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

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In the title compound, $C_{15}H_{17}BrN_2O$, the amide group is twisted out of π-conjugation with the indole double bond due to steric hindrance by the Br atom. The piperidine ring adopts a chair conformation.

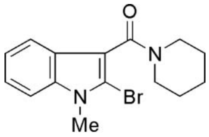
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.019 Å
R factor = 0.061
wR factor = 0.245
Data-to-parameter ratio = 13.7

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

Comment

The background to this study is set out in the preceding paper (Badenock *et al.* 2007). We report here the crystal structure of (I).



(I)

The X-ray crystallographic analysis confirms the proposed molecular structure and atom connectivity for (I) (Fig. 1). The indole ring system is planar. The sum of the angles around both the indole nitrogen N1 (360°) and amide nitrogen N2 (358.2°) indicate sp^2 hybridization, as expected. The piperidine ring adopts a chair conformation. The carbonyl oxygen, O1, is twisted away from the bromine atom, Br1, with a O1—C11—C3—C2 torsion angle of 58 (2)°, indicating little or no π-conjugation between the indole double bond and the carbonyl group. The C—Br bond length is 1.803 (9) Å.

The crystal packing of (I) is stabilized by C—H···Br and C—H···O intermolecular hydrogen bonds.

Experimental

To a stirred solution of 1-(1-methylindol-3-ylcarbonyl)piperidine (1.50 g, 6.21 mmol, 1 eq.) in tetrahydrofuran (35 ml) at 195 K was added ^{t}BuLi (4.30 ml, 7.01 mmol, 1.63 *M* in hexanes, 1.13 eq.) dropwise. The solution was stirred at 195 K for 1 h. At this time the anion was treated with 1,2-dibromo-1,1,2,2-tetrachloroethane (2.21 g, 6.78 mmol, 1.09 eq.) in tetrahydrofuran (20 ml). The solution was allowed to warm to room temperature slowly overnight. The reaction mixture was then poured on to ice–water (100 ml) and stirred for 2.5 h. The resulting white precipitate was collected by vacuum filtration and dried under vacuum to give 0.77 g (41%) of bromide (I). This was homologous by TLC and ^{1}H NMR. The filtrate was extracted with CH_2Cl_2 (4 × 75 ml) and the organic layers were combined. The yellow solution was washed with brine (1×100 ml), dried over Na_2SO_4 and concentrated *in vacuo* to give a yellow solid (1.25 g). This was purified using flash chromatography (1:1 hexanes–ethyl acetate) to give (I) (0.90 g, 48%) as a white solid; total yield of (I) was 1.67 g (84%). Crystals suitable for the X-ray study were grown from ethyl acetate [m.p. 379–380 K; literature value 378–380 K (Hassinger, 1999)].

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Crystal data

$C_{15}H_{17}BrN_2O$
$M_r = 321.22$
Triclinic, $P\overline{1}$
$a = 7.819$ (3) Å
$b = 8.218$ (3) Å
$c = 11.114$ (5) Å
$\alpha = 69.99$ (3)°
$\beta = 91.21$ (4)°
$\gamma = 111.16$ (3)°
$V = 621.5$ (4) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 3.30$ mm^{-1}
$T = 296$ K
$0.50 \times 0.30 \times 0.30$ mm

Data collection

Rigaku AFC-6S diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.289$, $T_{\max} = 0.438$ (expected range = 0.246–0.372)
2370 measured reflections
2370 independent reflections
750 reflections with $I > 2\sigma(I)$
3 standard reflections every 150 reflections intensity decay: 4.3%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.245$
$S = 0.95$
2370 reflections
173 parameters
All H-atom parameters refined
$\Delta\rho_{\max} = 0.47$ e Å^{-3}
$\Delta\rho_{\min} = -0.91$ e Å^{-3}

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
C7—H3$\cdots$O1^{i}	0.93	2.38	3.27 (2)	160
C10—H5$\cdots$O1^{ii}	0.96	2.36	3.200 (17)	145
C16—H16$\cdots$Br1^{iii}	0.97	2.73	3.464 (13)	133

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

H atoms were included in the riding-model approximation, with C—H = 0.93-0.97 Å and $U_{\rm iso}$(H) = 1.19 or $1.21U_{\rm eq}$(C). The ratio of observed to unique reflections is low (32%), as is the fraction of reflections measured (83%), due to the poor diffraction quality of the crystal. This is a structure of relatively 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*.

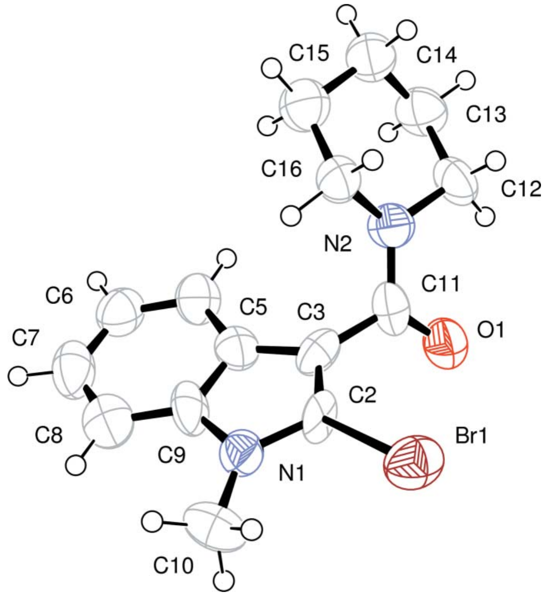

Figure 1
The molecular structure of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids.

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