



# Crystal structure and spectroscopic properties of (*E*)-1,3-dimethyl-2-[3-(4-nitrophenyl)triaz-2-enylidene]-2,3-dihydro-1*H*-imidazole

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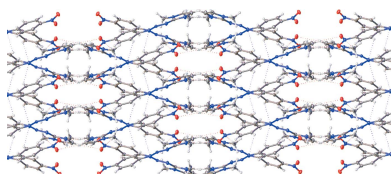
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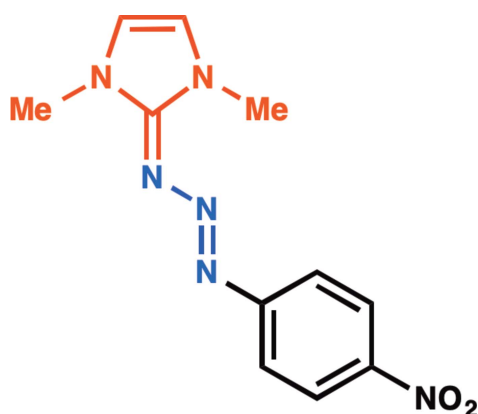
The title compound (*E*)-1,3-dimethyl-2-[3-(4-nitrophenyl)triaz-2-enylidene]-2,3-dihydro-1*H*-imidazole, C<sub>11</sub>H<sub>12</sub>N<sub>6</sub>O<sub>2</sub>, has monoclinic (*C*2/*c*) symmetry at 100 K. This triazene derivative was synthesized by the coupling reaction of 1,3-dimethylimidazolium iodide with 1-azido-4-nitro benzene in the presence of sodium hydride (60% in mineral oil) and characterized by <sup>1</sup>H NMR, <sup>13</sup>C NMR, IR, mass spectrometry, and single-crystal X-ray diffraction. The molecule consists of six-membered and five-membered rings, which are connected by a triazene moiety (–N=N–N–). In the solid-state, the molecule is found to be planar due to conjugation throughout the molecule. The extended structure shows two layers of molecules, which present weak intermolecular interactions that facilitate the stacked arrangement of the molecules forming the extended structure. Furthermore, there are several weak pseudo-cyclical interactions between the nitro oxygen atoms and symmetry-adjacent H atoms, which help to arrange the molecules.

## 1. Chemical context

Triazenes are versatile compounds in preparative chemistry because of their stable and highly modular nature (Patil & Bugarin, 2016). Triazene derivatives have been studied for their potential anticancer properties (Rouzer *et al.*, 1996; Connors *et al.*, 1976), used as a protecting group in natural product synthesis (Nicolaou *et al.*, 1999) and combinatorial chemistry (Brase *et al.*, 2000), incorporated into polymers (Jones *et al.*, 1997) and oligomer synthesis (Moore, 1997), and used to prepare heterocycles (Wirschun *et al.*, 1998). Their modular nature allows triazenes to be converted into a different functional group after treatment with the appropriate reagents. For example, aryl triazenes can be transformed to useful cross-coupling reagents (iodoarenes) *via* iodomethane-induced decomposition (Zollinger, 1994). Further, aryl triazenes have been studied for their unique structural and chemical properties (Cornali *et al.*, 2016; Knyazeva *et al.*, 2017). They have been used in medicinal, combinatorial chemistry, in natural synthesis and as organometallic ligands (Kimball *et al.*, 2002). In chemical biology, masked diazonium ions (triazabutadienes) have found use on protein surfaces for identifying host proteins that interact

