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1,10-Diazonia-18-crown-6 dichloride

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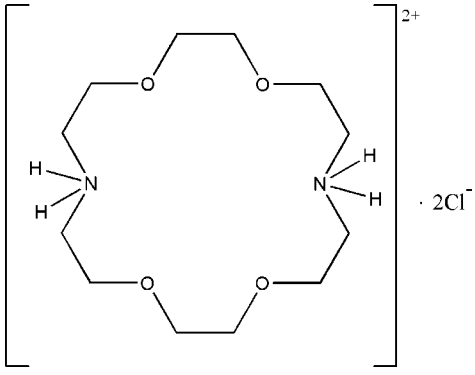
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.066; data-to-parameter ratio = 18.8.

The asymmetric unit of the title compound, $C_{12}H_{28}N_2O_4^{+}\cdot 2Cl^{-}$, contains one half of a centrosymmetric cation and one anion. In the crystal structure, intermolecular N—H···Cl hydrogen bonds result in the formation of a supramolecular structure.

Related literature

For related literature, see: Smith *et al.* (1999); Zafar *et al.* (2000); Chekhlov *et al.* (1994); Chekhlov & Martynov (1998); Chekhlov (2000, 2001, 2005); Simonov *et al.* (2003); Fonari *et al.* (2004); Moers *et al.* (2000).



Experimental

Crystal data

$C_{12}H_{28}N_2O_4^{2+}\cdot 2Cl^{-}$
M_r = 335.26
Monoclinic, $P2_1/n$
a = 9.5461 (19) Å
b = 5.6297 (11) Å
c = 15.688 (3) Å
β = 90.70 (3)°
V = 843.0 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 120 (2) K
0.35 × 0.10 × 0.05 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: numerical (shape of crystal determined optically)
T_{min} = 0.950, T_{max} = 0.980
6928 measured reflections
2012 independent reflections
1811 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.066
S = 1.13
2012 reflections
107 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···Cl1	0.89 (2)	2.23 (2)	3.1144 (12)	175.7 (14)
N1—H1D···Cl1^{i}	0.93 (2)	2.19 (2)	3.1142 (12)	174.0 (2)

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

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: HK2305).

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supplementary materials

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1,10-Diazonia-18-crown-6 dichloride

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Comment

In recent years, there has been considerable interest in proton transfer systems and their structures (Smith *et al.*, 1999; Zafar *et al.*, 2000). Several proton transfer systems using 1,10-diaza-18-crown-6, with proton donor molecules, such as [(H_2DA18C6)I_2·2H_2O], (II), (Chekhlov, 2005), [(H_2DA18C6)(C_2HO_4)$_2$], (III), and [(H_2DA18C6)$_2$(C_2O_4)$_2$·2H_2O], (IV), (Chekhlov, 2000), [(H_2DA18C6)(picrate)$_2$], (V), (Chekhlov, 2001), [(H_2DA18C6)(HPTD)$_2$], (VI), (Simonov *et al.*, 2003), [(H_2DA18C6)(PD)$_2$·(H_2O)$_4$], (VII), and [(H_2DA18C6)(PS)$_2$·(H_2O)$_2$], (VIII), (Fonari *et al.*, 2004), [(H_2DA18C6)(CCl_3COO)$_2$(CCl_3COOH)$_2$], (IX), (Chekhlov *et al.*, 1994), [(H_2DA18C6)(CCl_3COO)$_2$], (*X*), (Chekhlov & Martynov, 1998) and {[H_2DA18C6][($ArSO_2$)$_2$N]$_2$}, (XI), (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. We report herein the synthesis and crystal structure of the title compound, (I).

The asymmetric unit of (I), (Fig. 1), contains one half centrosymmetric cation and one anion. The bond lengths and angles in good agreement with the corresponding values in (II), (IX) and (*X*).

In the crystal structure, intermolecular N—H···Cl [H1c···Cl1^{i} = 2.23 (2) Å, N1···Cl1^{i} = 3.11 (1) Å, N1—H1c···Cl1^{i} = 176.0 (3)° and H1dc···Cl1^{ii} = 2.19 (2) Å, N1···Cl1^{ii} = 3.11 (2) Å, N1—H1d···Cl1^{ii} = 174.0 (2)°; symmetry codes: (i) 3/2 − *x*, 1/2 + *y*, 1/2 − *z*; (ii) *x*, 1 + *y*, *z*] hydrogen bonds seem to be effective in the stabilization of the structure, resulting in the formation of a supramolecular structure (Fig. 2).

