

## 3,12-Diaza-6,9-diazonia-2,13-dioxotetra-decane bis(perchlorate)

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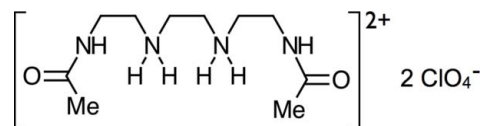
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Key indicators: single-crystal X-ray study;  $T = 150$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.039;  $wR$  factor = 0.087; data-to-parameter ratio = 17.6.

The crystal structure of the title diprotonated diacetyltriethylenetetramine (DAT) perchlorate salt,  $\text{C}_{10}\text{H}_{24}\text{N}_4\text{O}_2^{2+} \cdot 2\text{ClO}_4^-$ , can be described as a three-dimensional assembly of alternating layers consisting of diprotonated diacetyltriethylenetetramine ( $\text{H}_2\text{DAT}$ )<sup>2+</sup> strands along [100] and the anionic species  $\text{ClO}_4^-$ . The ( $\text{H}_2\text{DAT}$ )<sup>2+</sup> cations in the strands are connected *via*  $\text{N}-\text{H} \cdots \text{O}$  hydrogen bonding between the acetyl groups and the amine groups of neighbouring ( $\text{H}_2\text{DAT}$ )<sup>2+</sup> cations. Layers of ( $\text{H}_2\text{DAT}$ )<sup>2+</sup> strands and perchlorate anions are connected by a network of hydrogen bonds between the NH and  $\text{NH}_2$  groups and the O atoms of the perchlorate anion. The asymmetric unit consists of one perchlorate anion in a general position, as well as of one cation that is located on a center of inversion.

### Related literature

For background to pharmaceutical chelating agents in the treatment of diabetes, see: Cooper *et al.* (2004); Gong *et al.* (2006, 2008); Jüllig *et al.* (2007); Lu *et al.* (2010). For the detection of a new group of TETA metabolites, see: Lu *et al.* (2007). For the preparation and characterization of DAT mono- and dihydrochloride salts, see: Jonas *et al.* (2006); Wichmann *et al.* (2011). For related structures, see: Elaoud *et al.* (1999); Fu *et al.* (2005); Ilioudis *et al.* (2000, 2002); Ilioudis & Steed (2003); Wichmann *et al.* (2007).



### Experimental

#### Crystal data

$\text{C}_{10}\text{H}_{24}\text{N}_4\text{O}_2^{2+} \cdot 2\text{ClO}_4^-$   
 $M_r = 431.23$   
 Monoclinic,  $P2_1/c$   
 $a = 6.0888$  (5) Å  
 $b = 10.9415$  (9) Å  
 $c = 14.8160$  (11) Å  
 $\beta = 110.846$  (6)°

$V = 922.44$  (13) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.41$  mm<sup>-1</sup>  
 $T = 150$  K  
 $0.45 \times 0.35 \times 0.17$  mm

#### Data collection

Stoe IPDS II diffractometer  
 Absorption correction: numerical  
 ( $X\text{-RED32}$ ; Stoe & Cie, 2001)  
 $T_{\min} = 0.837$ ,  $T_{\max} = 0.936$

11528 measured reflections  
 2113 independent reflections  
 1624 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.114$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.087$   
 $S = 1.03$   
 2113 reflections

120 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.42$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N3}-\text{H3} \cdots \text{O4}$	0.88	2.12	2.989 (2)	167
$\text{N6}-\text{H6A} \cdots \text{O1}^i$	0.92	1.77	2.6745 (19)	168
$\text{N6}-\text{H6B} \cdots \text{O2}^{ii}$	0.92	2.13	2.9265 (17)	145
$\text{N6}-\text{H6B} \cdots \text{O5}^{ii}$	0.92	2.40	3.2141 (19)	147

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

Data collection: *X-Area* (Stoe & Cie, 2002); cell refinement: *X-Area*; data reduction: *X-Area*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *VESTA* (Momma & Izumi, 2011); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2261).

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## supporting information

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## 3,12-Diaza-6,9-diazonia-2,13-dioxotetradecane bis(perchlorate)

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### S1. Comment

As part of a larger project focusing on TETA and its metabolites as pharmaceutical chelating agents in Diabetes treatment (Cooper *et al.*, 2004, Gong *et al.* 2006, 2008, Jüllig *et al.* 2007, Lu *et al.* 2010), we previously published the detection of a new group of TETA metabolites, *N*<sup>1</sup>-Monoacetyltriethylenetetramine (MAT) and the *N*<sup>1</sup>, *N*<sup>10</sup>-Diacetyltriethylenetetramine (DAT) (Lu *et al.* 2007), as well as just recently the development of a new selective synthetic route and the characterization of the DAT mono- and dihydrochloride salts (Wichmann *et al.*, 2011).

