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N-(5-Bromo-2-iodophenyl)-*N*-methylcyclopentanecarboxamide

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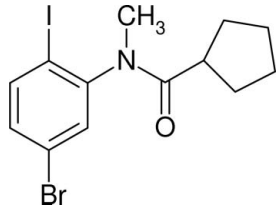
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.039; wR factor = 0.102; data-to-parameter ratio = 16.7.

The title compound, $C_{13}H_{15}BrINO$, contains two molecules in the asymmetric unit which are linked into dimeric associations by way of very short C—I···O interactions [I···O = 2.998 (4) and 3.044 (4) Å]. The cyclopentane rings of both molecules are disordered; the site occupancy ratios are *ca* 0.54:0.46 and 0.59:0.41.

Related literature

For a related structure, see Slawin *et al.* (2007). For background on C—I···O interactions, see: Allen *et al.* (1997); Glidewell *et al.* (2005). For crystallographic reference data, see: Bondi (1964); Allen *et al.* (1987).



Experimental

Crystal data

$C_{13}H_{15}BrINO$
M_r = 408.07
Triclinic, $P\overline{1}$
a = 9.0116 (14) Å
b = 11.8073 (18) Å
c = 13.697 (2) Å
α = 86.430 (12)°
β = 85.395 (13)°
γ = 78.849 (11)°
V = 1423.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.04 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} = 0.313, T_{max} = 0.633
9341 measured reflections
5099 independent reflections
4616 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.102
S = 1.02
5099 reflections
305 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.28 e Å^{-3}
$\Delta\rho_{min}$ = −1.51 e Å^{-3}

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2427).

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supplementary materials

Acta Cryst. (2007). E**63**, o3407 [**doi:10.1107/S1600536807032370**]

N-(5-Bromo-2-iodophenyl)-*N*-methylcyclopentanecarboxamide

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Comment

The title compound, (I), complements the recently reported cyclohexanecarboxylic acid (5-bromo-2-iodo-phenyl)-methyl-amide, (II), (Slawin *et al.*, 2007), with a cyclopentane ring in (I) replacing a cyclohexane ring in (II).

There are two molecules in the asymmetric unit of (I) and their geometrical parameters fall within the expected ranges, allowing for the uncertainties arising from the disordered cyclopentane rings (Allen *et al.*, 1995).

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 85.4 (2)°. A dihedral angle of 82.35 (17)° occurs for the equivalent atoms in the second (C14) molecule. In both molecules the methyl C atom and the O atom of the amide are in *cis* conformation.

In the crystal, the molecules of (I) form dimers (Fig. 1) by way of two very short C—I···O interactions (Allen *et al.*, 1997; Glidewell *et al.*, 2005) with the iodine···oxygen separations for C1—I1···O2 and C14—I2···O1 being 3.044 (4)Å and 2.998 (4) Å, respectively. The Bondi (1964) van der Waals' separation for I and O is 3.50 Å. The C1—I1···O2 and C14—I2···O1 angles are 171.12 (14)° and 174.44 (14), respectively.

A dimerization of the two asymmetric molecules *via* two very short C—I···O interactions [I···O = 3.038 (4) and 3.082 (4) Å]. also occurs in (II) (Slawin *et al.*, 2007).

Experimental

2-Iodo-5-bromoaniline (596 mg, 2.55 mmol) was added to DCM (5 ml) with triethylamine (0.7 ml, 5 mmol), and the mixture stirred magnetically whilst chilled in an ice bath. Once cool, cyclopentane carbonyl chloride (0.4 ml, 3 mmol) was added dropwise, and the mixture stirred for 2 hr during which time a precipitate was produced. Water (10 ml) was added to the flask, then the DCM layer was 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 to yield a pure white filtrate and a yellow liquor which was dried ($MgSO_4$), and the solvent removed to yield colourless plates of cyclopentanecarboxylic acid (5-bromo-2-iodo-phenyl)-amide, (III).

Compound (III) (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 left stirring overnight. Ammonium carbonate solution (10 ml) was then added and the mixture allowed to stir 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).

