

# Crystal structure of *tert*-butyl 3,6-diiodocarbazole-9-carboxylate

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Received 28 February 2023

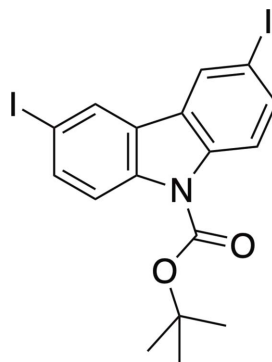
Accepted 8 March 2023

Edited by M. Weil, Vienna University of  
Technology, Austria**Keywords:** crystal structure; carbazole;  
halogen–halogen interaction;  $\pi$ – $\pi$  interaction.**CCDC reference:** 2247288**Supporting information:** this article has  
supporting information at journals.iucr.org/e

The molecular structure of *tert*-butyl 3,6-diiodocarbazole-9-carboxylate, C<sub>17</sub>H<sub>15</sub>I<sub>2</sub>NO<sub>2</sub>, features a nearly planar 13-membered carbazole ring with C–I bond lengths of 2.092 (4) and 2.104 (4) Å. The carbamate group has key bond lengths of 1.404 (6) Å (N–C), 1.330 (5) Å (O–C), and 1.201 (6) Å (C=O). The crystal contains intermolecular  $\pi$ – $\pi$  interactions, as well as both type I and type II intermolecular I··I interactions.

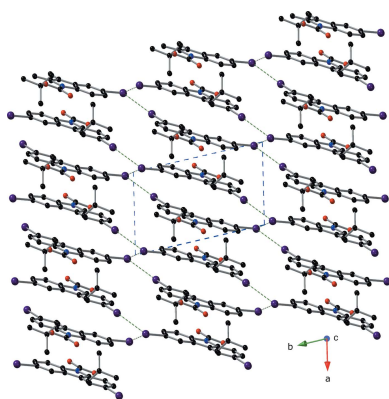
## 1. Chemical context

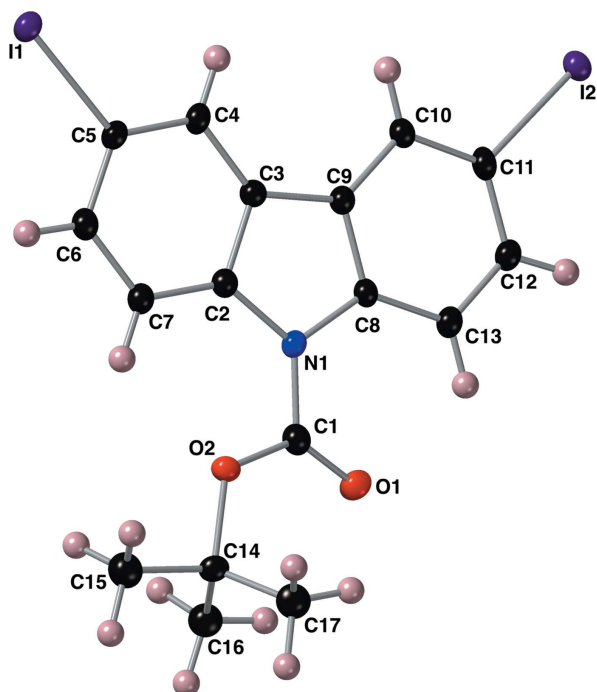
Derivatives of the carbazole ring system have been used in a wide variety of applications ranging from organic light-emitting diodes (Uoyama *et al.*, 2012) to cell membrane targeting fluorescent probes (Wnag *et al.*, 2023) to compounds that are able to influence the supramolecular structure of G-rich DNA sequences (Debnath *et al.*, 2016). Our group's interest in this molecular entity was inspired by the work of de Bettencourt-Dias and co-workers who have used carbazole derivatives as antennas to sensitize the luminescence of lanthanide metals (Monteiro *et al.*, 2017, 2018, 2020, 2022). Our group was working to derivatize carbazole for use in related lanthanide luminescence applications when compound **I**, a synthetic intermediate in our work, serendipitously crystallized in an NMR tube.



