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**Structure Reports**
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**(E)-tert-Butyl 4-(N'-hydroxycarbamimidoyl)piperazine-1-carboxylate**
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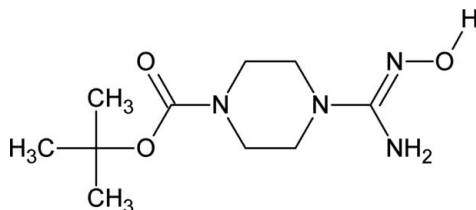
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 Key indicators: single-crystal X-ray study;  $T = 300$  K; mean  $\sigma(C-C) = 0.003$  Å;  $R$  factor = 0.044;  $wR$  factor = 0.130; data-to-parameter ratio = 13.7.

In the title compound,  $C_{10}H_{20}N_4O_3$ , the piperazine ring adopts a chair conformation. The molecule adopts an *E* conformation across the  $C=N$  double bond, with the  $-OH$  group and the piperazine ring *trans* to one another. Further, the H atom of the hydroxy group is directed away from the  $NH_2$  group. An intramolecular  $N-H \cdots O$  contact occurs involving the  $NH_2$  group and the oxime O atom. In the crystal, molecules are linked *via* strong  $N-H \cdots O$  and  $O-H \cdots N$  hydrogen bonds with alternating  $R_2^2(6)$  and  $C(9)$  motifs into tetrameric units forming  $R_4^4(28)$  motifs.

**Related literature**

For the synthesis, characterization and biological activity of piperazine and its derivatives, see: Gan *et al.* (2009*a,b*); Willems & Ilzerman (2010). For a related structure, see: Gowda *et al.* (2009). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter (1990).


**Experimental**
*Crystal data*
 $C_{10}H_{20}N_4O_3$ 
 $M_r = 244.3$ 

Triclinic,  $P\bar{1}$   
 $a = 8.1923$  (17) Å  
 $b = 8.7859$  (16) Å  
 $c = 9.714$  (2) Å  
 $\alpha = 109.451$  (7)°  
 $\beta = 99.540$  (7)°  
 $\gamma = 96.474$  (7)°

$V = 639.5$  (2) Å<sup>3</sup>  
 $Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 300$  K  
 $0.22 \times 0.16 \times 0.1$  mm

*Data collection*

Bruker SMART X2S diffractometer  
6407 measured reflections  
2218 independent reflections

1632 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.027$

*Refinement*

$R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.130$   
 $S = 1.05$   
2218 reflections  
162 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{max} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.20$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N2-H2D \cdots O1$	0.94 (3)	2.08 (3)	2.538 (2)	108.3 (19)
$N2-H2C \cdots O2^i$	0.89 (3)	2.10 (3)	2.988 (2)	173 (3)
$O1-H1 \cdots N1^{ii}$	0.82	2.04	2.764 (3)	147

 Symmetry codes: (i)  $x + 1, y, z$ ; (ii)  $-x + 1, -y + 3, -z + 2$ .

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZJ2096).

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## supporting information

*Acta Cryst.* (2012). E68, o3347 [doi:10.1107/S1600536812046223]

**(E)-tert-Butyl 4-(N'-hydroxycarbamimidoyl)piperazine-1-carboxylate**

S. Sreenivasa, K. E. Manojkumar, P. A. Suchetan, N. R. Mohan and B. S. Palakshamurthy

**S1. Comment**

Numerous piperazine derivatives like aryl amide, sulphonamides, Mannich bases, Schiff's bases, thiazolidinones, azetidinones, imidazolinones have shown a wide spectrum of biological activities *viz.* antiinflammatory, antibacterial, antimalarial, anticonvulsant, antipyretic, antitumor, anthelmintics, analgesic, antidepressant, antifungal, antitubercular, anticancer, antidiabetic *etc.* In this view, we synthesized the title compound to study its crystal structure. The molecule crystallizes in triclinic P-1 space group. The piperazine ring in the molecule adopts chair conformation and the molecule prefers E configuration across the C—N double bond, as the OH group and the piperazine ring are in the opposite side of the double bond (Figure 1). The hydrogen atom of the hydroxyl group is directed away from the NH<sub>2</sub> group. This results in stabilizing the structure through a strong intermolecular O—H···N(1) and an intramolecular N(2)—H···O hydrogen bonds. In addition to this, the molecule also exhibits a strong N(2)—H···O(C) intermolecular hydrogen bond. The molecules are connected through alternate  $R_2^2(6)$  ring and C(9) chain hydrogen bond patterns into tetrameric units exhibiting  $R_4^4(28)$  ring patterns (Figure 2). The average N—C bond length in the piperazine ring is 1.466 Å indicating the single bond nature. While, the N4—C(O) bond length is 1.359 (2) Å indicating the delocalization of the nitrogen lone pair of electrons into  $\pi$  system of the carbonyl group. The N(1)—C(1) bond length is 1.290 Å due to its double bond nature, but the N(3)—C(1) and N(2)—C(1) bond lengths are closer to N—C(O) lengths indicating the partial double bond nature of these bonds.

