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Crystal structures and hydrogen-bonding analysis of a series of solvated ammonium salts of molybdenum(II) chloride clusters

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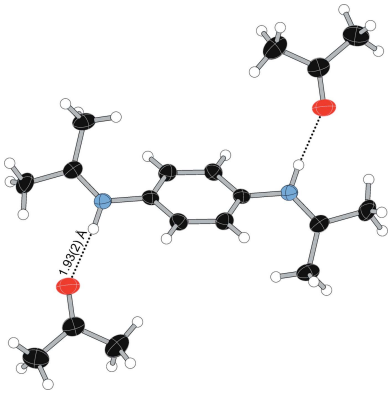
Charge-assisted hydrogen bonding plays a significant role in the crystal structures of solvates of ionic compounds, especially when the cation or cations are primary ammonium salts. We report the crystal structures of four ammonium salts of molybdenum halide cluster solvates where we observe significant hydrogen bonding between the solvent molecules and cations. The crystal structures of bis(anilinium) octa-μ_3-chlorido-hexachlorido-*octahedro*-hexamolybdate *N*,*N*-dimethylformamide tetrasolvate, $(C_6H_8N)_2[Mo_6Cl_8Cl_6]\cdot 4C_3H_7NO$, (**I**), *p*-phenylenediammonium octa-μ_3-chlorido-hexachlorido-*octahedro*-hexamolybdate *N*,*N*-dimethylformamide hexasolvate, $(C_6H_{10}N_2)[Mo_6Cl_8Cl_6]\cdot 6C_3H_7NO$, (**II**), *N*,*N*′-(1,4-phenylene)bis(propan-2-iminium) octa-μ_3-chlorido-hexachlorido-*octahedro*-hexamolybdate acetone trisolvate, $(C_{12}H_{18}N_2)[Mo_6Cl_8Cl_6]\cdot 3C_3H_6O$, (**III**), and 1,1′-dimethyl-4,4′-bipyridinium octa-μ_3-chlorido-hexachlorido-*octahedro*-hexamolybdate *N*,*N*-dimethylformamide tetrasolvate, $(C_{12}H_{14}N_2)[Mo_6Cl_8Cl_6]\cdot 4C_3H_7NO$, (**IV**), are reported and described. In (**I**), the anilinium cations and *N*,*N*-dimethylformamide (DMF) solvent molecules form a cyclic $R_4^2(8)$ hydrogen-bonded motif centered on a crystallographic inversion center with an additional DMF molecule forming a $D(2)$ interaction. The *p*-phenylenediammonium cation in (**II**) forms three $D(2)$ interactions between the three N—H bonds and three independent *N*,*N*-dimethylformamide molecules. The dication in (**III**) is a protonated Schiff base solvated by acetone molecules. Compound (**IV**) contains a methyl viologen dication with *N*,*N*-dimethylformamide molecules forming close contacts with both aromatic and methyl H atoms.

1. Chemical context

The unique photochemistry of the molybdenum and tungsten halide clusters $[M_6X_8Y_6]^{2-}$ (M = Mo, W; X, Y = Cl, Br, I) has been known for over 30 years (Maverick *et al.*, 1983) and researchers continue to explore the tunabilty of the redox potentials, crystal structures and photochemical properties of cluster-containing compounds *via* variation of the bridging and terminal ligands and the counter-ion (Mikhailov *et al.*, 2016; Saito *et al.*, 2017; Akagi *et al.*, 2018). Metal clusters, such as molybdenum halides, consist of an inner $[Mo_6X_8]^{4+}$ core surrounded by six axial ligands which are more labile than the core ligands, making the preparation of mixed-ligand cluster complexes relatively straightforward.

Charge-assisted hydrogen bonds (CAHBs) are particularly strong among hydrogen bonds (Gilli & Gilli, 2009) and can be a significant factor in the design and formation of supramolecular complexes. CAHBs have been exploited in the formation of supramolecular organic–inorganic uranyl materials (de Groot *et al.*, 2014), noncovalent macrocycles and catenanes (Pop *et al.*, 2016), molecular switches (Gurbanov *et*



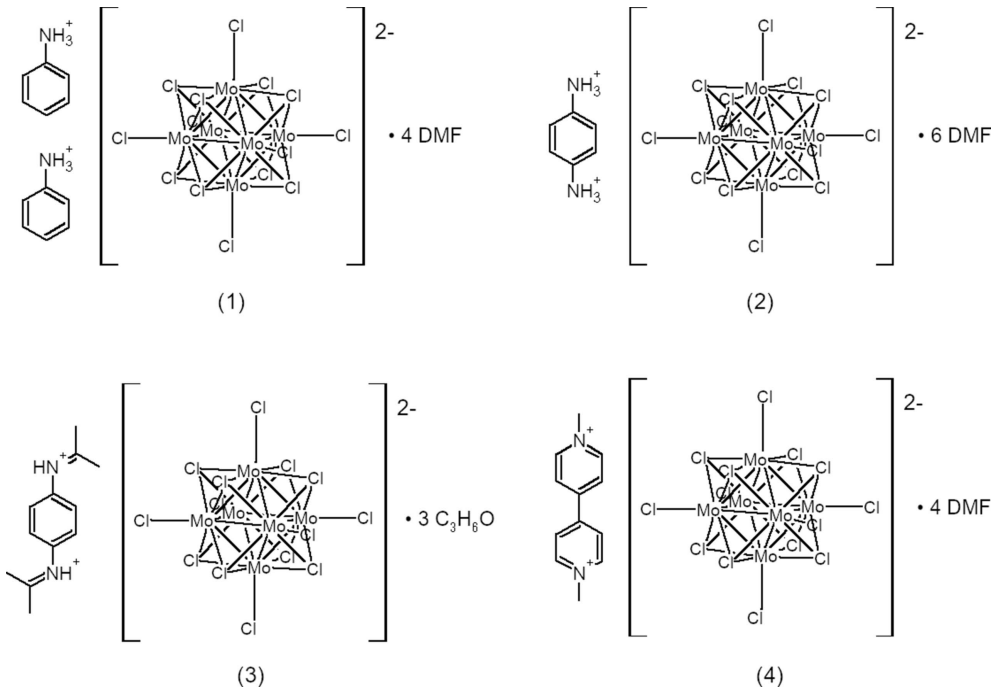


Figure 1
The structures of (I)–(IV).

al., 2017), and CAHB networks (Ward, 2009). Protonated diamines are a common motif found in hydrogen-bonded materials (Brozdowska & Chojnacki, 2017; Zick & Geiger, 2018). Examination of the nature and range of hydrogen bonding for solvates can provide information about the stability and physical properties of molecular solids (Brychczynska *et al.*, 2012).

We have prepared a series of ammonium salts of the $[Mo_6Cl_8Cl_6]^{2-}$ complex anion, each containing cations 'solvated' by either dimethylformamide or acetone through strong CAHBs.

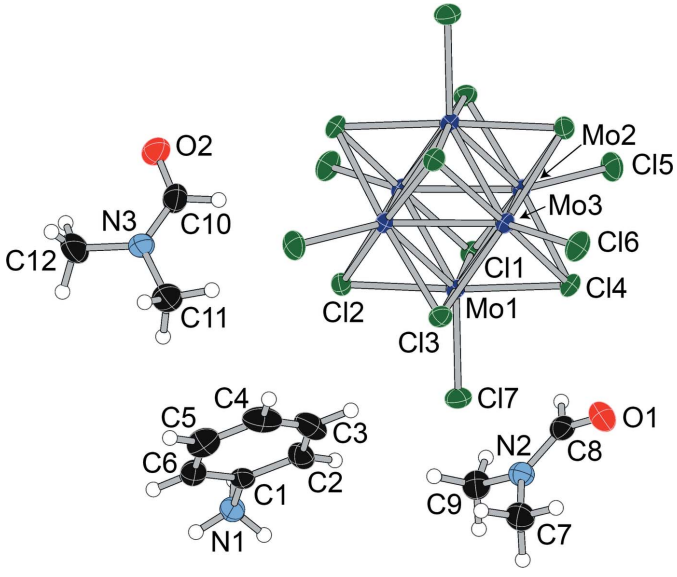


Figure 2
Displacement ellipsoid plot and atom-numbering scheme for (**I**), with ellipsoids drawn at the 50% probability level.

2. Structural commentary

The asymmetric unit of dianilinium salt (**I**) (Fig. 1) contains half a cluster unit, one anilinium cation, and two independent *N*,*N*-dimethylformamide (DMF) molecules. The structure with the atom-numbering scheme is shown in Fig. 2. The $[Mo_6Cl_8Cl_6]^{2-}$ cluster unit resides on a crystallographic inversion center, as it does in all four structures. In compound (**II**), the asymmetric unit contains half a cluster unit, half a *p*-phenylenediammonium cation, and three independent DMF molecules. The *p*-phenylenediammonium cation is disordered

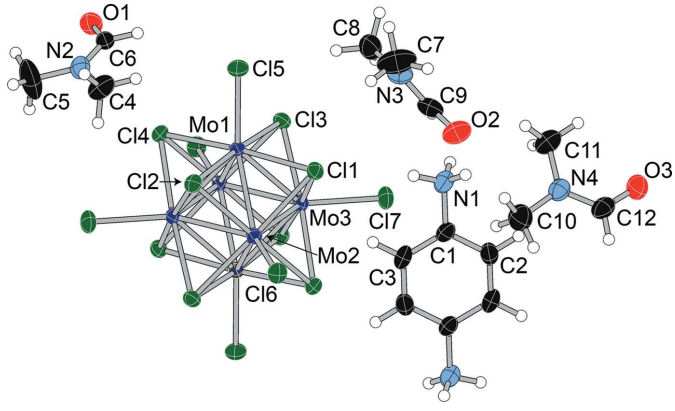


Figure 3
Displacement ellipsoid plot and atom-numbering scheme for (**II**), with ellipsoids drawn at the 50% probability level. The minor component of the disordered *p*-phenylenediammonium cation is not shown for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.89 (3)	2.01 (3)	2.827 (3)	152 (2)
N1—H1B···O2^{ii}	0.91 (3)	1.94 (3)	2.833 (3)	168 (3)
N1—H1C···O1^{iii}	0.91 (3)	1.82 (3)	2.715 (3)	166 (3)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.92 (2)	1.76 (2)	2.672 (4)	171 (4)
N1—H1B···O3^{ii}	0.93 (2)	1.79 (2)	2.710 (4)	173 (4)
N1—H1C···O1^{iii}	0.92 (2)	1.81 (2)	2.727 (4)	175 (4)

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

over two positions (rotation of 70.6° about the N—N axis), with a refined occupancy of 0.918 (4) for the primary orientation. The structure with the atom-numbering scheme is shown in Fig. 3.

The asymmetric unit of Schiff base salt (**III**) contains half a cluster unit, half a Schiff base cation, and two independent acetone molecules. The structure with the atom-numbering scheme is shown in Fig. 4. One acetone molecule is disordered over an inversion center. The Schiff base cation, presumably formed from the reaction between a *p*-phenylenediammonium cation and two acetone molecules, shows strong similarities to the cation found in the bismuthate structure reported by Shestimerova *et al.* (2018).

For comparison, a dicationic salt incapable of conventional hydrogen bonding (methyl viologen) was prepared and structurally characterized. The asymmetric unit of (**IV**), as in

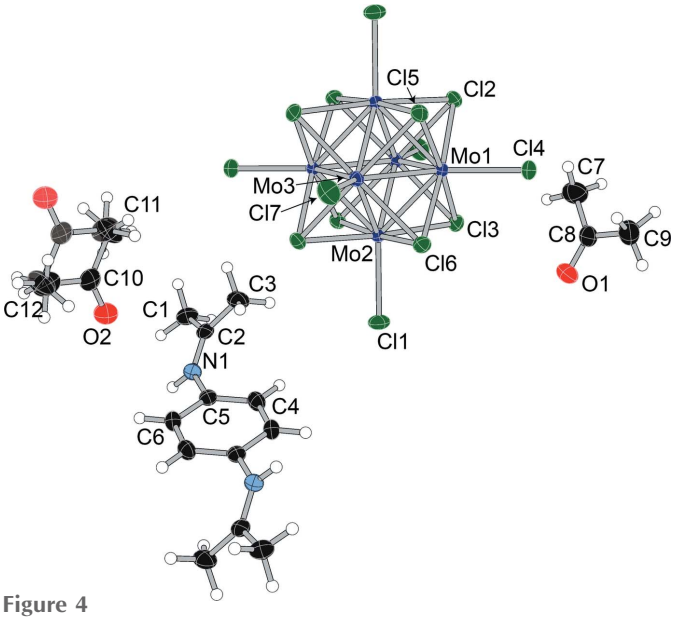


Figure 4
Displacement ellipsoid plot and atom-numbering scheme for (**III**), with ellipsoids drawn at the 50% probability level.

Table 3
Hydrogen-bond geometry (Å, °) for (**III**).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.87 (2)	1.93 (2)	2.791 (4)	172 (3)

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

Table 4
Hydrogen-bond geometry (Å, °) for (**IV**).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O1^{i}	0.95	2.23	3.063 (4)	145
C6—H6C···O2^{ii}	0.98	2.31	3.088 (4)	136

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

the other structures, contains half of the cluster unit, half of the methyl viologen dication, and two independent DMF molecules. The structure with the atom-numbering scheme is shown in Fig. 5.

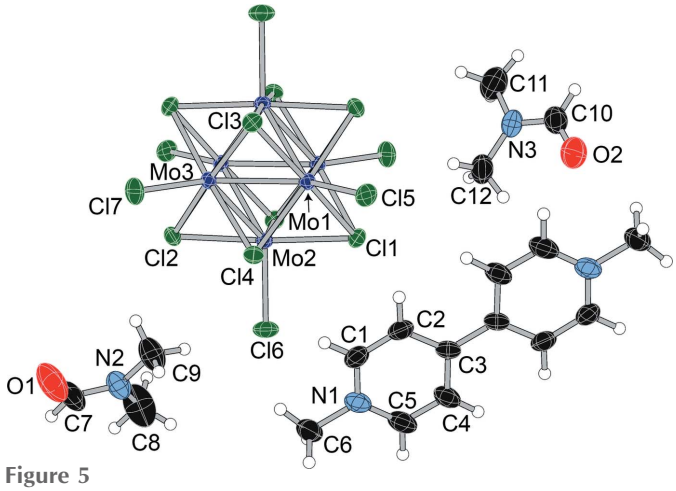


Figure 5
Displacement ellipsoid plot and atom-numbering scheme for (**IV**), with ellipsoids drawn at the 50% probability level.

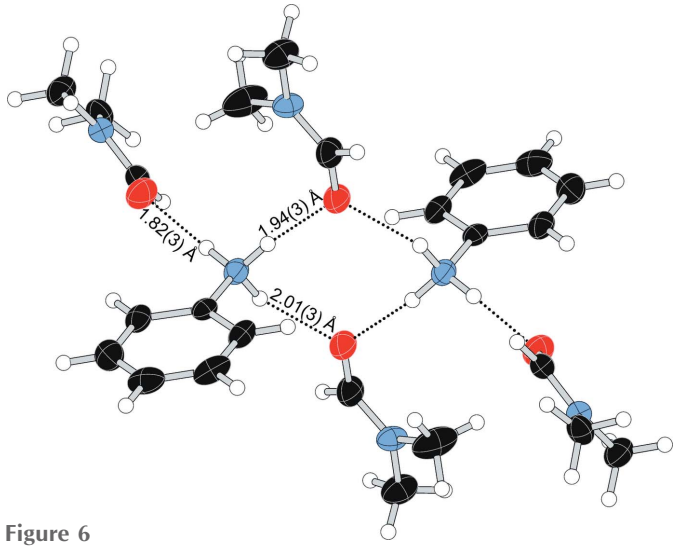


Figure 6
The cationic hydrogen-bonded dimer formed by anilinium cations and DMF molecules in (**I**).