TETA and its metabolites belong into the polyamine family, ambivalent and multidentate ligands, which are well known for their ability to form a variety of interesting open-chain, macrocyclic and three-dimensional architectures. TETA salts exist in variable protonation states with different anionic species (Ilioudis, *et al.* 2000, 2002, 2003, Elaoud *et al.* 1999, Fu *et al.* 2005, Wichmann *et al.* 2007). Therefore, we investigated the metabolite forms MAT and DAT towards their protonation and complexation behaviour (Wichmann *et al.*, 2011). The obtained crystal structure of the new DAT salt [(H<sub>2</sub>DAT) \* 2 ClO<sub>4</sub>] is described in this paper.

The (H<sub>2</sub>DAT)<sup>2+</sup> cations are arranged as a linear symmetric chain with the terminal NH—CO—CH<sub>3</sub> groups in *trans*-position to each other (Fig. 1).

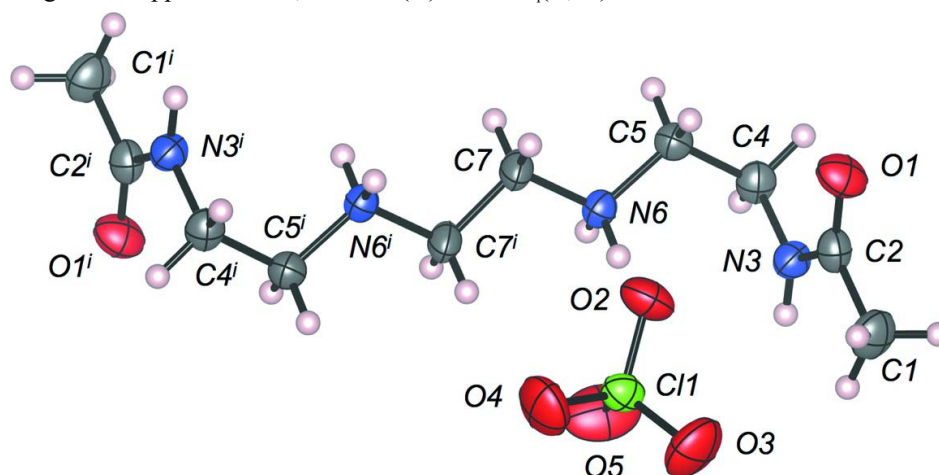
The crystal structure consists of a three-dimensional-network, containing alternating assembly of two-dimensional-layers of (H<sub>2</sub>DAT)<sup>2+</sup> cations (Fig. 2) and the ClO<sub>4</sub><sup>-</sup> anions. The (H<sub>2</sub>DAT)<sup>2+</sup> cations form linear strands along [100] (Fig. 3), connected *via* hydrogen bonding between the acetyl groups and the amine groups of neighbouring (H<sub>2</sub>DAT)<sup>2+</sup> cations, with a C2=O1 ... H6A/N6 distance of 1.767 (1) Å. These linear strands of the (H<sub>2</sub>DAT)<sup>2+</sup> cations form two-dimensional-layers in the (001) plane. However, the two-dimensional-layers of (H<sub>2</sub>DAT)<sup>2+</sup> cations and the perchlorate anions were stabilized by a network of intermolecular hydrogen bonds between the NH- and NH<sub>2</sub>-groups and the oxygen atoms of the perchlorate anion, with N6—H6B...O2—O4—O4...H3—N3 between 2.126 (1) Å and 2.125 (1) Å, (Table 1). The terminal NH-groups of a (H<sub>2</sub>DAT)<sup>2+</sup> cation binds to an O-atom of a perchlorate anion, which itself bound to an internal NH-group of another (H<sub>2</sub>DAT)<sup>2+</sup> cation and *vice versa*. Therefore each (H<sub>2</sub>DAT)<sup>2+</sup> cation is connected to four different (H<sub>2</sub>DAT)<sup>2+</sup> cations, two from the above and two from the below layer.