## 2. Structural commentary

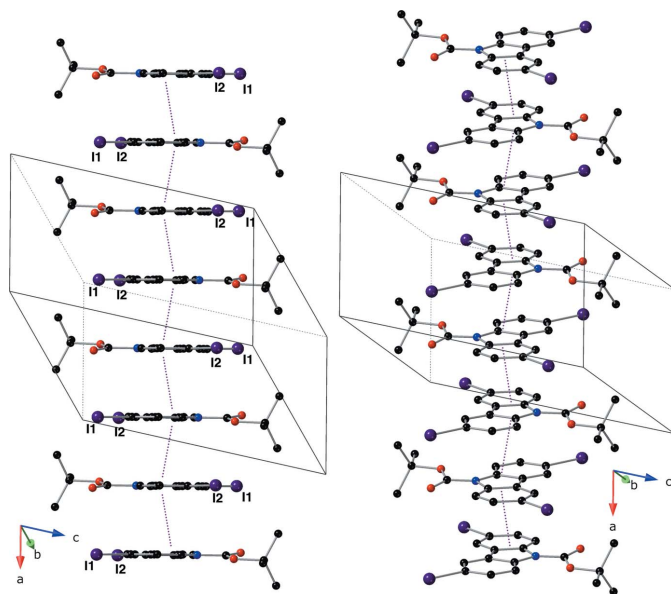
The molecular structure of compound **I** is shown in Fig. 1 along with the atom-numbering scheme. The structure of this substituted carbazole has C–I bond lengths of 2.092 (4) and 2.104 (4) Å. The carbamate group has bond lengths of 1.404 (6) Å for N1–C1, 1.330 (5) Å for O2–C1, and





**Figure 1**  
The molecular structure of compound **I**, with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level using standard CPK colors (I = purple).

1.201 (6) Å for the carbonyl C1=O1. The N1–C1–O2 bond angle is 110.3 (4)°, and these atoms are roughly coplanar with



**Figure 2**  
A depiction of the supramolecular pillars using a ball-and-stick model with standard CPK colors (I = purple). The left portion of the figure shows the pillars with a view that is aligned with the plane of the aromatic carbazole system, the right portion of the figure shows the same molecules tilted slightly along the *b* axis. The  $\pi$ - $\pi$  interactions described in the text are depicted with purple dotted lines and the unit cell is drawn with a solid black line.

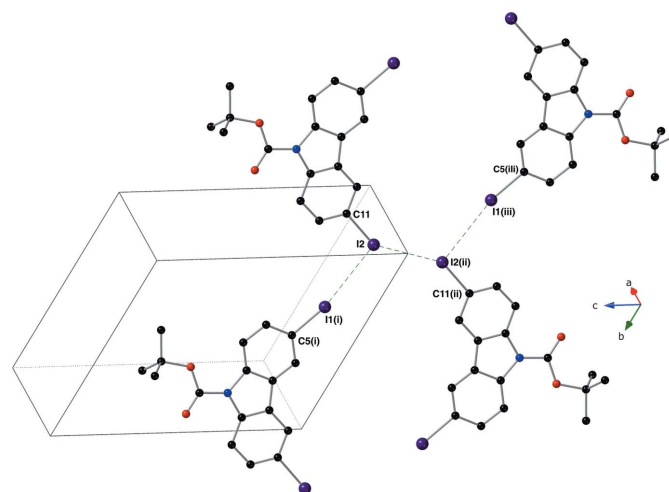
the atoms of the aromatic system with a C8–N1–C1–O2 torsion angle of  $-7.3$  (7)°. The 13-membered aromatic carbazole ring approaches planarity with an r.m.s. deviation of 0.007 Å where the atom C8 deviates the most from the calculated least-squares plane by 0.019 (4) Å.