Experimental

1,10-diaza-18-crown-6 (0.12 g, 0.45 mmol) was added to a solution of HCl (0.1 *M*, 10 ml) and the resulting colorless solution was stirred at 323 K for 2 h. Then, it was left to evaporate slowly at room temperature. The milky precipitated product was recrystallized from EtOH in two weeks (yield; 0.13 g, 84.7%, m.p. 468–471 K).

Refinement

H6A, H6B (for CH_2) and H1C, H1D (for NH_2) were located in difference syntheses and refined isotropically [C—H = 0.970 (17) and 0.979 (16) Å, U_{iso}(H) = 0.26 (4) and 0.19 (4) Å^2; N—H = 0.888 (18) and 0.926 (18) Å, U_{iso}(H) = 0.26 (4) and 0.31 (4) Å^2]. The remaining H atoms were positioned geometrically, with C—H = 0.97 Å for methylene H, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

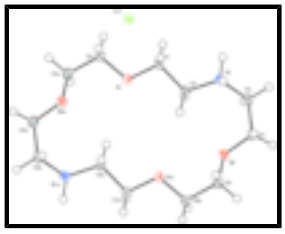

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [symmetry code a: 2 − *x*, −*y*, 1 − *z*].

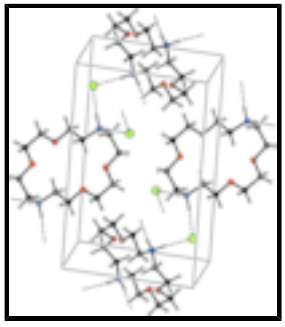

Fig. 2. A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

1,10-Diazonia-18-crown-6 dichloride

Crystal data

$C_{12}H_{28}N_2O_4^{2+}\cdot 2Cl^-$	F_{000} = 360
M_r = 335.26	D_x = 1.321 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2000 reflections
a = 9.5461 (19) Å	θ = 2.5–22.5º
b = 5.6297 (11) Å	μ = 0.40 mm^{-1}
c = 15.688 (3) Å	T = 120 (2) K
β = 90.70 (3)º	Needle, colorless
V = 843.0 (3) Å^3	0.35 × 0.10 × 0.05 mm
Z = 2	

Data collection

Stoe IPDSII diffractometer	2012 independent reflections
Radiation source: fine-focus sealed tube	1811 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.027
Detector resolution: 0.15 mm pixels mm^{-1}	θ_{max} = 27.9º
T = 120(2) K	θ_{min} = 2.5º
rotation method scans	h = −12→12
Absorption correction: numerical (shape of crystal determined optically)	k = −7→7
T_{min} = 0.950, T_{max} = 0.980	l = −20→20

6928 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
$wR(F^2) = 0.066$	$w = 1/[\sigma^2(F_o^2) + (0.0217P)^2 + 0.3926P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.13$	$(\Delta/\sigma)_{max} = 0.011$
2012 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
107 parameters	$\Delta\rho_{min} = -0.18$ 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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	0.57203 (3)	0.11647 (5)	0.317180 (18)	0.01755 (9)
O1	1.11725 (9)	−0.01497 (16)	0.39337 (5)	0.01855 (19)
O2	0.77044 (9)	0.43864 (16)	0.54459 (5)	0.01715 (19)
N1	0.83570 (11)	0.42244 (18)	0.35935 (6)	0.0150 (2)
H1C	0.7629 (18)	0.332 (3)	0.3449 (10)	0.026 (4)*
H1D	0.8705 (18)	0.479 (3)	0.3084 (11)	0.031 (4)*
C1	1.02583 (13)	0.1255 (2)	0.34187 (7)	0.0171 (2)
H1A	0.9627	0.0256	0.3088	0.021*
H1B	1.0788	0.2243	0.3031	0.021*
C2	0.94521 (12)	0.2770 (2)	0.40406 (8)	0.0159 (2)
H2A	0.9009	0.1753	0.4458	0.019*
H2B	1.0096	0.3816	0.4342	0.019*
C3	0.78311 (13)	0.6312 (2)	0.40907 (8)	0.0177 (2)
H3A	0.7316	0.7361	0.3710	0.021*
H3B	0.8624	0.7189	0.4321	0.021*
C4	0.68918 (12)	0.5579 (2)	0.48138 (8)	0.0180 (2)

