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1,4,10,13-Tetraoxa-7,16-diazoniacyclooctadecane bis[tetrachloridoaurate(III)] dihydrate

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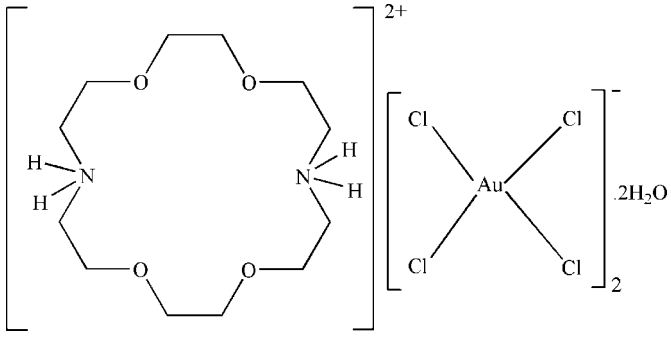
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.006 Å; R factor = 0.018; wR factor = 0.046; data-to-parameter ratio = 16.6.

The asymmetric unit of the title compound, $(C_{12}H_{28}N_2O_4)[AuCl_4]_2\cdot 2H_2O$, contains one half-cation, one anion and one water molecule; the cation is centrosymmetric. The Au ion has a square-planar coordination. In the crystal structure, intramolecular N—H···O and O—H···O, and intermolecular N—H···O, O—H···Cl and N—H···Cl hydrogen bonds link the ions and water molecules, forming a supramolecular structure.

Related literature

For related literature, see: Calleja *et al.* (2001); Chekhlov (2000, 2001, 2005); Chekhlov & Martynov (1998); Chekhlov *et al.* (1994); Fonari *et al.* (2004); Hasan *et al.* (1999); Johnson & Steed (1998); Moers *et al.* (2000); Simonov *et al.* (2003); Yap *et al.* (1995); Yousefi, Amani & Khavasi (2007); Yousefi, Teimouri *et al.* (2007); Zhang *et al.* (2006).



Experimental

Crystal data

$(C_{12}H_{28}N_2O_4)[AuCl_4]_2\cdot 2H_2O$
M_r = 977.94
Triclinic, $P\overline{1}$
a = 8.0168 (10) Å
b = 8.3359 (9) Å
c = 11.2989 (15) Å
α = 73.063 (11)°
β = 75.965 (10)°
γ = 74.929 (9)°
V = 686.02 (15) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 11.49 mm^{-1}
T = 120 (2) K
0.32 × 0.22 × 0.20 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED*; Stoe & Cie, 2005)
T_{min} = 0.065, T_{max} = 0.108
4188 measured reflections
2390 independent reflections
2381 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.045
S = 1.18
2390 reflections
144 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.67 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—Au1	2.2796 (11)	Cl3—Au1	2.2912 (11)
Cl2—Au1	2.2877 (10)	Cl4—Au1	2.2751 (11)
Cl4—Au1—Cl1	90.20 (4)	Cl4—Au1—Cl3	90.30 (4)
Cl4—Au1—Cl2	176.52 (4)	Cl1—Au1—Cl3	176.79 (3)
Cl1—Au1—Cl2	89.96 (4)	Cl2—Au1—Cl3	89.74 (4)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*C*···O1^{i}	0.90	2.49	2.791 (5)	100
N1—H1*C*···O3	0.90	1.98	2.844 (3)	160
N1—H1*D*···Cl1^{ii}	0.90	2.81	3.540 (4)	139
N1—H1*D*···Cl2^{ii}	0.90	2.49	3.262 (3)	143
O3—H3*C*···O1	0.76 (6)	2.14 (6)	2.858 (4)	158 (6)
O3—H3*C*···O2	0.76 (6)	2.51 (6)	3.057 (3)	130 (5)
O3—H3*D*···Cl3^{iii}	0.81 (7)	2.59 (6)	3.378 (4)	167.00

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2465).

