



Crystal structure of 1-[(1-methyl-5-nitro-1*H*-imidazol-2-yl)methyl]pyridinium iodide

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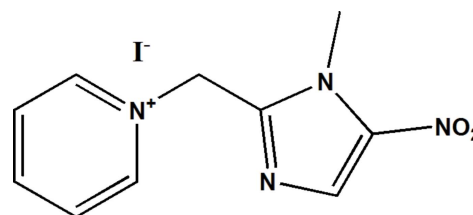
In the title salt, $C_{10}H_{11}N_4O_2^+ \cdot I^-$, the asymmetric unit consists of a pyridinium cation bearing a (1-methyl-5-nitro-1*H*-imidazol-2-yl)methyl group at the N position and an iodide anion. The imidazole ring is quasiplanar, with a maximum deviation of 0.0032 (16) Å, and forms a dihedral angle of 67.39 (6)° with the plane of the pyridinium ring. The crystal packing can be described as alternating zigzag layers of cations parallel to the (001) plane, which are sandwiched by the iodide ions. The structure features two types of hydrogen bonds (C—H...O and C—H...I), viz. cation–anion and cation–cation, which lead to the formation of a three-dimensional network.

Keywords: crystal structure; imidazole; pyridinium; iodide; hydrogen bonding.

CCDC reference: 1045139

1. Related literature

For the synthesis and applications of imidazole derivatives, see: Upcroft & Upcroft (2001); Çelik & Ateş (2006); Boyer (1986); Olender *et al.* (2009); Gaonkar *et al.* (2009); Larina & Lopyrev (2009). For our previous work on this type of chemistry, see: Zama *et al.* (2013); Alliouche *et al.* (2014); Bahnous *et al.* (2012). For the synthesis of the title compound, see: Albright & Shepherd (1973).



2. Experimental

2.1. Crystal data

$C_{10}H_{11}N_4O_2^+ \cdot I^-$
 $M_r = 346.13$
Monoclinic, $P2_1/c$
 $a = 11.035$ (7) Å
 $b = 9.073$ (6) Å
 $c = 12.859$ (8) Å
 $\beta = 91.69$ (2)°

$V = 1286.8$ (14) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 2.49$ mm⁻¹
 $T = 295$ K
0.14 × 0.12 × 0.11 mm

2.2. Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan
(SADABS; Sheldrick, 2002)
 $T_{\min} = 0.615$, $T_{\max} = 0.745$

22502 measured reflections
6134 independent reflections
3669 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.067$
 $S = 0.99$
6134 reflections

155 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 1.14$ e Å⁻³
 $\Delta\rho_{\min} = -0.89$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10—H10...O2 ⁱ	0.93	2.51	3.138 (3)	125
C5—H5A...I1 ⁱⁱ	0.97	3.04	3.807 (3)	137
C7—H7...I1 ⁱⁱⁱ	0.93	3.04	3.854 (3)	147

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 2, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x, -y + \frac{1}{2}, z - \frac{1}{2}$.

Data collection: APEX2 (Bruker, 2011); cell refinement: SAINT (Bruker, 2011); data reduction: SAINT; program(s) used to solve structure: SIR2002 (Burla *et al.*, 2003); program(s) used to refine structure: SHELXL97 (Sheldrick, 2015); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012) and DIAMOND (Brandenburg, 2006); software used to prepare material for publication: WinGX (Farrugia, 2012).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HG5425).