### 3. Supramolecular features

In the crystal, molecules of the title compound form pillars *via* intermolecular  $\pi$ - $\pi$  interactions that propagate parallel to the *a* axis; the centroid of the five-membered ring (N1/C2/C3/C9/C8) is denoted as *Cg*. These interactions have *Cg*···*Cg* distances of 3.484 (3) and 3.589 (3) Å with slippages of 1.028 and 1.376 Å and angles of 0.00 (3)° (Fig. 2; symmetry codes:  $-x + 1, -y + 1, -z + 1$  and  $-x + 2, -y + 1, -z + 1$ ). The supramolecular pillars are held together *via* both type I and type II intermolecular I···I interactions (Pedireddi *et al.*, 1994; Figs. 3 and 4). The type I halogen–halogen interaction has a *trans* arrangement and exists between atoms C11–I2···I2( $-x + 2, -y, -z + 2$ ) with an angle of 147.68 (13)° and an I···I distance of 3.6630 (5) Å. The type II interaction is found between atoms C11–I2···I1( $x + 1, y - 1, z$ ) with an I···I distance of 3.8332 (5) Å and an angle of 46.69 (13)°.

### 4. Database survey

A search of the Cambridge Structure Database (CSD version 5.43 with updates through June 2022; Groom *et al.*, 2016) for structures containing the carbazole ring system substituted with any halogen atom at the C5 and C11 positions (as numbered in Fig. 1) returned 101 hits. The structures CEYXAI (Malecki, 2018) and FUMLIK (Radula-Janik *et al.*, 2015) are closely related to that of compound **I** with iodine atoms at the C5 and C11 positions, but where the nitrogen atom has been alkylated with either a butyl or benzyl group.



**Figure 3**  
A figure showing the halogen–halogen interactions (depicted as green, dashed lines) present in the crystal of compound **I** using a ball-and-stick model with standard CPK colors (I = purple). The unit cell is drawn with a solid black line. [Symmetry codes: (i)  $-x + 1, -y + 1, -z + 2$ , (ii)  $-x + 2, -y, -z + 2$ , (iii)  $x + 1, y - 1, z$ ].

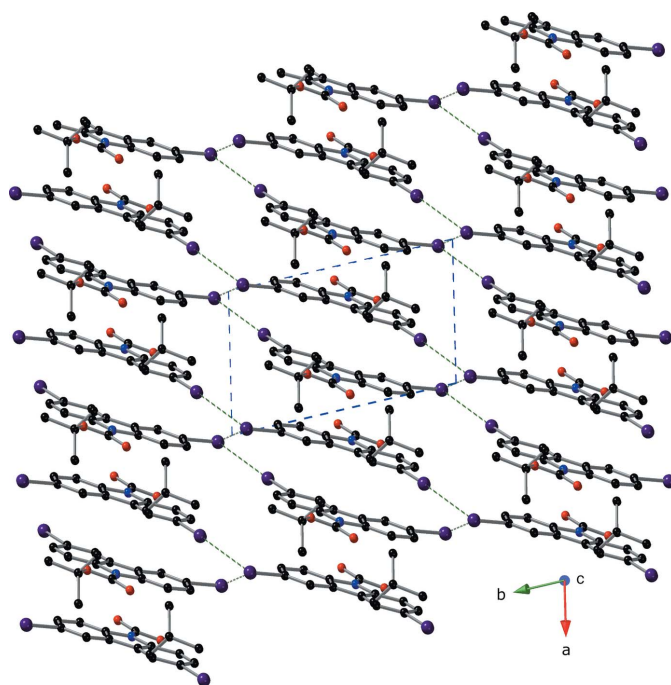


Figure 4

A view down the  $c$  axis showing the supramolecular pillars found in the crystal of compound **I** as well as the I–I interactions (depicted as green, dashed lines) that hold them together using a ball-and-stick model with standard CPK colors (I = purple). The unit cell is drawn with a blue, dashed line and the  $\pi$ – $\pi$  interactions are not shown for clarity.

Structure ECUNUM bears bromine atoms at the C5 and C11 positions with a phenylcarbamate group on the nitrogen atom (Duan *et al.*, 2006). A derivative of compound **I** that bears two iodine atoms in the same positions and a hydrogen atom bonded to the nitrogen atom has been solved as structure YAYDUZ (Xie *et al.*, 2012). Lastly, the di-iodo carbazole has been used as a ligand in a copper(I) complex as demonstrated by Kim and co-workers (ZASYUQ; Kim *et al.*, 2017).