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1,4,10,13-Tetraoxa-7,16-diazoniacyclooctadecane bis[tetrachloridoaurate(III)] dihydrate

Leila Hojjat Kashani, Mohammad Yousefi, Vahid Amani and Hamid Reza Khavasi

S1. Comment

Recently, we reported the synthesis and crystal structure of the [(H_2DA18C6)Cl_2], (II), (Yousefi, Amani & Khavasi, 2007) and [(H_2DA18C6)][$PtCl_6$].2H_2O, (III), (Yousefi, Teimouri *et al.*, 2007) [where H_2DA18C6 is 1,10-Diazonia-18-crown-6]. Several proton transfer systems using 1,10-diaza-18-crown-6, with proton donor molecules, such as [(H_2DA18C6)I_2.2H_2O], (IV), (Chekhlov, 2005), [(H_2DA18C6)(C_2HO_4)$_2$], (V), and [(H_2DA18C6)$_2$(C_2O_4)$_2$.2H_2O], (VI), (Chekhlov, 2000), [(H_2DA18C6)(picrate)$_2$], (VII), (Chekhlov, 2001), [(H_2DA18C6)(HPTD)$_2$], (VIII), (Simonov *et al.*, 2003), [(H_2DA18C6)(PD)$_2$.(H_2O)$_4$], (IX), and [(H_2DA18C6)(PS)$_2$.(H_2O)$_2$], (*X*), (Fonari *et al.*, 2004), [(H_2DA18C6)(CCl_3COO)$_2$(CCl_3COOH)$_2$], (XI), (Chekhlov *et al.*, 1994), [(H_2DA18C6)(CCl_3COO)$_2$], (XII), (Chekhlov & Martynov, 1998), and {[H_2DA18C6][($ArSO_2$)$_2$N]$_2$}, (XIII), (Moers *et al.*, 2000) [where C_2O_4 is oxalate, HPTD is (4*Z*,5E)-pyrimidine-2,4,5,6(1*H*,3H) -tetraone 4,5-dioxime anion, PD is 2-(2-methylphenyl)-2*H*-[1,2,3]- triazolo[4,5-*d*]pyrimidine-5,7(4*H*,6H)-dione 3-oxide anion, PS is 6-amino-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-ylsulfamate and ($ArSO_2$)$_2$N is bis(4-chlorobenzenesulfonyl)imide] have been synthesized and characterized by single-crystal X-ray diffraction methods.

There are also several proton transfer systems using $HAuCl_4$ with proton acceptor molecules, such as [EMI][$AuCl_4$] (XIV) and [BMI]$_2$[$AuCl_4$].2H_2O, (XV), (Hasan *et al.*, 1999), [H_2bipy][$AuCl_4$][Cl], (XVI), (Zhang *et al.*, 2006), [H_7O_3][15-crown-5][$AuCl_4$], (XVII) and [H_5O_2][benzo-15-crown-5] $_2$[$AuCl_4$], (XVIII), (Johnson & Steed, 1998), [H_5O_2]$_2$[12-crown-4]$_2$ [$AuCl_4$]$_2$, (XIX), [H_3O][18-crown-6][$AuCl_4$], (XX) and [H_3O] [4-nitrobenzo-18-crown-6][$AuCl_4$], (XXI), (Calleja *et al.*, 2001) and [DPpy.H][$AuCl_4$], (XXII), (Yap *et al.*, 1995) [where EMI is 1-ethyl-3-methylimidazolium, BMI is 1-butyl-3-methylimidazolium, H_2bipy is 2,2′-bipyridinium and DPpy.H is 2,6-Diphenylpyridinium] have been synthesized and characterized by single-crystal X-ray diffraction methods. We report herein the synthesis and crystal structure of the title compound, (I).

The asymmetric unit of (I), (Fig. 1) contains one half-cation, one anion and one water molecule; the cation is centrosymmetric. The Au ion has a square-planar coordination (Table 1). The bond lengths and angles, in cation, are in good agreement with the corresponding values in (II), (III) and (IV). Also, the Au-Cl bond lengths and angles (Table 1) are within normal range [XXII].