### S2. Experimental

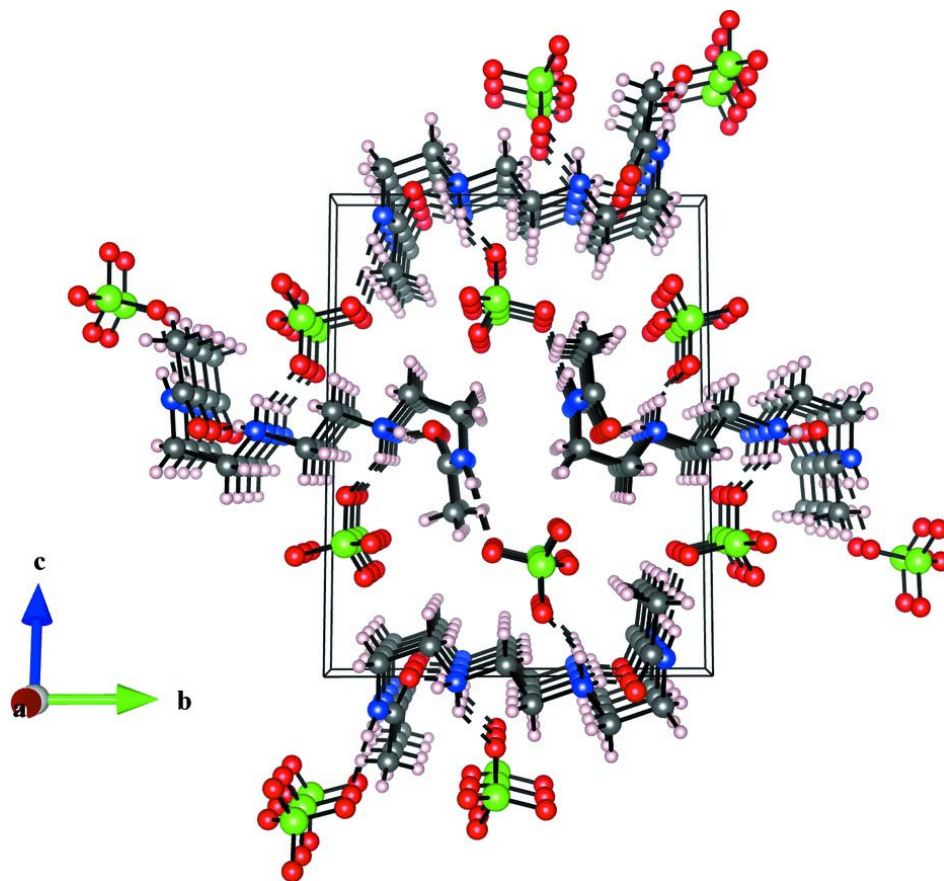
The DAT \* 2 HCl powder material was synthesized by CarboGen, Switzerland according to literature procedure (Jonas *et al.* 2006, Wichmann *et al.* 2011). Cu(ClO<sub>4</sub>)<sub>2</sub> is commercially available and was used as received. Crystals of the title compound were grown by slow evaporation of an aqueous solution of DAT \* 2 HCl and Cu(ClO<sub>4</sub>)<sub>2</sub> in stoichiometric ratio in water over a period of 6 weeks.

**S3. Refinement**

H atoms bonded to C and N atoms were positioned geometrically (C—H = 0.98–0.99 Å, N—H = 0.88–0.92 Å) and refined using a riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C}, \text{N})$ .

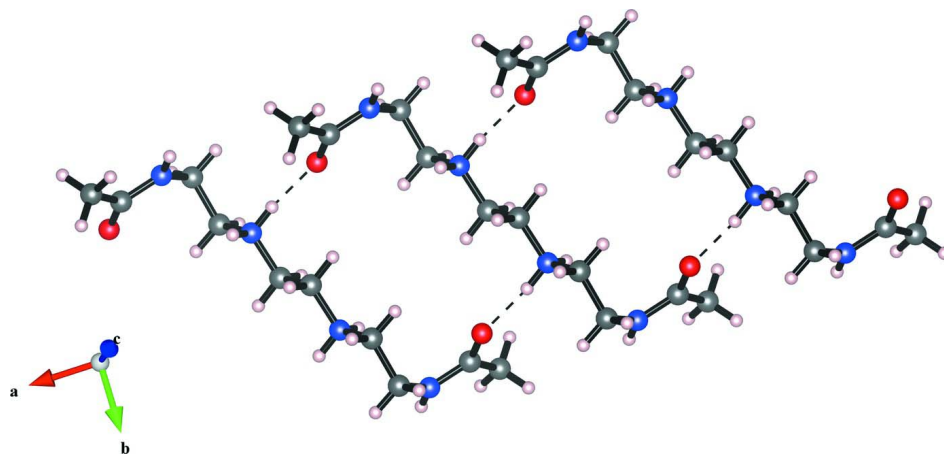
**Figure 1**

Crystal structure of the title compound with labeling and displacement ellipsoids drawn at the 75% probability level. [symmetry code: (i)  $-x, -y - z + 1$ ].