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supporting information

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Crystal structure of 1-[(1-methyl-5-nitro-1*H*-imidazol-2-yl)methyl]pyridinium iodide

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S1. Comment

1. Chemical Context

Nitroheterocyclic drugs have drawn a continuing interest over the years due to efficient use in the treatment of various anaerobic pathogenic bacterial and protozoal infections (Upcroft & Upcroft, 2001; Çelik & Ates, 2006). Nitroimidazole derivatives have been the subject of much research because of their properties. Depending on the nature and the position of substituents or the nitro group, the nitroimidazole derivatives can possess various pharmacological actions (Boyer, 1986). Nitroimidazoles, such as metronidazole, misonidazole, ornidazole, secnidazole and etamidazole, are commonly used as therapeutic agents against a variety of protozoan and bacterial infections of humans and animals (Olender *et al.*, 2009; Gaonkar *et al.* 2009; Larina & Lopyrev 2009). In previous work, we have reported the synthesis and structure determination of some new heterocyclic compounds bearing a nitroimidazole entity (Zama *et al.*, 2013; Alliouche *et al.*, 2014; Bahnous *et al.*, 2012). Herein, we report the synthesis and single-crystal X-ray structure of 1-[(1-methyl-5-nitro-1*H*-imidazol-2-yl)methyl]pyridinium iodide, (I).

2. Structural commentary

The molecule structure of (I), and the atomic numbering used, is illustrated in Fig. 1. The asymmetric unit of (I) consists of pyridinium cation bearing a 1-methyl-5-nitro-1*H*-imidazol-2-yl)methyl group at N position, and the iodide anion. The imidazol ring is quasiplanar with maximum deviation of 0.0032 (16) Å at C1 atom; and forms a dihedral angle of 67.39 (6)° with pyridinium ring. The crystal packing can be described by alternating layers in zigzag parallel to (001) plane of cations group, which are sandwiched by iodide ions (Fig. 2).

3. Supramolecular features

The crystal packing is mostly governed by classical hydrogen bonds (Fig. 3). Atoms C2, C5, C7, C10 and O2 of the cation participate in the formation of intramolecular [C—H...O and C—H...I] hydrogen bonds (Table 1). In this structure, we observe two types of hydrogen bonds, *viz.* cation-anion, cation-cation which form a three-dimensional network. The intramolecular hydrogen bond interactions C—H...O are also observed in cation moieties. However, the centroid to centroid distance between the phenyl rings are too long (4.430 (3) Å) for considering π - π interactions. These interactions link the molecules within the layers and also link the layers together and reinforcing the cohesion of the ionic structure.

S2. Experimental

The 1-[(1-methyl-5-nitro-1*H*-imidazol-2-yl)methyl]pyridinium iodide, I, was prepared from 1,2-dimethyl-5-nitro-1*H*-imidazole in presence of iodine and pyridine as solvent according to described procedure (Albright & Shepherd, 1973). The colorless crystals of the title compound used for the X-ray diffraction study were obtained from aqueous solution of I.

S3. Refinement

The H atoms were localized on Fourier maps but introduced in calculated positions and treated as riding on their parent atom (C) with C—H = 0.93 Å (aromatic), C—H = 0.97 Å (methylene) and C—H = 0.96 Å (methyl) with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{C})$.

