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1,10-Diazonia-18-crown-6 hexachloridoplatinate(IV) dihydrate

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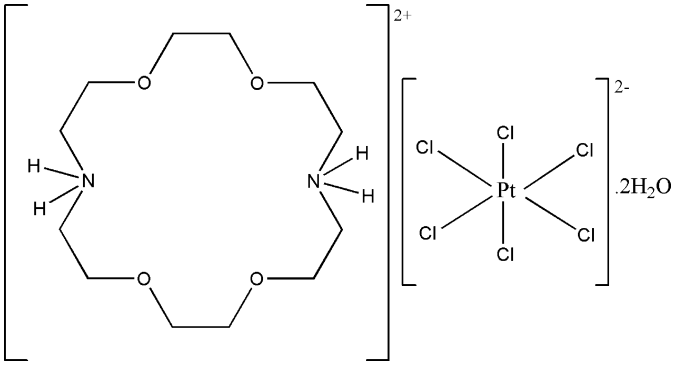
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.013 Å; R factor = 0.030; wR factor = 0.085; data-to-parameter ratio = 24.0.

The asymmetric unit of the title compound, $(C_{12}H_{28}N_2O_4)[PtCl_6]\cdot 2H_2O$, contains one half-cation, one half-anion and one water molecule, both ions lying on inversion centres. The Pt ion has an octahedral coordination. In the crystal structure, intermolecular O—H···Cl, N—H···O and O—H···O hydrogen bonds result in the formation of a supramolecular structure.

Related literature

For related literature, see: Bencini *et al.* (1992); Chekhlov (2000, 2001, 2005); Chekhlov & Martynov (1998); Chekhlov *et al.* (1994); Fonari *et al.* (2004); Hasan *et al.* (2001); Hu *et al.* (2003); Juan *et al.* (1998); Li & Liu (2003); Moers *et al.* (2000); Simonov *et al.* (2003); Terzis & Mentzafos (1983); Yousefi *et al.* (2007); Zordan & Brammer (2004).



Experimental

Crystal data

$(C_{12}H_{28}N_2O_4)[PtCl_6]\cdot 2H_2O$
M_r = 708.18
Monoclinic, $P2_1/c$
a = 9.3668 (12) Å
b = 12.5688 (11) Å
c = 10.9546 (15) Å
β = 112.384 (9)°
V = 1192.5 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 6.59 mm^{-1}
T = 298 (2) K
0.25 × 0.18 × 0.10 mm

Data collection

Stoe IPDS II diffractometer
Absorption correction: numerical (*X-RED*; Stoe & Cie, 2005)
T_{min} = 0.250, T_{max} = 0.510
8277 measured reflections
3168 independent reflections
2493 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.085
S = 1.22
3168 reflections
132 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pt1—Cl2	2.3257 (12)	Pt1—Cl1	2.3262 (12)
Pt1—Cl3	2.3259 (12)		
Cl2^i—Pt1—Cl2	180	Cl2^i—Pt1—Cl1	89.66 (5)
Cl2^i—Pt1—Cl3	89.30 (4)	Cl2—Pt1—Cl1	90.34 (5)
Cl2—Pt1—Cl3	90.70 (4)	Cl3—Pt1—Cl1	89.98 (5)
Cl3—Pt1—Cl3^i	180	Cl1^i—Pt1—Cl1	180
Cl3—Pt1—Cl1^i	90.02 (5)		

Symmetry code: (i) $-x+1, -y+2, -z+1$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···O2	0.90	2.55	2.803 (9)	97
N1—H1C···O3	0.90	2.21	2.936 (7)	138
N1—H1D···O1	0.90	2.57	2.852 (8)	99
O3—H3C···Cl2	0.79 (6)	2.39 (6)	3.169 (4)	173 (7)
O3—H3D···O2	0.69 (13)	2.48 (10)	3.103 (7)	152 (10)
N1—H1D···O3^{ii}	0.90	1.93	2.817 (6)	169
O3—H3D···O1^{ii}	0.69 (13)	2.53 (12)	2.961 (7)	123 (12)

Symmetry code: (ii) $-x, -y+2, -z$.

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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); 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: HK2322).