H4A	0.6454	0.6972	0.5059	0.022*
H4B	0.6158	0.4536	0.4601	0.022*
C5	0.69032 (13)	0.2846 (2)	0.59700 (8)	0.0191 (3)
H5A	0.6390	0.1716	0.5619	0.023*
H5B	0.6235	0.3759	0.6296	0.023*
C6	0.78945 (13)	0.1554 (2)	0.65629 (8)	0.0188 (3)
H6A	0.7348 (17)	0.054 (3)	0.6930 (10)	0.026 (4)*
H6B	0.8432 (16)	0.267 (3)	0.6918 (10)	0.019 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01650 (14)	0.01926 (15)	0.01685 (14)	−0.00181 (11)	−0.00086 (9)	−0.00161 (11)
O1	0.0203 (4)	0.0203 (4)	0.0151 (4)	0.0079 (4)	0.0013 (3)	0.0012 (3)
O2	0.0145 (4)	0.0191 (4)	0.0179 (4)	−0.0001 (3)	−0.0012 (3)	0.0042 (3)
N1	0.0140 (5)	0.0162 (5)	0.0149 (5)	−0.0006 (4)	−0.0024 (4)	0.0021 (4)
C1	0.0186 (5)	0.0179 (6)	0.0149 (5)	0.0031 (5)	−0.0003 (4)	0.0021 (5)
C2	0.0152 (5)	0.0172 (6)	0.0153 (5)	0.0033 (5)	−0.0021 (4)	0.0003 (5)
C3	0.0197 (6)	0.0132 (5)	0.0201 (6)	0.0028 (5)	−0.0026 (4)	0.0019 (5)
C4	0.0152 (5)	0.0190 (6)	0.0197 (6)	0.0048 (5)	−0.0012 (4)	0.0000 (5)
C5	0.0149 (5)	0.0205 (6)	0.0221 (6)	0.0013 (5)	0.0042 (4)	0.0029 (5)
C6	0.0186 (6)	0.0213 (6)	0.0166 (5)	0.0039 (5)	0.0053 (5)	0.0019 (5)

Geometric parameters (Å, °)

C1—O1	1.4223 (15)	C4—H4A	0.9700
C1—C2	1.5132 (16)	C4—H4B	0.9700
C1—H1A	0.9700	C5—O2	1.4243 (15)
C1—H1B	0.9700	C5—C6	1.5060 (18)
C2—N1	1.4958 (15)	C5—H5A	0.9700
C2—H2A	0.9700	C5—H5B	0.9700
C2—H2B	0.9700	C6—O1^{i}	1.4290 (14)
C3—N1	1.5003 (16)	C6—H6A	0.970 (17)
C3—C4	1.5119 (17)	C6—H6B	0.979 (16)
C3—H3A	0.9700	N1—H1C	0.888 (18)
C3—H3B	0.9700	N1—H1D	0.926 (18)
C4—O2	1.4203 (15)	O1—C6^{i}	1.4290 (14)
O1—C1—C2	105.10 (9)	C3—C4—H4B	109.8
O1—C1—H1A	110.7	H4A—C4—H4B	108.3
C2—C1—H1A	110.7	O2—C5—C6	108.23 (10)
O1—C1—H1B	110.7	O2—C5—H5A	110.1
C2—C1—H1B	110.7	C6—C5—H5A	110.1
H1A—C1—H1B	108.8	O2—C5—H5B	110.1
N1—C2—C1	111.36 (10)	C6—C5—H5B	110.1
N1—C2—H2A	109.4	H5A—C5—H5B	108.4
C1—C2—H2A	109.4	O1^{i}—C6—C5	108.75 (10)
N1—C2—H2B	109.4	O1^{i}—C6—H6A	109.9 (10)
C1—C2—H2B	109.4	C5—C6—H6A	108.3 (10)