**Figure 2**

Crystal structure of the title compound with view along the *a* axis. Hydrogen bonding interactions are shown as dashed lines.



**Figure 3**

The strands of  $(\text{H}_2\text{DAT})^{2+}$  cations viewed along the *a* axis. The dashed bonds indicate the hydrogen bonds.

**3,12-Diaza-6,9-diazonia-2,13-dioxotetradecane bis(perchlorate)***Crystal data*C<sub>10</sub>H<sub>24</sub>N<sub>4</sub>O<sub>2</sub><sup>2+</sup>·2ClO<sub>4</sub><sup>-</sup> $M_r = 431.23$ Monoclinic,  $P2_1/c$ 

Hall symbol: -P 2ybc

 $a = 6.0888$  (5) Å $b = 10.9415$  (9) Å $c = 14.8160$  (11) Å $\beta = 110.846$  (6)° $V = 922.44$  (13) Å<sup>3</sup> $Z = 2$  $F(000) = 452$  $D_x = 1.553$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 12054 reflections

 $\theta = 4.7$ – $59.0$ ° $\mu = 0.41$  mm<sup>-1</sup> $T = 150$  K

Not regular, colourless

 $0.45 \times 0.35 \times 0.17$  mm*Data collection*

Stoe IPDS II

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Image plate detector scans

Absorption correction: numerical

 $(X\text{-RED32; Stoe \& Cie, 2001})$  $T_{\min} = 0.837$ ,  $T_{\max} = 0.936$ 

11528 measured reflections

2113 independent reflections

1624 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.114$  $\theta_{\max} = 27.5$ °,  $\theta_{\min} = 2.4$ ° $h = -7 \rightarrow 6$  $k = -14 \rightarrow 14$  $l = -19 \rightarrow 19$ *Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.039$  $wR(F^2) = 0.087$  $S = 1.03$ 

2113 reflections

120 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

 $w = 1/[\sigma^2(F_o^2) + (0.0435P)^2 + 0.0412P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 0.38$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.42$  e Å<sup>-3</sup>Extinction correction: *SHELXL97* (Sheldrick,2008),  $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$ 

Extinction coefficient: 0.0082 (19)

*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 (Å<sup>2</sup>)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.7492 (2)	-0.27758 (14)	0.49858 (8)	0.0292 (3)
N3	0.4849 (2)	-0.35281 (14)	0.55667 (8)	0.0203 (3)
H3	0.4552	-0.3783	0.6075	0.030*

N6	0.1286 (2)	-0.15588 (13)	0.49690 (8)	0.0180 (3)
H6A	0.0102	-0.2008	0.5058	0.027*
H6B	0.2493	-0.1497	0.5556	0.027*
C1	0.8854 (3)	-0.3315 (2)	0.66635 (12)	0.0306 (4)
H1A	0.9869	-0.4013	0.6669	0.046*
H1B	0.8082	-0.3455	0.7133	0.046*
H1C	0.9805	-0.2570	0.6835	0.046*
C2	0.7025 (3)	-0.31761 (17)	0.56731 (10)	0.0212 (3)
C4	0.2944 (3)	-0.35009 (17)	0.46258 (10)	0.0218 (4)
H4A	0.1589	-0.3965	0.4668	0.033*
H4B	0.3476	-0.3914	0.4145	0.033*
C5	0.2149 (3)	-0.22157 (17)	0.42777 (10)	0.0211 (3)
H5A	0.3478	-0.1757	0.4205	0.032*
H5B	0.0875	-0.2255	0.3637	0.032*
C7	0.0392 (3)	-0.03159 (17)	0.46280 (11)	0.0230 (4)
H7A	-0.0947	-0.0376	0.4008	0.035*
H7B	0.1647	0.0169	0.4518	0.035*
Cl1	0.43802 (6)	-0.55724 (4)	0.77065 (2)	0.02264 (14)
O2	0.4296 (2)	-0.55967 (14)	0.86654 (8)	0.0337 (3)
O3	0.2285 (3)	-0.60503 (17)	0.70295 (10)	0.0478 (4)
O4	0.4650 (3)	-0.43253 (15)	0.74660 (10)	0.0461 (4)
O5	0.6382 (3)	-0.62675 (18)	0.77258 (10)	0.0507 (5)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0253 (6)	0.0377 (9)	0.0282 (5)	-0.0049 (6)	0.0139 (5)	-0.0020 (5)
N3	0.0192 (6)	0.0211 (8)	0.0200 (6)	0.0012 (6)	0.0062 (5)	0.0024 (5)
N6	0.0164 (6)	0.0183 (7)	0.0180 (5)	0.0012 (6)	0.0044 (4)	0.0009 (5)
C1	0.0222 (8)	0.0346 (12)	0.0290 (8)	0.0027 (8)	0.0020 (6)	0.0014 (8)
C2	0.0204 (7)	0.0180 (9)	0.0251 (7)	0.0018 (7)	0.0081 (6)	-0.0024 (6)
C4	0.0192 (7)	0.0211 (9)	0.0226 (7)	-0.0001 (7)	0.0041 (5)	-0.0031 (6)
C5	0.0214 (7)	0.0231 (9)	0.0185 (6)	0.0032 (7)	0.0069 (5)	0.0001 (6)
C7	0.0251 (8)	0.0201 (9)	0.0265 (7)	0.0083 (7)	0.0124 (6)	0.0060 (6)
Cl1	0.0200 (2)	0.0276 (2)	0.01913 (18)	0.00163 (17)	0.00557 (13)	0.00246 (15)
O2	0.0382 (7)	0.0424 (9)	0.0237 (6)	0.0036 (7)	0.0150 (5)	0.0075 (5)
O3	0.0355 (8)	0.0518 (11)	0.0400 (7)	-0.0072 (8)	-0.0063 (6)	-0.0065 (7)
O4	0.0564 (9)	0.0378 (10)	0.0441 (7)	-0.0072 (8)	0.0180 (7)	0.0166 (7)
O5	0.0381 (8)	0.0661 (13)	0.0463 (8)	0.0256 (8)	0.0130 (6)	-0.0092 (8)