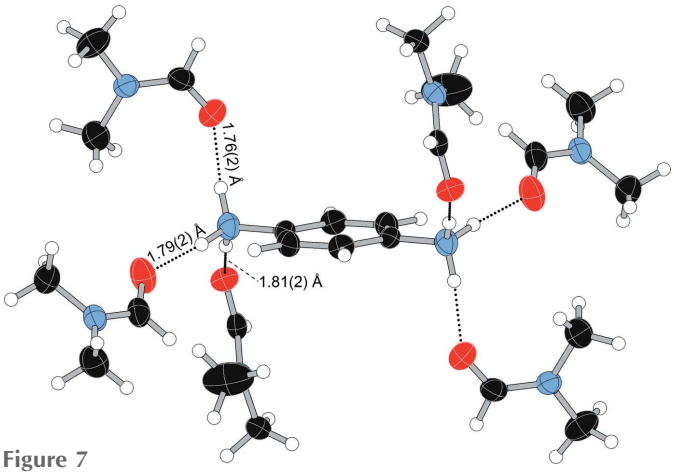


Figure 7
Hydrogen bonding in the DMF-solvated *p*-phenylenediammonium dication in (**II**). The minor component of the disordered *p*-phenylenediammonium cation is not shown for clarity.

3. Hydrogen-bonding analysis

In compound (**I**), the anilinium cation and DMF molecules form a cyclic $R_4^2(8)$ hydrogen-bonded motif centered on a crystallographic inversion center, with an additional DMF forming a $D(2)$ interaction, as illustrated in Fig. 6. Although similar to some motifs discussed by Loehlin & Okasako (2007), the hydrogen-bonding network in (**I**) does not represent an example of saturated hydrogen bonding, as one DMF molecule has an additional lone pair that is not involved in hydrogen bonding (Table 1). The DMF molecules in compound (**II**) form three unique $D(2)$ interactions with the three N—H bonds on each end of the *p*-phenylenediammonium cations, as shown in Fig. 7 (Table 2). In compound (**III**), one acetone molecule forms a hydrogen-bonding interaction with the N—H group of the Schiff base, as shown in Fig. 8 (Table 3).

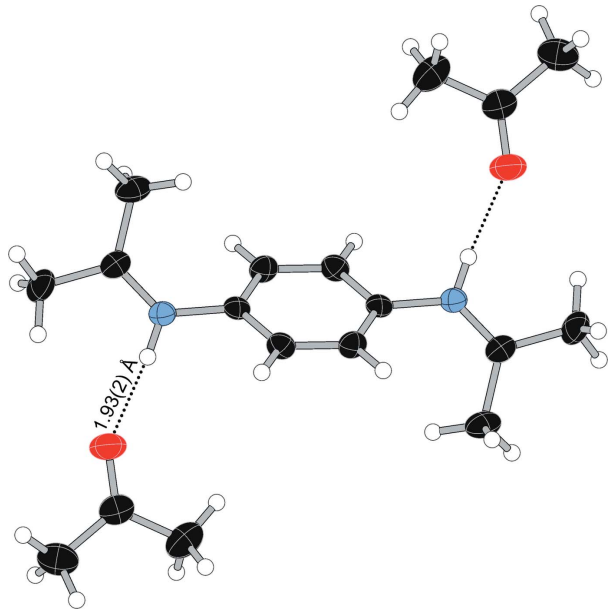


Figure 8
Hydrogen bonding in the acetone-solvated Schiff base dication in (**III**).

In spite of the lack of conventional hydrogen bonding in compound (**IV**), the methyl viologen cation forms several C—H···O contacts, with the O atoms of the two independent DMF molecules forming close contacts with the H atoms of the aromatic ring (O···H = 2.23 Å) and the methyl group (O···H = 2.31 Å) (Table 4).

Analysis of the hydrogen bonding and close contacts *via* Hirshfeld surfaces and fingerprint plots was conducted using *CrystalExplorer* (Spackman & Jayatilaka, 2009) and the results are shown in Fig. 9. Compound (**II**) has the strongest hydrogen-bonding interactions, with similar, but slightly weaker, interactions for (**I**) and (**III**). All four compounds show very similar H(cation)···Cl(cluster anion) interactions.

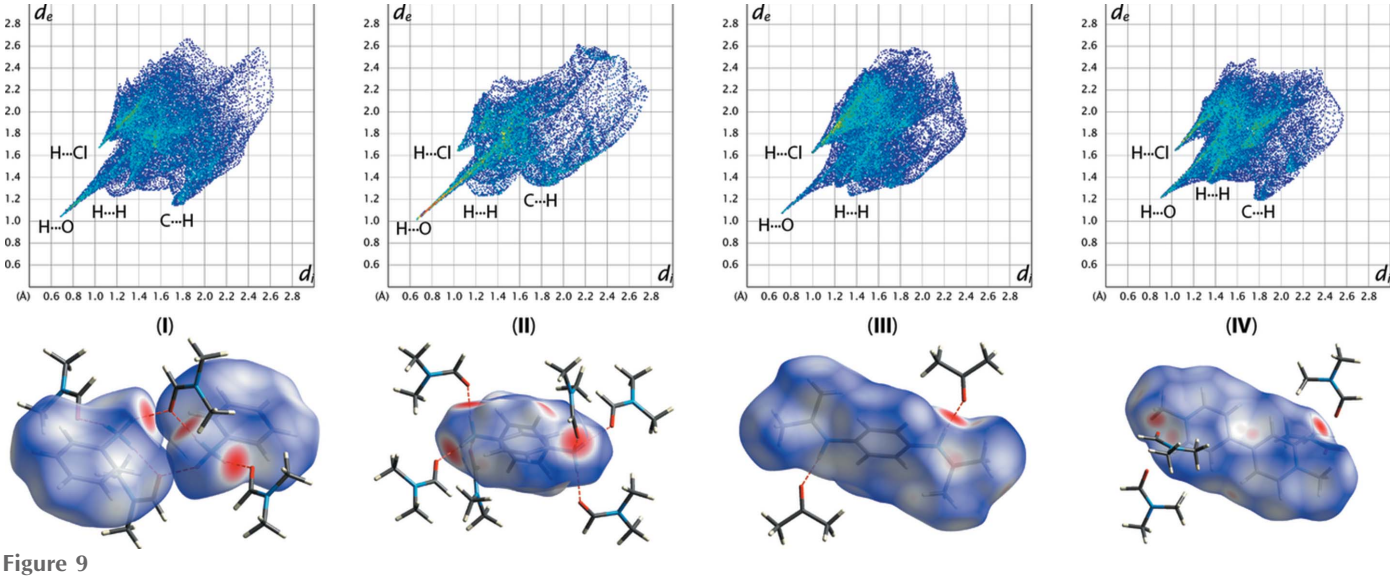


Figure 9
Fingerprint plots and Hirshfeld surfaces for (**I**)–(**IV**). For (**II**), only the major component of the disordered *p*-phenylenediammonium cation was included in the generation of the fingerprint plot.

Table 5
Experimental details.

	(**I**)	(**II**)	(**III**)	(**IV**)
Crystal data				
Chemical formula	$(C_6H_8N)_2[Mo_6Cl_8Cl_6]\cdot 4C_3H_7NO$	$(C_6H_{10}N_2)[Mo_6Cl_8Cl_6]\cdot 6C_3H_7NO$	$(C_{12}H_{18}N_2)[Mo_6Cl_8Cl_6]\cdot 3C_3H_6O$	$(C_{12}H_{14}N_2)[Mo_6Cl_8Cl_6]\cdot 4C_3H_7NO$
M_r	1552.59	1620.67	1436.46	1550.57
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	200	200	200	200
a, b, c (Å)	9.9813 (11), 10.6074 (13), 12.1686 (15)	10.1752 (16), 10.3227 (16), 13.736 (2)	9.451 (2), 11.236 (3), 11.712 (3)	9.8252 (11), 10.0933 (11), 12.6319 (15)
α, β, γ (°)	104.606 (3), 90.709 (3), 103.146 (3)	95.204 (4), 111.483 (4), 101.973 (4)	64.933 (6), 71.174 (6), 75.440 (6)	107.395 (3), 91.881 (3), 93.309 (3)
V (Å^3)	1210.7 (3)	1291.1 (3)	1056.7 (5)	1191.8 (2)
Z	1	1	1	1
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.32	2.18	2.64	2.35
Crystal size (mm)	0.48 × 0.46 × 0.12	0.50 × 0.13 × 0.13	0.55 × 0.33 × 0.20	0.32 × 0.30 × 0.28
Data collection				
Diffractometer	Bruker SMART X2S benchtop	Bruker SMART X2S benchtop	Bruker SMART X2S benchtop	Bruker SMART X2S benchtop
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)	Multi-scan (*SADABS*; Bruker, 2012)	Multi-scan (*SADABS*; Bruker, 2012)	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.498, 0.745	0.552, 0.745	0.490, 0.745	0.815, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11709, 4245, 3834	12459, 4504, 3666	10036, 3692, 3220	11498, 4187, 3743
R_{int}	0.026	0.035	0.030	0.024
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.597	0.595	0.598	0.597
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.019, 0.046, 1.07	0.026, 0.062, 1.03	0.025, 0.068, 1.05	0.020, 0.049, 1.02
No. of reflections	4245	4504	3692	4187
No. of parameters	258	285	235	250
No. of restraints	0	144	13	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.54	0.66, −0.56	0.96, −0.82	0.42, −0.44

Computer programs: *APEX2* (Bruker, 2012), *SAINT* (Bruker, 2012), *SHELXS* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015), *CrystalMaker* (Palmer, 2019), *CrystalExplorer* (Spackman & Jayatilaka, 2009), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

The C—H···O contacts in (**IV**), especially with the aromatic C—H group of the methyl viologen, can be clearly identified on the Hirshfeld surface.

4. Database survey

Interest in molybdenum(II) halide clusters and related compounds have led to numerous structural studies, with 45 entries in the Cambridge Structural Database (CSD, Version 5.40; Groom *et al.*, 2016) containing the $[Mo_6Cl_{14}]^{2-}$ dianion and almost 200 structures containing the $[Mo_6X_8]^{4+}$ core. Similarly, one can find over 50 structures in the Inorganic Crystal Structure Database (ICSD, Version 4.2.0; Hellenbrandt, 2004) containing the same molybdenum halide core structure. The structures of the $[Mo_6Cl_{14}]^{2-}$ cluster anions in this study are unremarkable and do not differ significantly from previous studies.

The anilinium cluster dihydrate structure published by Flemström (2003) has some similarities to (**I**). In that structure, the three N—H bonds of the anilinium cation serve as hydrogen-bond donors to one water molecule (hydrate) and two terminal Cl atoms on two discrete cluster anions. The N—H···Cl interactions create $C_4^4(15)$ chains. The water molecules create $R_4^4(14)$ rings involving two water molecules and two cluster units, as well as $C_2^2(8)$ and $C_2^2(7)$ chains.

While DMF-solvated ammonium salts appear to be relatively uncommon, a series of molybdenum halide cluster salts have been prepared with dimethylformamide-coordinated metal cations serving as the counter-cation (Khutornoi *et al.*, 2002; Kozhomuratova *et al.*, 2007; Liu *et al.*, 2006). The complexes prepared and characterized include the $[Mo_6Cl_8Cl_6]^{2-}$, $[Mo_6Br_8Cl_6]^{2-}$, and $[Mo_6Br_8(NCS)_6]^{2-}$ cluster anions as salts with $[M(DMF)]^{2+}$ cations, where M = Ca^{2+}, Mn^{2+}, and Co^{2+}. A similar set of rhenium chalcogenide cluster salts with DMF-solvated calcium and a series of lanthanides has been prepared by Perruchas *et al.* (2002) and Yarovoi *et al.* (2006).

A separate search of the CSD for structures with similar hydrogen-bonded networks containing anilinium and *p*-phenylenediammonium cations yielded a large number of hits due to their propensity for forming significant hydrogen-bonding networks. In the structure of anilinium dihydrogen phosphate (Kaman *et al.*, 2012), each of the three independent

ammonium groups forms four different hydrogen bonds to the O atoms of nearby dihydrogen phosphate moieties. A very similar set of hydrogen-bonding interactions and layered organic/inorganic structural arrangements are found in the structures of *p*-phenylenediammonium bis(dihydrogen phosphate) (Mrad *et al.*, 2006*a*) and *p*-phenylenediammonium dihydrogen diphosphate (Mrad *et al.*, 2006*b*). While less closely related to the current report, the structure of *p*-phenylenediammonium tetrachloridozincate(II) (Bringley & Rajeswaran, 2006) also displays alternating organic and inorganic layers and strong hydrogen bonding between the tetrachloridozinc(II) anions and the *p*-phenylenediammonium cations.

A dimethyl sulfoxide (DMSO)-solvated *p*-phenylenediammonium salt of an iodidobismuthate reported by Shestimerova *et al.* (2018) displays strong structural similarities to (**II**) in the way the DMSO solvates the *p*-phenylenediammonium cation. Three unique DMSO molecules also form *D*(2) interactions with each end of the *p*-phenylenediammonium. One of the three DMSO molecules simultaneously coordinates to one of the Bi atoms.

5. Synthesis and crystallization

All reagents were used as received from the manufacturer.

5.1. Cluster synthesis, metathesis, and crystallization of (I), (II), and (IV)

The hydronium salt of the $[Mo_6Cl_8Cl_6]^{2-}$ anion was prepared by the method of Hay *et al.* (2004) and then metathesized to the appropriate ammonium salt by combining an ethanolic solution of $(H_3O)_2[Mo_6Cl_8Cl_6]\cdot 6H_2O$ with a slight stoichiometric excess (~2.5 times) of the respective ammonium chloride salt (anilinium chloride, *p*-phenylenediamine hydrochloride, and methyl viologen dichloride). The bright-yellow precipitate that formed was isolated by filtration and the product was recrystallized by vapor diffusion of diethyl ether into a dimethylformamide solution of the cluster salt.

5.2. Synthesis and crystallization of Schiff base salt (III)

The cluster in compound (**III**) was prepared and metathesized to the diammonium salt *via* the same procedure as above using the *p*-phenylenediammonium chloride to isolate a yellow precipitate. The salt was then redissolved in acetone and allowed to evaporate. The acetone inadvertently formed a Schiff base dication in a reaction with the *p*-phenylenediammonium cation (Kolb & Bahadir, 1994).

6. Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 5. All H atoms were located in a difference map. All carbon-bonded H atoms were placed in idealized positions using a riding model, with aromatic and amide C—H = 0.95 Å and methyl C—H = 0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(C) (aromatic and amide) or U_{iso}(H) = 1.5U_{eq}(C) (methyl). The positions of all H atoms bonded to N atoms were refined with N—H distances restrained to 0.91 (2) (NH_3) or 0.88 (2) Å (Schiff base), and with U_{iso}(H) = 1.5U_{eq}(N).

All four structures were refined in the space group $P\overline{1}$ and the $[Mo_6Cl_{14}]^{2-}$ dianion sits on an inversion center in every case. The dications in (**II**), (**III**), and (**IV**) are also each located on an inversion center. The *p*-phenylenediammonium cation in (**II**) is disordered over two orientations with an occupancy of 0.918 (4) for the major component. One of the two acetone molecules in (**III**) is disordered over an inversion center.

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supporting information

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Crystal structures and hydrogen-bonding analysis of a series of solvated ammonium salts of molybdenum(II) chloride clusters

Dean H. Johnston and Ikponmwosa Agho

Computing details

For all structures, data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015); molecular graphics: *CrystalMaker* (Palmer, 2019) and *CrystalExplorer* (Spackman & Jayatilaka, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

Bis(anilinium) octa-μ_3-chlorido-hexachlorido-*octahedro*-hexamolybdate *N*,*N*-dimethylformamide tetrasolvate (**1**)

Crystal data

$(C_6H_8N)_2[Mo_6Cl_8Cl_6]\cdot 4C_3H_7NO$
$M_r = 1552.59$
Triclinic, $P\overline{1}$
$a = 9.9813$ (11) Å
$b = 10.6074$ (13) Å
$c = 12.1686$ (15) Å
$\alpha = 104.606$ (3)°
$\beta = 90.709$ (3)°
$\gamma = 103.146$ (3)°
$V = 1210.7$ (3) Å^3
$Z = 1$
$F(000) = 752$
$D_x = 2.129$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7006 reflections
$\theta = 2.3$–$25.1°$
$\mu = 2.32$ mm^{-1}
$T = 200$ K
Plate, clear orangish yellow
0.48 × 0.46 × 0.12 mm

Data collection

Bruker SMART X2S benchtop diffractometer
Radiation source: sealed microfocus source, XOS X-beam microfocus source
Graphite monochromator
Detector resolution: 8.3330 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
$T_{min} = 0.498$, $T_{max} = 0.745$
11709 measured reflections
4245 independent reflections
3834 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 25.1°$, $\theta_{min} = 2.3°$
$h = -11\rightarrow11$
$k = -12\rightarrow12$
$l = -14\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.046$
$S = 1.07$
4245 reflections
258 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0123P)^2 + 0.5808P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0096 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo1	0.33000 (2)	0.50142 (2)	0.43962 (2)	0.01755 (7)
Mo2	0.42505 (2)	0.33816 (2)	0.52988 (2)	0.01753 (7)
Mo3	0.45872 (2)	0.58737 (2)	0.64151 (2)	0.01766 (7)
Cl1	0.30660 (6)	0.26453 (5)	0.33717 (5)	0.02208 (12)
Cl2	0.44848 (6)	0.57046 (5)	0.27978 (5)	0.02298 (13)
Cl3	0.37196 (6)	0.73926 (5)	0.55008 (5)	0.02352 (13)
Cl4	0.22876 (5)	0.43149 (5)	0.60532 (5)	0.02318 (13)
Cl5	0.32774 (6)	0.12583 (6)	0.57003 (5)	0.03234 (15)
Cl6	0.39836 (6)	0.69501 (6)	0.82888 (5)	0.03331 (15)
Cl7	0.10839 (6)	0.50315 (6)	0.35713 (5)	0.03118 (14)
N1	0.1138 (2)	0.6861 (2)	−0.02394 (19)	0.0291 (5)
H1A	0.144 (3)	0.612 (3)	−0.048 (2)	0.044*
H1B	0.026 (3)	0.651 (3)	−0.010 (2)	0.044*
H1C	0.111 (3)	0.732 (3)	−0.078 (2)	0.044*
C1	0.1999 (2)	0.7715 (2)	0.0778 (2)	0.0262 (5)
C2	0.1890 (3)	0.7327 (3)	0.1777 (2)	0.0352 (6)
H2	0.125907	0.652029	0.180955	0.042*
C3	0.2713 (3)	0.8128 (3)	0.2728 (2)	0.0439 (7)
H3	0.264848	0.787222	0.342295	0.053*
C4	0.3621 (3)	0.9288 (3)	0.2678 (3)	0.0454 (7)
H4	0.418475	0.983410	0.333788	0.055*
C5	0.2894 (3)	0.8879 (2)	0.0709 (2)	0.0326 (6)
H5	0.294613	0.913873	0.001580	0.039*
C6	0.3718 (3)	0.9666 (3)	0.1671 (2)	0.0411 (7)
H6	0.435330	1.046973	0.163742	0.049*
O1	0.0921 (2)	0.85206 (19)	0.84303 (16)	0.0431 (5)
N2	0.0472 (2)	0.88716 (19)	0.67193 (17)	0.0287 (5)
C7	0.0074 (3)	1.0112 (3)	0.7195 (2)	0.0408 (7)
H7A	0.004355	1.025920	0.802189	0.061*
H7B	−0.083924	1.006277	0.685843	0.061*
H7C	0.074846	1.085755	0.702938	0.061*
C8	0.0860 (2)	0.8186 (3)	0.7380 (2)	0.0325 (6)
H8	0.111034	0.737608	0.702115	0.039*

C9	0.0410 (3)	0.8403 (3)	0.5486 (2)	0.0364 (6)
H9A	0.057014	0.749801	0.526886	0.055*
H9B	0.111888	0.901063	0.519142	0.055*
H9C	−0.050269	0.838466	0.516599	0.055*
O2	0.85598 (19)	0.57853 (18)	0.05179 (17)	0.0418 (5)
N3	0.7318 (2)	0.7329 (2)	0.05377 (17)	0.0320 (5)
C10	0.7698 (3)	0.6417 (3)	0.0921 (2)	0.0397 (7)
H10	0.727510	0.621423	0.157083	0.048*
C11	0.6338 (3)	0.8071 (3)	0.1095 (2)	0.0460 (7)
H11A	0.607218	0.779492	0.178708	0.069*
H11B	0.551612	0.788223	0.057583	0.069*
H11C	0.676785	0.903485	0.129568	0.069*
C12	0.7879 (4)	0.7684 (4)	−0.0466 (3)	0.0621 (10)
H12A	0.866691	0.846144	−0.023497	0.093*
H12B	0.716767	0.790546	−0.089431	0.093*
H12C	0.818232	0.692417	−0.094617	0.093*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01465 (11)	0.01876 (11)	0.01888 (11)	0.00353 (8)	0.00272 (8)	0.00463 (8)
Mo2	0.01627 (11)	0.01691 (10)	0.01871 (11)	0.00234 (8)	0.00337 (8)	0.00474 (8)
Mo3	0.01646 (12)	0.01852 (11)	0.01703 (11)	0.00379 (8)	0.00436 (8)	0.00307 (8)
Cl1	0.0205 (3)	0.0202 (3)	0.0218 (3)	0.0011 (2)	0.0008 (2)	0.0022 (2)
Cl2	0.0233 (3)	0.0256 (3)	0.0208 (3)	0.0045 (2)	0.0022 (2)	0.0086 (2)
Cl3	0.0231 (3)	0.0211 (3)	0.0272 (3)	0.0086 (2)	0.0041 (2)	0.0046 (2)
Cl4	0.0169 (3)	0.0267 (3)	0.0247 (3)	0.0029 (2)	0.0066 (2)	0.0066 (2)
Cl5	0.0345 (3)	0.0242 (3)	0.0369 (3)	−0.0020 (3)	0.0024 (3)	0.0134 (3)
Cl6	0.0350 (4)	0.0359 (3)	0.0241 (3)	0.0078 (3)	0.0112 (3)	−0.0010 (3)
Cl7	0.0199 (3)	0.0356 (3)	0.0397 (3)	0.0061 (3)	−0.0019 (3)	0.0132 (3)
N1	0.0306 (12)	0.0255 (11)	0.0341 (12)	0.0094 (10)	0.0055 (10)	0.0104 (10)
C1	0.0273 (13)	0.0252 (12)	0.0296 (13)	0.0132 (11)	0.0041 (11)	0.0072 (10)
C2	0.0437 (16)	0.0307 (13)	0.0379 (15)	0.0162 (12)	0.0069 (13)	0.0142 (12)
C3	0.063 (2)	0.0469 (17)	0.0309 (15)	0.0328 (16)	0.0019 (14)	0.0097 (13)
C4	0.0505 (18)	0.0396 (16)	0.0433 (17)	0.0259 (15)	−0.0084 (14)	−0.0073 (13)
C5	0.0372 (15)	0.0263 (13)	0.0383 (15)	0.0145 (11)	0.0117 (12)	0.0089 (11)
C6	0.0412 (17)	0.0277 (14)	0.0495 (18)	0.0115 (12)	0.0063 (14)	−0.0016 (13)
O1	0.0496 (12)	0.0479 (11)	0.0392 (11)	0.0149 (10)	0.0045 (9)	0.0219 (9)
N2	0.0270 (11)	0.0275 (11)	0.0326 (11)	0.0065 (9)	0.0007 (9)	0.0094 (9)
C7	0.0462 (17)	0.0355 (15)	0.0465 (17)	0.0170 (13)	0.0084 (14)	0.0144 (13)
C8	0.0248 (14)	0.0313 (13)	0.0428 (16)	0.0048 (11)	0.0040 (12)	0.0140 (12)
C9	0.0334 (15)	0.0362 (14)	0.0332 (14)	−0.0008 (12)	−0.0040 (12)	0.0062 (12)
O2	0.0359 (11)	0.0345 (10)	0.0614 (13)	0.0134 (9)	0.0149 (10)	0.0192 (9)
N3	0.0309 (12)	0.0407 (12)	0.0301 (11)	0.0169 (10)	0.0066 (10)	0.0120 (10)
C10	0.0391 (16)	0.0390 (15)	0.0470 (17)	0.0112 (13)	0.0145 (13)	0.0197 (13)
C11	0.0527 (19)	0.0527 (18)	0.0427 (17)	0.0301 (15)	0.0149 (15)	0.0142 (14)
C12	0.073 (2)	0.099 (3)	0.0438 (18)	0.053 (2)	0.0248 (17)	0.0425 (19)

Geometric parameters (Å, º)

Mo1—Mo2^{i}	2.6034 (4)	C3—H3	0.9500
Mo1—Mo2	2.6051 (3)	C3—C4	1.368 (4)
Mo1—Mo3^{i}	2.6068 (3)	C4—H4	0.9500
Mo1—Mo3	2.6032 (4)	C4—C6	1.381 (4)
Mo1—Cl1	2.4636 (6)	C5—H5	0.9500
Mo1—Cl2	2.4697 (6)	C5—C6	1.383 (4)
Mo1—Cl3	2.4782 (6)	C6—H6	0.9500
Mo1—Cl4	2.4700 (6)	O1—C8	1.234 (3)
Mo1—Cl7	2.4235 (6)	N2—C7	1.444 (3)
Mo2—Mo3	2.5922 (4)	N2—C8	1.319 (3)
Mo2—Mo3^{i}	2.6065 (3)	N2—C9	1.453 (3)
Mo2—Cl1	2.4687 (6)	C7—H7A	0.9800
Mo2—Cl2^{i}	2.4772 (6)	C7—H7B	0.9800
Mo2—Cl3^{i}	2.4756 (6)	C7—H7C	0.9800
Mo2—Cl4	2.4758 (6)	C8—H8	0.9500
Mo2—Cl5	2.4138 (6)	C9—H9A	0.9800
Mo3—Cl1^{i}	2.4754 (6)	C9—H9B	0.9800
Mo3—Cl2^{i}	2.4639 (6)	C9—H9C	0.9800
Mo3—Cl3	2.4695 (6)	O2—C10	1.239 (3)
Mo3—Cl4	2.4652 (6)	N3—C10	1.299 (3)
Mo3—Cl6	2.4288 (6)	N3—C11	1.461 (3)
N1—H1A	0.89 (3)	N3—C12	1.449 (3)
N1—H1B	0.91 (3)	C10—H10	0.9500
N1—H1C	0.91 (3)	C11—H11A	0.9800
N1—C1	1.465 (3)	C11—H11B	0.9800
C1—C2	1.376 (3)	C11—H11C	0.9800
C1—C5	1.371 (3)	C12—H12A	0.9800
C2—H2	0.9500	C12—H12B	0.9800
C2—C3	1.378 (4)	C12—H12C	0.9800
Mo2^{i}—Mo1—Mo2	89.753 (11)	Cl3—Mo3—Mo1^{i}	118.261 (15)
Mo2—Mo1—Mo3^{i}	60.013 (9)	Cl3—Mo3—Mo2^{i}	58.306 (14)
Mo2^{i}—Mo1—Mo3^{i}	59.675 (10)	Cl3—Mo3—Mo2	118.588 (17)
Mo3—Mo1—Mo2^{i}	60.080 (8)	Cl3—Mo3—Cl1^{i}	89.67 (2)
Mo3—Mo1—Mo2	59.698 (9)	Cl4—Mo3—Mo1^{i}	118.636 (17)
Mo3—Mo1—Mo3^{i}	89.786 (11)	Cl4—Mo3—Mo1	58.254 (14)
Cl1—Mo1—Mo2^{i}	118.020 (15)	Cl4—Mo3—Mo2	58.556 (14)
Cl1—Mo1—Mo2	58.214 (16)	Cl4—Mo3—Mo2^{i}	118.205 (16)
Cl1—Mo1—Mo3	117.899 (15)	Cl4—Mo3—Cl1^{i}	175.592 (19)
Cl1—Mo1—Mo3^{i}	58.365 (14)	Cl4—Mo3—Cl3	89.93 (2)
Cl1—Mo1—Cl2	89.683 (19)	Cl6—Mo3—Mo1	134.228 (19)
Cl1—Mo1—Cl3	175.251 (19)	Cl6—Mo3—Mo1^{i}	135.485 (18)
Cl1—Mo1—Cl4	89.963 (19)	Cl6—Mo3—Mo2^{i}	137.228 (18)
Cl2—Mo1—Mo2^{i}	58.387 (15)	Cl6—Mo3—Mo2	132.798 (17)
Cl2—Mo1—Mo2	117.999 (16)	Cl6—Mo3—Cl1^{i}	94.40 (2)
Cl2—Mo1—Mo3	118.444 (17)	Cl6—Mo3—Cl2^{i}	90.84 (2)