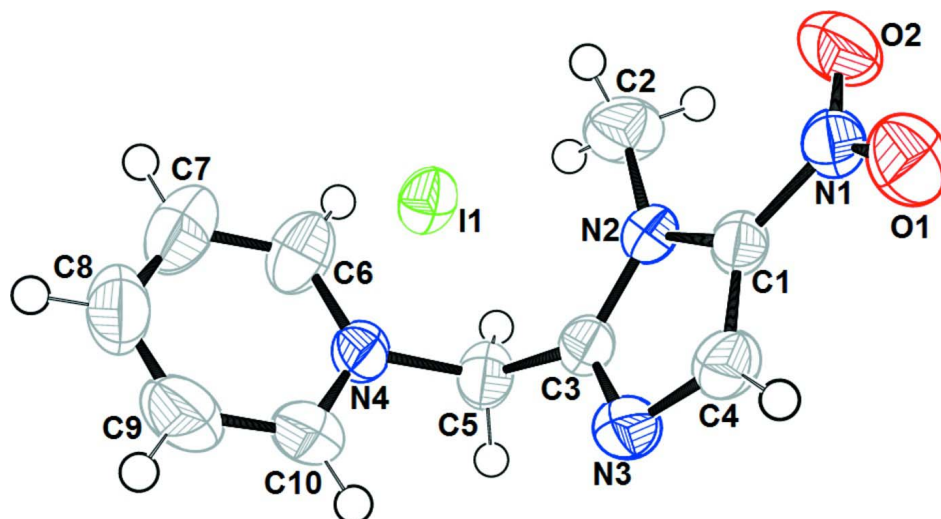


Figure 1

(Farrugia, 2012). The molecule structure of the title compound with the atomic labelling scheme. Displacement are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radius.

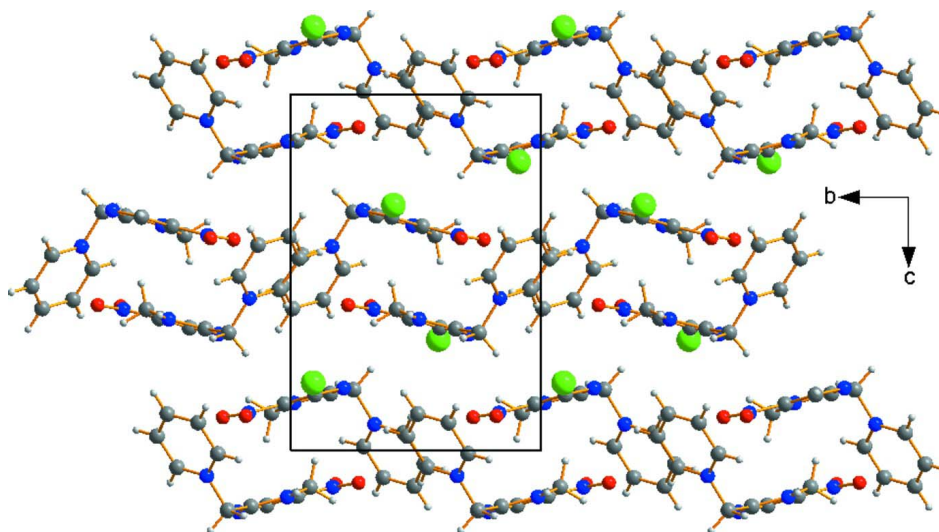
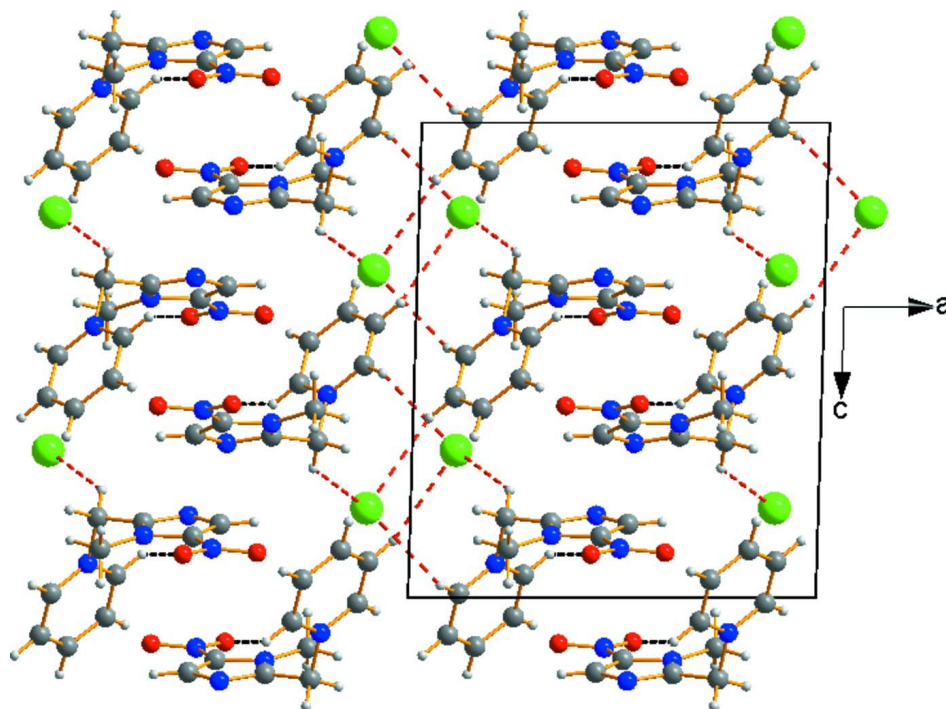


