}BrN_2O$, the pyrrolidine ring adopts a twist conformation. The amide group is twisted out of π-conjugation with the pyrrole double bond due to steric hindrance by the Br atom.

Comment

In view of our interest in the synthesis and chemistry of halogenated indoles (Gribble *et al.*, 1992; Liu & Gribble, 2000, 2001, 2002*a*,*b*; Gribble & Liu, 2001), many of which are naturally occurring (Gribble, 2003), we have prepared several 2-bromo-3-carboxamides such as the title compound, (I), to craft the rare indolo[2,3-*a*]pyrrolizidine ring system *via* the generation of an indole C-2 radical, followed by a 1,5-radical translocation process and subsequent 5-endo-trig cyclization (Gribble *et al.*, 2001). We now describe the crystal structure of (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The indole ring system is planar. The sum of the angles around both the indole nitrogen N1 (360.0°) and amide nitrogen N2 (359.0°) indicate sp^2 hybridization. The pyrrolidine ring adopts a twist conformation. The carbonyl oxygen is twisted away from the bromine with a O1—C11—C3—C2 torsion angle of −51.3 (16)°, indicating little or no π-conjugation between the pyrrole double bond and the carbonyl group. The C—Br bond length is 1.836 (8) Å.

The molecules are linked into a chain along the *b* axis by C7—H3···O1(*x*, 1 + *y*, *z*) hydrogen bonds [H3···O1 = 2.48, C7···O = 3.381 (15) Å and C7—H3···O1 = 163°].

Experimental

A solution of 1-(1-methylindol-3-ylcarbonyl)pyrrolidine (49.3 mg, 0.20 mmol) in tetrahydrofuran (5 ml) was treated with ^{t}BuLi (0.1 ml, 0.22 mmol, 1.7 *M* in hexanes) at 195 K and the solution immediately turned bright yellow. The solution was stirred at this temperature for 1 h, then warmed to room temperature over a period of 30 min. The solution was then cooled to 273 K and quenched with a solution of 1,2-dibromo-1,1,2,2-tetrachloroethane (71.7 mg, 0.22 mmol) in tetrahydrofuran (5 ml). The reaction was then warmed to room temperature overnight, quenched with water (1 ml), and concen-

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trated *in vacuo* to give a yellow residue. The residue was purified by preparative chromatography (1:1 hexanes–ethyl acetate) to give 57.9 mg of (I) as a yellow solid (94%). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethyl acetate solution (m.p. 373-378 K).

Crystal data

$C_{14}H_{15}BrN_2O$
M_r = 307.19
Triclinic, $P\bar{1}$
a = 7.327 (2) Å
b = 9.387 (3) Å
c = 10.412 (4) Å
α = 96.14 (3)°
β = 100.94 (3)°
γ = 109.32 (3)°
V = 652.3 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.14 mm^{-1}
T = 296 K
0.40 × 0.30 × 0.20 mm

Data collection

Rigaku AFC-6S diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.367, T_{max} = 0.572 (expected range = 0.342–0.534)
2850 measured reflections
2850 independent reflections
833 reflections with $I > 2\sigma(I)$
3 standard reflections every 150 reflections intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.277
S = 1.07
2850 reflections
164 parameters
18 restraints
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

H atoms were included in the riding-model approximation, with C—H = 0.93-0.97 Å and U_{iso}(H) = 1.18 or 1.20U_{eq}(C). The displacement parameters of atoms C6, C7 and C8 were restrained to approximately isotropic behaviour. Owing to the poor diffraction quality of the crystal, the ratio of observed to unique reflections is low (0.29) and this is a structure of low precision.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *CrystalStructure* (Rigaku/MSC, 2005); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

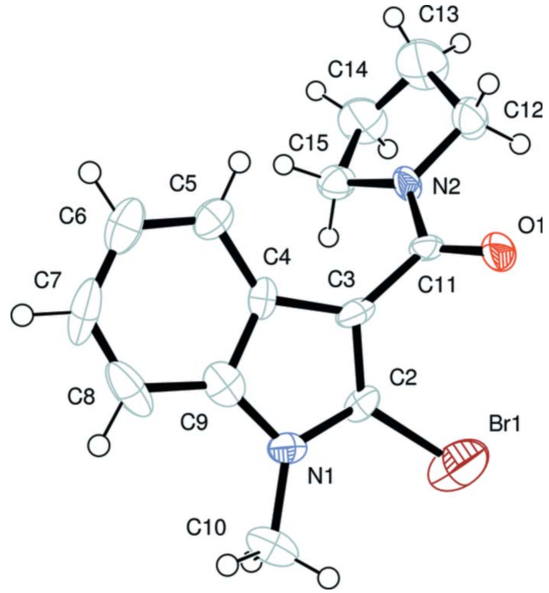

Figure 1
The molecular structure of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids.

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