Cl2—Mo1—Mo3^{i}	57.995 (14)	Cl6—Mo3—Cl3	93.06 (2)
Cl2—Mo1—Cl3	90.38 (2)	Cl6—Mo3—Cl4	90.01 (2)
Cl2—Mo1—Cl4	175.624 (19)	Mo1—Cl1—Mo2	63.764 (15)
Cl3—Mo1—Mo2^{i}	58.247 (14)	Mo1—Cl1—Mo3^{i}	63.713 (15)
Cl3—Mo1—Mo2	117.771 (17)	Mo2—Cl1—Mo3^{i}	63.630 (15)
Cl3—Mo1—Mo3^{i}	117.898 (15)	Mo1—Cl2—Mo2^{i}	63.507 (16)
Cl3—Mo1—Mo3	58.092 (14)	Mo3^{i}—Cl2—Mo1	63.792 (15)
Cl4—Mo1—Mo2^{i}	118.144 (16)	Mo3^{i}—Cl2—Mo2^{i}	63.287 (16)
Cl4—Mo1—Mo2	58.325 (14)	Mo2^{i}—Cl3—Mo1	63.408 (14)
Cl4—Mo1—Mo3^{i}	118.319 (15)	Mo3—Cl3—Mo1	63.489 (16)
Cl4—Mo1—Mo3	58.077 (16)	Mo3—Cl3—Mo2^{i}	63.616 (15)
Cl4—Mo1—Cl3	89.62 (2)	Mo1—Cl4—Mo2	63.569 (15)
Cl7—Mo1—Mo2	135.582 (17)	Mo3—Cl4—Mo1	63.669 (15)
Cl7—Mo1—Mo2^{i}	134.650 (17)	Mo3—Cl4—Mo2	63.286 (16)
Cl7—Mo1—Mo3	135.962 (17)	H1A—N1—H1B	101 (2)
Cl7—Mo1—Mo3^{i}	134.249 (18)	H1A—N1—H1C	114 (3)
Cl7—Mo1—Cl1	92.00 (2)	H1B—N1—H1C	109 (2)
Cl7—Mo1—Cl2	91.25 (2)	C1—N1—H1A	108.4 (18)
Cl7—Mo1—Cl3	92.75 (2)	C1—N1—H1B	113.6 (18)
Cl7—Mo1—Cl4	93.12 (2)	C1—N1—H1C	111.0 (18)
Mo1^{i}—Mo2—Mo1	90.246 (11)	C2—C1—N1	119.1 (2)
Mo1^{i}—Mo2—Mo3^{i}	59.955 (8)	C5—C1—N1	119.1 (2)
Mo1—Mo2—Mo3^{i}	60.026 (9)	C5—C1—C2	121.8 (2)
Mo3—Mo2—Mo1^{i}	60.228 (8)	C1—C2—H2	120.6
Mo3—Mo2—Mo1	60.114 (10)	C1—C2—C3	118.8 (3)
Mo3—Mo2—Mo3^{i}	90.033 (11)	C3—C2—H2	120.6
Cl1—Mo2—Mo1	58.022 (15)	C2—C3—H3	119.7
Cl1—Mo2—Mo1^{i}	118.250 (15)	C4—C3—C2	120.5 (3)
Cl1—Mo2—Mo3^{i}	58.310 (15)	C4—C3—H3	119.7
Cl1—Mo2—Mo3	118.122 (15)	C3—C4—H4	120.0
Cl1—Mo2—Cl2^{i}	175.448 (18)	C3—C4—C6	120.0 (3)
Cl1—Mo2—Cl3^{i}	89.68 (2)	C6—C4—H4	120.0
Cl1—Mo2—Cl4	89.71 (2)	C1—C5—H5	120.7
Cl2^{i}—Mo2—Mo1^{i}	58.106 (14)	C1—C5—C6	118.6 (3)
Cl2^{i}—Mo2—Mo1	118.200 (16)	C6—C5—H5	120.7
Cl2^{i}—Mo2—Mo3	58.105 (14)	C4—C6—C5	120.2 (3)
Cl2^{i}—Mo2—Mo3^{i}	118.038 (16)	C4—C6—H6	119.9
Cl3^{i}—Mo2—Mo1^{i}	58.346 (16)	C5—C6—H6	119.9
Cl3^{i}—Mo2—Mo1	118.097 (16)	C7—N2—C9	117.3 (2)
Cl3^{i}—Mo2—Mo3	118.549 (15)	C8—N2—C7	121.2 (2)
Cl3^{i}—Mo2—Mo3^{i}	58.076 (14)	C8—N2—C9	121.5 (2)
Cl3^{i}—Mo2—Cl2^{i}	90.26 (2)	N2—C7—H7A	109.5
Cl3^{i}—Mo2—Cl4	175.629 (18)	N2—C7—H7B	109.5
Cl4—Mo2—Mo1	58.105 (15)	N2—C7—H7C	109.5
Cl4—Mo2—Mo1^{i}	118.367 (16)	H7A—C7—H7B	109.5
Cl4—Mo2—Mo3	58.157 (14)	H7A—C7—H7C	109.5
Cl4—Mo2—Mo3^{i}	118.112 (15)	H7B—C7—H7C	109.5
Cl4—Mo2—Cl2^{i}	90.00 (2)	O1—C8—N2	124.7 (2)

Cl5—Mo2—Mo1^{i}	134.531 (18)	O1—C8—H8	117.6
Cl5—Mo2—Mo1	135.222 (19)	N2—C8—H8	117.6
Cl5—Mo2—Mo3^{i}	135.276 (17)	N2—C9—H9A	109.5
Cl5—Mo2—Mo3	134.689 (19)	N2—C9—H9B	109.5
Cl5—Mo2—Cl1	92.63 (2)	N2—C9—H9C	109.5
Cl5—Mo2—Cl2^{i}	91.92 (2)	H9A—C9—H9B	109.5
Cl5—Mo2—Cl3^{i}	92.04 (2)	H9A—C9—H9C	109.5
Cl5—Mo2—Cl4	92.31 (2)	H9B—C9—H9C	109.5
Mo1—Mo3—Mo1^{i}	90.214 (10)	C10—N3—C11	122.7 (2)
Mo1—Mo3—Mo2^{i}	59.965 (10)	C10—N3—C12	121.0 (2)
Mo2—Mo3—Mo1^{i}	60.098 (8)	C12—N3—C11	116.3 (2)
Mo2^{i}—Mo3—Mo1^{i}	59.961 (8)	O2—C10—N3	126.1 (3)
Mo2—Mo3—Mo1	60.188 (8)	O2—C10—H10	116.9
Mo2—Mo3—Mo2^{i}	89.968 (10)	N3—C10—H10	116.9
Cl1^{i}—Mo3—Mo1^{i}	57.923 (15)	N3—C11—H11A	109.5
Cl1^{i}—Mo3—Mo1	118.008 (15)	N3—C11—H11B	109.5
Cl1^{i}—Mo3—Mo2	118.001 (15)	N3—C11—H11C	109.5
Cl1^{i}—Mo3—Mo2^{i}	58.058 (14)	H11A—C11—H11B	109.5
Cl2^{i}—Mo3—Mo1^{i}	58.214 (14)	H11A—C11—H11C	109.5
Cl2^{i}—Mo3—Mo1	118.777 (16)	H11B—C11—H11C	109.5
Cl2^{i}—Mo3—Mo2	58.608 (16)	N3—C12—H12A	109.5
Cl2^{i}—Mo3—Mo2^{i}	118.165 (15)	N3—C12—H12B	109.5
Cl2^{i}—Mo3—Cl1^{i}	89.55 (2)	N3—C12—H12C	109.5
Cl2^{i}—Mo3—Cl3	176.071 (19)	H12A—C12—H12B	109.5
Cl2^{i}—Mo3—Cl4	90.56 (2)	H12A—C12—H12C	109.5
Cl3—Mo3—Mo1	58.419 (15)	H12B—C12—H12C	109.5
N1—C1—C2—C3	179.4 (2)	C3—C4—C6—C5	0.4 (4)
N1—C1—C5—C6	−178.9 (2)	C5—C1—C2—C3	−0.5 (4)
C1—C2—C3—C4	0.0 (4)	C7—N2—C8—O1	0.0 (4)
C1—C5—C6—C4	−0.8 (4)	C9—N2—C8—O1	−179.2 (2)
C2—C1—C5—C6	0.9 (4)	C11—N3—C10—O2	−177.1 (3)
C2—C3—C4—C6	0.1 (4)	C12—N3—C10—O2	1.5 (5)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{ii}	0.89 (3)	2.01 (3)	2.827 (3)	152 (2)
N1—H1*B*···O2^{iii}	0.91 (3)	1.94 (3)	2.833 (3)	168 (3)
N1—H1*C*···O1^{iv}	0.91 (3)	1.82 (3)	2.715 (3)	166 (3)

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

p-Phenylenediammonium octa-μ_3-chlorido-hexachlorido-*octahedro*-hexamolybdate *N*,*N*-dimethylformamide hexasolvate (2)

Crystal data

$(C_6H_{10}N_2)[Mo_6Cl_8Cl_6]\cdot 6C_3H_7NO$
$M_r = 1620.67$
Triclinic, $P\overline{1}$
$a = 10.1752$ (16) Å
$b = 10.3227$ (16) Å
$c = 13.736$ (2) Å
$\alpha = 95.204$ (4)°
$\beta = 111.483$ (4)°
$\gamma = 101.973$ (4)°
$V = 1291.1$ (3) Å^3
$Z = 1$
$F(000) = 790$
$D_x = 2.084$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4690 reflections
$\theta = 2.2$–$25.0°$
$\mu = 2.18$ mm^{-1}
$T = 200$ K
Needle, yellow
0.50 × 0.13 × 0.13 mm

Data collection

Bruker SMART X2S benchtop diffractometer
Radiation source: sealed microfocus source, XOS X-beam microfocus source
Graphite monochromator
Detector resolution: 8.3330 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
$T_{min} = 0.552$, $T_{max} = 0.745$
12459 measured reflections
4504 independent reflections
3666 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.4°$
$h = -12\rightarrow12$
$k = -12\rightarrow12$
$l = -16\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.062$
$S = 1.03$
4504 reflections
285 parameters
144 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0182P)^2 + 0.7035P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.66$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0019 (2)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Mo1	0.38576 (3)	0.32569 (3)	0.46504 (2)	0.01984 (9)	
Mo2	0.38226 (3)	0.53457 (3)	0.37203 (2)	0.01942 (9)	
Mo3	0.61719 (3)	0.44772 (3)	0.43759 (2)	0.01961 (9)	
Cl1	0.39098 (9)	0.31721 (8)	0.28606 (6)	0.02470 (19)	
Cl2	0.16889 (8)	0.41769 (8)	0.40411 (7)	0.02485 (19)	

Cl3	0.61432 (9)	0.25211 (8)	0.52889 (7)	0.02446 (19)	
Cl4	0.39296 (9)	0.35261 (8)	0.64765 (6)	0.02436 (19)	
Cl5	0.23637 (10)	0.09538 (8)	0.42076 (7)	0.0329 (2)	
Cl6	0.22927 (9)	0.58540 (9)	0.20362 (7)	0.0303 (2)	
Cl7	0.77350 (10)	0.38668 (9)	0.35511 (7)	0.0340 (2)	
N1	0.6572 (4)	0.7709 (3)	0.0311 (3)	0.0349 (8)	
H1A	0.593 (4)	0.816 (4)	−0.008 (3)	0.052*	
H1B	0.735 (3)	0.773 (4)	0.010 (3)	0.052*	
H1C	0.702 (4)	0.814 (4)	0.1013 (17)	0.052*	
C1	0.5771 (4)	0.6310 (4)	0.0161 (3)	0.0300 (8)	
C2	0.4838 (4)	0.5982 (4)	0.0660 (3)	0.0346 (10)	0.918 (4)
H2	0.473071	0.666286	0.111672	0.041*	0.918 (4)
C2A	0.614 (4)	0.562 (3)	0.098 (2)	0.0346 (10)	0.082 (4)
H2A	0.690135	0.603414	0.164923	0.041*	0.082 (4)
C3	0.5946 (4)	0.5334 (4)	−0.0500 (3)	0.0368 (11)	0.918 (4)
H3	0.659871	0.556614	−0.084221	0.044*	0.918 (4)
C3A	0.465 (2)	0.570 (3)	−0.0792 (18)	0.0368 (11)	0.082 (4)
H3A	0.440687	0.620672	−0.134626	0.044*	0.082 (4)
O1	0.2262 (3)	0.1030 (3)	0.7614 (2)	0.0397 (7)	
N2	0.0590 (3)	0.1987 (3)	0.6536 (2)	0.0345 (7)	
C4	−0.0193 (4)	0.2197 (4)	0.5464 (3)	0.0471 (11)	
H4A	0.012128	0.173444	0.496723	0.071*	
H4B	0.001881	0.316351	0.544827	0.071*	
H4C	−0.124667	0.183480	0.525552	0.071*	
C5	0.0344 (6)	0.2671 (6)	0.7385 (4)	0.089 (2)	
H5A	0.105377	0.355397	0.767114	0.133*	
H5B	0.045962	0.213536	0.795020	0.133*	
H5C	−0.064912	0.279168	0.711359	0.133*	
C6	0.1511 (4)	0.1229 (3)	0.6738 (3)	0.0310 (9)	
H6	0.160825	0.078393	0.614092	0.037*	
O2	0.4557 (3)	−0.1235 (3)	0.9024 (2)	0.0528 (8)	
N3	0.5360 (3)	−0.0093 (3)	0.7928 (2)	0.0365 (8)	
C7	0.6841 (5)	0.0426 (5)	0.8704 (4)	0.0737 (16)	
H7A	0.742668	−0.019070	0.863107	0.111*	
H7B	0.684310	0.050996	0.942109	0.111*	
H7C	0.726072	0.131344	0.858773	0.111*	
C8	0.5004 (5)	0.0294 (4)	0.6892 (3)	0.0521 (12)	
H8A	0.516051	0.127409	0.697400	0.078*	
H8B	0.397800	−0.014548	0.643792	0.078*	
H8C	0.563372	0.001622	0.656359	0.078*	
C9	0.4359 (4)	−0.0854 (4)	0.8172 (4)	0.0401 (10)	
H9	0.339326	−0.113538	0.763903	0.048*	
O3	1.1120 (3)	0.2000 (3)	0.0265 (2)	0.0531 (8)	
N4	0.9538 (3)	0.2480 (3)	0.0950 (2)	0.0348 (7)	
C10	0.8672 (5)	0.3384 (4)	0.1084 (4)	0.0542 (12)	
H10A	0.910401	0.386797	0.182173	0.081*	
H10B	0.767006	0.286161	0.091710	0.081*	
H10C	0.865666	0.403223	0.060362	0.081*	