H2A—C2—H2B	108.0	O1^{i}—C6—H6B	109.8 (9)
N1—C3—C4	112.45 (10)	C5—C6—H6B	111.3 (9)
N1—C3—H3A	109.1	H6A—C6—H6B	108.8 (13)
C4—C3—H3A	109.1	C2—N1—C3	114.92 (9)
N1—C3—H3B	109.1	C2—N1—H1C	110.3 (11)
C4—C3—H3B	109.1	C3—N1—H1C	108.3 (11)
H3A—C3—H3B	107.8	C2—N1—H1D	109.8 (11)
O2—C4—C3	109.21 (10)	C3—N1—H1D	107.6 (11)
O2—C4—H4A	109.8	H1C—N1—H1D	105.4 (15)
C3—C4—H4A	109.8	C1—O1—C6^{i}	112.34 (9)
O2—C4—H4B	109.8	C4—O2—C5	113.48 (9)
O1—C1—C2—N1	−175.53 (9)	C4—C3—N1—C2	−73.04 (13)
N1—C3—C4—O2	68.74 (13)	C2—C1—O1—C6^{i}	−174.95 (10)
O2—C5—C6—O1^{i}	−62.08 (13)	C3—C4—O2—C5	−156.73 (10)
C1—C2—N1—C3	−161.13 (10)	C6—C5—O2—C4	175.61 (10)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1C···Cl1	0.89 (2)	2.23 (2)	3.1144 (12)	175.7 (14)
N1—H1D···Cl1^{ii}	0.93 (2)	2.19 (2)	3.1142 (12)	174.0 (2)

Symmetry codes: (ii) −*x*+3/2, *y*+1/2, −*z*+1/2.

Fig. 1

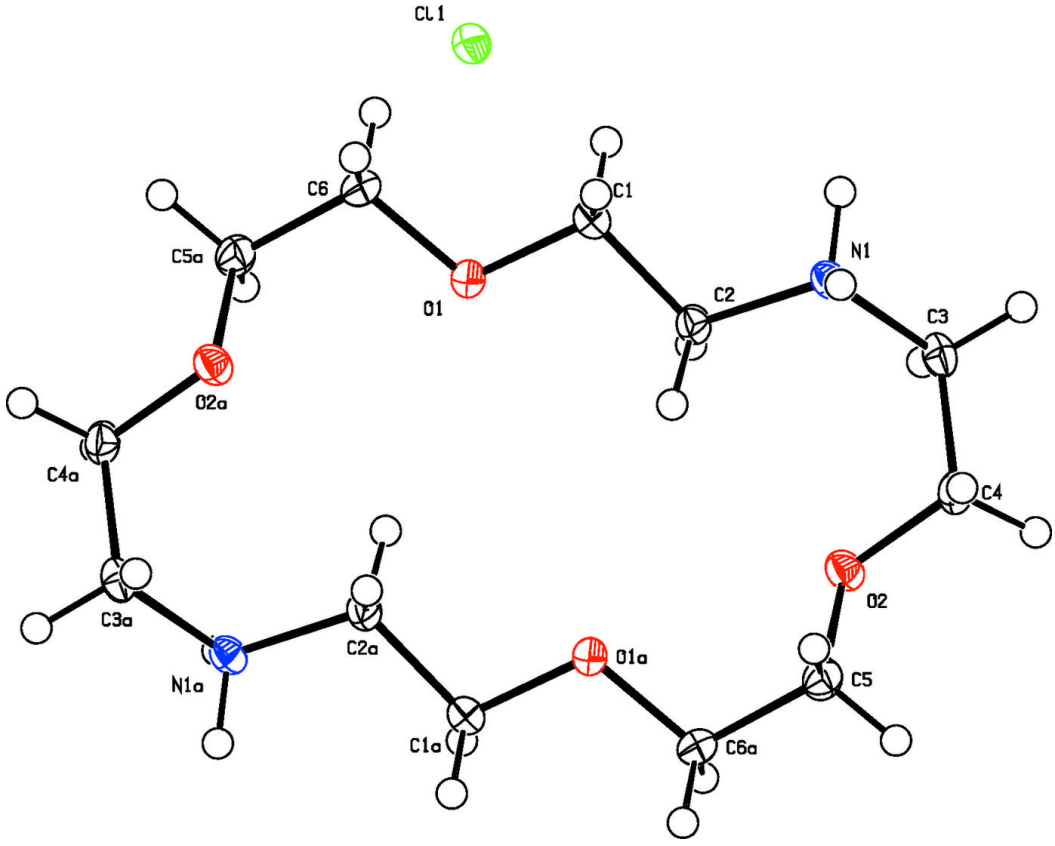

Fig. 2