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2-Aminomethyl-15-crown[5] hydrochloride dihydrate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.087
Data-to-parameter ratio = 18.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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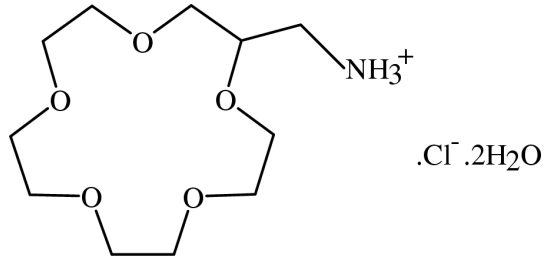
Positively charged methylamine-substituted 15-crown[5] co-crystallizes with one chloride counter-anion and two water molecules, $C_{11}H_{24}NO_5^+\cdot Cl^-\cdot 2H_2O$. A supramolecular array of crown ether dimers, linked *via* water–chloride clusters, is formed.

Comment

The title compound, (I), was the sole crystalline product from an attempted substitution reaction of hexachlorotriphosphazatriene with 2-aminomethyl-15-crown[5] and is shown to be (Fig. 1) the protonated crown ether complexed with a chloride anion and two waters of crystallization.



(I)

The bond lengths and angles conform to standard values as observed in the Cambridge Structural Database (Allen & Kennard, 1993). A search of the CSD revealed there to be no such similar monosubstituted 15-crown[5] structures.

The macrocycle adopts a conformation such that the maximum deviation from the mean ring plane is 0.928 (6) Å, with the amine (N1) situated 1.92 (3) Å above this plane. The average cross-ring O···O separation is 2.840 (5) Å, which is of a similar order of magnitude to that of 2.793 (6) and 2.805 (5) Å in uncoordinated 15-crown[5] found in the CSD (CSD refcodes CIGSAM and DUCNEU, respectively).

Interestingly, the water molecules and chloride ion are not complexed by the crown ether; however, an extensive supramolecular array is formed by means of numerous hydrogen bonds (Fig. 2). These intermolecular interactions are detailed in Table 2. The crown ethers associate solely with each other, forming dimers linked by a methylene–oxygen and two amine–oxygen interactions. These dimers are connected together, to form a three-dimensional structure, *via* clusters of water molecules hydrogen bonded to chloride anions. A total of three water molecules are coordinated by two chloride ions. Each chloride is involved in the formation of four hydrogen bonds (three to H_2O and one to the ether). Atom O6 of a

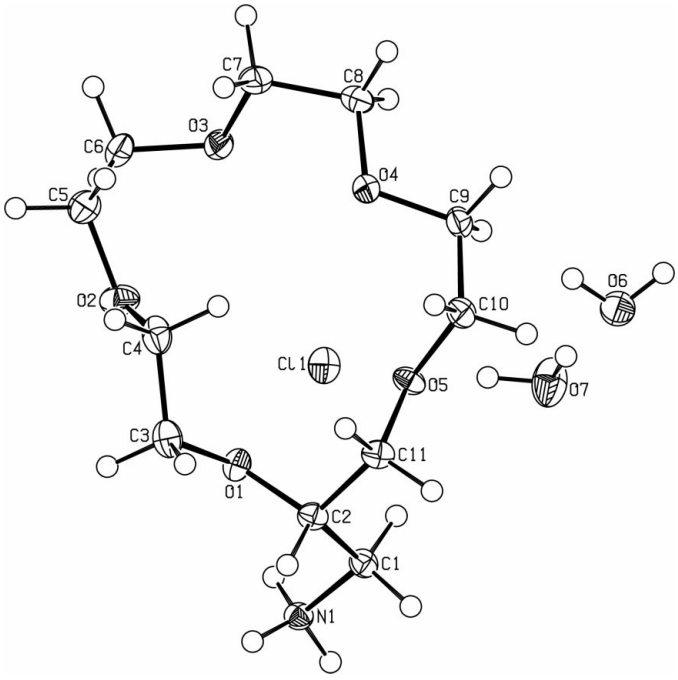


Figure 1
The molecular structure of (I) (50% probability displacement ellipsoids).