Refinement

The disordered atoms were refined isotropically. The hydrogen atoms were geometrically placed (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 1.20Å from H25A and the deepest difference hole is 0.84Å from I1.

Figures

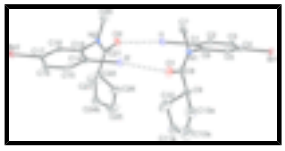


Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids (H atoms omitted for clarity) showing the C—I···O interactions as double dashed lines. Only one disorder component of each cyclopentane ring is shown.

N-(5-Bromo-2-iodophenyl)-*N*-methylcyclopentanecarboxamide

Crystal data

$C_{13}H_{15}BrINO$	V = 1423.6 (4) Å^3
M_r = 408.07	Z = 4
Triclinic, $P\overline{1}$	F_{000} = 784
Hall symbol: -P 1	D_x = 1.904 Mg m^{-3}
a = 9.0116 (14) Å	Mo $K\alpha$ radiation λ = 0.71073 Å
b = 11.8073 (18) Å	μ = 5.04 mm^{-1}
c = 13.697 (2) Å	T = 93 (2) K
α = 86.430 (12)º	Block, colourless
β = 85.395 (13)º	0.30 × 0.20 × 0.10 mm
γ = 78.849 (11)º	

Data collection

Rigaku Mercury CCD diffractometer	5099 independent reflections
Radiation source: rotating anode	4616 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.033
T = 93(2) K	θ_{max} = 26.0º
ω and φ scans	θ_{min} = 2.3º
Absorption correction: multi-scan (CrystalClear; Rigaku, 2004)	h = −9→11
T_{min} = 0.313, T_{max} = 0.633	k = −14→14
9341 measured reflections	l = −13→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.102$

$S = 1.02$

5099 reflections

305 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 4.5633P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 1.28$ e Å^{-3}

$\Delta\rho_{min} = -1.51$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	1.1153 (5)	−0.1887 (4)	0.2547 (3)	0.0124 (9)	
C2	1.2706 (5)	−0.2157 (4)	0.2354 (4)	0.0161 (10)	
H2	1.3310	−0.1606	0.2462	0.019*	
C3	1.3396 (5)	−0.3226 (4)	0.2003 (3)	0.0160 (10)	
H3	1.4463	−0.3408	0.1868	0.019*	
C4	1.2494 (5)	−0.4020 (4)	0.1854 (3)	0.0132 (9)	
C5	1.0937 (5)	−0.3760 (4)	0.2046 (3)	0.0121 (9)	
H5	1.0338	−0.4317	0.1945	0.015*	
C6	1.0248 (5)	−0.2686 (4)	0.2387 (3)	0.0115 (9)	
C7	0.8129 (6)	−0.2720 (5)	0.3635 (4)	0.0272 (12)	
H7A	0.9010	−0.2976	0.4023	0.041*	
H7B	0.7536	−0.3335	0.3650	0.041*	
H7C	0.7497	−0.2027	0.3910	0.041*	
C8	0.7602 (5)	−0.2074 (4)	0.1952 (4)	0.0161 (10)	
C9	0.8155 (6)	−0.1777 (5)	0.0919 (4)	0.0285 (12)	
H9	0.9286	−0.1913	0.0913	0.034*	
C10	0.7655 (14)	−0.0507 (6)	0.0590 (6)	0.086 (4)	
H10A	0.6569	−0.0246	0.0793	0.103*	
H10B	0.8246	−0.0026	0.0908	0.103*	
C11	0.787 (2)	−0.0374 (16)	−0.0402 (13)	0.058 (5)*	0.54 (2)
H11A	0.8888	−0.0190	−0.0589	0.069*	0.54 (2)
H11B	0.7094	0.0256	−0.0663	0.069*	0.54 (2)

