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Ethylenediammonium dichloride bis(1,10-phenanthroline) tetrahydrate

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.103
Data-to-parameter ratio = 12.7

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

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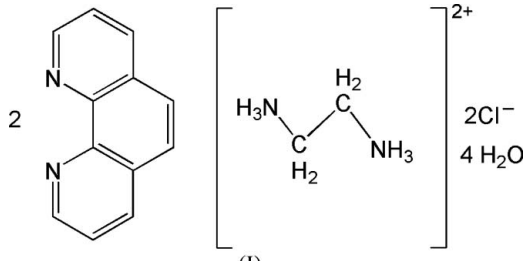
The title compound, $C_2H_{10}N_2^{2+}\cdot 2Cl^-\cdot 2C_{12}H_8N_2\cdot 4H_2O$, has an inversion centre located midway between the two C atoms of the ethylenediammonium cation. The 1,10-phenanthroline molecules stack along the *a* axis, and are linked by π–π stacking interactions to form one-dimensional chains. These are bridged by ethylenediammonium cations, *via* hydrogen bonds, forming two-dimensional supramolecular sheets parallel to the *ac* plane. Hydrogen bonds to chloride anions and water molecules connect these two-dimensional sheets, resulting in the formation of a three-dimensional supramolecular network.

Comment

The assembling of small organic or inorganic molecules into a supramolecular architecture through noncovalent interactions, such as hydrogen bonds and π···π interactions, has drawn a great deal of interest. This is not only due to the interesting structures thus obtained, but also because of the novel properties exhibited by such assemblies (Hu & Ruckenstein, 2006; Endo *et al.*, 1995; Veciana *et al.*, 1996; Lehn, 1988; Etter, 1990). We report here the synthesis and X-ray crystal structure of the title compound, (I).



(I)

Fig. 1 shows that the title compound consists of two 1,10-phenanthroline molecules, one ethylenediammonium cation, two chloride anions and four water molecules. An inversion centre is located midway between the two C atoms of the ethylenediammonium cation. The 1,10-phenanthroline molecules align parallel to each other along the *a* axis, with alternating perpendicular distances between them of 3.611 and 3.548 Å and alternating centroid-to-centroid separations of 3.887 and 3.802 Å for the corresponding pyridyl rings. These values indicate significant π–π stacking interactions between the 1,10-phenanthroline molecules. One-dimensional chains (Fig. 2) are thus formed parallel to the *a* axis. These chains are bridged by ethylenediammonium cations *via* hydrogen bonds, N3—H···N1*B* and N3—H···N2*B* [symmetry code: (*B*) $-x$, $-y+1$, $-z+2$], forming two-dimensional supramolecular

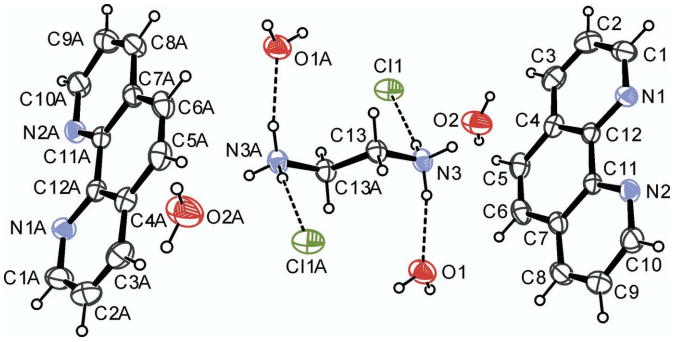


Figure 1
A view of the structure of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds. [Symmetry code: (*A*) $-x, -y+1, -z+1$.]

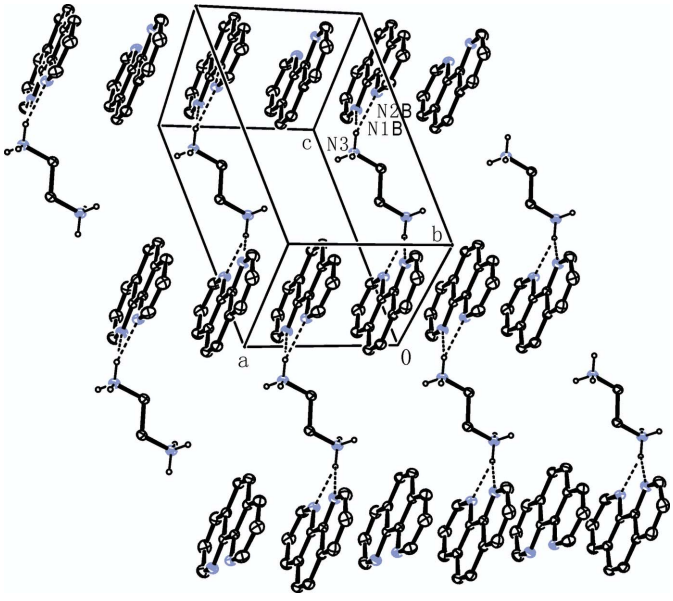


Figure 2
Two-dimensional sheets parallel to the *ac* plane assembled from ethylenediammonium cations and one-dimensional chains of 1,10-phenanthroline molecules. Dashed lines indicate hydrogen bonds. [Symmetry code: (*B*) $-x, -y+1, -z+2$.] H atoms not involved in the interactions shown have been omitted for clarity.