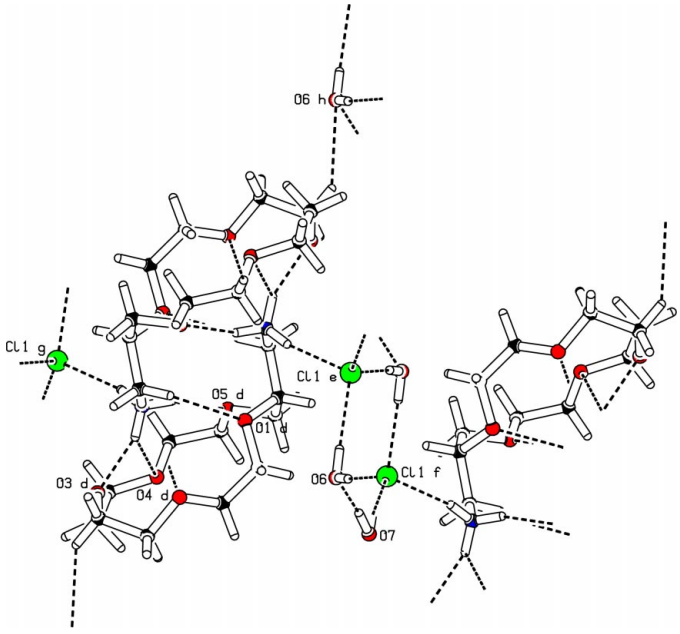


Figure 2
The supramolecular array formed by (I)

water molecule participates in three, and O7 in two classical hydrogen-bonding interactions.

Experimental

The title compound crystallized as a result of an attempted reaction between 2-aminomethyl-15-crown[5] and $N_3P_3Cl_6$ in tetrahydrofuran solvent.

Crystal data

$C_{11}H_{24}NO_5^+\cdot Cl^-\cdot 2H_2O$
$M_r = 321.79$
Monoclinic, $C2/c$
$a = 21.4218$ (4) Å
$b = 7.8864$ (2) Å
$c = 19.7794$ (4) Å
$\beta = 109.328$ (1)°
$V = 3153.22$ (12) Å^3
$Z = 8$
$D_x = 1.356$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11 239 reflections
$\theta = 2.9$–27.5°
$\mu = 0.27$ mm^{-1}
$T = 120$ (2) K
Rod, colourless
0.24 × 0.10 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (Blessing, 1997)
$T_{min} = 0.938$, $T_{max} = 0.989$
14 261 measured reflections
3595 independent reflections
3010 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.5°$
$h = -27 \rightarrow 25$
$k = -10 \rightarrow 10$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.087$
$S = 1.04$
3595 reflections
199 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 1.8513P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0021 (4)

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.4919 (16)	C7—O3	1.4306 (16)
C2—O1	1.4321 (15)	C8—O4	1.4269 (16)
C3—O1	1.4369 (15)	C9—O4	1.4239 (16)
C4—O2	1.4189 (17)	C10—O5	1.4326 (15)
C5—O2	1.4363 (18)	C11—O5	1.4279 (15)
C6—O3	1.4276 (16)		
C2—O1—C3	113.76 (9)	C9—O4—C8	112.77 (10)
C4—O2—C5	113.96 (11)	C11—O5—C10	111.80 (9)
C6—O3—C7	114.13 (10)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H01···Cl1	0.85 (2)	2.31 (2)	3.1633 (12)	178 (2)
O6—H02···Cl1^{i}	0.84 (2)	2.44 (2)	3.2711 (12)	174.7 (18)
N1—H1C···O1^{ii}	0.91	2.52	3.1063 (14)	122
N1—H1C···O5^{ii}	0.91	2.13	2.9783 (13)	154
N1—H1D···O3^{ii}	0.91	1.91	2.7772 (14)	158
N1—H1D···O4^{ii}	0.91	2.58	3.0662 (14)	114
N1—H1E···Cl1	0.91	2.27	3.1640 (11)	168
O7—H03···Cl1	0.89 (2)	2.39 (2)	3.2757 (13)	172.9 (18)
O7—H04···O6^{i}	0.92 (2)	1.93 (2)	2.8397 (16)	171.6 (18)
C1—H1B···O1^{ii}	0.99	2.40	3.1418 (16)	131
C9—H9B···O2^{iii}	0.99	2.54	3.4810 (19)	159

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

The H atoms on the crown ether were placed in idealized positions with their displacement parameters riding on the value of the parent atom, whilst the water H atoms were experimentally located and allowed to freely refine.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990).

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References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–429.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter and R. M. Sweet, pp. 307–326. London: Academic Press.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.