C12	0.773 (2)	−0.1645 (12)	−0.0832 (10)	0.042 (4)*	0.54 (2)
H12A	0.6762	−0.1593	−0.1138	0.051*	0.54 (2)
H12B	0.8583	−0.1915	−0.1317	0.051*	0.54 (2)
C13	0.7807 (15)	−0.2456 (8)	0.0118 (7)	0.018 (3)*	0.54 (2)
H13A	0.6825	−0.2706	0.0277	0.022*	0.54 (2)
H13B	0.8608	−0.3151	0.0025	0.022*	0.54 (2)
C11A	0.748 (2)	−0.0505 (15)	−0.0543 (13)	0.042 (4)*	0.46 (2)
H11C	0.6714	0.0165	−0.0759	0.051*	0.46 (2)
H11D	0.8456	−0.0482	−0.0922	0.051*	0.46 (2)
C12A	0.704 (3)	−0.1467 (18)	−0.0650 (16)	0.063 (6)*	0.46 (2)
H12C	0.7723	−0.1910	−0.1152	0.075*	0.46 (2)
H12D	0.6005	−0.1296	−0.0888	0.075*	0.46 (2)
C13A	0.702 (3)	−0.2148 (16)	0.0228 (12)	0.045 (5)*	0.46 (2)
H13C	0.7340	−0.2977	0.0090	0.054*	0.46 (2)
H13D	0.5984	−0.2030	0.0552	0.054*	0.46 (2)
N1	0.8644 (4)	−0.2453 (3)	0.2616 (3)	0.0134 (8)	
O1	0.6240 (4)	−0.1954 (3)	0.2193 (3)	0.0228 (8)	
Br1	1.33872 (5)	−0.54869 (4)	0.13779 (3)	0.01845 (13)	
I1	1.01734 (3)	−0.02584 (2)	0.30608 (2)	0.01834 (11)	
C14	0.4091 (5)	0.1964 (4)	0.3579 (3)	0.0116 (9)	
C15	0.2554 (5)	0.2366 (4)	0.3541 (3)	0.0132 (9)	
H15	0.1942	0.1892	0.3296	0.016*	
C16	0.1887 (5)	0.3460 (4)	0.3858 (3)	0.0148 (9)	
H16	0.0826	0.3734	0.3837	0.018*	
C17	0.2814 (5)	0.4141 (4)	0.4205 (3)	0.0105 (9)	
C18	0.4349 (5)	0.3751 (4)	0.4247 (3)	0.0111 (9)	
H18	0.4959	0.4227	0.4491	0.013*	
C19	0.5009 (5)	0.2656 (4)	0.3930 (3)	0.0115 (9)	
C20	0.7014 (6)	0.1548 (4)	0.4922 (4)	0.0197 (10)	
H20A	0.6094	0.1398	0.5298	0.030*	
H20B	0.7564	0.1966	0.5321	0.030*	
H20C	0.7661	0.0813	0.4745	0.030*	
C21	0.7703 (5)	0.2563 (4)	0.3387 (3)	0.0137 (9)	
C22	0.7215 (5)	0.3257 (4)	0.2463 (4)	0.0177 (10)	
H22	0.6125	0.3639	0.2557	0.021*	
C23	0.8186 (10)	0.4167 (6)	0.2159 (5)	0.059 (2)	
H23A	0.7582	0.4958	0.2221	0.071*	
H23B	0.9065	0.4074	0.2566	0.071*	
C24A	0.870 (2)	0.3936 (16)	0.1089 (11)	0.038 (5)*	0.41 (2)
H24A	0.8656	0.4677	0.0701	0.045*	0.41 (2)
H24B	0.9766	0.3510	0.1041	0.045*	0.41 (2)
C24B	0.7931 (14)	0.4481 (10)	0.1136 (7)	0.031 (3)*	0.59 (2)
H24C	0.8806	0.4769	0.0795	0.038*	0.59 (2)
H24D	0.7003	0.5077	0.1070	0.038*	0.59 (2)
C25	0.7743 (9)	0.3274 (7)	0.0718 (5)	0.0525 (18)	
H25A	0.8259	0.2816	0.0164	0.063*	
H25B	0.6797	0.3768	0.0503	0.063*	
C26	0.7441 (10)	0.2519 (6)	0.1590 (4)	0.055 (2)	
H26A	0.8308	0.1871	0.1667	0.066*	