In the crystal structure, intramolecular N-H···O and O-H···O and intermolecular N-H···O, O-H···Cl and N-H···Cl hydrogen bonds (Table 2) link the molecules to form a supramolecular structure (Fig. 2), in which they may be effective in the stabilization of the structure.

S2. Experimental

For the preparation of the title compound, (I), a solution of 1,10-diaza-18 -crown-6 (0.10 g, 0.37 mmol) in EtOH (20 ml) was added to a solution of $HAuCl_4.3H_2O$, (0.29 g, 0.74 mmol) in water (30 ml) and the resulting yellow solution was stirred for 10 min at 313 K. Then, it was left to evaporate slowly at room temperature. After one week, yellow prismatic crystals of (I) were isolated (yield; 0.26 g; 72.0%).

S3. Refinement

H atoms (for H_2O) were located in a difference syntheses and refined [O-H = 0.71 (6) and 0.76 (6) Å; U_{iso}(H) = 0.019 (15) and 0.034 (17) Å^2]. The remaining H atoms were positioned geometrically, with N-H = 0.90 Å (for NH_2) and C-H = 0.97 Å for methylene H and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C,N).

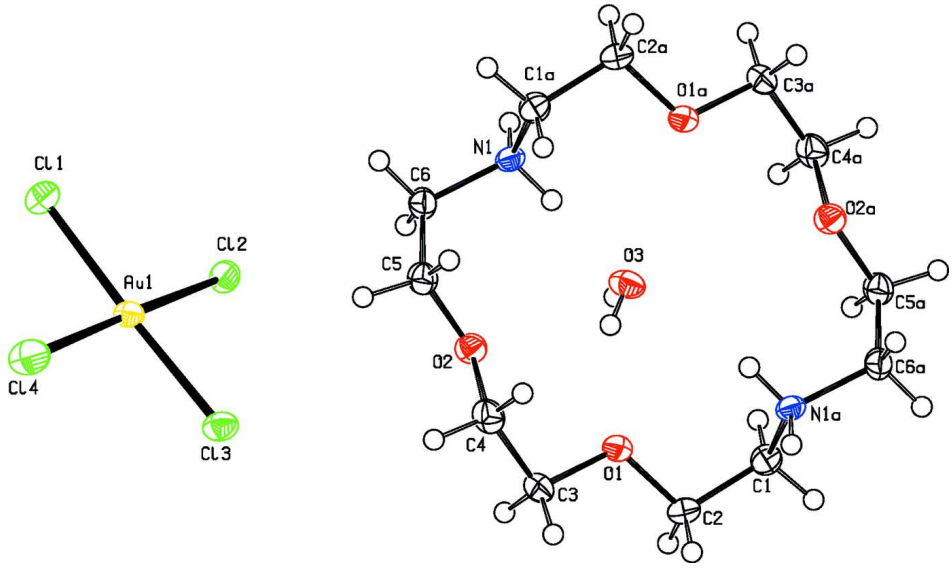

Figure 1

The asymmetric unit of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level [symmetry code: (a) 1 - x, 1 - y, -z].

