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# *N*-(5-Bromo-2-iodophenyl)-*N*-methylcyclohexanecarboxamide

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**Key indicators**

Single-crystal X-ray study
*T* = 93 K
Mean σ(C–C) = 0.008 Å
*R* factor = 0.040
*wR* factor = 0.108
Data-to-parameter ratio = 15.3

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

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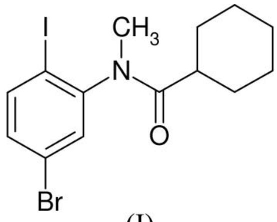
The title compound, $C_{14}H_{17}BrINO$, contains two molecules in the asymmetric unit which are linked into dimeric associations by way of very short C—I···O interactions [I···O = 3.038 (4) and 3.082 (4) Å].

## Comment

The title compound, (I), was prepared as part of our ongoing studies of intramolecular ring-forming reactions (Percival *et al.*, 2007).



(I)

There are two molecules in the asymmetric unit of (I) (Fig. 1) and their geometrical parameters fall within the expected ranges (Allen *et al.*, 1987). For the first (C1) molecule, the dihedral angle between the mean planes of the aromatic ring and the methylated amide (C7/C8/N1/O1) group is 82.1 (3)°. A dihedral angle of 82.6 (3)° occurs for the equivalent atoms in the second (C15) molecule. In both molecules, the methyl C atom and the O atom of the amide are in *cis* conformations. The chair conformations of the cyclohexane rings for both molecules are normal.

When superimposed (Fig. 2), the only significant difference between the two molecules is the degree of twisting of the cyclohexane ring about the C8—C9 and C22—C23 bonds in the first and second molecules, respectively. This is reflected in