H26B	0.6522	0.2197	0.1519	0.066*
N2	0.6596 (4)	0.2249 (3)	0.4027 (3)	0.0113 (8)
O2	0.9042 (4)	0.2256 (3)	0.3547 (3)	0.0205 (7)
Br2	0.19457 (5)	0.56264 (4)	0.46655 (3)	0.01497 (13)
I2	0.50529 (3)	0.03337 (2)	0.30769 (2)	0.01627 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.011 (2)	0.0097 (19)	0.015 (2)	0.0024 (17)	−0.0031 (18)	0.0017 (17)
C2	0.012 (2)	0.016 (2)	0.021 (2)	−0.0048 (19)	−0.0006 (19)	−0.0012 (19)
C3	0.010 (2)	0.019 (2)	0.019 (2)	−0.0029 (19)	−0.0014 (19)	−0.0002 (19)
C4	0.015 (2)	0.012 (2)	0.012 (2)	−0.0010 (18)	−0.0008 (18)	−0.0021 (17)
C5	0.010 (2)	0.015 (2)	0.012 (2)	−0.0055 (18)	−0.0017 (18)	0.0001 (17)
C6	0.010 (2)	0.016 (2)	0.008 (2)	−0.0016 (17)	−0.0004 (17)	0.0027 (17)
C7	0.021 (3)	0.035 (3)	0.020 (3)	0.003 (2)	0.006 (2)	0.012 (2)
C8	0.011 (3)	0.016 (2)	0.022 (2)	−0.0010 (18)	−0.006 (2)	−0.0051 (19)
C9	0.017 (3)	0.050 (3)	0.014 (2)	0.007 (2)	−0.004 (2)	−0.006 (2)
C10	0.183 (11)	0.039 (4)	0.042 (4)	−0.056 (6)	0.039 (6)	−0.013 (3)
N1	0.0090 (19)	0.0192 (19)	0.0115 (19)	−0.0042 (15)	0.0019 (15)	0.0050 (15)
O1	0.0085 (18)	0.0267 (18)	0.034 (2)	−0.0042 (14)	−0.0009 (15)	−0.0075 (16)
Br1	0.0197 (3)	0.0146 (2)	0.0195 (3)	0.00165 (19)	−0.0020 (2)	−0.00340 (18)
I1	0.01572 (19)	0.01515 (17)	0.02395 (19)	−0.00073 (13)	−0.00382 (13)	−0.00309 (12)
C14	0.011 (2)	0.012 (2)	0.011 (2)	−0.0020 (17)	0.0024 (18)	0.0001 (17)
C15	0.011 (2)	0.016 (2)	0.014 (2)	−0.0075 (18)	0.0018 (18)	−0.0025 (18)
C16	0.008 (2)	0.024 (2)	0.013 (2)	−0.0030 (18)	0.0013 (18)	0.0006 (18)
C17	0.010 (2)	0.014 (2)	0.006 (2)	−0.0004 (17)	0.0021 (17)	0.0011 (16)
C18	0.009 (2)	0.015 (2)	0.010 (2)	−0.0051 (18)	−0.0003 (17)	−0.0005 (17)
C19	0.011 (2)	0.016 (2)	0.008 (2)	−0.0048 (18)	0.0004 (17)	0.0015 (17)
C20	0.020 (3)	0.023 (2)	0.017 (2)	−0.005 (2)	−0.008 (2)	0.006 (2)
C21	0.009 (2)	0.014 (2)	0.019 (2)	−0.0034 (18)	0.0004 (19)	−0.0053 (18)
C22	0.012 (2)	0.021 (2)	0.019 (2)	−0.0021 (19)	0.0021 (19)	0.0022 (19)
C23	0.097 (6)	0.047 (4)	0.051 (4)	−0.051 (4)	−0.024 (4)	0.012 (3)
C25	0.061 (5)	0.063 (5)	0.029 (4)	−0.007 (4)	0.007 (3)	0.001 (3)
C26	0.106 (6)	0.033 (3)	0.022 (3)	0.004 (4)	−0.019 (4)	−0.003 (3)
N2	0.0058 (19)	0.0151 (18)	0.0124 (18)	−0.0011 (15)	−0.0011 (15)	0.0016 (15)
O2	0.0095 (17)	0.0194 (17)	0.033 (2)	−0.0031 (13)	−0.0052 (15)	−0.0016 (15)
Br2	0.0117 (3)	0.0145 (2)	0.0182 (2)	0.00015 (18)	−0.00163 (19)	−0.00392 (18)
I2	0.01425 (19)	0.01533 (16)	0.02016 (18)	−0.00486 (12)	−0.00063 (13)	−0.00245 (12)

