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Azepinyl 2-bromo-1-methyl-1*H*-indol-3-yl ketone

Heidi L. Fraser,[a] Jeanese C. Badenock,[a] Gordon W. Gribble[a] and Jerry P. Jasinski[b]*

[a]Department of Chemistry, Dartmouth College, Hanover, NH 03755-3564, USA, and [b]Department of Chemistry, Keene State College, Keene, NH 03435-2001, USA

Correspondence e-mail: jjasinski@keene.edu

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.012 Å
R factor = 0.073
wR factor = 0.231
Data-to-parameter ratio = 18.5

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

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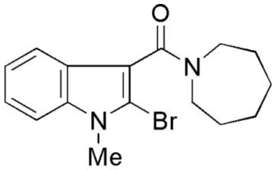
The crystal structure of the title compound, $C_{16}H_{19}BrN_2O$, reveals that the amide group is twisted out of π-conjugation with the indole double bond due to steric hindrance by the Br atom. The azepane ring adopts a twist-chair conformation.

Comment

The background to this study is set out in Badenock *et al.* (2007). We now describe the X-ray crystal structure determination of (I).



(I)

In the molecular structure of (I) (Fig. 1), the indole ring system is planar. The sum of the bond angles around both the indole nitrogen N1 (360.0°) and amide nitrogen N2 (359.2°) indicate sp^2 hybridization. The azepane ring adopts a twist-chair conformation. The carbonyl oxygen O1 is twisted away from the bromine atom Br1, with an O1—C11—C3—C2 torsion angle of 76.0 (8)°, indicating little or no π-conjugation between the indole double bond and the carbonyl group. The C—Br bond length is 1.861 (7) Å.

Experimental

To a stirred solution of 1-azepinyl-(1-methyl-1*H*-indol-3-yl)methanone (632 mg, 2.47 mmol, 1 equivalent) in tetrahydrofuran (40 ml) was added ^{t}BuLi (1.8 ml, 2.96 mmol, 1.7 *M* in hexanes, 1.2 equivalents) at 195 K. The solution was stirred at 195 K for 1.5 h, treated with dried 1,2-dibromo-1,1,2,2-tetrachloroethane (0.97 g, 2.96 mmol, 1.2 equivalents) in tetrahydrofuran (20 ml) and allowed to warm to room temperature overnight. The reaction mixture was poured on to ice–water (100 ml) and stirred for 30 min. After repeated extraction with CH_2Cl_2, the organic layers were combined, washed with brine, dried over Na_2SO_4, and concentrated *in vacuo*. Purification using flash column chromatography (1:1 hexanes–ethyl acetate) gave compound (I) (yield 0.75 g, 90%) as a white solid. An analytical sample of (I) was obtained by several recrystallizations from ethyl acetate. Single crystals suitable for X-diffraction study were grown in ethyl acetate (m.p. 398-399 K).

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Crystal data

$C_{16}H_{19}BrN_2O$
$M_r = 335.24$
Triclinic, $P\overline{1}$
$a = 6.9348\ (9)$ Å
$b = 10.7030\ (19)$ Å
$c = 10.882\ (3)$ Å
$\alpha = 108.384\ (17)^\circ$
$\beta = 97.601\ (16)^\circ$
$\gamma = 100.395\ (13)^\circ$
$V = 738.3\ (3)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 2.78$ mm^{-1}
$T = 296$ K
$0.50 \times 0.40 \times 0.30$ mm

Data collection

Rigaku AFC-6S diffractometer
Absorption correction: ψ scan
(North *et al.*, 1968)
$T_{\min} = 0.337$, $T_{\max} = 0.489$
(expected range = 0.299–0.434)
3376 measured reflections
3376 independent reflections
1471 reflections with $I > 2\sigma(I)$
3 standard reflections
every 150 reflections
intensity decay: 1.4%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.231$
$S = 1.00$
3376 reflections
182 parameters
All H-atom parameters refined
$\Delta\rho_{\max} = 0.75$ e Å^{-3}
$\Delta\rho_{\min} = -1.97$ e Å^{-3}

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 1.21$U_{\rm eq}$(C). Owing to the large number of weak high-angle reflections, the ratio of observed to unique reflections is low (44%).

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*.

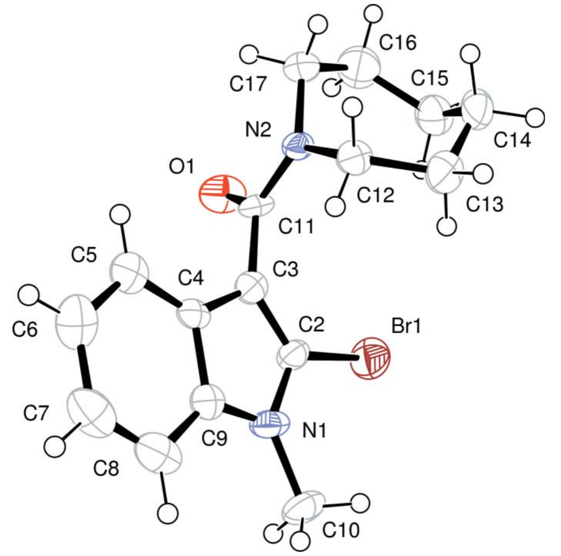

Figure 1
The molecular structure of (I), showing the atomic numbering and 50% probability displacement ellipsoids.

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