**S2. Experimental**

To a solution of N-boc-piperazine (10.6 mmol) in 20 ml of acetonitrile was added cyanogen bromide (10.7 mmol) and K<sub>2</sub>CO<sub>3</sub> (21.2 mmol) at -10°C. The reaction mixture was stirred for 18 h at room temperature under nitrogen atmosphere. N-Cyano-4-boc-piperazine was obtained. To N-cyano-4-boc-piperazine (4.6 mmol) in methanol was added NH<sub>2</sub>OH.HCl (9.3 mmol) and stirred for 30 min at room temperature. The solvent was removed under reduced pressure and the crude product was washed with cold water and dried to yield white solid product. Single crystals employed in X-ray diffraction studies were obtained from slow evaporation of the solution of the compound in methanol.

**S3. Refinement**

The H atoms were positioned with idealized geometry using a riding model with C—H = 0.93 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the  $U_{eq}$  of the parent atom).

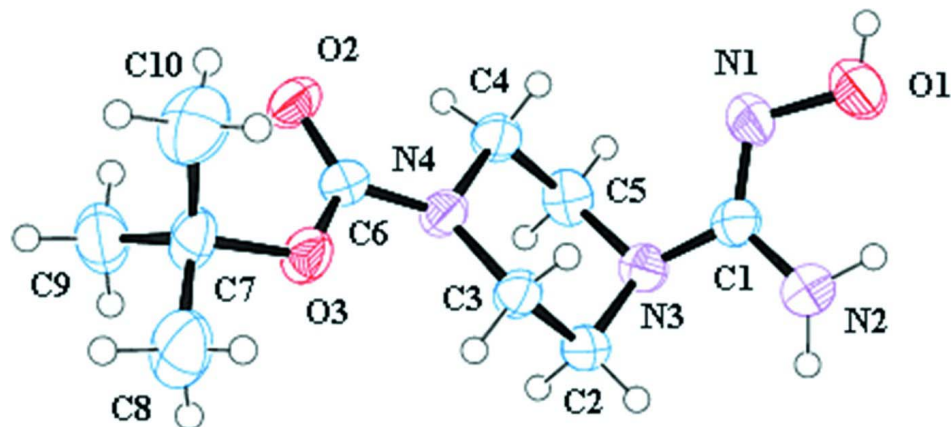


Figure 1

Molecular Structure of the title compound, Showing the atom-labeled Scheme.