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supplementary materials

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1,10-Diazonia-18-crown-6 hexachloridoplatinate(IV) dihydrate

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Comment

Recently, we reported the synthesis and crystal structure of [(H_2DA18C6)Cl_2], (II), (Yousefi *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], (III), (Chekhlov, 2005), [(H_2DA18C6)(C_2HO_4)$_2$], (IV), and [(H_2DA18C6)$_2$(C_2O_4)$_2$·2H_2O], (V), (Chekhlov, 2000), [(H_2DA18C6)(picrate)$_2$], (VI), (Chekhlov, 2001), [(H_2DA18C6)(HPTD)$_2$], (VII), (Simonov *et al.*, 2003), [(H_2DA18C6)(PD)$_2$·(H_2O)$_4$], (VIII), and [(H_2DA18C6)(PS)$_2$·(H_2O)$_2$], (IX), (Fonari *et al.*, 2004), [(H_2DA18C6)(CCl_3COO)$_2$(CCl_3COOH)$_2$], (*X*), (Chekhlov *et al.*, 1994), [(H_2DA18C6)(CCl_3COO)$_2$], (XI), (Chekhlov & Martynov, 1998), and {[H_2DA18C6][($ArSO_2$)$_2$N]$_2$}, (XII), (Moers *et al.*, 2000) [where H_2DA18C6 is 1,10-Diazonia-18-crown-6, 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-yl- sulfamate 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 H_2[$PtCl_6$] with proton acceptor molecules, such as [Hpy-Br-3]$_2$[$PtCl_6$]·2H_2O, (XIII), and [HpyI-3]$_2$[$PtCl_6$]·2H_2O, (XIV), (Zordan & Brammer, 2004), [BMIM]$_2$[$PtCl_6$], (XV), and [EMIM]$_2$[$PtCl_6$], (XVI), (Hasan *et al.*, 2001), {(DABCO)H_2[$PtCl_6$]}, (XVI), (Juan *et al.*, 1998), {*p*-C_6H_4(CH_2ImMe)$_2$[$PtCl_6$]}, (XVIII), (Li & Liu, 2003), [het][$PtCl_6$]·2H_2O, (XIX), (Hu *et al.*, 2003), [9-MeGuaH]$_2$[$PtCl_6$]·2H_2O, (XX), (Terzis & Mentzafos, 1983) and [H_{10}[30]aneN_{10}][$PtCl_6$]$_2$$Cl_6$·2$H_2O$, (XXI), (Bencini *et al.*, 1992) [Where BMIM^+ is 1-*n*-butyl-3-methylimidazolium, EMIM^+ is 1-ethyl-3-methyl- imidazolium, DABCO is 1,4-diazabicyclooctane, het is 2-(?-hydroxyethyl) thiamine and 9-MeGuaH is 9-methylguaninium] 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 half-anion and one water molecule. The Pt ion has an octahedral coordination. The bond lengths and angles, in cation, are in good agreement with the corresponding values in (II) and (III). Also, the Pt—Cl bond lengths and angles (Table 1) are within normal ranges, as in [H_{10}[30]ane][$PtCl_6$]$_2$$Cl_6$·2$H_2O$, (XXII), (Bencini *et al.*, 1992).

In the crystal structure, the intermolecular O—H···Cl, N—H···O and O—H···O hydrogen bonds (Table 2) seem to be effective in the stabilization of the crystal structure, resulting in the formation of a supramolecular structure (Fig. 2).

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 water (50 ml) was added to a solution of H_2PtCl_6·2H_2O (0.20 g, 0.37 mmol) in water (30 ml) and the resulting yellow solution was stirred

for 30 min at 333 K. Then, it was left to evaporate slowly at room temperature. After 24 h, yellow prismatic crystals of (I) were isolated (yield; 0.22 g, 84%, m.p. 470–472 K).

Refinement

H3C and H3D (for OH_2) were located in difference syntheses and refined isotropically [O—H = 0.69 (10) and 0.79 (7) Å and U_{iso}(H) = 0.062 (19) and 0.08 (3) Å^2]. The remaining H atoms were positioned geometrically, with N—H = 0.90 Å 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*).

Figures

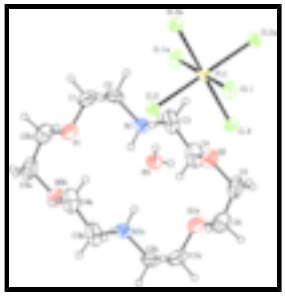


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [symmetry codes: (*a*) 1 − *x*, 2 − *y*, 1 − *z*; (*b*) −*x*, 2 − *y*, −*z*].

