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Bis(2-ammonio-5-chloropyridine) tetradecachlorohexamolybdate

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In the title compound, $(C_5H_6ClN_2)_2[(Mo_6Cl_8)Cl_6]$, the 2-ammonio-5-chloropyridine salt of the Mo^{II} chloride cluster shows a well defined N—H···Cl hydrogen-bond network. The anion is centrosymmetric.

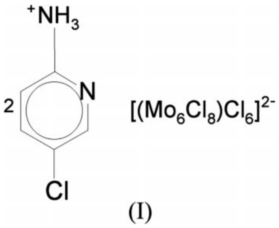
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.024
wR factor = 0.087
Data-to-parameter ratio = 17.8

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

Comment

The title compound, (I), is the 2-ammonio-5-chloropyridine salt of the centrosymmetric tetradecachloromolybdate dianion (Figs. 1 and 2). It has been investigated as part of a project aimed at the synthesis of extended molecular assemblies based on $[(Mo_6Cl_8)Cl_6]^{2-}$ clusters (Flemström *et al.*, 2004). On crystallization of (I), the pH of the mother liquor was ~ 1.5, which implies that equilibrium should be shifted towards protonation of the amine groups to give ammonium.



(I)

In (I), the geometric parameters for both the cation (Kvick & Backeus, 1974) and anion (Flemström *et al.*, 2004) are normal (Table 1).

The N—H···Cl hydrogen-bonding pattern (Table 2) is shown in Figs. 3 and 4. In the *bc* plane, the constituent species are in a sheet-like arrangement, and the stabilizing forces between the sheets are pure van der Waals attractions.

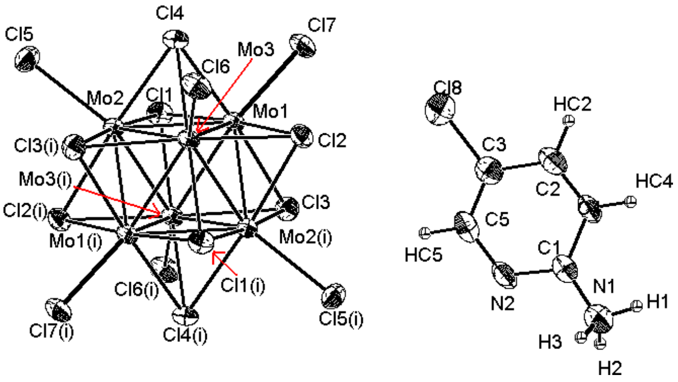


Figure 1
A view of the centrosymmetric anion and the unique cation of (I), with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $-x$, $-y$, $1-z$.]

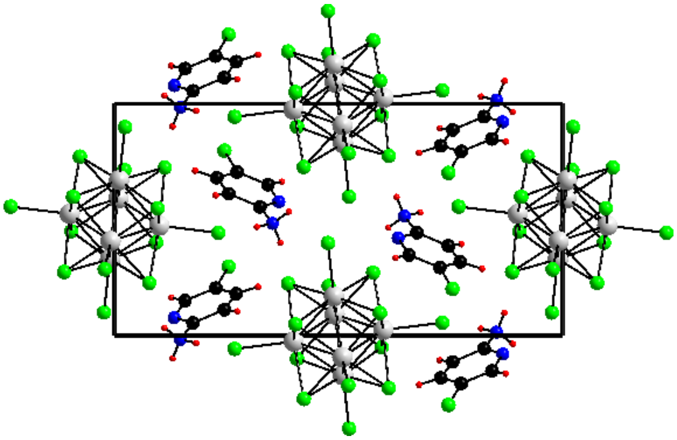


Figure 2
The unit-cell packing in (I), viewed along [100]. Blue spheres denote N atoms, black C, green Cl, grey Mo and red H.

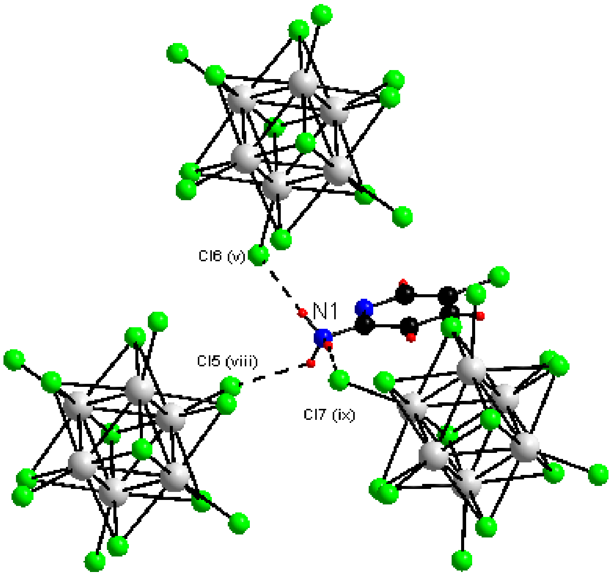


Figure 3
The N—H···Cl hydrogen-bond arrangements in (I), indicated by dashed lines, showing the Mo^{II} cluster arrangement around 2-ammonio-5-chloropyridine. [Symmetry codes: (v) $1 - x, -y, 1 + z$; ; (viii) $1 + x, y, z - 1$; (ix) $1 + x, \frac{1}{2} - y, z - \frac{1}{2}$.]