C11	0.9567 (5)	0.1356 (4)	0.1512 (3)	0.0513 (11)
H11A	0.986243	0.066079	0.116745	0.077*
H11B	0.859138	0.097880	0.149607	0.077*
H11C	1.026857	0.166773	0.225219	0.077*
C12	1.0327 (4)	0.2695 (4)	0.0379 (3)	0.0415 (10)
H12	1.027845	0.344630	0.002820	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01844 (16)	0.01776 (16)	0.02281 (18)	0.00192 (12)	0.00933 (13)	0.00253 (12)
Mo2	0.01778 (16)	0.01982 (16)	0.02082 (18)	0.00380 (12)	0.00849 (13)	0.00326 (12)
Mo3	0.01810 (16)	0.01988 (16)	0.02220 (18)	0.00434 (12)	0.01006 (13)	0.00274 (12)
Cl1	0.0253 (4)	0.0231 (4)	0.0234 (5)	0.0034 (3)	0.0097 (4)	−0.0003 (3)
Cl2	0.0174 (4)	0.0274 (4)	0.0279 (5)	0.0026 (3)	0.0088 (4)	0.0041 (4)
Cl3	0.0252 (4)	0.0212 (4)	0.0287 (5)	0.0079 (3)	0.0115 (4)	0.0047 (4)
Cl4	0.0249 (4)	0.0248 (4)	0.0264 (5)	0.0041 (3)	0.0142 (4)	0.0067 (4)
Cl5	0.0336 (5)	0.0222 (4)	0.0385 (5)	−0.0019 (4)	0.0151 (4)	0.0018 (4)
Cl6	0.0290 (5)	0.0361 (5)	0.0254 (5)	0.0108 (4)	0.0087 (4)	0.0072 (4)
Cl7	0.0331 (5)	0.0407 (5)	0.0373 (5)	0.0137 (4)	0.0223 (4)	0.0051 (4)
N1	0.0345 (19)	0.039 (2)	0.0338 (19)	0.0056 (16)	0.0181 (16)	0.0081 (16)
C1	0.028 (2)	0.036 (2)	0.029 (2)	0.0097 (17)	0.0132 (17)	0.0136 (17)
C2	0.039 (2)	0.038 (2)	0.032 (2)	0.015 (2)	0.018 (2)	0.0055 (19)
C2A	0.039 (2)	0.038 (2)	0.032 (2)	0.015 (2)	0.018 (2)	0.0055 (19)
C3	0.035 (2)	0.049 (3)	0.033 (2)	0.010 (2)	0.021 (2)	0.012 (2)
C3A	0.035 (2)	0.049 (3)	0.033 (2)	0.010 (2)	0.021 (2)	0.012 (2)
O1	0.0426 (16)	0.0405 (16)	0.0385 (17)	0.0171 (13)	0.0145 (14)	0.0111 (13)
N2	0.0296 (17)	0.0395 (19)	0.040 (2)	0.0109 (15)	0.0191 (15)	0.0086 (15)
C4	0.030 (2)	0.060 (3)	0.054 (3)	0.013 (2)	0.015 (2)	0.027 (2)
C5	0.109 (5)	0.126 (5)	0.065 (4)	0.083 (4)	0.045 (3)	0.016 (4)
C6	0.032 (2)	0.0247 (19)	0.039 (2)	0.0027 (17)	0.0197 (19)	0.0024 (17)
O2	0.0527 (19)	0.0526 (19)	0.052 (2)	0.0122 (15)	0.0172 (16)	0.0249 (16)
N3	0.0329 (18)	0.0305 (18)	0.040 (2)	0.0080 (15)	0.0073 (15)	0.0089 (15)
C7	0.048 (3)	0.060 (3)	0.082 (4)	−0.008 (3)	−0.001 (3)	0.027 (3)
C8	0.076 (3)	0.054 (3)	0.041 (3)	0.032 (3)	0.029 (2)	0.016 (2)
C9	0.033 (2)	0.027 (2)	0.051 (3)	0.0096 (18)	0.007 (2)	0.003 (2)
O3	0.0490 (18)	0.0476 (18)	0.071 (2)	0.0059 (15)	0.0388 (17)	0.0033 (16)
N4	0.0308 (17)	0.0370 (19)	0.0326 (19)	0.0017 (15)	0.0122 (15)	0.0045 (15)
C10	0.047 (3)	0.057 (3)	0.064 (3)	0.012 (2)	0.028 (2)	0.008 (2)
C11	0.051 (3)	0.055 (3)	0.048 (3)	0.009 (2)	0.021 (2)	0.020 (2)
C12	0.039 (2)	0.040 (2)	0.037 (2)	−0.004 (2)	0.014 (2)	0.0043 (19)

Geometric parameters (Å, º)

Mo1—Mo2^{i}	2.6065 (5)	C3A—H3A	0.9500
Mo1—Mo2	2.6040 (5)	O1—C6	1.229 (4)
Mo1—Mo3	2.6039 (5)	N2—C4	1.456 (5)
Mo1—Mo3^{i}	2.5984 (5)	N2—C5	1.435 (5)

Mo1—Cl1	2.4724 (9)	N2—C6	1.312 (4)
Mo1—Cl2	2.4764 (9)	C4—H4A	0.9800
Mo1—Cl3	2.4727 (9)	C4—H4B	0.9800
Mo1—Cl4	2.4708 (9)	C4—H4C	0.9800
Mo1—Cl5	2.4277 (9)	C5—H5A	0.9800
Mo2—Mo3^{i}	2.6027 (5)	C5—H5B	0.9800
Mo2—Mo3	2.6055 (5)	C5—H5C	0.9800
Mo2—Cl1	2.4729 (9)	C6—H6	0.9500
Mo2—Cl2	2.4644 (9)	O2—C9	1.228 (5)
Mo2—Cl3^{i}	2.4682 (9)	N3—C7	1.442 (5)
Mo2—Cl4^{i}	2.4629 (9)	N3—C8	1.450 (5)
Mo2—Cl6	2.4436 (9)	N3—C9	1.316 (5)
Mo3—Cl1	2.4781 (9)	C7—H7A	0.9800
Mo3—Cl2^{i}	2.4751 (9)	C7—H7B	0.9800
Mo3—Cl3	2.4724 (9)	C7—H7C	0.9800
Mo3—Cl4^{i}	2.4632 (9)	C8—H8A	0.9800
Mo3—Cl7	2.4116 (9)	C8—H8B	0.9800
N1—H1A	0.922 (18)	C8—H8C	0.9800
N1—H1B	0.927 (18)	C9—H9	0.9500
N1—H1C	0.924 (18)	O3—C12	1.227 (5)
N1—C1	1.458 (5)	N4—C10	1.453 (5)
C1—C2	1.367 (5)	N4—C11	1.450 (5)
C1—C2A	1.361 (16)	N4—C12	1.314 (5)
C1—C3	1.377 (5)	C10—H10A	0.9800
C1—C3A	1.366 (16)	C10—H10B	0.9800
C2—H2	0.9500	C10—H10C	0.9800
C2—C3^{ii}	1.378 (5)	C11—H11A	0.9800
C2A—H2A	0.9500	C11—H11B	0.9800
C2A—C3A^{ii}	1.380 (15)	C11—H11C	0.9800
C3—H3	0.9500	C12—H12	0.9500

Mo2—Mo1—Mo2^{i}	90.126 (16)	Cl4^{i}—Mo3—Cl3	175.60 (3)
Mo3—Mo1—Mo2^{i}	59.936 (14)	Cl7—Mo3—Mo1	136.77 (3)
Mo3—Mo1—Mo2	60.038 (12)	Cl7—Mo3—Mo1^{i}	133.17 (3)
Mo3^{i}—Mo1—Mo2^{i}	60.074 (13)	Cl7—Mo3—Mo2^{i}	135.56 (3)
Mo3^{i}—Mo1—Mo2	60.036 (15)	Cl7—Mo3—Mo2	134.21 (3)
Mo3^{i}—Mo1—Mo3	89.946 (14)	Cl7—Mo3—Cl1	92.98 (3)
Cl1—Mo1—Mo2^{i}	118.30 (2)	Cl7—Mo3—Cl2^{i}	91.41 (3)
Cl1—Mo1—Mo2	58.24 (2)	Cl7—Mo3—Cl3	93.96 (3)
Cl1—Mo1—Mo3^{i}	118.26 (2)	Cl7—Mo3—Cl4^{i}	90.44 (3)
Cl1—Mo1—Mo3	58.37 (2)	Mo1—Cl1—Mo2	63.55 (2)
Cl1—Mo1—Cl2	89.61 (3)	Mo1—Cl1—Mo3	63.47 (2)
Cl1—Mo1—Cl3	90.01 (3)	Mo2—Cl1—Mo3	63.50 (2)
Cl2—Mo1—Mo2	57.97 (2)	Mo2—Cl2—Mo1	63.61 (2)
Cl2—Mo1—Mo2^{i}	118.37 (2)	Mo2—Cl2—Mo3^{i}	63.59 (2)
Cl2—Mo1—Mo3^{i}	58.32 (2)	Mo3^{i}—Cl2—Mo1	63.31 (2)
Cl2—Mo1—Mo3	117.99 (2)	Mo2^{i}—Cl3—Mo1	63.68 (2)
Cl3—Mo1—Mo2^{i}	58.08 (2)	Mo2^{i}—Cl3—Mo3	63.58 (2)

Cl3—Mo1—Mo2	118.24 (2)	Mo3—Cl3—Mo1	63.55 (2)
Cl3—Mo1—Mo3^{i}	118.13 (2)	Mo2^{i}—Cl4—Mo1	63.78 (2)
Cl3—Mo1—Mo3	58.22 (2)	Mo2^{i}—Cl4—Mo3^{i}	63.86 (2)
Cl3—Mo1—Cl2	175.50 (3)	Mo3^{i}—Cl4—Mo1	63.56 (2)
Cl4—Mo1—Mo2^{i}	57.96 (2)	H1A—N1—H1B	112 (4)
Cl4—Mo1—Mo2	118.09 (2)	H1A—N1—H1C	110 (4)
Cl4—Mo1—Mo3	117.88 (2)	H1B—N1—H1C	103 (3)
Cl4—Mo1—Mo3^{i}	58.08 (2)	C1—N1—H1A	108 (3)
Cl4—Mo1—Cl1	175.46 (3)	C1—N1—H1B	109 (3)
Cl4—Mo1—Cl2	90.28 (3)	C1—N1—H1C	114 (3)
Cl4—Mo1—Cl3	89.74 (3)	C2—C1—N1	119.9 (3)
Cl5—Mo1—Mo2^{i}	134.38 (3)	C2—C1—C3	120.7 (4)
Cl5—Mo1—Mo2	135.49 (3)	C2A—C1—N1	119.6 (15)
Cl5—Mo1—Mo3	135.18 (2)	C2A—C1—C3A	120.3 (10)
Cl5—Mo1—Mo3^{i}	134.87 (3)	C3—C1—N1	119.4 (3)
Cl5—Mo1—Cl1	92.58 (3)	C3A—C1—N1	120.1 (16)
Cl5—Mo1—Cl2	92.57 (3)	C1—C2—H2	119.9
Cl5—Mo1—Cl3	91.93 (3)	C1—C2—C3^{ii}	120.1 (4)
Cl5—Mo1—Cl4	91.96 (3)	C3^{ii}—C2—H2	119.9
Mo1—Mo2—Mo1^{i}	89.873 (16)	C1—C2A—H2A	121.4
Mo1—Mo2—Mo3	59.978 (12)	C1—C2A—C3A^{ii}	117 (3)
Mo3^{i}—Mo2—Mo1^{i}	59.981 (14)	C3A^{ii}—C2A—H2A	121.4
Mo3^{i}—Mo2—Mo1	59.875 (13)	C1—C3—C2^{ii}	119.2 (4)
Mo3—Mo2—Mo1^{i}	59.809 (14)	C1—C3—H3	120.4
Mo3^{i}—Mo2—Mo3	89.819 (14)	C2^{ii}—C3—H3	120.4
Cl1—Mo2—Mo1^{i}	118.14 (2)	C1—C3A—C2A^{ii}	122 (3)
Cl1—Mo2—Mo1	58.22 (2)	C1—C3A—H3A	118.8
Cl1—Mo2—Mo3^{i}	118.08 (2)	C2A^{ii}—C3A—H3A	118.8
Cl1—Mo2—Mo3	58.34 (2)	C5—N2—C4	117.3 (3)
Cl2—Mo2—Mo1^{i}	118.37 (2)	C6—N2—C4	122.3 (3)
Cl2—Mo2—Mo1	58.42 (2)	C6—N2—C5	120.4 (3)
Cl2—Mo2—Mo3	118.38 (2)	N2—C4—H4A	109.5
Cl2—Mo2—Mo3^{i}	58.40 (2)	N2—C4—H4B	109.5
Cl2—Mo2—Cl1	89.88 (3)	N2—C4—H4C	109.5
Cl2—Mo2—Cl3^{i}	89.95 (3)	H4A—C4—H4B	109.5
Cl3^{i}—Mo2—Mo1^{i}	58.25 (2)	H4A—C4—H4C	109.5
Cl3^{i}—Mo2—Mo1	118.15 (2)	H4B—C4—H4C	109.5
Cl3^{i}—Mo2—Mo3^{i}	58.29 (2)	N2—C5—H5A	109.5
Cl3^{i}—Mo2—Mo3	118.04 (2)	N2—C5—H5B	109.5
Cl3^{i}—Mo2—Cl1	175.56 (3)	N2—C5—H5C	109.5
Cl4^{i}—Mo2—Mo1	118.04 (2)	H5A—C5—H5B	109.5
Cl4^{i}—Mo2—Mo1^{i}	58.26 (2)	H5A—C5—H5C	109.5
Cl4^{i}—Mo2—Mo3	58.07 (2)	H5B—C5—H5C	109.5
Cl4^{i}—Mo2—Mo3^{i}	118.22 (2)	O1—C6—N2	127.2 (4)
Cl4^{i}—Mo2—Cl1	89.81 (3)	O1—C6—H6	116.4
Cl4^{i}—Mo2—Cl2	175.77 (3)	N2—C6—H6	116.4
Cl4^{i}—Mo2—Cl3^{i}	90.03 (3)	C7—N3—C8	117.8 (4)
Cl6—Mo2—Mo1^{i}	133.87 (3)	C9—N3—C7	120.7 (4)