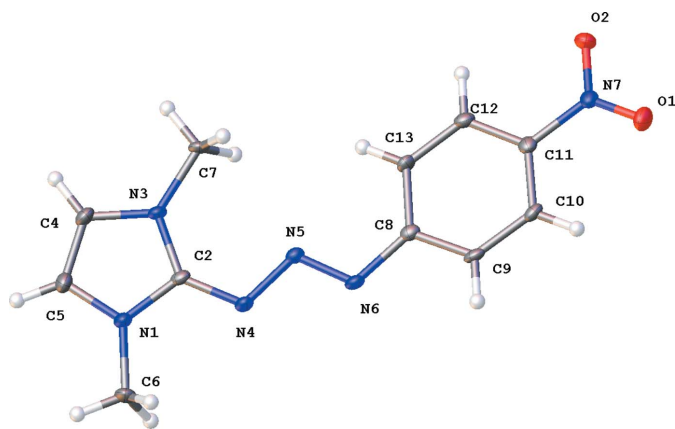


during early stages of viral entities (Jensen *et al.*, 2016; Shadmehr *et al.*, 2018). In addition, triazabutadienes have shown tunable reactivity under specific conditions. For example, unique transformations of triazabutadienes have been affected *via* water solubility, pH (Kimani & Jewett, 2015; Guzman *et al.*, 2016; He *et al.*, 2017), and photoinduced isomerization (He *et al.*, 2015). In synthesis, these triazenes have been used as starting materials for aldehydes, ketones, ethers, and sulfides, under mild reaction conditions (Barragan & Bugarin, 2017; Cornali *et al.*, 2016). Furthermore, natural sunlight has been utilized to activate those triazenes to produce bisaryls and anilides (Noonikara-Poyil *et al.*, 2019; Barragan *et al.*, 2020).

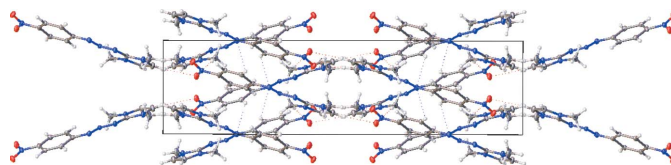


## 2. Structural commentary

The title compound **1** crystallizes in the monoclinic  $C2/c$  space group with a single moiety in the asymmetric unit (see Fig. 1). The molecule is nearly planar with a dihedral angle of  $7.36$  ( $9^\circ$ ) between the imidazole and benzene rings. Plane **I** (N1/N3/C2/C4–C7) makes dihedral angles of  $9.70$  ( $3^\circ$ ) and  $2.40$  ( $6^\circ$ ), respectively, with planes **II** (N7/C8–C13) and **III** (N4–N6) while plane **II** makes a dihedral angle of  $7.36$  ( $4^\circ$ ) with plane **III**.



**Figure 1**  
ORTEP diagram of compound **1** showing the atom-labeling scheme with 50% probability displacement ellipsoids.



**Figure 2**  
Perspective view of the molecular packing of **1** (there are no hydrogen atoms involved in hydrogen-bonding interactions. Dotted lines represent weak non-covalent C–H...N and C–H...O interactions, which direct the packing.

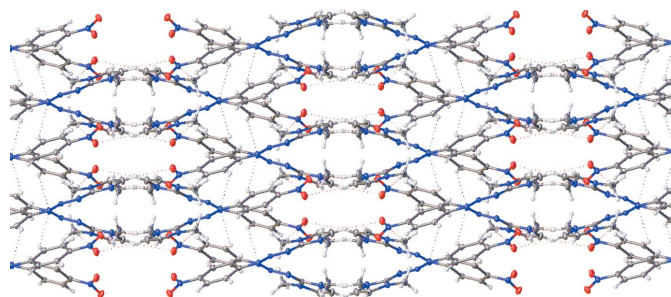
There are no lattice-held water molecules or organic solvent molecules in the unit cell of the determined structure, a potential issue since the starting material 1,3-dimethylimidazolium iodide is hygroscopic. The bond lengths and angles in **1** are similar to those reported for analogous structures (Khrarov & Bielawski, 2005; Jishkariani *et al.*, 2013). The C–C bond lengths in the phenyl rings are in the normal range of  $1.33$ – $1.40$  Å, which is characteristic of delocalized phenyl rings. The C–C–C bond angles are around  $120^\circ$ , with the variation being less than  $2^\circ$ , which is characteristic of  $sp^2$ -hybridized carbons.

## 3. Supramolecular features

Figs. 2 and 3 show a perspective view of the crystal packing of the title compound. The packing diagram shows two layers of molecules, which are independently arranged in the unit cell without intra- and inter molecular hydrogen bonds. In each layer, the molecules are alternately parallel.