Geometric parameters (Å, °)

C1—C2	1.382 (7)	C12A—H12D	0.9900
C1—C6	1.398 (6)	C13A—H13C	0.9900
C1—I1	2.093 (4)	C13A—H13D	0.9900
C2—C3	1.391 (7)	C14—C15	1.379 (6)
C2—H2	0.9500	C14—C19	1.399 (6)
C3—C4	1.388 (6)	C14—I2	2.086 (4)
C3—H3	0.9500	C15—C16	1.395 (6)

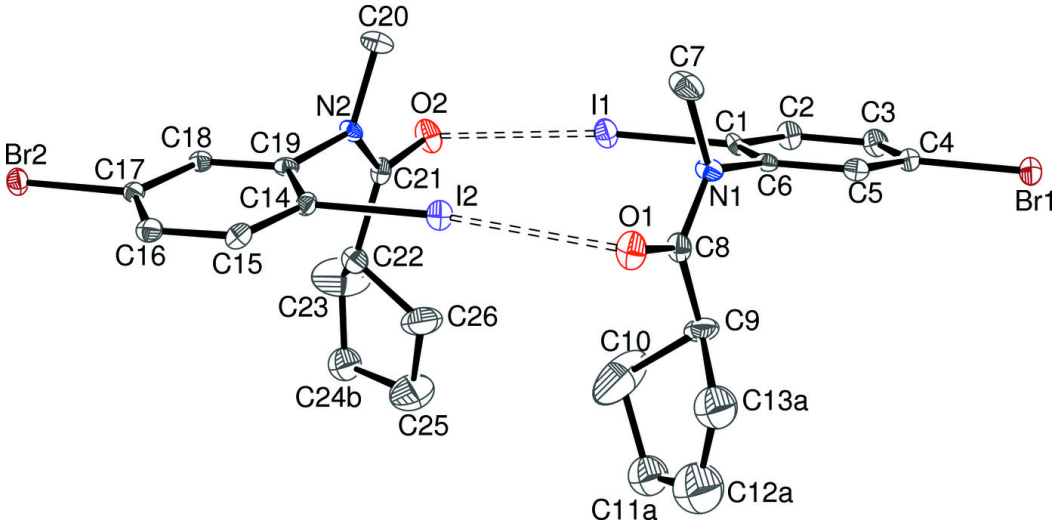
C4—C5	1.384 (7)	C15—H15	0.9500
C4—Br1	1.895 (4)	C16—C17	1.394 (6)
C5—C6	1.391 (6)	C16—H16	0.9500
C5—H5	0.9500	C17—C18	1.375 (6)
C6—N1	1.431 (6)	C17—Br2	1.901 (4)
C7—N1	1.470 (6)	C18—C19	1.394 (6)
C7—H7A	0.9800	C18—H18	0.9500
C7—H7B	0.9800	C19—N2	1.432 (6)
C7—H7C	0.9800	C20—N2	1.469 (6)
C8—O1	1.228 (6)	C20—H20A	0.9800
C8—N1	1.353 (6)	C20—H20B	0.9800
C8—C9	1.506 (7)	C20—H20C	0.9800
C9—C13	1.486 (10)	C21—O2	1.225 (6)
C9—C10	1.530 (10)	C21—N2	1.365 (6)
C9—C13A	1.586 (17)	C21—C22	1.512 (6)
C9—H9	1.0000	C22—C26	1.501 (8)
C10—C11	1.361 (19)	C22—C23	1.531 (7)
C10—C11A	1.57 (2)	C22—H22	1.0000
C10—H10A	0.9900	C23—C24B	1.451 (12)
C10—H10B	0.9900	C23—C24A	1.527 (18)
C11—C12	1.68 (2)	C23—H23A	0.9900
C11—H11A	0.9900	C23—H23B	0.9900
C11—H11B	0.9900	C24A—C25	1.415 (16)
C12—C13	1.565 (16)	C24A—H24A	0.9900
C12—H12A	0.9900	C24A—H24B	0.9900
C12—H12B	0.9900	C24B—C25	1.612 (13)
C13—H13A	0.9900	C24B—H24C	0.9900
C13—H13B	0.9900	C24B—H24D	0.9900
C11A—C12A	1.29 (3)	C25—C26	1.485 (9)
C11A—H11C	0.9900	C25—H25A	0.9900
C11A—H11D	0.9900	C25—H25B	0.9900
C12A—C13A	1.40 (2)	C26—H26A	0.9900
C12A—H12C	0.9900	C26—H26B	0.9900
C2—C1—C6	120.3 (4)	C12A—C13A—H13D	110.1
C2—C1—I1	119.1 (3)	C9—C13A—H13D	110.1
C6—C1—I1	120.6 (3)	H13C—C13A—H13D	108.4
C1—C2—C3	120.8 (4)	C8—N1—C6	124.5 (4)
C1—C2—H2	119.6	C8—N1—C7	119.2 (4)
C3—C2—H2	119.6	C6—N1—C7	116.2 (4)
C4—C3—C2	118.7 (4)	C15—C14—C19	120.2 (4)
C4—C3—H3	120.6	C15—C14—I2	119.7 (3)
C2—C3—H3	120.6	C19—C14—I2	120.1 (3)
C5—C4—C3	121.1 (4)	C14—C15—C16	120.8 (4)
C5—C4—Br1	118.8 (3)	C14—C15—H15	119.6
C3—C4—Br1	120.1 (3)	C16—C15—H15	119.6
C4—C5—C6	120.1 (4)	C17—C16—C15	118.4 (4)
C4—C5—H5	120.0	C17—C16—H16	120.8
C6—C5—H5	120.0	C15—C16—H16	120.8
C5—C6—C1	119.0 (4)	C18—C17—C16	121.4 (4)