## 5. Synthesis and crystallization

The title compound was prepared according to the procedure published by Lee and co-workers (Moon *et al.*, 2007). The compound was dissolved in  $\text{CDCl}_3$  and the crystals studied here grew as the solvent slowly evaporated from the NMR tube.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All hydrogen atoms bonded to carbon atoms were placed in calculated positions and refined as riding: C–H = 0.95–1.00 Å with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for aromatic hydrogen atoms and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for the hydrogen atoms of the methyl group.

## Acknowledgements

We thank the GVSU Chemistry Department Weldon Fund for financial support, as well as the GVSU Library Open Access

Table 1

Experimental details.

Crystal data	
Chemical formula	$\text{C}_{17}\text{H}_{15}\text{I}_2\text{NO}_2$
$M_r$	519.10
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	100
$a, b, c$ (Å)	6.9611 (3), 11.9737 (5), 12.0697 (4)
$\alpha, \beta, \gamma$ (°)	65.618 (4), 78.588 (3), 74.826 (4)
$V$ (Å <sup>3</sup> )	879.71 (7)
$Z$	2
Radiation type	Cu $K\alpha$
$\mu$ (mm <sup>-1</sup> )	28.13
Crystal size (mm)	0.11 × 0.06 × 0.02
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Multi-scan ( <i>CrysAlis PRO</i> ; Oxford Diffraction, 2006)
$T_{\text{min}}, T_{\text{max}}$	0.756, 1.000
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	10800, 3717, 3209
$R_{\text{int}}$	0.038
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.639
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.033, 0.090, 1.08
No. of reflections	3717
No. of parameters	202
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.74, -1.17

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2006), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *CrystalMaker* (Palmer, 2007), *OLEX2* (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015).

Fund. We are grateful to Dr Susan Mendoza (GVSU CUSE) for her unwavering support as well as Dr Bruce (GVSU) for inspiring conversations. Many thanks to Dr de Bettencourt-Dias for giving us the opportunity to contribute to her work in the area of lanthanide luminescence.

## Funding information

Funding for this research was provided by: National Science Foundation, Directorate for Mathematical and Physical Sciences (grant No. RUI CHE-2102576 to SB at GVSU; grant No. MRI CHE-1725699 to SB at GVSU; grant No. MRI CHE-1919565 to RJS at MSU); Grand Valley State University (grant No. McNair Fellowship to ENS; grant No. Kindshi Fellowship to ENS).

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## supporting information

*Acta Cryst.* (2023). E79, 345-348 [https://doi.org/10.1107/S205698902300230X]

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## Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2006); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2006); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2006); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *CrystalMaker* (Palmer, 2007); software used to prepare material for publication: *Olex2* (Dolomanov *et al.*, 2009; Bourhis *et al.*, 2015).

*tert*-Butyl 3,6-diiodocarbazole-9-carboxylate

## Crystal data

$C_{17}H_{15}I_2NO_2$

$M_r = 519.10$

Triclinic,  $P\bar{1}$

$a = 6.9611$  (3) Å

$b = 11.9737$  (5) Å

$c = 12.0697$  (4) Å

$\alpha = 65.618$  (4)°

$\beta = 78.588$  (3)°

$\gamma = 74.826$  (4)°

$V = 879.71$  (7) Å<sup>3</sup>

$Z = 2$

$F(000) = 492$

$D_x = 1.960$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54184$  Å

Cell parameters from 6514 reflections

$\theta = 4.1\text{--}79.3^\circ$

$\mu = 28.13$  mm<sup>-1</sup>

$T = 100$  K

Irregular, colourless

$0.11 \times 0.06 \times 0.02$  mm

## Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer

Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan

(*CrysAlisPro*; Oxford Diffraction, 2006)

$T_{\min} = 0.756$ ,  $T_{\max} = 1.000$

10800 measured reflections

3717 independent reflections

3209 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 80.0^\circ$ ,  $\theta_{\min} = 4.0^\circ$

$h = -8 \rightarrow 8$

$k = -15 \rightarrow 15$

$l = -15 \rightarrow 12$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.090$

$S = 1.08$

3717 reflections

202 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 2.1415P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.74$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -1.17$  e Å<sup>-3</sup>