Experimental

Compound (I) was synthesized by mixing a saturated solution of $(H_3O)_2[(Mo_6Cl_8)Cl_6]\cdot 7H_2O$ (20 ml; Flemström *et al.*, 2002) in 95% ethanol (Kemtyl) with 95% ethanol (10 ml) saturated with commercial 2-amino-5-chloropyridin (Merck). Immediately, yellow block-shaped crystals of (I) precipitated. A well shaped crystal was chosen and glued on to a glass fibre.

Crystal data

$(C_5H_6ClN_2)_2[Mo_6Cl_{14}]$
$M_r = 1331.08$
Monoclinic, $P2_1/c$
$a = 9.186$ (6) Å
$b = 18.238$ (8) Å
$c = 9.614$ (6) Å
$\beta = 100.81$ (8)°
$V = 1582.0$ (16) Å^3
$Z = 2$
$D_x = 2.794$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5000 reflections
$\theta = 4.3$–52.0°
$\mu = 3.67$ mm^{-1}
$T = 293$ K
Blockx, yellow
0.18 × 0.18 × 0.10 mm

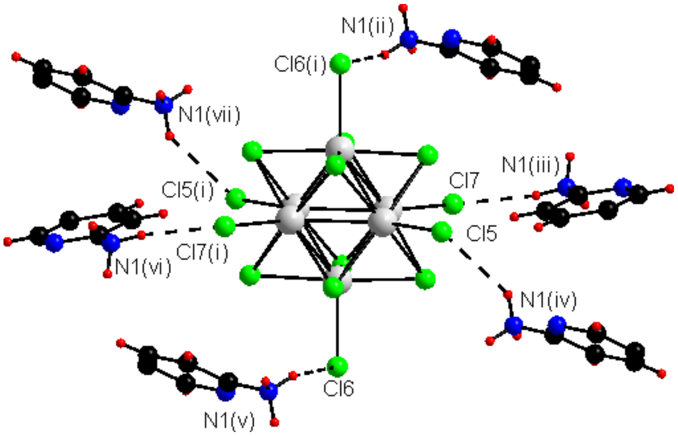


Figure 4
The N—H···Cl hydrogen bonding in (I), indicated by dashed lines, showing the 2-ammonio-5-chloropyridine arrangement around the Mo^{II} cluster. [Symmetry codes: (i) $-x, -y, 1 - z$; (ii) $x - 1, y, z$; (iii) $x - 1, \frac{1}{2} - y, \frac{1}{2} + z$; (iv) $x - 1, y, 1 + z$; (v) $1 - x, -y, 1 + z$; (vi) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (vii) $1 - x, -y, -z$..]

Data collection

Stoe IPDS area-detector diffractometer
φ scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1996)
$T_{min} = 0.486$, $T_{max} = 0.652$
12 075 measured reflections
2926 independent reflections
2486 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 26.0°$
$h = -10 \rightarrow 10$
$k = -22 \rightarrow 22$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.087$
$S = 1.18$
2926 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.04$ e Å^{-3}
$\Delta\rho_{min} = -1.64$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mo1—Mo3^i	2.6052 (13)	Mo2—Cl5	2.444 (2)
Mo1—Mo3	2.6057 (15)	Mo2—Cl2^i	2.4590 (16)
Mo1—Mo2	2.6112 (13)	Mo2—Cl3^i	2.4703 (18)
Mo1—Mo2^i	2.6151 (11)	Mo2—Cl1	2.4709 (17)
Mo2—Mo3	2.621 (2)	Mo2—Cl4	2.4735 (16)
Mo2—Mo3^i	2.6096 (17)	Mo3—Cl6	2.446 (2)
Mo1—Cl7	2.4243 (16)	Mo3—Cl4	2.4713 (15)
Mo1—Cl4	2.4754 (19)	Mo3—Cl2	2.4732 (16)
Mo1—Cl3	2.4757 (19)	Mo3—Cl1^i	2.4761 (15)
Mo1—Cl2	2.480 (2)	Mo3—Cl3^i	2.4860 (16)
Mo1—Cl1	2.495 (2)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H2···Cl5^x	0.89	2.75	3.472 (5)	140
N1—H3···Cl6^{xi}	0.89	2.61	3.359 (5)	143
N1—H1···Cl7^{xii}	0.89	2.39	3.274 (5)	171

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

The H-atoms were positioned geometrically and refined riding on their carrier atoms, with C—H = 0.93 and N—H = 0.89 Å, and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C,N), allowing for free rotation about the C···N vector of the C—NH_3^+ moiety.

Data collection: *IPDS* (Stoe & Cie, 1997); cell refinement: *IPDS*; data reduction: *IPDS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997) and *JANA*2000 (Petříček & Dušek, 2000); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997) and *JANA*2000; molecular graphics: *DIAMOND* (Bergerhoff, 1999); software used to prepare material for publication: *SHELXL*97 and *JANA*2000.

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