## 4. Database survey

The first X-ray structure of a  $\pi$ -conjugated triazene was reported by Khrarov & Bielawski (2005). The current WebCSD structural database includes the structures of only 18  $\pi$ -conjugated triazenes. However, there is only one structure, reported by our research group (Patil & Bugarin, 2014), that utilizes the small molecule (1,3-dimethylimidazolium iodide) as the carbene coupling partner, and one more that bears an electron-deficient aryl group in combination with the small



**Figure 3**  
Extended network of the structure of compound **1** viewed from (001). There are two layers of molecules, which are arranged independently in the unit cell without intra- and/or intermolecular hydrogen bonds. Dotted lines represent weak non-covalent C–H...N and C–H...O interactions, which direct the packing.

carbene precursor (Patil *et al.*, 2014). Those characteristics highlight the novelty and uniqueness of the compound reported herein.

## 5. Synthesis and crystallization

1-Azido-4-nitro benzene (Siddiki *et al.*, 2013) and 1,3-dimethylimidazolium iodide (Oertel *et al.*, 2011) were prepared according to literature procedures. For the synthesis of the title compound, 1-azido-4-nitro benzene (131.2 mg, 0.8 mmol) and 1,3-dimethylimidazolium iodide (89.5 mg, 0.4 mmol) were stirred at room temperature for 5 min. in dry THF (5 mL). NaH (16 mg, 0.4 mmol, 60% in mineral oil) was added in one portion and stirring was continued at room temperature overnight. A red precipitate formed, which was collected by filtration and dried under reduced pressure, giving the pure product (*E*)-1,3-dimethyl-2-[(4-nitrophenyl)triaz-2-enylidene]-2,3-dihydro-1*H*-imidazole as a red crystalline solid (88.2 mg, 85%). Crystals suitable for X-ray analysis were grown from the slow evaporation of a THF/hexane mixture yielding air-stable, red-colored crystals.

IR (neat)  $\nu$  3435, 1572, 1382, 1360, 1233  $\text{cm}^{-1}$ .  $^1\text{H}$  NMR (400 MHz, DMSO-*d*6):  $\delta$  8.12 (*d*,  $J = 9.2$  Hz, 2 H, Ph-H), 7.43 (*d*,  $J = 9.2$  Hz, 2 H, Ph-H), 7.13 (*s*, 2 H, NCH), 3.62 (*s*, 6 H, N-CH<sub>3</sub>).  $^{13}\text{C}$  NMR (100 MHz, DMSO-*d*6):  $\delta$  159.1, 150.7, 143.2, 125.5, 120.8, 118.9, 35.7. UV/Vis (0.1  $\mu\text{M}$ , CH<sub>2</sub>Cl<sub>2</sub>):  $\lambda$  ( $\epsilon$ ) = 450 nm. HRMS (ESI, N<sub>2</sub>): *m/z* calculated for C<sub>11</sub>H<sub>12</sub>N<sub>6</sub>O<sub>2</sub>Na [*M* + Na]<sup>+</sup> 283.0914, found 283.0918.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were included in calculated positions and treated as riding atoms: C–H = 0.95–0.98 Å with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{C-methyl})$ .

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**Table 1**  
Experimental details.

Crystal data	
Chemical formula	C <sub>11</sub> H <sub>12</sub> N <sub>6</sub> O <sub>2</sub>
<i>M<sub>r</sub></i>	260.27
Crystal system, space group	Monoclinic, C2/c
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	27.7311 (18), 7.1747 (9), 11.7849 (14)
$\beta$ (°)	94.101 (4)
<i>V</i> (Å <sup>3</sup> )	2338.7 (4)
<i>Z</i>	8
Radiation type	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.11
Crystal size (mm)	0.11 × 0.05 × 0.02
Data collection	
Diffractometer	Bruker AXS D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2018)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.655, 0.746
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	23709, 2807, 2206
<i>R<sub>int</sub></i>	0.077
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.659
Refinement	
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , <i>S</i>	0.070, 0.158, 1.16
No. of reflections	2807
No. of parameters	174
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$ , $\Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.36, -0.37

Computer programs: *APEX3* and *S SAINT* (Bruker, 2018), *SHELXT2018/2* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