Figure 2

(Brandenburg, 2006). Alternating layers parallel to (001) plane of (I) sandwiched by iodide ions viewed *via a* axis

**Figure 3**

(Brandenburg, 2006). Crystal packing of (I) viewed *via* *b* axis showing hydrogen bond as dashed lines [C—H...I in red and C—H...O in black]

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Crystal data

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 $c = 12.859$ (8) Å
 $\beta = 91.69$ (2)°
 $V = 1286.8$ (14) Å³
 $Z = 4$

$F(000) = 672$
 $D_x = 1.787$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 6343 reflections
 $\theta = 2.8$ – 29.3 °
 $\mu = 2.49$ mm⁻¹
 $T = 295$ K
 Prism, colorless
 $0.14 \times 0.12 \times 0.11$ mm

Data collection

Bruker APEXII
 diffractometer
 Radiation source: Enraf–Nonius FR590
 Graphite monochromator
 CCD rotation images, thick slices scans
 Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 2002)
 $T_{\min} = 0.615$, $T_{\max} = 0.745$

22502 measured reflections
 6134 independent reflections
 3669 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$
 $\theta_{\max} = 36.5$ °, $\theta_{\min} = 2.9$ °
 $h = -18 \rightarrow 17$
 $k = -14 \rightarrow 14$
 $l = -21 \rightarrow 21$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.067$
 $S = 0.99$
 6134 reflections
 155 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0275P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.006$
 $\Delta\rho_{\max} = 1.14 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.89 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.53170 (14)	-0.02548 (17)	0.12964 (12)	0.0288 (3)
C2	0.75852 (17)	-0.0764 (2)	0.10429 (18)	0.0469 (5)
H2A	0.8317	-0.0235	0.1217	0.07*
H2B	0.755	-0.0965	0.031	0.07*
H2C	0.7577	-0.1676	0.1421	0.07*
C3	0.65440 (15)	0.15859 (18)	0.15888 (12)	0.0299 (3)
C4	0.46650 (16)	0.09581 (18)	0.15569 (14)	0.0361 (4)
H4	0.3826	0.0995	0.1603	0.043*
C5	0.76681 (16)	0.25171 (19)	0.16974 (12)	0.0353 (4)
H5A	0.7584	0.3202	0.227	0.042*
H5B	0.8361	0.189	0.1855	0.042*
C6	0.87603 (16)	0.2915 (2)	0.01029 (14)	0.0458 (4)
H6	0.9214	0.2079	0.0268	0.055*
C7	0.8991 (2)	0.3696 (3)	-0.07726 (17)	0.0563 (5)
H7	0.9604	0.3395	-0.1206	0.068*
C8	0.8319 (3)	0.4928 (3)	-0.10157 (16)	0.0633 (6)
H8	0.8489	0.5479	-0.1603	0.076*
C9	0.7398 (3)	0.5340 (2)	-0.03922 (18)	0.0698 (7)
H9	0.6922	0.6156	-0.0562	0.084*
C10	0.7181 (2)	0.4539 (2)	0.04885 (16)	0.0520 (5)
H10	0.6556	0.481	0.092	0.062*
N1	0.48460 (14)	-0.16821 (16)	0.10430 (11)	0.0361 (3)
N2	0.65316 (11)	0.01303 (14)	0.13202 (9)	0.0278 (3)
N3	0.54387 (13)	0.21140 (15)	0.17398 (11)	0.0377 (3)
N4	0.78797 (12)	0.33517 (15)	0.07265 (10)	0.0323 (3)