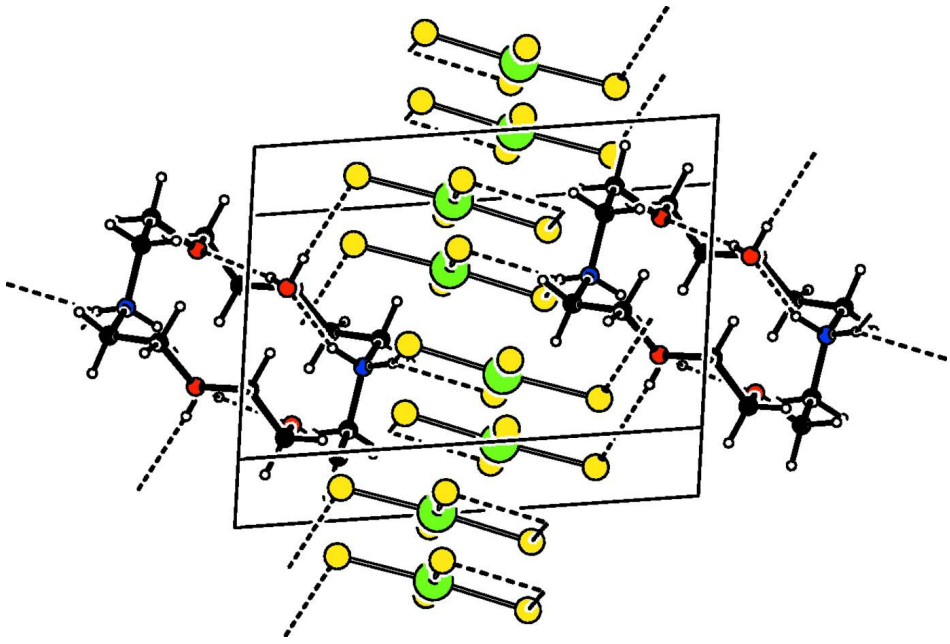

Figure 2

A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

1,4,10,13-Tetraoxa-7,16-diazoniacyclooctadecane bis[tetrachloridoaurate(III)] dihydrate

Crystal data

$(C_{12}H_{28}N_2O_4)[AuCl_4]_2\cdot 2H_2O$
$M_r = 977.94$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.0168\ (10)$ Å
$b = 8.3359\ (9)$ Å
$c = 11.2989\ (15)$ Å
$\alpha = 73.063\ (11)°$
$\beta = 75.965\ (10)°$
$\gamma = 74.929\ (9)°$
$V = 686.02\ (15)$ Å^3
$Z = 1$
$F(000) = 460$
$D_x = 2.367$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1139 reflections
$\theta = 1.9–25.2°$
$\mu = 11.49$ mm^{-1}
$T = 120$ K
Block, yellow
0.32 × 0.22 × 0.20 mm

Data collection

Stoe IPDSII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0.15 mm pixels mm^{-1}
rotation method scans
Absorption correction: numerical shape of crystal determined optically
$T_{min} = 0.065$, $T_{max} = 0.108$
4188 measured reflections
2390 independent reflections
2381 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.2°$, $\theta_{min} = 1.9°$
$h = -9\rightarrow 8$
$k = -9\rightarrow 8$
$l = -12\rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.017$
$wR(F^2) = 0.045$
$S = 1.18$
2390 reflections
144 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0216P)^2 + 1.1824P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.018$
$\Delta\rho_{max} = 0.59$ e Å^{-3}
$\Delta\rho_{min} = -0.67$ e Å^{-3}

Special details

Experimental. (X-SHAPE and X-RED; Stoe & Cie, 2005)

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 $F^2 > 2$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^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Au1	0.774200 (17)	0.949008 (17)	0.436516 (13)	0.01917 (8)
Cl1	0.65158 (14)	0.91699 (13)	0.64419 (10)	0.0271 (2)
Cl2	0.77516 (14)	0.66859 (13)	0.45325 (10)	0.0249 (2)
Cl3	0.88196 (14)	0.98407 (13)	0.22535 (10)	0.0257 (2)
Cl4	0.79013 (16)	1.22269 (14)	0.42240 (12)	0.0362 (3)
O1	0.8055 (4)	0.5576 (4)	−0.1123 (3)	0.0230 (6)
O2	0.6857 (4)	0.6728 (3)	0.1006 (3)	0.0227 (6)
O3	0.6136 (4)	0.3371 (5)	0.0862 (4)	0.0259 (7)
H3C	0.666 (7)	0.406 (7)	0.049 (5)	0.019 (15)*
H3D	0.689 (8)	0.264 (8)	0.118 (5)	0.034 (17)*
N1	0.3824 (4)	0.5189 (4)	0.2598 (3)	0.0193 (7)
H1C	0.4424	0.4823	0.1909	0.023*
H1D	0.3672	0.4259	0.3230	0.023*
C1	0.7938 (5)	0.3778 (5)	−0.2343 (4)	0.0231 (9)
H1A	0.7787	0.2856	−0.1594	0.028*
H1B	0.8544	0.3272	−0.3043	0.028*
C2	0.9019 (5)	0.4874 (5)	−0.2162 (4)	0.0228 (9)
H2A	1.0152	0.4194	−0.1983	0.027*
H2B	0.9210	0.5783	−0.2913	0.027*
C3	0.8743 (5)	0.6921 (5)	−0.0984 (4)	0.0232 (9)
H3A	0.9062	0.7677	−0.1798	0.028*
H3B	0.9779	0.6447	−0.0597	0.028*
C4	0.7321 (6)	0.7883 (5)	−0.0161 (4)	0.0259 (9)