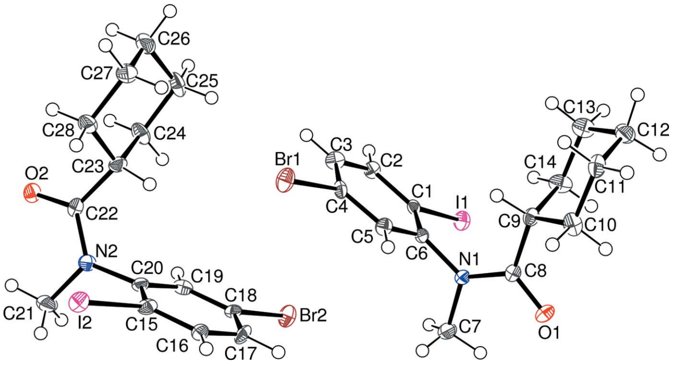


**Figure 1**
The asymmetric unit of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

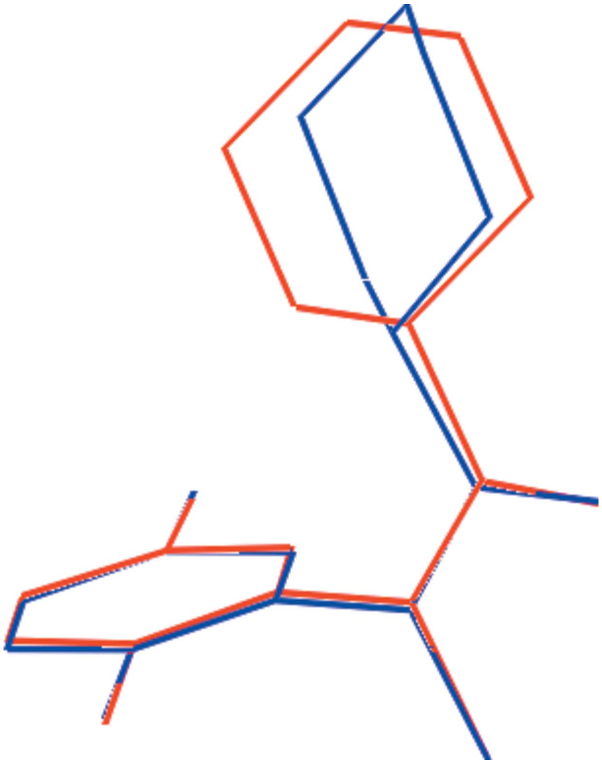


**Figure 2**
Superposition of the C1 (red) and C15 (blue) molecules of (I), with H atoms omitted for clarity.

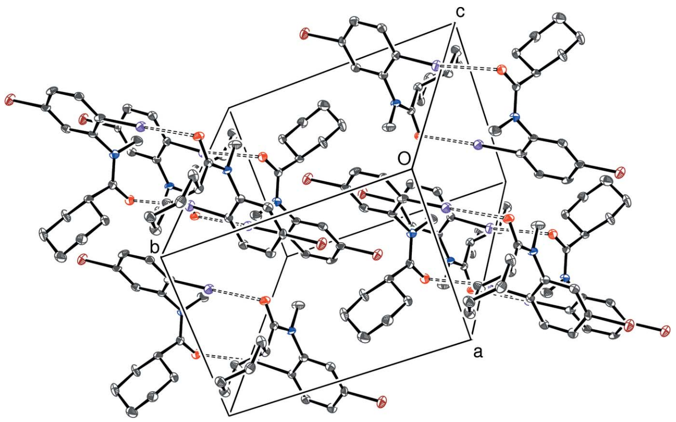


**Figure 3**
The packing of (I), with H atoms omitted for clarity and I···O interactions shown as dashed lines.

the differing O1—C8—C9—C10 [−32.7 (7)°] and O2—C22—C23—C24 [−65.6 (7)°] torsion angles.

In the crystal structure, the molecules of (I) interact by way of very short C—I···O interactions (Glidewell *et al.*, 2005) with an iodine–oxygen separation of 3.082 (4) Å for C1—I1···O2$^{i}$ [symmetry code: (i) $x - 1$, $y + 1$, $z$], some 0.42 Å shorter than the Bondi (1964) van der Waals separation. The C1—I1···O2$^{i}$ angle is 169.28 (17)°. Likewise, C15—I2···O1$^{ii}$ [symmetry code: (ii) $x + 1$, $y - 1$, $z$] features an equivalent separation of 3.038 (4) Å and an angle of 173.03 (17)°. These somewhat enigmatic interactions (Allen *et al.*, 1997) lead to dimeric associations of the C1 and C15 molecules in the crystal structure of (I) (Fig. 3).

## Experimental

5-Bromo-2-iodoaniline (596 mg, 2.55 mmol) was added to dichloromethane (DCM, 5 ml) with triethylamine (0.7 ml, 5 mmol), and the mixture stirred magnetically while chilled in an ice bath. Once cool, cyclohexane carbonyl chloride (0.4 ml, 3 mmol) was added dropwise, and the mixture stirred for 2 h during which time a precipitate was produced. Water (10 ml) was added to the flask and the DCM layer was then separated and washed with saturated sodium hydrogen carbonate (15 ml) and brine (15 ml), during which time the mixture emulsified. The DCM layer was filtered, yielding a pure white filtrate and a yellow liquor which was dried ($MgSO_4$), and the solvent removed to yield colourless plates of cyclohexanecarboxylic acid (5-bromo-2-iodophenyl)amide, (II).

Compound (II) (750 mg, 1.8 mmol) was dissolved in dry THF (10 ml), then injected into a pre-dried flask containing sodium hydride (40 mg, 1.8 mmol) in dry THF (10 ml) and the mixture stirred magnetically. When bubbling of the mixture ceased, methyl iodide (0.12 ml, 1.98 mmol) was added and the reaction mixture stirred overnight. Ammonium carbonate solution (10 ml) was then added and the mixture was stirred for 10 min, during which time a white precipitate formed which redissolved on the addition of water (15 ml). An extraction was performed into ethyl acetate (3 × 20 ml). Purification by flash column chromatography (10:1 *v*/*v* hexane–ethyl acetate) yielded 385 mg (51%) of the title compound with $R_F = 0.20$. Recrystallization from ethyl acetate afforded colourless prisms of (I).

*Crystal data*

$C_{14}H_{17}BrINO$
$M_r = 422.10$
Triclinic, $P\overline{1}$
$a = 9.0837$ (10) Å
$b = 12.2016$ (17) Å
$c = 13.702$ (2) Å
$\alpha = 88.495$ (8)°
$\beta = 84.782$ (8)°
$\gamma = 79.050$ (8)°
$V = 1484.8$ (3) Å$^3$
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 4.84$ mm$^{-1}$
$T = 93$ (2) K
0.30 × 0.20 × 0.10 mm

*Data collection*

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2004)
$T_{min} = 0.325$, $T_{max} = 0.643$
9422 measured reflections
4997 independent reflections
4376 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$

*Refinement*

$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.108$
$S = 1.07$
4997 reflections
327 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 1.08$ e Å$^{-3}$
$\Delta\rho_{min} = -1.14$ e Å$^{-3}$

The H atoms were positioned geometrically (C—H = 0.95–0.99 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C). The highest difference peak is 1.20 Å from H25*A* and the deepest difference hole is 0.84 Å from I1.

Data collection: *CrystalClear* (Rigaku, 2004); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

**o2118** Slawin *et al.* • $C_{14}H_{17}BrINO$ *Acta Cryst.* (2007). E**63**, o2117–o2119

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*Acta Cryst.* (2007). E**63**, o2117–o2119 Slawin *et al.* • $C_{14}H_{17}BrINO$ **o2119**