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## supporting information

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## Crystal structure and spectroscopic properties of (*E*)-1,3-dimethyl-2-[3-(4-nitrophenyl)triaz-2-enylidene]-2,3-dihydro-1*H*-imidazole

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### Computing details

Data collection: *APEX3* (Bruker, 2018); cell refinement: *SAINT* (Bruker, 2018); data reduction: *SAINT* (Bruker, 2018); program(s) used to solve structure: *SHELXT2018/2* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

### (*E*)-1,3-Dimethyl-2-[3-(4-nitrophenyl)triaz-2-enylidene]-2,3-dihydro-1*H*-imidazole

#### Crystal data

$C_{11}H_{12}N_6O_2$

$M_r = 260.27$

Monoclinic, *C2/c*

$a = 27.7311$  (18) Å

$b = 7.1747$  (9) Å

$c = 11.7849$  (14) Å

$\beta = 94.101$  (4)°

$V = 2338.7$  (4) Å<sup>3</sup>

$Z = 8$

$F(000) = 1088$

$D_x = 1.478$  Mg m<sup>-3</sup>

Mo *K*α radiation,  $\lambda = 0.71073$  Å

Cell parameters from 4851 reflections

$\theta = 3.0$ – $27.8$ °

$\mu = 0.11$  mm<sup>-1</sup>

$T = 100$  K

Plates, red

$0.11 \times 0.05 \times 0.02$  mm

#### Data collection

Bruker AXS D8 Quest  
diffractometer with PhotonII charge-integrating  
pixel array detector (CPAD)

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Bruker, 2018)

$T_{\min} = 0.655$ ,  $T_{\max} = 0.746$

23709 measured reflections

2807 independent reflections

2206 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.077$

$\theta_{\max} = 27.9$ °,  $\theta_{\min} = 2.9$ °

$h = -36 \rightarrow 36$

$k = -9 \rightarrow 9$

$l = -15 \rightarrow 15$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.070$

$wR(F^2) = 0.158$

$S = 1.16$

2807 reflections

174 parameters

0 restraints

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 4.2467P]$

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

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

$\Delta\rho_{\max} = 0.36$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.37$  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>	$U_{\text{iso}}^*/U_{\text{eq}}$
O2	0.90725 (6)	0.6192 (3)	0.42545 (14)	0.0208 (4)
O1	0.91792 (6)	0.7705 (3)	0.58417 (15)	0.0231 (4)
N5	0.67583 (7)	0.4345 (3)	0.54983 (15)	0.0122 (4)
N3	0.60385 (7)	0.2903 (3)	0.39246 (15)	0.0133 (4)
N6	0.70494 (7)	0.4980 (3)	0.63041 (15)	0.0144 (4)
N1	0.55532 (7)	0.2913 (3)	0.52968 (16)	0.0163 (4)
N7	0.89267 (7)	0.6760 (3)	0.51636 (16)	0.0156 (4)
N4	0.63305 (7)	0.3932 (3)	0.58753 (15)	0.0147 (4)
C2	0.60118 (8)	0.3293 (3)	0.50481 (18)	0.0132 (5)
C8	0.75071 (8)	0.5395 (3)	0.59381 (18)	0.0131 (5)
C13	0.76739 (8)	0.4927 (3)	0.48705 (19)	0.0138 (5)
H13	0.746476	0.430092	0.432205	0.017*
C10	0.82858 (8)	0.6785 (3)	0.64889 (18)	0.0140 (5)
H10	0.849717	0.740951	0.703362	0.017*
C12	0.81359 (8)	0.5371 (3)	0.46184 (19)	0.0150 (5)
H12	0.824761	0.505139	0.390018	0.018*
C9	0.78212 (8)	0.6344 (3)	0.67289 (18)	0.0140 (5)
H9	0.771038	0.668868	0.744390	0.017*
C11	0.84393 (8)	0.6295 (3)	0.54293 (19)	0.0148 (5)
C7	0.64554 (8)	0.3132 (3)	0.32329 (18)	0.0157 (5)
H7A	0.656635	0.442877	0.327693	0.023*
H7B	0.635989	0.281514	0.243959	0.023*
H7C	0.671761	0.230600	0.352109	0.023*
C4	0.55897 (8)	0.2268 (4)	0.34928 (19)	0.0184 (5)
H4	0.550771	0.189696	0.272952	0.022*
C5	0.52938 (8)	0.2270 (4)	0.4335 (2)	0.0200 (5)
H5	0.496428	0.189691	0.428140	0.024*
C6	0.53830 (9)	0.3056 (4)	0.6434 (2)	0.0249 (6)
H6A	0.553996	0.209729	0.692393	0.037*
H6B	0.503190	0.287618	0.639429	0.037*
H6C	0.546263	0.429166	0.674856	0.037*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O2	0.0169 (8)	0.0327 (10)	0.0134 (8)	−0.0025 (7)	0.0046 (6)	0.0010 (8)
O1	0.0202 (9)	0.0287 (10)	0.0195 (9)	−0.0084 (8)	−0.0042 (7)	−0.0001 (8)
N5	0.0143 (9)	0.0132 (9)	0.0092 (8)	0.0006 (7)	0.0006 (7)	0.0000 (7)
N3	0.0145 (9)	0.0185 (10)	0.0072 (8)	−0.0003 (8)	0.0022 (7)	0.0002 (7)