H4A	0.7733	0.8800	−0.0025	0.031*
H4B	0.6303	0.8385	−0.0566	0.031*
C5	0.5532 (5)	0.7528 (5)	0.1851 (4)	0.0242 (9)
H5A	0.4568	0.8215	0.1431	0.029*
H5B	0.5998	0.8274	0.2147	0.029*
C6	0.4895 (5)	0.6163 (5)	0.2940 (4)	0.0232 (9)
H6A	0.4190	0.6689	0.3610	0.028*
H6B	0.5900	0.5367	0.3257	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Au1	0.01758 (11)	0.01715 (11)	0.02133 (12)	−0.00433 (7)	−0.00015 (7)	−0.00455 (7)
Cl1	0.0296 (5)	0.0268 (5)	0.0227 (6)	−0.0041 (4)	−0.0003 (4)	−0.0077 (4)
Cl2	0.0334 (6)	0.0204 (5)	0.0217 (5)	−0.0106 (4)	−0.0006 (4)	−0.0055 (4)
Cl3	0.0271 (5)	0.0229 (5)	0.0236 (5)	−0.0074 (4)	0.0019 (4)	−0.0037 (4)
Cl4	0.0433 (7)	0.0205 (5)	0.0411 (7)	−0.0105 (5)	0.0085 (5)	−0.0118 (5)
O1	0.0223 (14)	0.0256 (15)	0.0229 (16)	−0.0110 (12)	0.0022 (12)	−0.0083 (12)
O2	0.0249 (15)	0.0196 (14)	0.0227 (16)	−0.0063 (12)	−0.0022 (12)	−0.0039 (12)
O3	0.0223 (17)	0.0229 (17)	0.0312 (19)	−0.0092 (16)	0.0008 (15)	−0.0049 (14)
N1	0.0197 (17)	0.0206 (17)	0.0162 (17)	−0.0061 (13)	0.0016 (14)	−0.0046 (14)
C1	0.020 (2)	0.023 (2)	0.027 (2)	−0.0009 (16)	−0.0022 (17)	−0.0108 (18)
C2	0.018 (2)	0.027 (2)	0.022 (2)	−0.0043 (17)	0.0019 (17)	−0.0077 (18)
C3	0.023 (2)	0.024 (2)	0.025 (2)	−0.0102 (17)	−0.0032 (17)	−0.0057 (18)
C4	0.032 (2)	0.019 (2)	0.027 (2)	−0.0099 (18)	−0.0062 (19)	−0.0021 (17)
C5	0.025 (2)	0.021 (2)	0.029 (2)	−0.0057 (17)	−0.0029 (18)	−0.0100 (18)
C6	0.022 (2)	0.026 (2)	0.026 (2)	−0.0046 (17)	−0.0044 (17)	−0.0123 (18)

Geometric parameters (Å, º)