Cl6—Mo2—Mo1	136.26 (3)	C9—N3—C8	121.5 (4)
Cl6—Mo2—Mo3^{i}	134.96 (3)	N3—C7—H7A	109.5
Cl6—Mo2—Mo3	135.19 (2)	N3—C7—H7B	109.5
Cl6—Mo2—Cl1	93.14 (3)	N3—C7—H7C	109.5
Cl6—Mo2—Cl2	92.74 (3)	H7A—C7—H7B	109.5
Cl6—Mo2—Cl3^{i}	91.30 (3)	H7A—C7—H7C	109.5
Cl6—Mo2—Cl4^{i}	91.49 (3)	H7B—C7—H7C	109.5
Mo1^{i}—Mo3—Mo1	90.053 (14)	N3—C8—H8A	109.5
Mo1^{i}—Mo3—Mo2^{i}	60.086 (12)	N3—C8—H8B	109.5
Mo1—Mo3—Mo2	59.984 (14)	N3—C8—H8C	109.5
Mo1^{i}—Mo3—Mo2	60.116 (12)	H8A—C8—H8B	109.5
Mo2^{i}—Mo3—Mo1	60.082 (11)	H8A—C8—H8C	109.5
Mo2^{i}—Mo3—Mo2	90.179 (13)	H8B—C8—H8C	109.5
Cl1—Mo3—Mo1^{i}	118.25 (2)	O2—C9—N3	126.1 (4)
Cl1—Mo3—Mo1	58.16 (2)	O2—C9—H9	116.9
Cl1—Mo3—Mo2	58.15 (2)	N3—C9—H9	116.9
Cl1—Mo3—Mo2^{i}	118.23 (2)	C11—N4—C10	117.9 (3)
Cl2^{i}—Mo3—Mo1	118.07 (2)	C12—N4—C10	121.7 (3)
Cl2^{i}—Mo3—Mo1^{i}	58.37 (2)	C12—N4—C11	120.3 (4)
Cl2^{i}—Mo3—Mo2	118.46 (2)	N4—C10—H10A	109.5
Cl2^{i}—Mo3—Mo2^{i}	58.00 (2)	N4—C10—H10B	109.5
Cl2^{i}—Mo3—Cl1	175.60 (3)	N4—C10—H10C	109.5
Cl3—Mo3—Mo1	58.23 (2)	H10A—C10—H10B	109.5
Cl3—Mo3—Mo1^{i}	118.20 (2)	H10A—C10—H10C	109.5
Cl3—Mo3—Mo2	118.20 (2)	H10B—C10—H10C	109.5
Cl3—Mo3—Mo2^{i}	58.13 (2)	N4—C11—H11A	109.5
Cl3—Mo3—Cl1	89.89 (3)	N4—C11—H11B	109.5
Cl3—Mo3—Cl2^{i}	89.61 (3)	N4—C11—H11C	109.5
Cl4^{i}—Mo3—Mo1	118.03 (2)	H11A—C11—H11B	109.5
Cl4^{i}—Mo3—Mo1^{i}	58.36 (2)	H11A—C11—H11C	109.5
Cl4^{i}—Mo3—Mo2^{i}	118.43 (2)	H11B—C11—H11C	109.5
Cl4^{i}—Mo3—Mo2	58.06 (2)	O3—C12—N4	125.0 (4)
Cl4^{i}—Mo3—Cl1	89.68 (3)	O3—C12—H12	117.5
Cl4^{i}—Mo3—Cl2^{i}	90.48 (3)	N4—C12—H12	117.5
N1—C1—C2—C3^{ii}	178.5 (3)	C3A—C1—C2A—C3A^{ii}	1.0 (17)
N1—C1—C2A—C3A^{ii}	−179.7 (9)	C4—N2—C6—O1	177.0 (4)
N1—C1—C3—C2^{ii}	−178.5 (3)	C5—N2—C6—O1	−1.6 (6)
N1—C1—C3A—C2A^{ii}	179.7 (9)	C7—N3—C9—O2	−2.6 (6)
C2—C1—C3—C2^{ii}	0.3 (6)	C8—N3—C9—O2	179.8 (4)
C2A—C1—C3A—C2A^{ii}	−1.1 (18)	C10—N4—C12—O3	177.3 (4)
C3—C1—C2—C3^{ii}	−0.3 (6)	C11—N4—C12—O3	0.4 (6)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{iii}	0.92 (2)	1.76 (2)	2.672 (4)	171 (4)

N1—H1*B*···O3^{iv}	0.93 (2)	1.79 (2)	2.710 (4)	173 (4)
N1—H1*C*···O1^{i}	0.92 (2)	1.81 (2)	2.727 (4)	175 (4)

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

N,*N*′-(1,4-Phenylene)bis(propan-2-iminium) octa-μ_3-chlorido-hexachlorido-*octahedro*-hexamolybdate acetone trisolvate (3)

Crystal data

$(C_{12}H_{18}N_2)[Mo_6Cl_8Cl_6]\cdot 3C_3H_6O$
M_r = 1436.46
Triclinic, $P\overline{1}$
a = 9.451 (2) Å
b = 11.236 (3) Å
c = 11.712 (3) Å
α = 64.933 (6)°
β = 71.174 (6)°
γ = 75.440 (6)°
V = 1056.7 (5) Å^3
Z = 1
F(000) = 690
D_x = 2.257 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5309 reflections
θ = 2.2–25.1°
μ = 2.64 mm^{-1}
T = 200 K
Block, clear light orange
0.55 × 0.33 × 0.20 mm

Data collection

Bruker SMART X2S benchtop diffractometer
Radiation source: sealed microfocus source, XOS X-beam microfocus source
Graphite monochromator
Detector resolution: 8.3330 pixels mm^{-1}
φ and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.490, T_{max} = 0.745
10036 measured reflections
3692 independent reflections
3220 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.2°, θ_{min} = 2.6°
h = −11→11
k = −13→13
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.068
S = 1.05
3692 reflections
235 parameters
13 restraints
Primary atom site location: heavy-atom method
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0347P)^2 + 0.3989P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.96 e Å^{-3}
$\Delta\rho_{min}$ = −0.82 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Mo1	0.54044 (3)	0.46936 (2)	0.65449 (2)	0.01669 (9)	
Mo2	0.46251 (3)	0.32776 (2)	0.56169 (2)	0.01681 (9)	
Mo3	0.29965 (3)	0.55064 (2)	0.56544 (2)	0.01718 (9)	

Cl1	0.41212 (10)	0.10003 (8)	0.64118 (8)	0.0324 (2)	
Cl2	0.76390 (8)	0.58538 (7)	0.52537 (7)	0.02273 (17)	
Cl3	0.69229 (9)	0.25958 (7)	0.64379 (7)	0.02413 (18)	
Cl4	0.59212 (10)	0.43277 (8)	0.85707 (7)	0.0303 (2)	
Cl5	0.38283 (9)	0.68264 (7)	0.65005 (7)	0.02236 (17)	
Cl6	0.31409 (9)	0.35417 (8)	0.76707 (7)	0.02377 (18)	
Cl7	0.03772 (9)	0.61748 (9)	0.65222 (9)	0.0360 (2)	
N1	0.2062 (3)	0.0615 (3)	0.2565 (3)	0.0242 (6)	
H1	0.196 (4)	0.021 (3)	0.211 (3)	0.036*	
C1	0.4140 (4)	0.1369 (4)	0.0787 (3)	0.0382 (9)	
H1A	0.431717	0.228439	0.022574	0.057*	
H1B	0.366916	0.102550	0.037660	0.057*	
H1C	0.510355	0.081919	0.091601	0.057*	
C2	0.3131 (4)	0.1337 (3)	0.2058 (3)	0.0265 (7)	
C3	0.3406 (4)	0.2109 (3)	0.2704 (4)	0.0370 (9)	
H3A	0.424810	0.163487	0.311157	0.056*	
H3B	0.249770	0.222227	0.337134	0.056*	
H3C	0.365572	0.298048	0.205817	0.056*	
C4	0.1507 (4)	−0.0037 (3)	0.4928 (3)	0.0281 (7)	
H4	0.253881	−0.006002	0.486934	0.034*	
C5	0.1019 (4)	0.0328 (3)	0.3819 (3)	0.0227 (7)	
C6	−0.0482 (4)	0.0368 (3)	0.3885 (3)	0.0274 (7)	
H6	−0.079966	0.062241	0.311317	0.033*	
O1	0.1737 (3)	0.9573 (3)	0.0897 (2)	0.0440 (7)	
C7	0.0245 (5)	0.7839 (4)	0.1942 (4)	0.0502 (11)	
H7A	0.012696	0.802221	0.271696	0.075*	
H7B	0.067673	0.691137	0.209401	0.075*	
H7C	−0.074289	0.800190	0.175911	0.075*	
C8	0.1262 (4)	0.8717 (4)	0.0818 (4)	0.0345 (8)	
C9	0.1705 (5)	0.8501 (4)	−0.0432 (4)	0.0468 (10)	
H9A	0.205823	0.930184	−0.115350	0.070*	
H9B	0.083109	0.830392	−0.056688	0.070*	
H9C	0.251527	0.775370	−0.039653	0.070*	
O2	0.0767 (6)	0.3001 (5)	0.0578 (5)	0.0461 (13)	0.5
C10	0.0375 (9)	0.4167 (7)	0.0246 (6)	0.0351 (17)	0.5
C11	0.143 (2)	0.512 (2)	−0.011 (2)	0.049 (5)	0.5
H11A	0.117815	0.548863	0.056809	0.073*	0.5
H11B	0.134909	0.584134	−0.094095	0.073*	0.5
H11C	0.246860	0.466249	−0.018685	0.073*	0.5
C12	−0.1156 (17)	0.473 (2)	0.006 (2)	0.044 (5)	0.5
H12A	−0.114886	0.495968	−0.085108	0.067*	0.5
H12B	−0.148442	0.553655	0.026059	0.067*	0.5
H12C	−0.185388	0.408443	0.063119	0.067*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01921 (17)	0.01543 (15)	0.01644 (15)	−0.00245 (12)	−0.00747 (11)	−0.00459 (11)

Mo2	0.01936 (17)	0.01344 (15)	0.01826 (15)	−0.00251 (11)	−0.00714 (11)	−0.00450 (11)
Mo3	0.01548 (17)	0.01715 (15)	0.01989 (15)	−0.00117 (11)	−0.00588 (11)	−0.00728 (12)
Cl1	0.0385 (5)	0.0179 (4)	0.0390 (5)	−0.0077 (4)	−0.0110 (4)	−0.0057 (3)
Cl2	0.0208 (4)	0.0243 (4)	0.0272 (4)	−0.0053 (3)	−0.0092 (3)	−0.0098 (3)
Cl3	0.0278 (4)	0.0172 (4)	0.0286 (4)	0.0030 (3)	−0.0158 (3)	−0.0065 (3)
Cl4	0.0429 (5)	0.0301 (4)	0.0223 (4)	−0.0098 (4)	−0.0159 (4)	−0.0053 (3)
Cl5	0.0248 (4)	0.0208 (4)	0.0247 (4)	−0.0008 (3)	−0.0075 (3)	−0.0118 (3)
Cl6	0.0266 (4)	0.0245 (4)	0.0186 (4)	−0.0084 (3)	−0.0029 (3)	−0.0057 (3)
Cl7	0.0189 (4)	0.0453 (5)	0.0457 (5)	0.0015 (4)	−0.0055 (4)	−0.0237 (4)
N1	0.0263 (16)	0.0219 (14)	0.0257 (14)	−0.0046 (12)	−0.0068 (12)	−0.0090 (11)
C1	0.040 (2)	0.036 (2)	0.0310 (19)	−0.0163 (18)	−0.0023 (16)	−0.0037 (16)
C2	0.0241 (19)	0.0158 (16)	0.0354 (18)	0.0018 (14)	−0.0126 (15)	−0.0045 (14)
C3	0.037 (2)	0.0235 (18)	0.054 (2)	−0.0030 (16)	−0.0155 (18)	−0.0152 (17)
C4	0.0213 (18)	0.0312 (18)	0.0346 (18)	−0.0018 (15)	−0.0099 (15)	−0.0136 (15)
C5	0.0251 (19)	0.0163 (15)	0.0275 (16)	0.0008 (13)	−0.0074 (14)	−0.0102 (13)
C6	0.028 (2)	0.0311 (18)	0.0265 (17)	−0.0021 (15)	−0.0113 (14)	−0.0115 (15)
O1	0.0565 (18)	0.0433 (16)	0.0429 (15)	−0.0198 (14)	−0.0152 (13)	−0.0166 (13)
C7	0.038 (2)	0.047 (2)	0.063 (3)	−0.012 (2)	−0.013 (2)	−0.014 (2)
C8	0.031 (2)	0.0322 (19)	0.043 (2)	0.0004 (16)	−0.0204 (17)	−0.0105 (16)
C9	0.057 (3)	0.043 (2)	0.050 (2)	−0.001 (2)	−0.028 (2)	−0.018 (2)
O2	0.051 (4)	0.037 (3)	0.052 (3)	−0.007 (3)	−0.016 (3)	−0.015 (3)
C10	0.046 (5)	0.038 (5)	0.020 (3)	−0.006 (4)	−0.003 (3)	−0.013 (3)
C11	0.048 (8)	0.054 (10)	0.061 (10)	−0.010 (7)	−0.008 (8)	−0.040 (8)
C12	0.043 (7)	0.042 (8)	0.036 (8)	0.000 (6)	0.006 (5)	−0.017 (7)

Geometric parameters (Å, º)

Mo1—Mo2^{i}	2.5943 (6)	C2—C3	1.480 (5)
Mo1—Mo2	2.6126 (5)	C3—H3A	0.9800
Mo1—Mo3	2.6038 (6)	C3—H3B	0.9800
Mo1—Mo3^{i}	2.6031 (7)	C3—H3C	0.9800
Mo1—Cl2	2.4695 (9)	C4—H4	0.9500
Mo1—Cl3	2.4627 (9)	C4—C5	1.381 (4)
Mo1—Cl4	2.4202 (9)	C4—C6^{ii}	1.372 (5)
Mo1—Cl5	2.4727 (9)	C5—C6	1.386 (4)
Mo1—Cl6	2.4729 (9)	C6—H6	0.9500
Mo2—Mo3^{i}	2.6069 (6)	O1—C8	1.206 (4)
Mo2—Mo3	2.5989 (7)	C7—H7A	0.9800
Mo2—Cl1	2.4391 (10)	C7—H7B	0.9800
Mo2—Cl2^{i}	2.4668 (9)	C7—H7C	0.9800
Mo2—Cl3	2.4727 (9)	C7—C8	1.480 (5)
Mo2—Cl5^{i}	2.4738 (9)	C8—C9	1.494 (5)
Mo2—Cl6	2.4616 (9)	C9—H9A	0.9800
Mo3—Cl2^{i}	2.4655 (8)	C9—H9B	0.9800
Mo3—Cl3^{i}	2.4680 (9)	C9—H9C	0.9800
Mo3—Cl5	2.4767 (8)	O2—C10	1.193 (8)
Mo3—Cl6	2.4714 (9)	C10—C11	1.488 (13)
Mo3—Cl7	2.4110 (10)	C10—C12	1.475 (13)

N1—H1	0.870 (18)	C11—H11A	0.9800
N1—C2	1.284 (4)	C11—H11B	0.9800
N1—C5	1.434 (4)	C11—H11C	0.9800
C1—H1A	0.9800	C12—H12A	0.9800
C1—H1B	0.9800	C12—H12B	0.9800
C1—H1C	0.9800	C12—H12C	0.9800
C1—C2	1.477 (5)		