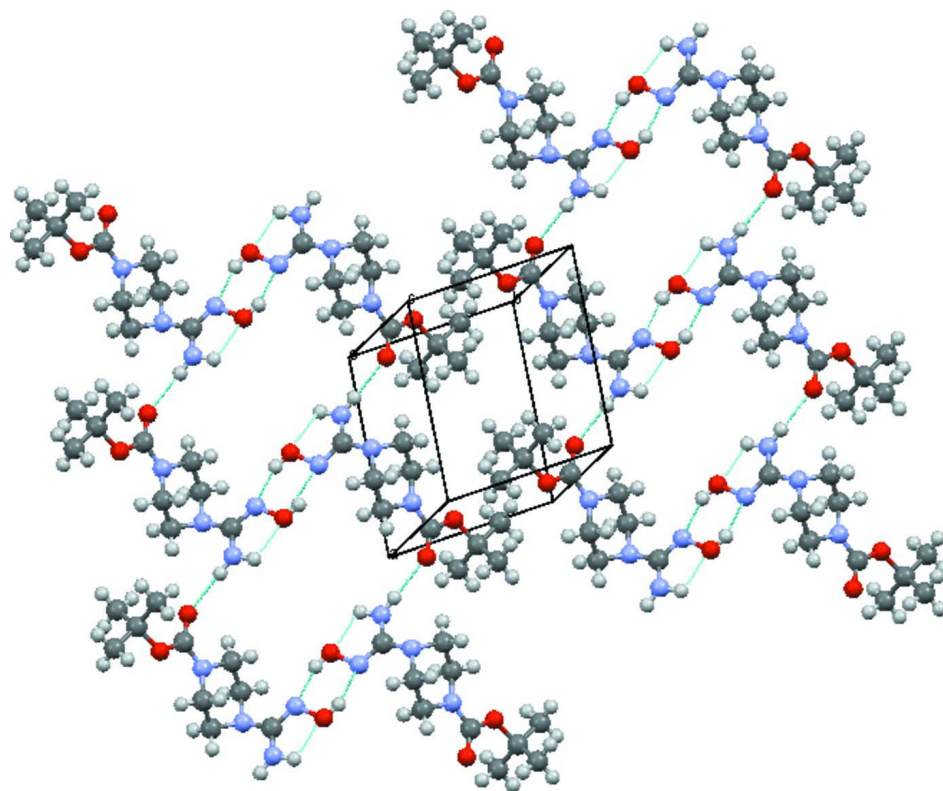


Figure 2

Molecular packing of the title compound, Hydrogen bonds are shown in dashed lines.

**(*E*)-*tert*-Butyl 4-(*N'*-hydroxycarbamimidoyl)piperazine-1-carboxylate**

*Crystal data*

$C_{10}H_{20}N_4O_3$

$M_r = 244.3$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 8.1923 (17) \text{ \AA}$

$b = 8.7859 (16) \text{ \AA}$

$c = 9.714 (2) \text{ \AA}$

$\alpha = 109.451 (7)^\circ$

$\beta = 99.540 (7)^\circ$

$\gamma = 96.474 (7)^\circ$

$V = 639.5 (2) \text{ \AA}^3$   
 $Z = 2$   
 $F(000) = 264$   
 prism  
 $D_x = 1.269 \text{ Mg m}^{-3}$   
 Melting point: 463 K  
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1632 reflections

$\theta = 25^\circ$   
 $\mu = 0.10 \text{ mm}^{-1}$   
 $T = 300 \text{ K}$   
 Prism, colorless  
 $0.22 \times 0.16 \times 0.1 \text{ mm}$

*Data collection*

Bruker SMART X2S  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 multi-scan  
 6407 measured reflections  
 2218 independent reflections

1632 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.027$   
 $\theta_{\text{max}} = 25.0^\circ$ ,  $\theta_{\text{min}} = 2.5^\circ$   
 $h = -9 \rightarrow 9$   
 $k = -10 \rightarrow 10$   
 $l = -11 \rightarrow 11$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.044$   
 $wR(F^2) = 0.130$   
 $S = 1.05$   
 2218 reflections  
 162 parameters  
 0 restraints  
 0 constraints  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0714P)^2 + 0.0663P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C8	-0.0609 (3)	0.4383 (3)	0.7235 (3)	0.0705 (7)
H8A	-0.1504	0.3592	0.7258	0.106*
H8B	0.0275	0.4655	0.8107	0.106*
H8C	-0.0177	0.3931	0.6354	0.106*
C7	-0.1275 (2)	0.5921 (2)	0.7215 (2)	0.0452 (5)
O3	0.02502 (15)	0.69800 (15)	0.71797 (16)	0.0449 (4)
C6	0.0186 (2)	0.8470 (2)	0.7099 (2)	0.0363 (4)
N4	0.17149 (18)	0.92339 (18)	0.70828 (17)	0.0379 (4)
C3	0.3293 (2)	0.8718 (2)	0.7533 (2)	0.0386 (5)
H3A	0.3094	0.7539	0.7291	0.046*