*Special details*

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

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.35523 (5)	0.84654 (3)	0.68070 (3)	0.02553 (10)
I2	1.02078 (5)	0.06482 (2)	0.83259 (2)	0.02519 (10)
O1	0.9081 (6)	0.4713 (3)	0.2038 (3)	0.0308 (8)
O2	0.6990 (5)	0.6592 (3)	0.1740 (3)	0.0229 (7)
N1	0.7721 (6)	0.5314 (3)	0.3644 (3)	0.0176 (7)
C1	0.8021 (7)	0.5484 (4)	0.2405 (4)	0.0216 (9)
C2	0.6704 (7)	0.6182 (4)	0.4183 (4)	0.0177 (8)
C3	0.6784 (7)	0.5579 (4)	0.5462 (4)	0.0178 (8)
C4	0.5909 (7)	0.6212 (4)	0.6236 (4)	0.0199 (9)
H4	0.596672	0.580826	0.709538	0.024*
C5	0.4954 (7)	0.7448 (4)	0.5712 (4)	0.0189 (8)
C6	0.4877 (7)	0.8063 (4)	0.4439 (4)	0.0206 (9)
H6	0.423151	0.891758	0.410340	0.025*
C7	0.5745 (7)	0.7422 (4)	0.3669 (4)	0.0194 (8)
H7	0.567988	0.782539	0.281049	0.023*
C8	0.8459 (6)	0.4167 (4)	0.4582 (4)	0.0169 (8)
C9	0.7881 (6)	0.4311 (4)	0.5706 (4)	0.0176 (8)
C10	0.8392 (7)	0.3303 (4)	0.6804 (4)	0.0190 (8)
H10	0.802819	0.338279	0.757291	0.023*
C11	0.9449 (7)	0.2191 (4)	0.6707 (4)	0.0201 (9)
C12	0.9994 (7)	0.2046 (4)	0.5597 (4)	0.0207 (9)
H12	1.070569	0.125606	0.558551	0.025*
C13	0.9521 (7)	0.3030 (4)	0.4505 (4)	0.0204 (9)
H13	0.989829	0.293776	0.374211	0.024*
C14	0.7063 (8)	0.6986 (4)	0.0389 (4)	0.0244 (10)
C15	0.5773 (9)	0.8287 (5)	0.0027 (5)	0.0332 (12)
H15A	0.442886	0.824282	0.045892	0.050*
H15B	0.568722	0.864481	−0.085716	0.050*
H15C	0.636703	0.881733	0.024436	0.050*
C16	0.9203 (9)	0.7015 (5)	−0.0200 (5)	0.0336 (12)
H16A	0.980140	0.748546	0.009296	0.050*
H16B	0.920443	0.742127	−0.109047	0.050*
H16C	0.998346	0.615734	0.001846	0.050*
C17	0.6131 (10)	0.6110 (5)	0.0144 (5)	0.0351 (12)
H17A	0.688431	0.525099	0.050166	0.053*
H17B	0.616737	0.635693	−0.074038	0.053*
H17C	0.474017	0.615706	0.051213	0.053*