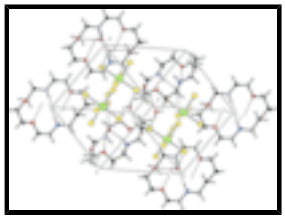


Fig. 2. A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

1,10-Diazonia-18-crown-6 hexachloridoplatinate(IV) dihydrate

Crystal data

$(C_{12}H_{28}N_2O_4)[PtCl_6]\cdot 2H_2O$	F_{000} = 692
M_r = 708.18	D_x = 1.972 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2000 reflections
a = 9.3668 (12) Å	θ = 2.4–29.3º
b = 12.5688 (11) Å	μ = 6.59 mm^{-1}
c = 10.9546 (15) Å	*T* = 298 (2) K
β = 112.384 (9)º	Prism, yellow
V = 1192.5 (3) Å^3	0.25 × 0.18 × 0.10 mm
Z = 2	

Data collection

Stoe IPDS II diffractometer	3168 independent reflections

Radiation source: fine-focus sealed tube	2493 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.036$
Detector resolution: 0.15 mm pixels mm^{-1}	$\theta_{max} = 29.3°$
$T = 298(2)$ K	$\theta_{min} = 2.4°$
rotation method scans	$h = -12 \rightarrow 12$
Absorption correction: numerical (X-RED; Stoe & Cie, 2005)	$k = -17 \rightarrow 13$
$T_{min} = 0.250$, $T_{max} = 0.510$	$l = -10 \rightarrow 15$
8277 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.030$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.085$	$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2 + 0.839P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.22$	$(\Delta/\sigma)_{max} = 0.011$
3168 reflections	$\Delta\rho_{max} = 1.27$ e $Å^{-3}$
132 parameters	$\Delta\rho_{min} = -0.66$ e $Å^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2$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}
C1	0.3080 (7)	0.8830 (6)	−0.0359 (7)	0.074 (2)
H1A	0.3526	0.9281	0.0412	0.089*
H1B	0.3907	0.8576	−0.0611	0.089*
C2	0.2280 (7)	0.7913 (5)	−0.0044 (7)	0.0660 (17)
H2A	0.1829	0.7470	−0.0822	0.079*
H2B	0.3025	0.7486	0.0643	0.079*
C3	0.0327 (10)	0.7382 (5)	0.0851 (8)	0.086 (2)
H3A	0.1064	0.7091	0.1667	0.103*

H3B	0.0038	0.6822	0.0192	0.103*
C4	−0.1087 (10)	0.7764 (6)	0.1067 (9)	0.088 (2)
H4A	−0.1832	0.8060	0.0257	0.105*
H4B	−0.1569	0.7178	0.1342	0.105*
C5	−0.1737 (10)	0.8963 (8)	0.2511 (10)	0.097 (3)
H5B	−0.2307	0.8373	0.2676	0.116*
H5A	−0.1197	0.9316	0.3348	0.116*
C6	−0.2797 (11)	0.9680 (9)	0.1662 (12)	0.108 (3)
H6A	−0.3505	0.9919	0.2061	0.130*
H6B	−0.3393	0.9326	0.0838	0.130*
N1	0.1050 (5)	0.8269 (3)	0.0404 (5)	0.0515 (10)
H1C	0.1456	0.8735	0.1072	0.062*
H1D	0.0317	0.8612	−0.0264	0.062*
O1	0.2010 (5)	0.9412 (4)	−0.1400 (5)	0.0696 (12)
O2	−0.0603 (5)	0.8538 (4)	0.2044 (5)	0.0697 (11)
O3	0.1170 (5)	1.0429 (4)	0.1461 (4)	0.0531 (9)
H3C	0.199 (8)	1.060 (6)	0.197 (7)	0.062 (19)*
H3D	0.076 (12)	1.015 (6)	0.176 (11)	0.08 (3)*
Pt1	0.5000	1.0000	0.5000	0.03249 (8)
Cl1	0.54112 (16)	1.13900 (10)	0.64977 (13)	0.0528 (3)
Cl2	0.45210 (13)	1.12134 (10)	0.32866 (12)	0.0489 (3)
Cl3	0.23954 (13)	0.99812 (10)	0.46689 (13)	0.0476 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.051 (3)	0.095 (5)	0.075 (4)	0.013 (3)	0.023 (3)	−0.023 (4)
C2	0.067 (4)	0.061 (3)	0.060 (4)	0.030 (3)	0.013 (3)	0.000 (3)
C3	0.165 (8)	0.035 (3)	0.073 (4)	−0.007 (4)	0.064 (5)	−0.004 (3)
C4	0.113 (6)	0.075 (5)	0.080 (5)	−0.045 (4)	0.043 (5)	−0.011 (4)
C5	0.103 (6)	0.115 (7)	0.103 (6)	−0.013 (5)	0.074 (6)	0.014 (6)
C6	0.072 (5)	0.143 (8)	0.131 (9)	0.000 (5)	0.063 (6)	0.036 (7)
N1	0.065 (3)	0.038 (2)	0.050 (2)	0.0104 (18)	0.019 (2)	−0.0004 (18)
O1	0.061 (2)	0.071 (3)	0.092 (3)	0.012 (2)	0.044 (2)	0.007 (3)
O2	0.082 (3)	0.053 (2)	0.090 (3)	−0.005 (2)	0.051 (3)	0.000 (2)
O3	0.044 (2)	0.063 (2)	0.046 (2)	−0.0040 (18)	0.0101 (17)	−0.002 (2)
Pt1	0.03113 (11)	0.03264 (11)	0.03280 (11)	−0.00666 (8)	0.01116 (8)	−0.00084 (10)
Cl1	0.0654 (7)	0.0460 (6)	0.0518 (7)	−0.0157 (5)	0.0276 (6)	−0.0151 (5)
Cl2	0.0440 (6)	0.0511 (7)	0.0459 (6)	−0.0080 (5)	0.0106 (5)	0.0102 (5)
Cl3	0.0354 (5)	0.0533 (6)	0.0552 (6)	−0.0044 (4)	0.0183 (4)	0.0045 (6)