O1	0.37309 (12)	-0.17941 (15)	0.09568 (11)	0.0516 (3)
O2	0.55464 (14)	-0.27106 (14)	0.09207 (13)	0.0594 (4)
I1	1.107377 (10)	0.089936 (13)	0.189052 (9)	0.04248 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0291 (8)	0.0313 (8)	0.0262 (7)	-0.0053 (6)	0.0025 (6)	0.0001 (6)
C2	0.0322 (10)	0.0468 (11)	0.0620 (12)	0.0067 (8)	0.0072 (9)	-0.0108 (9)
C3	0.0317 (8)	0.0315 (8)	0.0268 (7)	-0.0030 (6)	0.0042 (6)	0.0000 (6)
C4	0.0269 (8)	0.0405 (9)	0.0412 (9)	-0.0011 (7)	0.0070 (7)	-0.0027 (7)
C5	0.0376 (9)	0.0392 (9)	0.0291 (7)	-0.0098 (7)	0.0012 (7)	0.0012 (7)
C6	0.0322 (9)	0.0637 (12)	0.0416 (10)	-0.0018 (9)	0.0049 (8)	0.0067 (9)
C7	0.0492 (12)	0.0784 (15)	0.0417 (10)	-0.0130 (11)	0.0102 (9)	0.0030 (11)
C8	0.1028 (19)	0.0505 (12)	0.0368 (10)	-0.0304 (13)	0.0066 (11)	0.0034 (10)
C9	0.123 (2)	0.0316 (10)	0.0558 (13)	0.0119 (12)	0.0106 (14)	0.0052 (11)
C10	0.0778 (16)	0.0293 (8)	0.0498 (11)	0.0068 (10)	0.0161 (10)	-0.0007 (9)
N1	0.0418 (9)	0.0356 (7)	0.0311 (7)	-0.0086 (6)	0.0038 (6)	-0.0016 (6)
N2	0.0259 (7)	0.0309 (7)	0.0267 (6)	0.0000 (5)	0.0039 (5)	0.0005 (5)
N3	0.0354 (8)	0.0333 (7)	0.0449 (8)	-0.0005 (6)	0.0095 (6)	-0.0049 (6)
N4	0.0349 (8)	0.0312 (7)	0.0310 (6)	-0.0096 (6)	0.0028 (6)	-0.0037 (6)
O1	0.0413 (8)	0.0544 (8)	0.0589 (8)	-0.0164 (6)	-0.0041 (6)	-0.0063 (7)
O2	0.0606 (9)	0.0336 (7)	0.0847 (11)	-0.0003 (7)	0.0177 (8)	-0.0112 (7)
I1	0.03084 (7)	0.04881 (8)	0.04816 (8)	0.00945 (5)	0.00730 (5)	0.00972 (5)

Geometric parameters (Å, °)

C1—C4	1.362 (2)	C5—H5B	0.97
C1—N2	1.385 (2)	C6—N4	1.338 (2)
C1—N1	1.429 (2)	C6—C7	1.360 (3)
C2—N2	1.470 (2)	C6—H6	0.93
C2—H2A	0.96	C7—C8	1.373 (3)
C2—H2B	0.96	C7—H7	0.93
C2—H2C	0.96	C8—C9	1.365 (4)
C3—N3	1.330 (2)	C8—H8	0.93
C3—N2	1.365 (2)	C9—C10	1.373 (3)
C3—C5	1.504 (2)	C9—H9	0.93
C4—N3	1.368 (2)	C10—N4	1.355 (2)
C4—H4	0.93	C10—H10	0.93
C5—N4	1.484 (2)	N1—O2	1.225 (2)
C5—H5A	0.97	N1—O1	1.236 (2)
C4—C1—N2	107.98 (14)	C7—C6—H6	120
C4—C1—N1	126.65 (15)	C6—C7—C8	119.9 (2)
N2—C1—N1	125.37 (14)	C6—C7—H7	120
N2—C2—H2A	109.5	C8—C7—H7	120
N2—C2—H2B	109.5	C9—C8—C7	119.7 (2)
H2A—C2—H2B	109.5	C9—C8—H8	120.1