Mo2^{i}—Mo1—Mo2	89.98 (2)	Cl3^{i}—Mo3—Cl5	89.98 (3)
Mo2^{i}—Mo1—Mo3^{i}	60.005 (19)	Cl3^{i}—Mo3—Cl6	175.12 (3)
Mo2^{i}—Mo1—Mo3	60.202 (14)	Cl5—Mo3—Mo1	58.18 (2)
Mo3^{i}—Mo1—Mo2	59.977 (16)	Cl5—Mo3—Mo1^{i}	118.35 (2)
Mo3—Mo1—Mo2	59.763 (15)	Cl5—Mo3—Mo2^{i}	58.17 (2)
Mo3^{i}—Mo1—Mo3	89.98 (2)	Cl5—Mo3—Mo2	118.46 (3)
Cl2—Mo1—Mo2	118.05 (2)	Cl6—Mo3—Mo1	58.25 (2)
Cl2—Mo1—Mo2^{i}	58.24 (2)	Cl6—Mo3—Mo1^{i}	117.84 (3)
Cl2—Mo1—Mo3^{i}	58.09 (2)	Cl6—Mo3—Mo2	58.02 (2)
Cl2—Mo1—Mo3	118.42 (3)	Cl6—Mo3—Mo2^{i}	117.94 (2)
Cl2—Mo1—Cl5	90.29 (3)	Cl6—Mo3—Cl5	90.12 (3)
Cl2—Mo1—Cl6	175.36 (2)	Cl7—Mo3—Mo1	134.62 (3)
Cl3—Mo1—Mo2	58.22 (2)	Cl7—Mo3—Mo1^{i}	135.36 (2)
Cl3—Mo1—Mo2^{i}	118.22 (2)	Cl7—Mo3—Mo2^{i}	134.89 (3)
Cl3—Mo1—Mo3^{i}	58.23 (2)	Cl7—Mo3—Mo2	135.11 (3)
Cl3—Mo1—Mo3	117.98 (2)	Cl7—Mo3—Cl2^{i}	92.39 (3)
Cl3—Mo1—Cl2	89.80 (3)	Cl7—Mo3—Cl3^{i}	92.59 (3)
Cl3—Mo1—Cl5	175.65 (2)	Cl7—Mo3—Cl5	91.72 (3)
Cl3—Mo1—Cl6	89.39 (3)	Cl7—Mo3—Cl6	92.28 (3)
Cl4—Mo1—Mo2^{i}	134.02 (3)	Mo2^{i}—Cl2—Mo1	63.41 (2)
Cl4—Mo1—Mo2	136.00 (2)	Mo3^{i}—Cl2—Mo1	63.67 (2)
Cl4—Mo1—Mo3^{i}	135.46 (3)	Mo3^{i}—Cl2—Mo2^{i}	63.59 (2)
Cl4—Mo1—Mo3	134.55 (2)	Mo1—Cl3—Mo2	63.92 (2)
Cl4—Mo1—Cl2	91.90 (3)	Mo1—Cl3—Mo3^{i}	63.73 (2)
Cl4—Mo1—Cl3	93.21 (3)	Mo3^{i}—Cl3—Mo2	63.69 (2)
Cl4—Mo1—Cl5	91.13 (3)	Mo1—Cl5—Mo2^{i}	63.26 (2)
Cl4—Mo1—Cl6	92.71 (3)	Mo1—Cl5—Mo3	63.48 (2)
Cl5—Mo1—Mo2^{i}	58.39 (2)	Mo2^{i}—Cl5—Mo3	63.55 (2)
Cl5—Mo1—Mo2	118.08 (2)	Mo2—Cl6—Mo1	63.94 (2)
Cl5—Mo1—Mo3	58.33 (2)	Mo2—Cl6—Mo3	63.58 (3)
Cl5—Mo1—Mo3^{i}	118.37 (2)	Mo3—Cl6—Mo1	63.56 (2)
Cl5—Mo1—Cl6	90.17 (3)	C2—N1—H1	117 (2)
Cl6—Mo1—Mo2^{i}	118.37 (2)	C2—N1—C5	129.1 (3)
Cl6—Mo1—Mo2	57.82 (2)	C5—N1—H1	114 (2)
Cl6—Mo1—Mo3	58.19 (2)	H1A—C1—H1B	109.5
Cl6—Mo1—Mo3^{i}	117.79 (2)	H1A—C1—H1C	109.5
Mo1^{i}—Mo2—Mo1	90.02 (2)	H1B—C1—H1C	109.5
Mo1^{i}—Mo2—Mo3	60.165 (17)	C2—C1—H1A	109.5
Mo1^{i}—Mo2—Mo3^{i}	60.081 (15)	C2—C1—H1B	109.5
Mo3—Mo2—Mo1	59.951 (17)	C2—C1—H1C	109.5

Mo3^{i}—Mo2—Mo1	59.830 (17)	N1—C2—C1	118.1 (3)
Mo3—Mo2—Mo3^{i}	90.00 (2)	N1—C2—C3	122.8 (3)
Cl1—Mo2—Mo1	135.83 (3)	C1—C2—C3	119.1 (3)
Cl1—Mo2—Mo1^{i}	134.15 (2)	C2—C3—H3A	109.5
Cl1—Mo2—Mo3	134.88 (3)	C2—C3—H3B	109.5
Cl1—Mo2—Mo3^{i}	135.11 (2)	C2—C3—H3C	109.5
Cl1—Mo2—Cl2^{i}	91.56 (3)	H3A—C3—H3B	109.5
Cl1—Mo2—Cl3	93.08 (3)	H3A—C3—H3C	109.5
Cl1—Mo2—Cl5^{i}	91.61 (3)	H3B—C3—H3C	109.5
Cl1—Mo2—Cl6	92.55 (3)	C5—C4—H4	120.4
Cl2^{i}—Mo2—Mo1^{i}	58.35 (2)	C6^{ii}—C4—H4	120.4
Cl2^{i}—Mo2—Mo1	118.11 (2)	C6^{ii}—C4—C5	119.1 (3)
Cl2^{i}—Mo2—Mo3^{i}	118.40 (2)	C4—C5—N1	121.0 (3)
Cl2^{i}—Mo2—Mo3	58.18 (2)	C4—C5—C6	121.2 (3)
Cl2^{i}—Mo2—Cl3	175.35 (2)	C6—C5—N1	117.7 (3)
Cl2^{i}—Mo2—Cl5^{i}	90.33 (3)	C4^{ii}—C6—C5	119.6 (3)
Cl3—Mo2—Mo1	57.85 (2)	C4^{ii}—C6—H6	120.2
Cl3—Mo2—Mo1^{i}	118.13 (2)	C5—C6—H6	120.2
Cl3—Mo2—Mo3^{i}	58.07 (2)	H7A—C7—H7B	109.5
Cl3—Mo2—Mo3	117.79 (2)	H7A—C7—H7C	109.5
Cl3—Mo2—Cl5^{i}	89.93 (3)	H7B—C7—H7C	109.5
Cl5^{i}—Mo2—Mo1	118.09 (2)	C8—C7—H7A	109.5
Cl5^{i}—Mo2—Mo1^{i}	58.35 (2)	C8—C7—H7B	109.5
Cl5^{i}—Mo2—Mo3^{i}	58.28 (2)	C8—C7—H7C	109.5
Cl5^{i}—Mo2—Mo3	118.49 (2)	O1—C8—C7	121.9 (4)
Cl6—Mo2—Mo1	58.24 (2)	O1—C8—C9	120.5 (3)
Cl6—Mo2—Mo1^{i}	118.54 (3)	C7—C8—C9	117.7 (3)
Cl6—Mo2—Mo3^{i}	118.06 (2)	C8—C9—H9A	109.5
Cl6—Mo2—Mo3	58.39 (2)	C8—C9—H9B	109.5
Cl6—Mo2—Cl2^{i}	89.98 (3)	C8—C9—H9C	109.5
Cl6—Mo2—Cl3	89.42 (3)	H9A—C9—H9B	109.5
Cl6—Mo2—Cl5^{i}	175.82 (2)	H9A—C9—H9C	109.5
Mo1^{i}—Mo3—Mo1	90.025 (19)	H9B—C9—H9C	109.5
Mo1—Mo3—Mo2^{i}	59.719 (18)	O2—C10—C11	122.1 (9)
Mo1^{i}—Mo3—Mo2^{i}	60.193 (13)	O2—C10—C12	120.8 (9)
Mo2—Mo3—Mo1	60.287 (13)	C12—C10—C11	116.9 (14)
Mo2—Mo3—Mo1^{i}	59.830 (12)	C10—C11—H11A	109.5
Mo2—Mo3—Mo2^{i}	90.00 (2)	C10—C11—H11B	109.5
Cl2^{i}—Mo3—Mo1	118.50 (2)	C10—C11—H11C	109.5
Cl2^{i}—Mo3—Mo1^{i}	58.24 (2)	H11A—C11—H11B	109.5
Cl2^{i}—Mo3—Mo2	58.23 (2)	H11A—C11—H11C	109.5
Cl2^{i}—Mo3—Mo2^{i}	118.42 (2)	H11B—C11—H11C	109.5
Cl2^{i}—Mo3—Cl3^{i}	89.77 (3)	C10—C12—H12A	109.5
Cl2^{i}—Mo3—Cl5	175.88 (3)	C10—C12—H12B	109.5
Cl2^{i}—Mo3—Cl6	89.79 (3)	C10—C12—H12C	109.5
Cl3^{i}—Mo3—Mo1^{i}	58.03 (2)	H12A—C12—H12B	109.5
Cl3^{i}—Mo3—Mo1	117.94 (2)	H12A—C12—H12C	109.5
Cl3^{i}—Mo3—Mo2^{i}	58.24 (2)	H12B—C12—H12C	109.5

Cl3^{i}—Mo3—Mo2	117.85 (2)		
N1—C5—C6—C4^{ii}	176.9 (3)	C5—N1—C2—C1	174.4 (3)
C2—N1—C5—C4	−46.6 (5)	C5—N1—C2—C3	−4.4 (5)
C2—N1—C5—C6	136.5 (3)	C6^{ii}—C4—C5—N1	−176.7 (3)
C4—C5—C6—C4^{ii}	−0.1 (5)	C6^{ii}—C4—C5—C6	0.1 (5)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{iii}	0.87 (2)	1.93 (2)	2.791 (4)	172 (3)

Symmetry code: (iii) *x*, *y*−1, *z*.

1,1′-Dimethyl-4,4′-bipyridinium octa-μ_3-chlorido-hexachlorido-*octahedro*-hexamolybdate *N*,*N*-dimethylformamide tetrasolvate (4)

Crystal data

$(C_{12}H_{14}N_2)[Mo_6Cl_8Cl_6]\cdot 4C_3H_7NO$
M_r = 1550.57
Triclinic, $P\overline{1}$
a = 9.8252 (11) Å
b = 10.0933 (11) Å
c = 12.6319 (15) Å
α = 107.395 (3)°
β = 91.881 (3)°
γ = 93.309 (3)°
V = 1191.8 (2) Å^3
Z = 1
$F(000)$ = 750
D_x = 2.161 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6604 reflections
θ = 2.6–25.0°
μ = 2.35 mm^{-1}
T = 200 K
Block, orange
0.32 × 0.30 × 0.28 mm

Data collection

Bruker SMART X2S benchtop diffractometer
Radiation source: sealed microfocus source, XOS X-beam microfocus source
Graphite monochromator
Detector resolution: 8.3330 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.815, T_{max} = 1.000
11498 measured reflections
4187 independent reflections
3743 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 25.1°, θ_{min} = 2.3°
h = −11→11
k = −11→11
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.049
S = 1.02
4187 reflections
250 parameters
0 restraints
Primary atom site location: heavy-atom method
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0184P)^2 + 0.8263P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0075 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Mo1	0.34350 (2)	0.52105 (2)	0.42443 (2)	0.01878 (7)
Mo2	0.43880 (2)	0.61443 (2)	0.62913 (2)	0.01930 (7)
Mo3	0.41622 (2)	0.34763 (2)	0.52876 (2)	0.01916 (7)
Cl4	0.21336 (6)	0.48336 (6)	0.57792 (5)	0.02520 (14)
Cl2	0.51113 (6)	0.44527 (7)	0.72253 (5)	0.02551 (14)
Cl7	0.30745 (7)	0.14843 (7)	0.56719 (6)	0.03486 (17)
Cl6	0.35663 (7)	0.76331 (7)	0.79813 (6)	0.03874 (18)
Cl3	0.32970 (6)	0.26780 (6)	0.33243 (5)	0.02516 (14)
Cl1	0.37330 (6)	0.77313 (6)	0.52328 (5)	0.02510 (14)
Cl5	0.13401 (6)	0.54800 (7)	0.32618 (5)	0.03256 (16)
N1	0.0168 (2)	0.9093 (3)	0.7467 (2)	0.0392 (6)
C3	0.0031 (2)	0.9806 (3)	0.5519 (2)	0.0299 (6)
C6	0.0223 (4)	0.8700 (4)	0.8508 (3)	0.0560 (9)
H6A	0.092144	0.930439	0.903109	0.084*
H6B	0.045141	0.772867	0.834244	0.084*
H6C	−0.066769	0.880619	0.883944	0.084*
O1	0.0987 (4)	0.2529 (3)	0.9699 (2)	0.0947 (11)
N2	0.1930 (3)	0.4599 (3)	0.9693 (2)	0.0462 (7)
C7	0.1610 (5)	0.3616 (4)	1.0150 (3)	0.0661 (11)
H7	0.190127	0.378592	1.090665	0.079*
C9	0.1539 (4)	0.4414 (4)	0.8553 (3)	0.0642 (10)
H9A	0.191569	0.357250	0.808268	0.096*
H9B	0.189323	0.522299	0.834281	0.096*
H9C	0.054111	0.432088	0.845257	0.096*
C8	0.2613 (6)	0.5918 (5)	1.0316 (4)	0.0954 (17)
H8A	0.274154	0.595293	1.109641	0.143*
H8B	0.205703	0.667344	1.026178	0.143*
H8C	0.350353	0.602489	1.001246	0.143*
O2	0.2430 (3)	0.9764 (3)	0.1097 (2)	0.0629 (7)
N3	0.4369 (3)	0.9015 (3)	0.1660 (2)	0.0425 (6)
C10	0.3573 (4)	0.9392 (3)	0.0942 (3)	0.0479 (8)
H10	0.393572	0.936671	0.024855	0.058*
C12	0.3883 (4)	0.9057 (4)	0.2736 (3)	0.0567 (9)
H12A	0.311583	0.965299	0.289780	0.085*
H12B	0.462220	0.943321	0.330545	0.085*
H12C	0.358102	0.811373	0.273342	0.085*
C11	0.5716 (4)	0.8576 (4)	0.1409 (4)	0.0698 (11)
H11A	0.576876	0.762359	0.144373	0.105*
H11B	0.638360	0.920002	0.195053	0.105*