Geometric parameters (Å, °)

Pt1—Cl2^{i}	2.3257 (12)	C2—H2A	0.9700
Pt1—Cl2	2.3257 (12)	C2—H2B	0.9700
Pt1—Cl3	2.3259 (12)	C3—N1	1.482 (8)
Pt1—Cl3^{i}	2.3259 (12)	C3—C4	1.509 (12)
Pt1—Cl1^{i}	2.3262 (12)	C3—H3A	0.9700

Pt1—Cl1	2.3262 (12)	C3—H3B	0.9700
O1—C6^{ii}	1.444 (11)	C4—O2	1.389 (9)
O3—H3C	0.79 (7)	C4—H4A	0.9700
O3—H3D	0.69 (10)	C4—H4B	0.9700
N1—H1C	0.9000	C5—C6	1.400 (13)
N1—H1D	0.9000	C5—O2	1.446 (9)
C1—O1	1.404 (8)	C5—H5B	0.9700
C1—C2	1.485 (10)	C5—H5A	0.9700
C1—H1A	0.9700	C6—O1^{ii}	1.444 (11)
C1—H1B	0.9700	C6—H6A	0.9700
C2—N1	1.483 (8)	C6—H6B	0.9700
Cl2^{i}—Pt1—Cl2	180	N1—C2—C1	111.5 (5)
Cl2^{i}—Pt1—Cl3	89.30 (4)	N1—C2—H2A	109.3
Cl2—Pt1—Cl3	90.70 (4)	C1—C2—H2A	109.3
Cl2^{i}—Pt1—Cl3^{i}	90.70 (4)	N1—C2—H2B	109.3
Cl2—Pt1—Cl3^{i}	89.30 (4)	C1—C2—H2B	109.3
Cl3—Pt1—Cl3^{i}	180	H2A—C2—H2B	108.0
Cl2^{i}—Pt1—Cl1^{i}	90.34 (5)	N1—C3—C4	110.6 (5)
Cl2—Pt1—Cl1^{i}	89.66 (5)	N1—C3—H3A	109.5
Cl3—Pt1—Cl1^{i}	90.02 (5)	C4—C3—H3A	109.5
Cl3^{i}—Pt1—Cl1^{i}	89.98 (5)	N1—C3—H3B	109.5
Cl2^{i}—Pt1—Cl1	89.66 (5)	C4—C3—H3B	109.5
Cl2—Pt1—Cl1	90.34 (5)	H3A—C3—H3B	108.1
Cl3—Pt1—Cl1	89.98 (5)	O2—C4—C3	107.2 (6)
Cl3^{i}—Pt1—Cl1	90.02 (5)	O2—C4—H4A	110.3
Cl1^{i}—Pt1—Cl1	180	C3—C4—H4A	110.3
C1—O1—C6^{ii}	108.3 (6)	O2—C4—H4B	110.3
C4—O2—C5	117.6 (6)	C3—C4—H4B	110.3
H3C—O3—H3D	112 (10)	H4A—C4—H4B	108.5
C3—N1—C2	113.1 (5)	C6—C5—O2	115.7 (7)
C3—N1—H1C	109.0	C6—C5—H5B	108.4
C2—N1—H1C	109.0	O2—C5—H5B	108.4
C3—N1—H1D	109.0	C6—C5—H5A	108.4
C2—N1—H1D	109.0	O2—C5—H5A	108.4
H1C—N1—H1D	107.8	H5B—C5—H5A	107.4
O1—C1—C2	109.1 (5)	C5—C6—O1^{ii}	110.8 (7)
O1—C1—H1A	109.9	C5—C6—H6A	109.5
C2—C1—H1A	109.9	O1^{ii}—C6—H6A	109.5
O1—C1—H1B	109.9	C5—C6—H6B	109.5
C2—C1—H1B	109.9	O1^{ii}—C6—H6B	109.5
H1A—C1—H1B	108.3	H6A—C6—H6B	108.1
O1—C1—C2—N1	−62.0 (7)	C1—C2—N1—C3	−174.4 (6)
N1—C3—C4—O2	−61.1 (8)	C2—C1—O1—C6^{ii}	178.1 (7)
O2—C5—C6—O1^{ii}	−59.2 (13)	C3—C4—O2—C5	−174.7 (6)