*Geometric parameters (Å, °)*

O1—C2	1.231 (2)	C4—C5	1.517 (2)
N3—C2	1.334 (2)	C4—H4A	0.9900
N3—C4	1.4622 (17)	C4—H4B	0.9900
N3—H3	0.8800	C5—H5A	0.9900
N6—C7	1.485 (2)	C5—H5B	0.9900
N6—C5	1.492 (2)	C7—C7 <sup>i</sup>	1.515 (3)

N6—H6A	0.9200	C7—H7A	0.9900
N6—H6B	0.9200	C7—H7B	0.9900
C1—C2	1.501 (2)	C11—O3	1.4129 (13)
C1—H1A	0.9800	C11—O5	1.4284 (15)
C1—H1B	0.9800	C11—O4	1.4344 (16)
C1—H1C	0.9800	C11—O2	1.4401 (12)
C2—N3—C4	121.60 (13)	N3—C4—H4B	109.0
C2—N3—H3	119.2	C5—C4—H4B	109.0
C4—N3—H3	119.2	H4A—C4—H4B	107.8
C7—N6—C5	112.56 (12)	N6—C5—C4	111.17 (13)
C7—N6—H6A	109.1	N6—C5—H5A	109.4
C5—N6—H6A	109.1	C4—C5—H5A	109.4
C7—N6—H6B	109.1	N6—C5—H5B	109.4
C5—N6—H6B	109.1	C4—C5—H5B	109.4
H6A—N6—H6B	107.8	H5A—C5—H5B	108.0
C2—C1—H1A	109.5	N6—C7—C7 <sup>i</sup>	110.03 (16)
C2—C1—H1B	109.5	N6—C7—H7A	109.7
H1A—C1—H1B	109.5	C7 <sup>i</sup> —C7—H7A	109.7
C2—C1—H1C	109.5	N6—C7—H7B	109.7
H1A—C1—H1C	109.5	C7 <sup>i</sup> —C7—H7B	109.7
H1B—C1—H1C	109.5	H7A—C7—H7B	108.2
O1—C2—N3	121.11 (13)	O3—C11—O5	111.45 (11)
O1—C2—C1	122.39 (15)	O3—C11—O4	109.27 (10)
N3—C2—C1	116.49 (14)	O5—C11—O4	109.82 (11)
N3—C4—C5	113.08 (13)	O3—C11—O2	110.69 (9)
N3—C4—H4A	109.0	O5—C11—O2	107.47 (8)
C5—C4—H4A	109.0	O4—C11—O2	108.06 (10)

Symmetry code: (i)  $-x, -y, -z+1$ .

*Hydrogen-bond geometry* ( $\text{\AA}$ ,  $^\circ$ )

<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>
N3—H3 $\cdots$ O4	0.88	2.12	2.989 (2)	167
N6—H6A $\cdots$ O1 <sup>ii</sup>	0.92	1.77	2.6745 (19)	168
N6—H6B $\cdots$ O2 <sup>iii</sup>	0.92	2.13	2.9265 (17)	145
N6—H6B $\cdots$ O5 <sup>iii</sup>	0.92	2.40	3.2141 (19)	147

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