H11C	0.591481	0.860583	0.066069	0.105*
C2	0.0219 (3)	0.8454 (3)	0.5522 (2)	0.0407 (7)
H2	0.030656	0.774946	0.483942	0.049*
C1	0.0281 (3)	0.8128 (4)	0.6492 (3)	0.0450 (8)
H1	0.040636	0.719562	0.647548	0.054*
C4	−0.0086 (4)	1.0768 (3)	0.6542 (3)	0.0499 (9)
H4	−0.021238	1.170823	0.658585	0.060*
C5	−0.0023 (4)	1.0392 (3)	0.7493 (3)	0.0539 (9)
H5	−0.011812	1.107404	0.818648	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mo1	0.01652 (11)	0.02000 (13)	0.02050 (12)	0.00181 (8)	0.00057 (8)	0.00707 (9)
Mo2	0.01846 (11)	0.01990 (13)	0.01947 (12)	0.00228 (9)	0.00309 (8)	0.00547 (9)
Mo3	0.01755 (11)	0.01909 (13)	0.02210 (12)	0.00020 (8)	0.00183 (8)	0.00827 (9)
Cl4	0.0185 (3)	0.0295 (3)	0.0290 (3)	0.0021 (2)	0.0059 (2)	0.0103 (3)
Cl2	0.0272 (3)	0.0301 (4)	0.0223 (3)	0.0025 (3)	0.0018 (2)	0.0124 (3)
Cl7	0.0300 (3)	0.0327 (4)	0.0474 (4)	−0.0075 (3)	−0.0014 (3)	0.0226 (3)
Cl6	0.0430 (4)	0.0374 (4)	0.0305 (4)	0.0072 (3)	0.0131 (3)	0.0001 (3)
Cl3	0.0239 (3)	0.0227 (3)	0.0259 (3)	−0.0016 (2)	−0.0023 (2)	0.0038 (3)
Cl1	0.0240 (3)	0.0207 (3)	0.0318 (3)	0.0047 (2)	0.0021 (2)	0.0090 (3)
Cl5	0.0254 (3)	0.0383 (4)	0.0335 (4)	0.0070 (3)	−0.0058 (3)	0.0100 (3)
N1	0.0294 (12)	0.0537 (17)	0.0346 (13)	0.0046 (11)	0.0096 (10)	0.0125 (12)
C3	0.0193 (12)	0.0339 (16)	0.0316 (14)	−0.0056 (11)	0.0078 (11)	0.0031 (12)
C6	0.054 (2)	0.082 (3)	0.0421 (19)	0.0242 (19)	0.0164 (16)	0.0289 (19)
O1	0.166 (3)	0.0601 (18)	0.0529 (17)	−0.046 (2)	0.0022 (19)	0.0192 (15)
N2	0.0566 (17)	0.0467 (16)	0.0351 (14)	−0.0092 (13)	−0.0047 (12)	0.0153 (13)
C7	0.105 (3)	0.061 (3)	0.0329 (18)	−0.010 (2)	0.0046 (19)	0.0184 (19)
C9	0.078 (3)	0.070 (3)	0.047 (2)	−0.013 (2)	−0.0116 (19)	0.028 (2)
C8	0.132 (4)	0.080 (3)	0.067 (3)	−0.046 (3)	−0.033 (3)	0.026 (3)
O2	0.0563 (16)	0.0770 (18)	0.0618 (16)	0.0167 (14)	−0.0073 (13)	0.0295 (14)
N3	0.0483 (15)	0.0320 (14)	0.0497 (16)	0.0033 (12)	−0.0034 (13)	0.0165 (12)
C10	0.060 (2)	0.0404 (19)	0.0432 (18)	−0.0019 (16)	−0.0034 (16)	0.0146 (16)
C12	0.078 (3)	0.048 (2)	0.047 (2)	0.0120 (18)	−0.0094 (18)	0.0183 (17)
C11	0.056 (2)	0.064 (3)	0.100 (3)	0.0104 (19)	0.009 (2)	0.038 (2)
C2	0.0383 (16)	0.0433 (19)	0.0360 (16)	0.0171 (14)	0.0025 (13)	0.0024 (14)
C1	0.0423 (17)	0.049 (2)	0.0455 (19)	0.0226 (15)	0.0062 (14)	0.0125 (16)
C4	0.079 (2)	0.0295 (17)	0.0366 (17)	−0.0111 (16)	0.0227 (16)	0.0043 (14)
C5	0.080 (3)	0.039 (2)	0.0360 (18)	−0.0106 (17)	0.0218 (17)	0.0016 (15)

Geometric parameters (Å, º)

Mo1—Mo2^{i}	2.6043 (4)	C6—H6C	0.9800
Mo1—Mo2	2.5948 (4)	O1—C7	1.195 (4)
Mo1—Mo3	2.6026 (3)	N2—C7	1.318 (4)
Mo1—Mo3^{i}	2.5996 (4)	N2—C9	1.432 (4)
Mo1—Cl4	2.4671 (6)	N2—C8	1.442 (5)

Mo1—Cl2^{i}	2.4692 (6)	C7—H7	0.9500
Mo1—Cl3	2.4650 (7)	C9—H9A	0.9800
Mo1—Cl1	2.4716 (7)	C9—H9B	0.9800
Mo1—Cl5	2.4392 (7)	C9—H9C	0.9800
Mo2—Mo3^{i}	2.5949 (3)	C8—H8A	0.9800
Mo2—Mo3	2.6037 (4)	C8—H8B	0.9800
Mo2—Cl4	2.4760 (6)	C8—H8C	0.9800
Mo2—Cl2	2.4701 (6)	O2—C10	1.208 (4)
Mo2—Cl6	2.4088 (7)	N3—C10	1.331 (4)
Mo2—Cl3^{i}	2.4683 (6)	N3—C12	1.445 (4)
Mo2—Cl1	2.4720 (6)	N3—C11	1.435 (4)
Mo3—Cl4	2.4745 (6)	C10—H10	0.9500
Mo3—Cl2	2.4766 (7)	C12—H12A	0.9800
Mo3—Cl7	2.4061 (7)	C12—H12B	0.9800
Mo3—Cl3	2.4719 (7)	C12—H12C	0.9800
Mo3—Cl1^{i}	2.4689 (6)	C11—H11A	0.9800
N1—C6	1.484 (4)	C11—H11B	0.9800
N1—C1	1.334 (4)	C11—H11C	0.9800
N1—C5	1.327 (4)	C2—H2	0.9500
C3—C3^{ii}	1.477 (5)	C2—C1	1.361 (4)
C3—C2	1.388 (4)	C1—H1	0.9500
C3—C4	1.377 (4)	C4—H4	0.9500
C6—H6A	0.9800	C4—C5	1.363 (4)
C6—H6B	0.9800	C5—H5	0.9500

Mo2—Mo1—Mo2^{i}	89.921 (11)	Cl7—Mo3—Mo2^{i}	135.04 (2)
Mo2—Mo1—Mo3^{i}	59.942 (8)	Cl7—Mo3—Cl4	92.33 (2)
Mo2—Mo1—Mo3	60.128 (10)	Cl7—Mo3—Cl2	92.13 (2)
Mo3—Mo1—Mo2^{i}	59.784 (9)	Cl7—Mo3—Cl3	92.61 (2)
Mo3^{i}—Mo1—Mo2^{i}	60.045 (11)	Cl7—Mo3—Cl1^{i}	91.81 (2)
Mo3^{i}—Mo1—Mo3	89.992 (11)	Cl3—Mo3—Mo1	58.057 (16)
Cl4—Mo1—Mo2^{i}	118.129 (17)	Cl3—Mo3—Mo1^{i}	118.159 (16)
Cl4—Mo1—Mo2	58.503 (17)	Cl3—Mo3—Mo2	117.829 (17)
Cl4—Mo1—Mo3^{i}	118.428 (17)	Cl3—Mo3—Mo2^{i}	58.247 (16)
Cl4—Mo1—Mo3	58.357 (16)	Cl3—Mo3—Cl4	89.57 (2)
Cl4—Mo1—Cl2^{i}	175.80 (2)	Cl3—Mo3—Cl2	175.25 (2)
Cl4—Mo1—Cl1	90.24 (2)	Cl1^{i}—Mo3—Mo1	118.500 (16)
Cl2^{i}—Mo1—Mo2	118.347 (18)	Cl1^{i}—Mo3—Mo1^{i}	58.302 (17)
Cl2^{i}—Mo1—Mo2^{i}	58.200 (16)	Cl1^{i}—Mo3—Mo2^{i}	58.376 (16)
Cl2^{i}—Mo1—Mo3^{i}	58.426 (16)	Cl1^{i}—Mo3—Mo2	118.352 (17)
Cl2^{i}—Mo1—Mo3	117.975 (17)	Cl1^{i}—Mo3—Cl4	175.86 (2)
Cl2^{i}—Mo1—Cl1	90.06 (2)	Cl1^{i}—Mo3—Cl2	89.95 (2)
Cl3—Mo1—Mo2^{i}	58.199 (15)	Cl1^{i}—Mo3—Cl3	90.20 (2)
Cl3—Mo1—Mo2	118.426 (17)	Mo1—Cl4—Mo2	63.328 (16)
Cl3—Mo1—Mo3	58.314 (17)	Mo1—Cl4—Mo3	63.562 (15)
Cl3—Mo1—Mo3^{i}	118.235 (16)	Mo3—Cl4—Mo2	63.465 (17)
Cl3—Mo1—Cl4	89.90 (2)	Mo1^{i}—Cl2—Mo2	63.638 (16)
Cl3—Mo1—Cl2^{i}	89.50 (2)	Mo1^{i}—Cl2—Mo3	63.422 (17)

Cl3—Mo1—Cl1	175.83 (2)	Mo2—Cl2—Mo3	63.519 (17)
Cl1—Mo1—Mo2^{i}	118.234 (17)	Mo1—Cl3—Mo2^{i}	63.726 (16)
Cl1—Mo1—Mo2	58.347 (15)	Mo1—Cl3—Mo3	63.629 (16)
Cl1—Mo1—Mo3^{i}	58.203 (15)	Mo2^{i}—Cl3—Mo3	63.372 (16)
Cl1—Mo1—Mo3	118.456 (18)	Mo1—Cl1—Mo2	63.322 (17)
Cl5—Mo1—Mo2	134.428 (19)	Mo3^{i}—Cl1—Mo1	63.496 (16)
Cl5—Mo1—Mo2^{i}	135.644 (19)	Mo3^{i}—Cl1—Mo2	63.363 (16)
Cl5—Mo1—Mo3	134.380 (19)	C1—N1—C6	119.9 (3)
Cl5—Mo1—Mo3^{i}	135.62 (2)	C5—N1—C6	120.5 (3)
Cl5—Mo1—Cl4	91.17 (2)	C5—N1—C1	119.6 (3)
Cl5—Mo1—Cl2^{i}	93.01 (2)	C2—C3—C3^{ii}	122.0 (3)
Cl5—Mo1—Cl3	92.03 (2)	C4—C3—C3^{ii}	121.9 (3)
Cl5—Mo1—Cl1	92.13 (2)	C4—C3—C2	116.1 (3)
Mo1—Mo2—Mo1^{i}	90.078 (10)	N1—C6—H6A	109.5
Mo1—Mo2—Mo3^{i}	60.121 (11)	N1—C6—H6B	109.5
Mo1—Mo2—Mo3	60.084 (8)	N1—C6—H6C	109.5
Mo3—Mo2—Mo1^{i}	59.890 (9)	H6A—C6—H6B	109.5
Mo3^{i}—Mo2—Mo1^{i}	60.076 (9)	H6A—C6—H6C	109.5
Mo3^{i}—Mo2—Mo3	90.070 (10)	H6B—C6—H6C	109.5
Cl4—Mo2—Mo1^{i}	118.113 (18)	C7—N2—C9	120.4 (3)
Cl4—Mo2—Mo1	58.169 (15)	C7—N2—C8	122.3 (3)
Cl4—Mo2—Mo3	58.238 (16)	C9—N2—C8	117.2 (3)
Cl4—Mo2—Mo3^{i}	118.272 (17)	O1—C7—N2	125.8 (3)
Cl2—Mo2—Mo1	118.429 (18)	O1—C7—H7	117.1
Cl2—Mo2—Mo1^{i}	58.164 (15)	N2—C7—H7	117.1
Cl2—Mo2—Mo3	58.360 (17)	N2—C9—H9A	109.5
Cl2—Mo2—Mo3^{i}	118.230 (17)	N2—C9—H9B	109.5
Cl2—Mo2—Cl4	90.05 (2)	N2—C9—H9C	109.5
Cl2—Mo2—Cl1	175.86 (2)	H9A—C9—H9B	109.5
Cl6—Mo2—Mo1	134.52 (2)	H9A—C9—H9C	109.5
Cl6—Mo2—Mo1^{i}	135.40 (2)	H9B—C9—H9C	109.5
Cl6—Mo2—Mo3	134.68 (2)	N2—C8—H8A	109.5
Cl6—Mo2—Mo3^{i}	135.25 (2)	N2—C8—H8B	109.5
Cl6—Mo2—Cl4	91.70 (2)	N2—C8—H8C	109.5
Cl6—Mo2—Cl2	92.13 (2)	H8A—C8—H8B	109.5
Cl6—Mo2—Cl3^{i}	92.69 (2)	H8A—C8—H8C	109.5
Cl6—Mo2—Cl1	92.01 (3)	H8B—C8—H8C	109.5
Cl3^{i}—Mo2—Mo1^{i}	58.075 (17)	C10—N3—C12	119.7 (3)
Cl3^{i}—Mo2—Mo1	118.479 (16)	C10—N3—C11	122.2 (3)
Cl3^{i}—Mo2—Mo3^{i}	58.382 (16)	C11—N3—C12	118.1 (3)
Cl3^{i}—Mo2—Mo3	117.956 (16)	O2—C10—N3	125.6 (3)
Cl3^{i}—Mo2—Cl4	175.60 (2)	O2—C10—H10	117.2
Cl3^{i}—Mo2—Cl2	89.40 (2)	N3—C10—H10	117.2
Cl3^{i}—Mo2—Cl1	90.21 (2)	N3—C12—H12A	109.5
Cl1—Mo2—Mo1^{i}	118.322 (16)	N3—C12—H12B	109.5
Cl1—Mo2—Mo1	58.332 (17)	N3—C12—H12C	109.5
Cl1—Mo2—Mo3^{i}	58.262 (16)	H12A—C12—H12B	109.5
Cl1—Mo2—Mo3	118.397 (19)	H12A—C12—H12C	109.5

Cl1—Mo2—Cl4	90.03 (2)	H12B—C12—H12C	109.5
Mo1^{i}—Mo3—Mo1	90.008 (11)	N3—C11—H11A	109.5
Mo1^{i}—Mo3—Mo2	60.064 (8)	N3—C11—H11B	109.5
Mo1—Mo3—Mo2	59.788 (11)	N3—C11—H11C	109.5
Mo2^{i}—Mo3—Mo1	60.140 (9)	H11A—C11—H11B	109.5
Mo2^{i}—Mo3—Mo1^{i}	59.936 (10)	H11A—C11—H11C	109.5
Mo2^{i}—Mo3—Mo2	89.930 (10)	H11B—C11—H11C	109.5
Cl4—Mo3—Mo1^{i}	118.345 (18)	C3—C2—H2	119.6
Cl4—Mo3—Mo1	58.082 (15)	C1—C2—C3	120.8 (3)
Cl4—Mo3—Mo2	58.296 (16)	C1—C2—H2	119.6
Cl4—Mo3—Mo2^{i}	118.209 (17)	N1—C1—C2	121.2 (3)
Cl4—Mo3—Cl2	89.94 (2)	N1—C1—H1	119.4
Cl2—Mo3—Mo1	117.894 (18)	C2—C1—H1	119.4
Cl2—Mo3—Mo1^{i}	58.152 (15)	C3—C4—H4	119.4
Cl2—Mo3—Mo2^{i}	118.066 (17)	C5—C4—C3	121.1 (3)
Cl2—Mo3—Mo2	58.121 (16)	C5—C4—H4	119.4
Cl7—Mo3—Mo1	135.303 (19)	N1—C5—C4	121.2 (3)
Cl7—Mo3—Mo1^{i}	134.689 (19)	N1—C5—H5	119.4
Cl7—Mo3—Mo2	135.02 (2)	C4—C5—H5	119.4
C3^{ii}—C3—C2—C1	179.5 (3)	C8—N2—C7—O1	176.1 (5)
C3^{ii}—C3—C4—C5	−179.8 (3)	C12—N3—C10—O2	−1.1 (5)
C3—C2—C1—N1	−0.3 (5)	C11—N3—C10—O2	179.6 (3)
C3—C4—C5—N1	0.8 (5)	C2—C3—C4—C5	−0.3 (5)
C6—N1—C1—C2	179.3 (3)	C1—N1—C5—C4	−1.0 (5)
C6—N1—C5—C4	−179.5 (3)	C4—C3—C2—C1	0.0 (4)
C9—N2—C7—O1	−0.6 (7)	C5—N1—C1—C2	0.8 (5)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O1^{iii}	0.95	2.23	3.063 (4)	145
C6—H6*C*···O2^{ii}	0.98	2.31	3.088 (4)	136

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