C4—C3—N1—C2	−170.2 (6)	C6—C5—O2—C4	−75.3 (10)

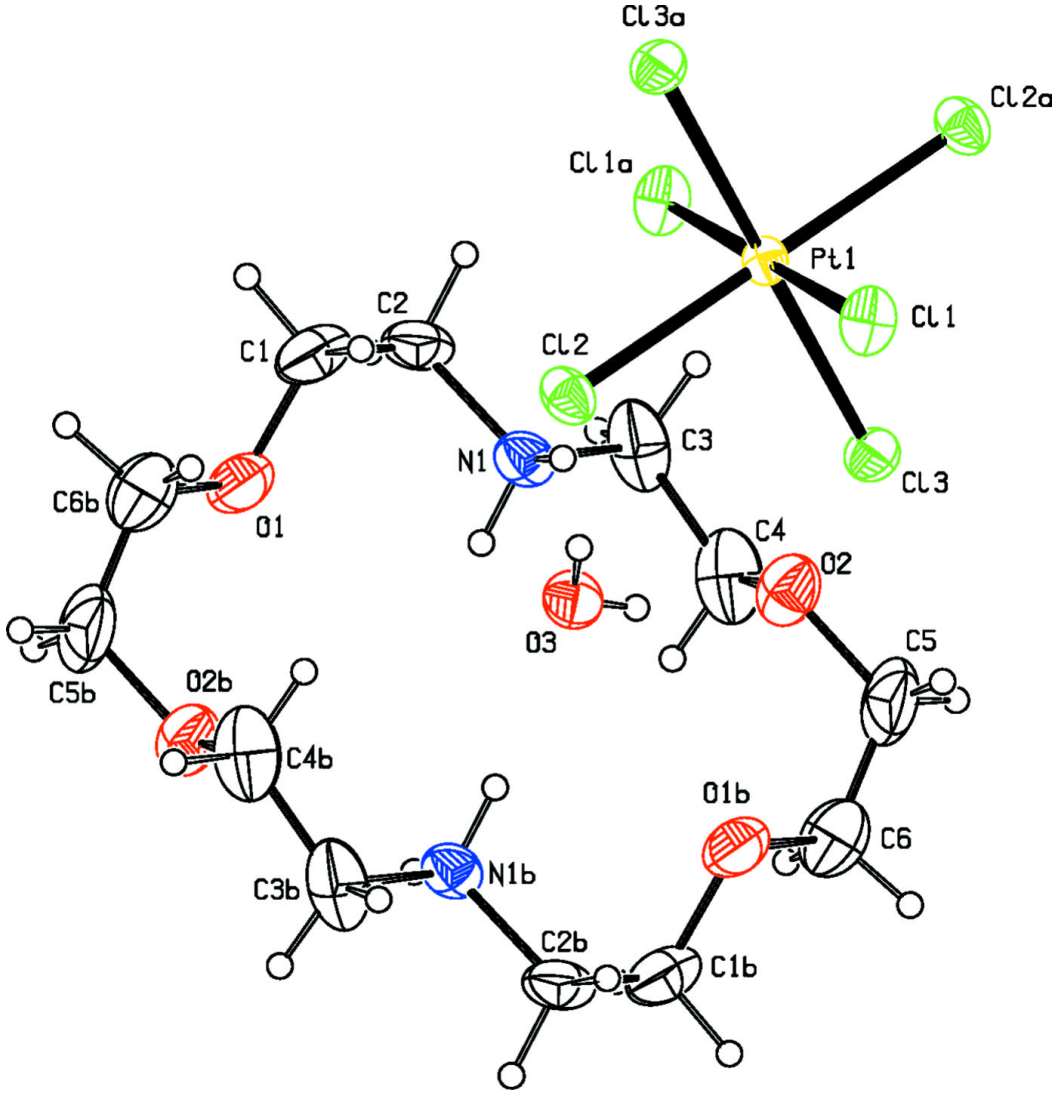
Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1; (ii) −*x*, −*y*+2, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1C···O2	0.90	2.55	2.803 (9)	97
N1—H1C···O3	0.90	2.21	2.936 (7)	138
N1—H1D···O1	0.90	2.57	2.852 (8)	99
O3—H3C···Cl2	0.79 (6)	2.39 (6)	3.169 (4)	173 (7)
O3—H3D···O2	0.69 (13)	2.48 (10)	3.103 (7)	152 (10)
N1—H1D···O3^{ii}	0.90	1.93	2.817 (6)	169
O3—H3D···O1^{ii}	0.69 (13)	2.53 (12)	2.961 (7)	123 (12)