H3B	0.3705	0.9241	0.8606	0.046*
C2	0.4601 (2)	0.9178 (2)	0.6738 (2)	0.0426 (5)
H2A	0.5652	0.8871	0.7081	0.051*
H2B	0.423	0.8574	0.5672	0.051*
N3	0.48827 (18)	1.09515 (18)	0.70205 (16)	0.0380 (4)
C1	0.5834 (2)	1.1978 (2)	0.8426 (2)	0.0372 (5)
C10	-0.1871 (3)	0.6755 (3)	0.8630 (3)	0.0710 (7)
H10A	-0.2858	0.6076	0.866	0.106*
H10B	-0.2133	0.7796	0.864	0.106*
H10C	-0.0999	0.6921	0.9486	0.106*
O2	-0.10816 (16)	0.90611 (16)	0.70148 (16)	0.0489 (4)
N2	0.7249 (2)	1.1515 (2)	0.8972 (2)	0.0500 (5)
C5	0.3302 (2)	1.1390 (2)	0.6480 (2)	0.0437 (5)
H5A	0.2963	1.083	0.5405	0.052*
H5B	0.3481	1.2561	0.6682	0.052*
C4	0.1884 (2)	1.0956 (2)	0.7193 (2)	0.0422 (5)
H4A	0.2114	1.1666	0.8236	0.051*
H4B	0.0835	1.1134	0.6693	0.051*
C9	-0.2609 (3)	0.5532 (3)	0.5819 (3)	0.0623 (6)
H9A	-0.3586	0.4846	0.5859	0.093*
H9B	-0.2185	0.4968	0.496	0.093*
H9C	-0.2907	0.6533	0.5747	0.093*
O1	0.6559 (2)	1.42316 (18)	1.05009 (17)	0.0655 (5)
H1	0.6236	1.5071	1.0951	0.098*
N1	0.5327 (2)	1.33013 (19)	0.91361 (19)	0.0483 (5)
H2C	0.776 (3)	1.084 (3)	0.835 (3)	0.061 (7)*
H2D	0.789 (3)	1.232 (3)	0.986 (3)	0.076 (8)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C8	0.0538 (14)	0.0579 (14)	0.113 (2)	0.0029 (12)	0.0172 (14)	0.0500 (15)
C7	0.0329 (10)	0.0476 (12)	0.0584 (13)	-0.0013 (9)	0.0116 (9)	0.0252 (10)
O3	0.0339 (7)	0.0396 (8)	0.0685 (9)	0.0052 (6)	0.0167 (7)	0.0264 (7)
C6	0.0348 (10)	0.0372 (10)	0.0377 (10)	0.0091 (8)	0.0113 (8)	0.0119 (8)
N4	0.0307 (8)	0.0338 (8)	0.0539 (10)	0.0079 (6)	0.0134 (7)	0.0191 (7)
C3	0.0327 (10)	0.0316 (10)	0.0541 (12)	0.0074 (8)	0.0106 (9)	0.0176 (9)
C2	0.0378 (11)	0.0360 (10)	0.0500 (12)	0.0067 (8)	0.0149 (9)	0.0077 (9)
N3	0.0399 (9)	0.0358 (9)	0.0411 (9)	0.0045 (7)	0.0144 (7)	0.0153 (7)
C1	0.0357 (10)	0.0344 (10)	0.0457 (11)	0.0046 (8)	0.0150 (9)	0.0173 (9)
C10	0.0646 (15)	0.0950 (19)	0.0633 (15)	0.0070 (14)	0.0278 (13)	0.0369 (14)
O2	0.0332 (8)	0.0476 (8)	0.0696 (10)	0.0143 (6)	0.0153 (7)	0.0214 (7)
N2	0.0422 (10)	0.0483 (11)	0.0554 (12)	0.0131 (9)	0.0093 (9)	0.0125 (9)
C5	0.0481 (12)	0.0424 (11)	0.0436 (11)	0.0037 (9)	0.0060 (9)	0.0225 (9)
C4	0.0390 (11)	0.0326 (10)	0.0571 (12)	0.0106 (8)	0.0091 (9)	0.0182 (9)
C9	0.0512 (13)	0.0536 (13)	0.0695 (16)	-0.0039 (11)	0.0003 (12)	0.0164 (12)
O1	0.0646 (10)	0.0469 (9)	0.0624 (10)	0.0096 (8)	-0.0060 (8)	0.0004 (7)
N1	0.0436 (10)	0.0355 (9)	0.0547 (11)	0.0041 (8)	0.0012 (8)	0.0073 (8)