*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
I1	0.03315 (18)	0.01775 (15)	0.02357 (16)	0.00301 (11)	-0.00265 (12)	-0.01080 (12)
I2	0.03258 (18)	0.01525 (15)	0.01960 (16)	0.00277 (11)	-0.00287 (11)	-0.00313 (11)
O1	0.044 (2)	0.0203 (16)	0.0229 (16)	0.0062 (15)	-0.0023 (15)	-0.0109 (14)
O2	0.0316 (18)	0.0177 (15)	0.0150 (14)	0.0022 (13)	-0.0047 (12)	-0.0053 (12)
N1	0.0192 (18)	0.0143 (16)	0.0183 (17)	-0.0010 (14)	-0.0004 (14)	-0.0071 (14)
C1	0.021 (2)	0.017 (2)	0.022 (2)	0.0003 (17)	-0.0045 (17)	-0.0055 (18)
C2	0.017 (2)	0.017 (2)	0.019 (2)	-0.0053 (16)	-0.0010 (16)	-0.0066 (16)
C3	0.017 (2)	0.016 (2)	0.018 (2)	0.0000 (16)	-0.0008 (16)	-0.0068 (16)
C4	0.021 (2)	0.016 (2)	0.024 (2)	-0.0005 (16)	-0.0029 (17)	-0.0093 (17)
C5	0.017 (2)	0.016 (2)	0.022 (2)	0.0012 (16)	0.0015 (16)	-0.0105 (17)
C6	0.020 (2)	0.016 (2)	0.023 (2)	-0.0018 (16)	-0.0012 (17)	-0.0066 (17)
C7	0.019 (2)	0.016 (2)	0.023 (2)	-0.0011 (16)	-0.0043 (16)	-0.0074 (17)
C8	0.0157 (19)	0.0137 (19)	0.019 (2)	-0.0014 (15)	-0.0013 (15)	-0.0054 (16)
C9	0.0150 (19)	0.016 (2)	0.021 (2)	-0.0013 (15)	0.0015 (15)	-0.0076 (16)
C10	0.018 (2)	0.0145 (19)	0.020 (2)	-0.0001 (16)	-0.0010 (16)	-0.0048 (17)
C11	0.021 (2)	0.0119 (19)	0.023 (2)	0.0024 (16)	-0.0087 (17)	-0.0025 (16)
C12	0.022 (2)	0.0156 (19)	0.023 (2)	0.0000 (16)	-0.0016 (17)	-0.0087 (17)
C13	0.019 (2)	0.017 (2)	0.025 (2)	-0.0011 (16)	-0.0008 (17)	-0.0094 (17)
C14	0.035 (3)	0.021 (2)	0.017 (2)	-0.0024 (19)	-0.0028 (18)	-0.0082 (18)
C15	0.040 (3)	0.023 (2)	0.029 (3)	0.006 (2)	-0.010 (2)	-0.007 (2)
C16	0.040 (3)	0.027 (3)	0.029 (3)	-0.003 (2)	0.005 (2)	-0.012 (2)
C17	0.052 (4)	0.028 (3)	0.027 (3)	-0.009 (2)	-0.011 (2)	-0.009 (2)

*Geometric parameters (Å, °)*

I1—C5	2.092 (4)	C9—C10	1.408 (6)
I2—C11	2.104 (4)	C10—H10	0.9500
O1—C1	1.201 (6)	C10—C11	1.382 (6)
O2—C1	1.330 (5)	C11—C12	1.388 (7)
O2—C14	1.493 (5)	C12—H12	0.9500
N1—C1	1.404 (6)	C12—C13	1.386 (6)
N1—C2	1.413 (6)	C13—H13	0.9500
N1—C8	1.416 (6)	C14—C15	1.512 (7)
C2—C3	1.414 (6)	C14—C16	1.521 (8)
C2—C7	1.385 (6)	C14—C17	1.516 (7)
C3—C4	1.391 (6)	C15—H15A	0.9800
C3—C9	1.446 (6)	C15—H15B	0.9800
C4—H4	0.9500	C15—H15C	0.9800
C4—C5	1.383 (6)	C16—H16A	0.9800
C5—C6	1.409 (6)	C16—H16B	0.9800
C6—H6	0.9500	C16—H16C	0.9800
C6—C7	1.393 (6)	C17—H17A	0.9800
C7—H7	0.9500	C17—H17B	0.9800
C8—C9	1.404 (6)	C17—H17C	0.9800
C8—C13	1.400 (6)		