sheets parallel to the *ac* plane (Fig. 2). These are further linked by hydrogen bonds between ethylenediammonium cations, chloride anions and water molecules (Table 1), which also connect neighbouring sheets to each other along the *b* axis, resulting in the formation of a three-dimensional supramolecular network (Fig. 3).

Experimental

$MnCl_2·4H_2O$ (0.1991 g, 1 mmol), 1,10-phenanthroline (0.1985 g, 1 mmol) and ethylenediamine (0.07 ml, 1 mmol) were placed in a 23 ml Teflon-lined autoclave. To this mixture were added ethanol (3 ml) and water (3 ml). The autoclave was heated at 433 K for 72 h and then cooled over a period of 48 h, and the reaction mixture was filtered. The filtrate was allowed to evaporate at ambient temperature to give yellow single crystals of (I) after 8 d.

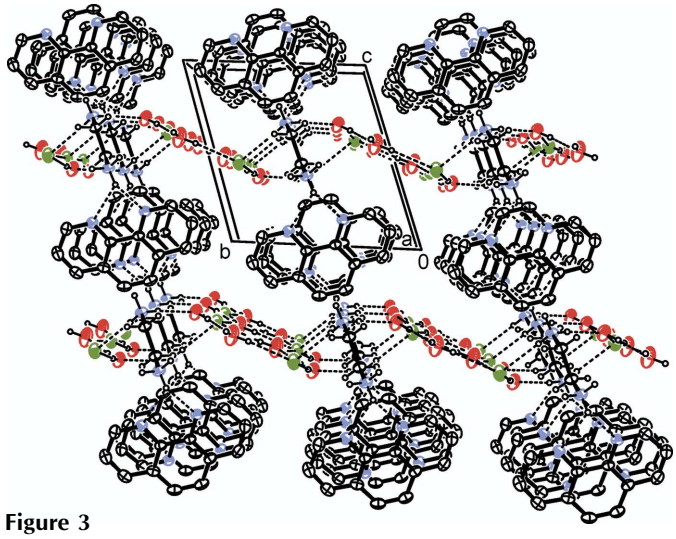


Figure 3
A view of the three-dimensional supramolecular network, showing hydrogen bonds between ethylenediammonium cations, chloride anions and water molecules, indicated by dashed lines. H atoms not involved in the interactions shown have been omitted for clarity.

Crystal data

$C_2H_{10}N_2^{2+}·2Cl^-·2C_{12}H_8N_2·4H_2O$
$M_r = 282.75$
Triclinic, $P\overline{1}$
$a = 7.6518$ (8) Å
$b = 9.8985$ (10) Å
$c = 10.6525$ (11) Å
$\alpha = 69.492$ (2)°
$\beta = 70.416$ (2)°
$\gamma = 80.602$ (2)°
$V = 711.02$ (13) Å^3
$Z = 2$
$D_x = 1.321$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.27$ mm^{-1}
$T = 291$ (2) K
Block, yellow
0.35 × 0.30 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.912$, $T_{max} = 0.967$
3211 measured reflections
2402 independent reflections
2013 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.103$
$S = 1.07$
2402 reflections
189 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 0.1558P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O1	0.89	1.94	2.816 (2)	168
N3—H3B···N2^{i}	0.89	2.05	2.870 (2)	153
N3—H3B···N1^{i}	0.89	2.43	3.023 (2)	125
N3—H3C···Cl1	0.89	2.35	3.2165 (16)	164
O1—H1W···Cl1^{ii}	0.85 (1)	2.31 (1)	3.1623 (16)	174 (2)
O1—H2W···O2^{ii}	0.85 (1)	1.86 (1)	2.705 (2)	176 (2)
O2—H3W···Cl1^{iii}	0.85 (1)	2.33 (1)	3.172 (2)	168 (3)
O2—H4W···Cl1^{iv}	0.85 (3)	2.40 (3)	3.238 (2)	171 (3)

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

H atoms of water molecules were located in a difference Fourier map and refined freely. H atoms on N and C atoms were placed in calculated positions, with N—H = 0.89 Å and C—H = 0.93 Å for the 1,10-phenanthroline molecule and 0.97 Å for the ethylenediammonium ion, and refined using a riding model, with $U_{iso}(H) = 1.5U_{eq}(N)$ and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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