C5—C6—N1	119.1 (4)	C18—C17—Br2	118.8 (3)
C1—C6—N1	121.7 (4)	C16—C17—Br2	119.7 (3)
N1—C7—H7A	109.5	C17—C18—C19	120.0 (4)
N1—C7—H7B	109.5	C17—C18—H18	120.0
H7A—C7—H7B	109.5	C19—C18—H18	120.0
N1—C7—H7C	109.5	C18—C19—C14	119.2 (4)
H7A—C7—H7C	109.5	C18—C19—N2	118.8 (4)
H7B—C7—H7C	109.5	C14—C19—N2	121.9 (4)
O1—C8—N1	120.8 (4)	N2—C20—H20A	109.5
O1—C8—C9	120.9 (4)	N2—C20—H20B	109.5
N1—C8—C9	118.3 (4)	H20A—C20—H20B	109.5
C13—C9—C8	118.1 (6)	N2—C20—H20C	109.5
C13—C9—C10	105.8 (6)	H20A—C20—H20C	109.5
C8—C9—C10	114.2 (5)	H20B—C20—H20C	109.5
C13—C9—C13A	27.6 (6)	O2—C21—N2	120.5 (4)
C8—C9—C13A	106.1 (7)	O2—C21—C22	121.8 (4)
C10—C9—C13A	90.8 (8)	N2—C21—C22	117.6 (4)
C13—C9—H9	106.0	C26—C22—C21	111.8 (4)
C8—C9—H9	106.0	C26—C22—C23	102.9 (5)
C10—C9—H9	106.0	C21—C22—C23	112.6 (4)
C13A—C9—H9	133.2	C26—C22—H22	109.8
C11—C10—C9	110.0 (9)	C21—C22—H22	109.8
C11—C10—C11A	16.7 (10)	C23—C22—H22	109.8
C9—C10—C11A	105.5 (8)	C24B—C23—C24A	33.2 (7)
C11—C10—H10A	109.7	C24B—C23—C22	105.7 (6)
C9—C10—H10A	109.7	C24A—C23—C22	104.0 (7)
C11A—C10—H10A	97.3	C24B—C23—H23A	80.0
C11—C10—H10B	109.7	C24A—C23—H23A	110.9
C9—C10—H10B	109.7	C22—C23—H23A	110.9
C11A—C10—H10B	125.4	C24B—C23—H23B	135.2
H10A—C10—H10B	108.2	C24A—C23—H23B	110.9
C10—C11—C12	105.3 (12)	C22—C23—H23B	110.9
C10—C11—H11A	110.7	H23A—C23—H23B	109.0
C12—C11—H11A	110.7	C25—C24A—C23	108.7 (11)
C10—C11—H11B	110.7	C25—C24A—H24A	110.0
C12—C11—H11B	110.7	C23—C24A—H24A	110.0
H11A—C11—H11B	108.8	C25—C24A—H24B	110.0
C13—C12—C11	102.4 (10)	C23—C24A—H24B	110.0
C13—C12—H12A	111.3	H24A—C24A—H24B	108.3
C11—C12—H12A	111.3	C23—C24B—C25	102.5 (7)
C13—C12—H12B	111.3	C23—C24B—H24C	111.3
C11—C12—H12B	111.3	C25—C24B—H24C	111.3
H12A—C12—H12B	109.2	C23—C24B—H24D	111.3
C9—C13—C12	106.7 (7)	C25—C24B—H24D	111.3
C9—C13—H13A	110.4	H24C—C24B—H24D	109.2
C12—C13—H13A	110.4	C24A—C25—C26	101.4 (8)
C9—C13—H13B	110.4	C24A—C25—C24B	31.9 (7)
C12—C13—H13B	110.4	C26—C25—C24B	105.9 (6)
H13A—C13—H13B	108.6	C24A—C25—H25A	111.5