N6	0.0159 (10)	0.0204 (10)	0.0070 (8)	0.0011 (8)	0.0010 (7)	-0.0003 (8)
N1	0.0133 (9)	0.0269 (11)	0.0089 (9)	-0.0009 (8)	0.0009 (7)	0.0009 (8)
N7	0.0147 (9)	0.0192 (10)	0.0126 (9)	-0.0016 (8)	-0.0004 (7)	0.0040 (8)
N4	0.0136 (9)	0.0226 (10)	0.0080 (8)	0.0006 (8)	0.0013 (7)	-0.0019 (8)
C2	0.0158 (11)	0.0162 (11)	0.0076 (10)	0.0026 (9)	0.0002 (8)	0.0020 (8)
C8	0.0161 (11)	0.0134 (10)	0.0097 (10)	0.0022 (9)	0.0002 (8)	0.0015 (8)
C13	0.0149 (11)	0.0172 (11)	0.0090 (10)	-0.0015 (9)	-0.0004 (8)	-0.0032 (8)
C10	0.0174 (11)	0.0156 (11)	0.0084 (10)	0.0001 (9)	-0.0035 (8)	0.0003 (8)
C12	0.0180 (11)	0.0183 (11)	0.0088 (10)	0.0005 (9)	0.0014 (8)	0.0017 (9)
C9	0.0184 (11)	0.0177 (11)	0.0059 (9)	0.0032 (9)	0.0003 (8)	0.0008 (9)
C11	0.0146 (11)	0.0179 (11)	0.0118 (10)	-0.0005 (9)	-0.0007 (8)	0.0052 (9)
C7	0.0164 (11)	0.0238 (12)	0.0073 (10)	-0.0006 (9)	0.0047 (8)	-0.0004 (9)
C4	0.0171 (11)	0.0281 (13)	0.0096 (10)	-0.0009 (10)	-0.0024 (8)	-0.0011 (10)
C5	0.0146 (11)	0.0308 (14)	0.0141 (11)	-0.0040 (10)	-0.0032 (9)	0.0027 (10)
C6	0.0169 (11)	0.0473 (17)	0.0112 (11)	-0.0049 (12)	0.0063 (9)	-0.0017 (11)