*Geometric parameters (Å, °)*

C8—C7	1.517 (3)	N3—C5	1.457 (2)
C8—H8A	0.96	C1—N1	1.292 (2)
C8—H8B	0.96	C1—N2	1.356 (3)
C8—H8C	0.96	C10—H10A	0.96
C7—O3	1.484 (2)	C10—H10B	0.96
C7—C9	1.506 (3)	C10—H10C	0.96
C7—C10	1.515 (3)	N2—H2C	0.89 (3)
O3—C6	1.343 (2)	N2—H2D	0.94 (3)
C6—O2	1.216 (2)	C5—C4	1.522 (3)
C6—N4	1.358 (2)	C5—H5A	0.97
N4—C3	1.465 (2)	C5—H5B	0.97
N4—C4	1.470 (2)	C4—H4A	0.97
C3—C2	1.514 (3)	C4—H4B	0.97
C3—H3A	0.97	C9—H9A	0.96
C3—H3B	0.97	C9—H9B	0.96
C2—N3	1.473 (2)	C9—H9C	0.96
C2—H2A	0.97	O1—N1	1.447 (2)
C2—H2B	0.97	O1—H1	0.82
N3—C1	1.398 (2)		
C7—C8—H8A	109.5	C5—N3—C2	108.79 (15)
C7—C8—H8B	109.5	N1—C1—N2	123.51 (19)
H8A—C8—H8B	109.5	N1—C1—N3	119.03 (17)
C7—C8—H8C	109.5	N2—C1—N3	117.45 (17)
H8A—C8—H8C	109.5	C7—C10—H10A	109.5
H8B—C8—H8C	109.5	C7—C10—H10B	109.5
O3—C7—C9	110.52 (16)	H10A—C10—H10B	109.5
O3—C7—C10	109.00 (17)	C7—C10—H10C	109.5
C9—C7—C10	112.76 (19)	H10A—C10—H10C	109.5
O3—C7—C8	101.95 (15)	H10B—C10—H10C	109.5
C9—C7—C8	110.19 (18)	C1—N2—H2C	119.9 (15)
C10—C7—C8	111.90 (18)	C1—N2—H2D	112.7 (15)
C6—O3—C7	121.19 (14)	H2C—N2—H2D	120 (2)
O2—C6—O3	124.82 (16)	N3—C5—C4	113.42 (14)
O2—C6—N4	123.46 (17)	N3—C5—H5A	108.9
O3—C6—N4	111.70 (15)	C4—C5—H5A	108.9
C6—N4—C3	123.10 (15)	N3—C5—H5B	108.9
C6—N4—C4	117.64 (15)	C4—C5—H5B	108.9
C3—N4—C4	115.06 (15)	H5A—C5—H5B	107.7
N4—C3—C2	110.37 (15)	N4—C4—C5	110.69 (15)
N4—C3—H3A	109.6	N4—C4—H4A	109.5
C2—C3—H3A	109.6	C5—C4—H4A	109.5
N4—C3—H3B	109.6	N4—C4—H4B	109.5
C2—C3—H3B	109.6	C5—C4—H4B	109.5
H3A—C3—H3B	108.1	H4A—C4—H4B	108.1
N3—C2—C3	111.33 (14)	C7—C9—H9A	109.5

N3—C2—H2A	109.4	C7—C9—H9B	109.5
C3—C2—H2A	109.4	H9A—C9—H9B	109.5
N3—C2—H2B	109.4	C7—C9—H9C	109.5
C3—C2—H2B	109.4	H9A—C9—H9C	109.5
H2A—C2—H2B	108	H9B—C9—H9C	109.5
C1—N3—C5	117.38 (15)	N1—O1—H1	109.5
C1—N3—C2	116.57 (15)	C1—N1—O1	109.58 (16)
C9—C7—O3—C6	60.5 (2)	C3—C2—N3—C5	-59.90 (19)
C10—C7—O3—C6	-64.0 (2)	C5—N3—C1—N1	-3.9 (2)
C8—C7—O3—C6	177.58 (17)	C2—N3—C1—N1	-135.54 (18)
C7—O3—C6—O2	-1.7 (3)	C5—N3—C1—N2	175.79 (15)
C7—O3—C6—N4	179.86 (15)	C2—N3—C1—N2	44.1 (2)
O2—C6—N4—C3	165.07 (18)	C1—N3—C5—C4	-77.5 (2)
O3—C6—N4—C3	-16.5 (2)	C2—N3—C5—C4	57.6 (2)
O2—C6—N4—C4	9.3 (3)	C6—N4—C4—C5	-154.43 (16)
O3—C6—N4—C4	-172.28 (15)	C3—N4—C4—C5	47.9 (2)
C6—N4—C3—C2	152.67 (16)	N3—C5—C4—N4	-51.3 (2)
C4—N4—C3—C2	-51.0 (2)	N2—C1—N1—O1	4.7 (3)
N4—C3—C2—N3	56.6 (2)	N3—C1—N1—O1	-175.65 (15)
C3—C2—N3—C1	75.6 (2)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N2—H2 <i>D</i> $\cdots$ O1	0.94 (3)	2.08 (3)	2.538 (2)	108.3 (19)
N2—H2 <i>C</i> $\cdots$ O2 <sup>i</sup>	0.89 (3)	2.10 (3)	2.988 (2)	173 (3)
O1—H1 $\cdots$ N1 <sup>ii</sup>	0.82	2.04	2.764 (3)	147

Symmetry codes: (i)  $x+1, y, z$ ; (ii)  $-x+1, -y+3, -z+2$ .