C12A—C11A—C10	103.7 (15)	C26—C25—H25A	111.5
C12A—C11A—H11C	111.0	C24B—C25—H25A	133.1
C10—C11A—H11C	111.0	C24A—C25—H25B	111.5
C12A—C11A—H11D	111.0	C26—C25—H25B	111.5
C10—C11A—H11D	111.0	C24B—C25—H25B	80.8
H11C—C11A—H11D	109.0	H25A—C25—H25B	109.3
C11A—C12A—C13A	112.4 (18)	C25—C26—C22	106.9 (5)
C11A—C12A—H12C	109.1	C25—C26—H26A	110.3
C13A—C12A—H12C	109.1	C22—C26—H26A	110.3
C11A—C12A—H12D	109.1	C25—C26—H26B	110.3
C13A—C12A—H12D	109.1	C22—C26—H26B	110.3
H12C—C12A—H12D	107.9	H26A—C26—H26B	108.6
C12A—C13A—C9	108.1 (13)	C21—N2—C19	123.6 (4)
C12A—C13A—H13C	110.1	C21—N2—C20	119.8 (4)
C9—C13A—H13C	110.1	C19—N2—C20	116.4 (3)
C6—C1—C2—C3	−0.3 (7)	C1—C6—N1—C8	98.8 (5)
I1—C1—C2—C3	−179.4 (3)	C5—C6—N1—C7	91.4 (5)
C1—C2—C3—C4	−0.3 (7)	C1—C6—N1—C7	−85.1 (5)
C2—C3—C4—C5	0.2 (7)	C19—C14—C15—C16	−0.5 (7)
C2—C3—C4—Br1	−179.9 (3)	I2—C14—C15—C16	−179.0 (3)
C3—C4—C5—C6	0.5 (7)	C14—C15—C16—C17	0.6 (7)
Br1—C4—C5—C6	−179.4 (3)	C15—C16—C17—C18	−0.6 (7)
C4—C5—C6—C1	−1.1 (7)	C15—C16—C17—Br2	−178.9 (3)
C4—C5—C6—N1	−177.7 (4)	C16—C17—C18—C19	0.5 (6)
C2—C1—C6—C5	1.0 (7)	Br2—C17—C18—C19	178.8 (3)
I1—C1—C6—C5	−180.0 (3)	C17—C18—C19—C14	−0.4 (6)
C2—C1—C6—N1	177.5 (4)	C17—C18—C19—N2	−176.9 (4)
I1—C1—C6—N1	−3.5 (6)	C15—C14—C19—C18	0.4 (6)
O1—C8—C9—C13	−63.9 (8)	I2—C14—C19—C18	178.9 (3)
N1—C8—C9—C13	117.6 (7)	C15—C14—C19—N2	176.8 (4)
O1—C8—C9—C10	61.4 (8)	I2—C14—C19—N2	−4.6 (6)
N1—C8—C9—C10	−117.2 (7)	O2—C21—C22—C26	76.3 (6)
O1—C8—C9—C13A	−37.0 (10)	N2—C21—C22—C26	−101.3 (6)