*Geometric parameters (Å, °)*

O2—N7	1.241 (3)	C13—C12	1.373 (3)
O1—N7	1.228 (3)	C10—H10	0.9500
N5—N6	1.285 (3)	C10—C9	1.375 (3)
N5—N4	1.330 (3)	C10—C11	1.393 (3)
N3—C2	1.360 (3)	C12—H12	0.9500
N3—C7	1.471 (3)	C12—C11	1.395 (3)
N3—C4	1.387 (3)	C9—H9	0.9500
N6—C8	1.402 (3)	C7—H7A	0.9800
N1—C2	1.353 (3)	C7—H7B	0.9800
N1—C5	1.378 (3)	C7—H7C	0.9800
N1—C6	1.456 (3)	C4—H4	0.9500
N7—C11	1.448 (3)	C4—C5	1.332 (3)
N4—C2	1.348 (3)	C5—H5	0.9500
C8—C13	1.412 (3)	C6—H6A	0.9800
C8—C9	1.405 (3)	C6—H6B	0.9800
C13—H13	0.9500	C6—H6C	0.9800
N6—N5—N4	111.13 (17)	C11—C12—H12	120.4
C2—N3—C7	128.02 (19)	C8—C9—H9	119.3
C2—N3—C4	108.35 (18)	C10—C9—C8	121.4 (2)
C4—N3—C7	123.61 (18)	C10—C9—H9	119.3
N5—N6—C8	112.51 (18)	C10—C11—N7	119.1 (2)
C2—N1—C5	109.43 (19)	C10—C11—C12	121.7 (2)
C2—N1—C6	123.82 (19)	C12—C11—N7	119.2 (2)
C5—N1—C6	126.6 (2)	N3—C7—H7A	109.5
O2—N7—C11	118.54 (19)	N3—C7—H7B	109.5
O1—N7—O2	122.5 (2)	N3—C7—H7C	109.5
O1—N7—C11	118.95 (19)	H7A—C7—H7B	109.5
N5—N4—C2	112.85 (18)	H7A—C7—H7C	109.5
N1—C2—N3	106.65 (19)	H7B—C7—H7C	109.5

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N4—C2—N3	134.0 (2)	N3—C4—H4	125.9
N4—C2—N1	119.30 (19)	C5—C4—N3	108.1 (2)
N6—C8—C13	125.8 (2)	C5—C4—H4	125.9
N6—C8—C9	115.53 (19)	N1—C5—H5	126.3
C9—C8—C13	118.6 (2)	C4—C5—N1	107.5 (2)
C8—C13—H13	119.7	C4—C5—H5	126.3
C12—C13—C8	120.5 (2)	N1—C6—H6A	109.5
C12—C13—H13	119.7	N1—C6—H6B	109.5
C9—C10—H10	120.7	N1—C6—H6C	109.5
C9—C10—C11	118.5 (2)	H6A—C6—H6B	109.5
C11—C10—H10	120.7	H6A—C6—H6C	109.5
C13—C12—H12	120.4	H6B—C6—H6C	109.5
C13—C12—C11	119.2 (2)		
O2—N7—C11—C10	174.5 (2)	C13—C12—C11—N7	179.9 (2)
O2—N7—C11—C12	-5.6 (3)	C13—C12—C11—C10	-0.1 (3)
O1—N7—C11—C10	-5.8 (3)	C9—C8—C13—C12	0.9 (3)
O1—N7—C11—C12	174.2 (2)	C9—C10—C11—N7	179.7 (2)
N5—N6—C8—C13	-9.6 (3)	C9—C10—C11—C12	-0.3 (3)
N5—N6—C8—C9	171.13 (19)	C11—C10—C9—C8	1.0 (3)
N5—N4—C2—N3	3.5 (4)	C7—N3—C2—N1	178.1 (2)
N5—N4—C2—N1	-176.5 (2)	C7—N3—C2—N4	-1.8 (4)
N3—C4—C5—N1	0.3 (3)	C7—N3—C4—C5	-178.5 (2)
N6—N5—N4—C2	178.84 (19)	C4—N3—C2—N1	-0.3 (3)
N6—C8—C13—C12	-178.4 (2)	C4—N3—C2—N4	179.8 (3)
N6—C8—C9—C10	178.0 (2)	C5—N1—C2—N3	0.5 (3)
N4—N5—N6—C8	178.84 (18)	C5—N1—C2—N4	-179.6 (2)
C2—N3—C4—C5	0.0 (3)	C6—N1—C2—N3	176.7 (2)
C2—N1—C5—C4	-0.5 (3)	C6—N1—C2—N4	-3.4 (4)
C8—C13—C12—C11	-0.2 (3)	C6—N1—C5—C4	-176.5 (2)
C13—C8—C9—C10	-1.3 (3)		

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