Cl1—Au1	2.2796 (11)	C2—H2B	0.9700
Cl2—Au1	2.2877 (10)	C3—O1	1.432 (5)
Cl3—Au1	2.2912 (11)	C3—C4	1.500 (6)
Cl4—Au1	2.2751 (11)	C3—H3A	0.9700
O3—H3C	0.71 (6)	C3—H3B	0.9700
O3—H3D	0.76 (6)	C4—O2	1.419 (5)
N1—C1^{i}	1.496 (5)	C4—H4A	0.9700
N1—H1C	0.9000	C4—H4B	0.9700
N1—H1D	0.9000	C5—O2	1.412 (5)
C1—C2	1.495 (6)	C5—C6	1.501 (6)
C1—N1^{i}	1.496 (5)	C5—H5A	0.9700
C1—H1A	0.9700	C5—H5B	0.9700
C1—H1B	0.9700	C6—N1	1.501 (5)
C2—O1	1.429 (5)	C6—H6A	0.9700
C2—H2A	0.9700	C6—H6B	0.9700
Cl4—Au1—Cl1	90.20 (4)	H2A—C2—H2B	108.6
Cl4—Au1—Cl2	176.52 (4)	O1—C3—C4	106.8 (3)

Cl1—Au1—Cl2	89.96 (4)	O1—C3—H3A	110.4
Cl4—Au1—Cl3	90.30 (4)	C4—C3—H3A	110.4
Cl1—Au1—Cl3	176.79 (3)	O1—C3—H3B	110.4
Cl2—Au1—Cl3	89.74 (4)	C4—C3—H3B	110.4
C2—O1—C3	113.3 (3)	H3A—C3—H3B	108.6
C5—O2—C4	112.8 (3)	O2—C4—C3	108.8 (3)
H3C—O3—H3D	103 (6)	O2—C4—H4A	109.9
C1^{i}—N1—C6	113.5 (3)	C3—C4—H4A	109.9
C1^{i}—N1—H1C	108.9	O2—C4—H4B	109.9
C6—N1—H1C	108.9	C3—C4—H4B	109.9
C1^{i}—N1—H1D	108.9	H4A—C4—H4B	108.3
C6—N1—H1D	108.9	O2—C5—C6	108.5 (3)
H1C—N1—H1D	107.7	O2—C5—H5A	110.0
C2—C1—N1^{i}	110.8 (3)	C6—C5—H5A	110.0
C2—C1—H1A	109.5	O2—C5—H5B	110.0
N1^{i}—C1—H1A	109.5	C6—C5—H5B	110.0
C2—C1—H1B	109.5	H5A—C5—H5B	108.4
N1^{i}—C1—H1B	109.5	C5—C6—N1	112.9 (3)
H1A—C1—H1B	108.1	C5—C6—H6A	109.0
O1—C2—C1	106.6 (3)	N1—C6—H6A	109.0
O1—C2—H2A	110.4	C5—C6—H6B	109.0
C1—C2—H2A	110.4	N1—C6—H6B	109.0
O1—C2—H2B	110.4	H6A—C6—H6B	107.8
C1—C2—H2B	110.4		

N1^{i}—C1—C2—O1	59.1 (4)	C1—C2—O1—C3	−168.3 (3)
O1—C3—C4—O2	58.5 (4)	C4—C3—O1—C2	162.2 (3)
O2—C5—C6—N1	−71.6 (4)	C6—C5—O2—C4	169.1 (3)
C5—C6—N1—C1^{i}	−70.3 (4)	C3—C4—O2—C5	179.4 (3)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···O1^{i}	0.90	2.49	2.791 (5)	100
N1—H1*C*···O3	0.90	1.98	2.844 (3)	160
N1—H1*D*···Cl1^{ii}	0.90	2.81	3.540 (4)	139
N1—H1*D*···Cl2^{ii}	0.90	2.49	3.262 (3)	143
O3—H3*C*···O1	0.76 (6)	2.14 (6)	2.858 (4)	158 (6)
O3—H3*C*···O2	0.76 (6)	2.51 (6)	3.057 (3)	130 (5)
O3—H3*D*···Cl3^{iii}	0.81 (7)	2.59 (6)	3.378 (4)	167.00

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*, *y*−1, *z*.