N1—C8—C9—C13A	144.5 (9)	O2—C21—C22—C23	−39.1 (7)
C13—C9—C10—C11	−33.6 (13)	N2—C21—C22—C23	143.3 (5)
C8—C9—C10—C11	−165.1 (12)	C26—C22—C23—C24B	41.2 (9)
C13A—C9—C10—C11	−57.1 (13)	C21—C22—C23—C24B	161.8 (7)
C13—C9—C10—C11A	−16.7 (11)	C26—C22—C23—C24A	6.8 (10)
C8—C9—C10—C11A	−148.2 (9)	C21—C22—C23—C24A	127.5 (9)
C13A—C9—C10—C11A	−40.2 (12)	C24B—C23—C24A—C25	−78.8 (15)
C9—C10—C11—C12	28.0 (16)	C22—C23—C24A—C25	18.5 (15)
C11A—C10—C11—C12	−49 (4)	C24A—C23—C24B—C25	56.7 (12)
C10—C11—C12—C13	−13.1 (16)	C22—C23—C24B—C25	−35.1 (9)
C8—C9—C13—C12	151.9 (8)	C23—C24A—C25—C26	−35.9 (14)
C10—C9—C13—C12	22.5 (10)	C23—C24A—C25—C24B	66.1 (14)
C13A—C9—C13—C12	82.0 (16)	C23—C24B—C25—C24A	−69.0 (14)
C11—C12—C13—C9	−6.8 (13)	C23—C24B—C25—C26	16.9 (10)
C11—C10—C11A—C12A	141 (5)	C24A—C25—C26—C22	40.7 (11)
C9—C10—C11A—C12A	32.5 (18)	C24B—C25—C26—C22	8.2 (9)

C10—C11A—C12A—C13A	−5(2)	C21—C22—C26—C25	−150.2 (5)
C11A—C12A—C13A—C9	−23 (2)	C23—C22—C26—C25	−29.0 (8)
C13—C9—C13A—C12A	−85.2 (19)	O2—C21—N2—C19	174.5 (4)
C8—C9—C13A—C12A	154.3 (13)	C22—C21—N2—C19	−7.9 (6)
C10—C9—C13A—C12A	38.8 (15)	O2—C21—N2—C20	0.7 (6)
O1—C8—N1—C6	175.9 (4)	C22—C21—N2—C20	178.4 (4)
C9—C8—N1—C6	−5.6 (7)	C18—C19—N2—C21	−80.3 (5)
O1—C8—N1—C7	−0.1 (7)	C14—C19—N2—C21	103.3 (5)
C9—C8—N1—C7	178.5 (4)	C18—C19—N2—C20	93.7 (5)
C5—C6—N1—C8	−84.7 (6)	C14—C19—N2—C20	−82.8 (5)

Fig. 1


C20
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C24b
C25
C13a
C11a
C12a