Symmetry codes: (ii) −*x*, −*y*+2, −*z*.

Fig. 1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Cl1
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C1
N1
C3
C6b
O1
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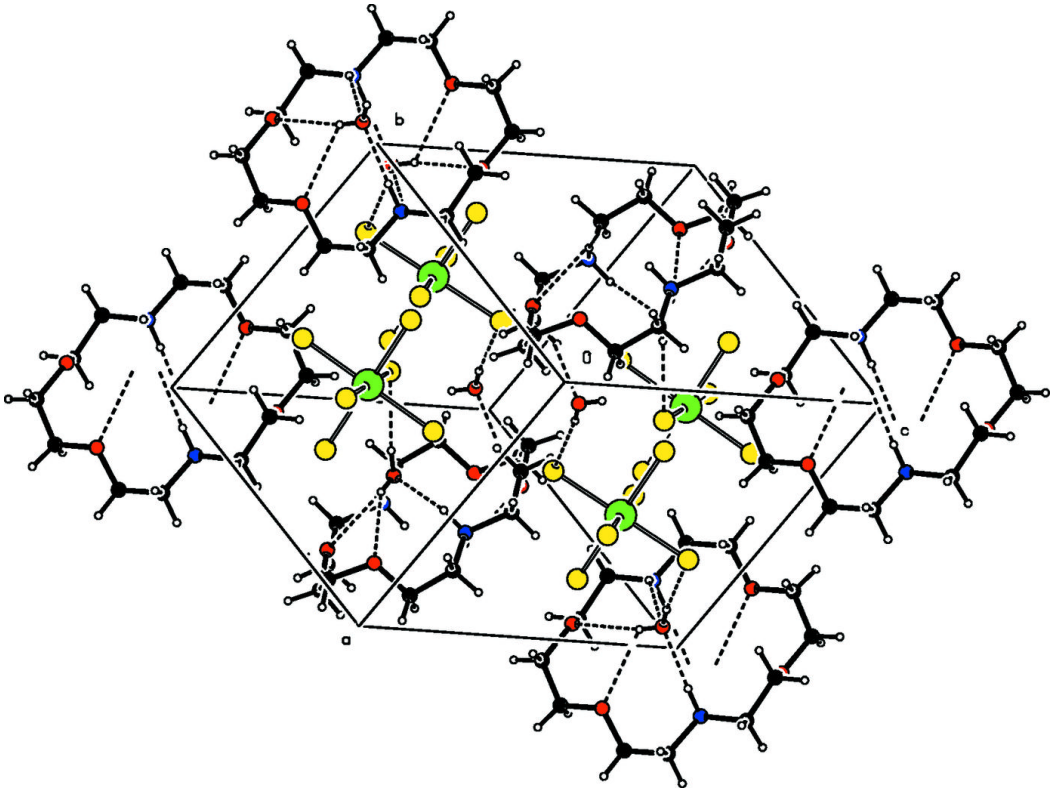
Fig. 2