N2—C2—H2C	109.5	C7—C8—H8	120.1
H2A—C2—H2C	109.5	C8—C9—C10	119.4 (2)
H2B—C2—H2C	109.5	C8—C9—H9	120.3
N3—C3—N2	112.53 (14)	C10—C9—H9	120.3
N3—C3—C5	122.78 (15)	N4—C10—C9	119.7 (2)
N2—C3—C5	124.69 (15)	N4—C10—H10	120.2
C1—C4—N3	109.28 (16)	C9—C10—H10	120.2
C1—C4—H4	125.4	O2—N1—O1	123.78 (15)
N3—C4—H4	125.4	O2—N1—C1	119.53 (15)
N4—C5—C3	110.99 (13)	O1—N1—C1	116.69 (15)
N4—C5—H5A	109.4	C3—N2—C1	104.61 (13)
C3—C5—H5A	109.4	C3—N2—C2	126.38 (14)
N4—C5—H5B	109.4	C1—N2—C2	128.85 (14)
C3—C5—H5B	109.4	C3—N3—C4	105.60 (14)
H5A—C5—H5B	108	C6—N4—C10	121.25 (16)
N4—C6—C7	119.9 (2)	C6—N4—C5	119.21 (15)
N4—C6—H6	120	C10—N4—C5	119.53 (15)
N2—C1—C4—N3	0.4 (2)	C5—C3—N2—C2	-3.1 (2)
N1—C1—C4—N3	-179.70 (15)	C4—C1—N2—C3	-0.60 (17)
N3—C3—C5—N4	-83.61 (19)	N1—C1—N2—C3	179.51 (14)
N2—C3—C5—N4	95.79 (18)	C4—C1—N2—C2	-176.19 (17)
N4—C6—C7—C8	-0.1 (3)	N1—C1—N2—C2	3.9 (3)
C6—C7—C8—C9	-1.8 (3)	N2—C3—N3—C4	-0.35 (19)
C7—C8—C9—C10	1.9 (4)	C5—C3—N3—C4	179.12 (15)
C8—C9—C10—N4	-0.1 (4)	C1—C4—N3—C3	-0.1 (2)
C4—C1—N1—O2	-173.00 (17)	C7—C6—N4—C10	2.1 (3)
N2—C1—N1—O2	6.9 (2)	C7—C6—N4—C5	-177.91 (17)
C4—C1—N1—O1	7.2 (2)	C9—C10—N4—C6	-2.0 (3)
N2—C1—N1—O1	-172.89 (15)	C9—C10—N4—C5	178.00 (19)
N3—C3—N2—C1	0.59 (17)	C3—C5—N4—C6	-105.28 (18)
C5—C3—N2—C1	-178.86 (14)	C3—C5—N4—C10	74.8 (2)
N3—C3—N2—C2	176.33 (16)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2C \cdots O2	0.96	2.50	2.861 (3)	102
C10—H10 \cdots O2 ⁱ	0.93	2.51	3.138 (3)	125
C5—H5A \cdots I1 ⁱⁱ	0.97	3.04	3.807 (3)	137
C7—H7 \cdots I1 ⁱⁱⁱ	0.93	3.04	3.854 (3)	147

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+2, y+1/2, -z+1/2$; (iii) $x, -y+1/2, z-1/2$.