C1—O2—C14	120.3 (4)	C10—C11—I2	118.1 (3)
C1—N1—C2	128.7 (4)	C10—C11—C12	122.9 (4)
C1—N1—C8	122.7 (4)	C12—C11—I2	119.0 (3)
C2—N1—C8	108.5 (3)	C11—C12—H12	119.4
O1—C1—O2	127.0 (4)	C13—C12—C11	121.3 (4)
O1—C1—N1	122.8 (4)	C13—C12—H12	119.4
O2—C1—N1	110.3 (4)	C8—C13—H13	121.6
N1—C2—C3	108.2 (4)	C12—C13—C8	116.7 (4)
C7—C2—N1	131.2 (4)	C12—C13—H13	121.6
C7—C2—C3	120.7 (4)	O2—C14—C15	101.9 (4)
C2—C3—C9	107.3 (4)	O2—C14—C16	110.3 (4)
C4—C3—C2	121.1 (4)	O2—C14—C17	108.4 (4)
C4—C3—C9	131.6 (4)	C15—C14—C16	111.4 (4)
C3—C4—H4	121.2	C15—C14—C17	111.3 (5)
C5—C4—C3	117.7 (4)	C17—C14—C16	113.0 (5)
C5—C4—H4	121.2	C14—C15—H15A	109.5
C4—C5—I1	120.4 (3)	C14—C15—H15B	109.5
C4—C5—C6	121.8 (4)	C14—C15—H15C	109.5
C6—C5—I1	117.8 (3)	H15A—C15—H15B	109.5
C5—C6—H6	119.9	H15A—C15—H15C	109.5
C7—C6—C5	120.3 (4)	H15B—C15—H15C	109.5
C7—C6—H6	119.9	C14—C16—H16A	109.5
C2—C7—C6	118.5 (4)	C14—C16—H16B	109.5
C2—C7—H7	120.8	C14—C16—H16C	109.5
C6—C7—H7	120.8	H16A—C16—H16B	109.5
C9—C8—N1	108.2 (4)	H16A—C16—H16C	109.5
C13—C8—N1	129.7 (4)	H16B—C16—H16C	109.5
C13—C8—C9	122.1 (4)	C14—C17—H17A	109.5
C8—C9—C3	107.9 (4)	C14—C17—H17B	109.5
C8—C9—C10	120.4 (4)	C14—C17—H17C	109.5
C10—C9—C3	131.8 (4)	H17A—C17—H17B	109.5
C9—C10—H10	121.7	H17A—C17—H17C	109.5
C11—C10—C9	116.6 (4)	H17B—C17—H17C	109.5
C11—C10—H10	121.7		
I1—C5—C6—C7	-178.5 (3)	C3—C4—C5—C6	-0.7 (7)
I2—C11—C12—C13	179.6 (4)	C3—C9—C10—C11	179.1 (5)
N1—C2—C3—C4	179.7 (4)	C4—C3—C9—C8	-179.1 (5)
N1—C2—C3—C9	0.0 (5)	C4—C3—C9—C10	1.1 (9)
N1—C2—C7—C6	-179.2 (4)	C4—C5—C6—C7	1.2 (7)
N1—C8—C9—C3	-0.8 (5)	C5—C6—C7—C2	-1.1 (7)
N1—C8—C9—C10	179.1 (4)	C7—C2—C3—C4	-0.1 (7)
N1—C8—C13—C12	-178.0 (4)	C7—C2—C3—C9	-179.8 (4)
C1—O2—C14—C15	-177.9 (4)	C8—N1—C1—O1	-7.3 (7)
C1—O2—C14—C16	-59.5 (6)	C8—N1—C1—O2	173.0 (4)
C1—O2—C14—C17	64.7 (6)	C8—N1—C2—C3	-0.5 (5)
C1—N1—C2—C3	179.7 (4)	C8—N1—C2—C7	179.2 (5)



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C1—N1—C2—C7	-0.6 (8)	C8—C9—C10—C11	-0.7 (7)
C1—N1—C8—C9	-179.3 (4)	C9—C3—C4—C5	179.7 (5)
C1—N1—C8—C13	-1.6 (7)	C9—C8—C13—C12	-0.5 (7)
C2—N1—C1—O1	172.5 (5)	C9—C10—C11—I2	-179.0 (3)
C2—N1—C1—O2	-7.2 (7)	C9—C10—C11—C12	-0.3 (7)
C2—N1—C8—C9	0.8 (5)	C10—C11—C12—C13	0.9 (8)
C2—N1—C8—C13	178.6 (5)	C11—C12—C13—C8	-0.5 (7)
C2—C3—C4—C5	0.2 (7)	C13—C8—C9—C3	-178.8 (4)
C2—C3—C9—C8	0.5 (5)	C13—C8—C9—C10	1.1 (7)
C2—C3—C9—C10	-179.4 (5)	C14—O2—C1—O1	0.5 (8)
C3—C2—C7—C6	0.6 (7)	C14—O2—C1—N1	-179.8 (4)
C3—C4—C5—I1	178.9 (3)		

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