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2-Bromo-*N*-*tert*-butyl-*N*,1-dimethyl-1*H*-indole-3-carboxamide

Jeanese C. Badenock,[a] Heidi L. Fraser,[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

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Key indicators

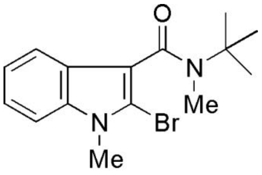
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.013 Å
R factor = 0.051
wR factor = 0.175
Data-to-parameter ratio = 14.6

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

The crystal structure of the title compound, $C_{15}H_{19}BrN_2O$, reveals that the amide group is twisted out of π-conjugation with the pyrrole double bond due to steric hindrance by the Br atom.

Comment

The background to this study is set out in Badenock *et al.* (2007). We now describe the X-crystal structure determination 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, with a maximum deviation of 0.025 (9) Å for atom C5. The sum of the bond angles around N1 and N2 are 360.1 and 358.4°, respectively, indicating sp^2 hybridization. However, the O1—C11—N2—C13 torsion angle of −18.9 (11)° reveals imperfect π-conjugation in the amide group due to repulsion between the *tert*-butyl group and the carbonyl oxygen. In addition, the carbonyl oxygen (O1) is severely twisted away from the bromine atom (Br1) with a O1—C11—C3—C2 torsion angle of 134.5 (9)°, indicating little or no π-conjugation between the pyrrole double bond and the carbonyl group. The C2—Br1 bond length is 1.862 (8) Å.

Experimental

To a stirred solution of *N*-*tert*-butyl-*N*,1-dimethyl-1H-indole-3-carboxamide (816 mg, 3.34 mmol, 1 equivalent) in tetrahydrofuran (40 ml) was added slowly ^{t}BuLi (3.0 ml, 5.01 mmol, 1.7 *M* in hexanes, 1.5 equivalents) at 195 K and the solution was stirred at 195 K for 1 h, then warmed to room temperature over 30 min before recooling to 195 K. At this time, the anion was treated with dried 1,2-dibromo-1,1,2,2-tetrachloroethane (1.63 g, 5.01 mmol, 1.5 equivalents) in tetrahydrofuran (20 ml). The solution was warmed to room temperature overnight, then poured on to ice–water (100 ml) and stirred for 2 h. No precipitate formed upon stirring. The aqueous layer was extracted with CH_2Cl_2 (4 × 50 ml). The organic layers were combined, washed with brine (1 × 100 ml), dried (Na_2SO_4), and concentrated *in vacuo* to give a brown oil. Purification using flash column chromatography (2:1 hexanes–ethyl acetate) gave compound

(I) as a yellow solid (887 mg, 86%). An analytical sample of (I) was obtained by several recrystallizations from diethyl ether. Single crystals suitable for X-diffraction study were grown in diethyl ether (m.p. 378–379 K).

Crystal data

$C_{15}H_{19}BrN_2O$
$M_r = 323.23$
Monoclinic, $P2_1/n$
$a = 7.373$ (7) Å
$b = 21.453$ (7) Å
$c = 9.132$ (9) Å
$\beta = 96.06$ (9)°
$V = 1436$ (2) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 2.86$ mm^{-1}
$T = 296$ (2) K
0.50 × 0.40 × 0.30 mm

Data collection

Rigaku AFC-6S diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.329$, $T_{\max} = 0.481$ (expected range = 0.291–0.425)
2526 measured reflections
2526 independent reflections
992 reflections with $I > 2\sigma(I)$
3 standard reflections every 150 reflections intensity decay: 1.4%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.175$
$S = 1.02$
2526 reflections
173 parameters
All H-atom parameters refined
$\Delta\rho_{\max} = 0.51$ e Å^{-3}
$\Delta\rho_{\min} = -0.45$ e Å^{-3}

H atoms were included in the riding-model approximation, with C—H = 0.93–0.96 Å and $U_{\rm iso}$(H) = 1.18 or 1.20$U_{\rm eq}$(C). Owing to the large number of weak high-angle reflections, the ratio of observed to unique reflections is low (39%), 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*.

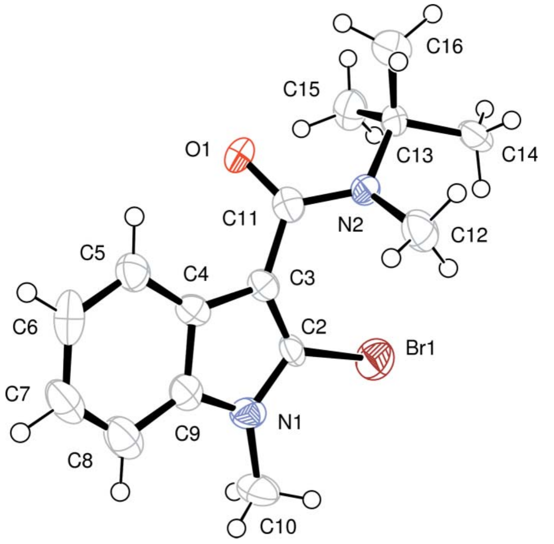


Figure 1
The molecular structure of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids.

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