Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Tripyridinium *cis*-tetrachloridodioxidomolybdate(VI) chloride

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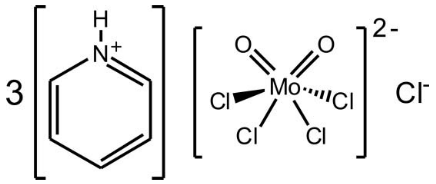
Received 6 June 2010; accepted 20 June 2010

Key indicators: single-crystal X-ray study; $T$ = 150 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.038; $wR$ factor = 0.074; data-to-parameter ratio = 43.9.

In the title compound, $(C_5H_6N)_3[MoCl_4O_2]Cl$, the pyridinium cations are N—H⋯Cl hydrogen bonded to the anionic $[MoCl_4O_2]^{2-}$ complexes and to the two crystallographically independent chloride anions (located on *C*2 axes). The $Mo^{6+}$ centre adopts a highly distorted octahedral geometry, being surrounded by four chloride and two terminal oxide groups. The oxide ligands are mutually *cis*.

## Related literature

For a related structure, see: Luan *et al.* (2008). For previous studies by our group on dioxidomolybdenum complexes, see: Monteiro *et al.* (2010); Gago *et al.* (2009); Pereira *et al.* (2007); Cunha-Silva *et al.* (2007); Bruno *et al.* (2007). For graph-set notation for hydrogen-bonded aggregates, see: Grell *et al.* (1999). For a description of the Cambridge Structural Database, see: Allen (2002).



## Experimental

### Crystal data

$(C_5H_6N)_3[MoCl_4O_2]Cl$
$M_r$ = 545.51
Trigonal, $P3_121$
$a$ = 11.3972 (2) Å
$c$ = 29.4265 (9) Å
$V$ = 3310.28 (13) Å$^3$
$Z$ = 6
Mo $K\alpha$ radiation
$\mu$ = 1.21 mm$^{-1}$
$T$ = 150 K
0.18 × 0.12 × 0.10 mm

### Data collection

Bruker X8 Kappa CCD APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998) $T_{min}$ = 0.811, $T_{max}$ = 0.888
33230 measured reflections
10395 independent reflections
8105 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.074
$S$ = 1.05
10395 reflections
237 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.68 e Å$^{-3}$
Absolute structure: Flack (1983), 4490 Friedel pairs
Flack parameter: 0.03 (4)

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Mo1—O2 | 1.6988 (13) | Mo1—Cl1 | 2.3995 (6) |
| Mo1—O1 | 1.7004 (13) | Mo1—Cl3 | 2.5746 (5) |
| Mo1—Cl2 | 2.3750 (6) | Mo1—Cl4 | 2.5953 (5) |
| | | | |
| O2—Mo1—O1 | 102.11 (7) | Cl2—Mo1—Cl3 | 85.57 (2) |
| O2—Mo1—Cl2 | 94.45 (6) | Cl1—Mo1—Cl3 | 83.91 (2) |
| O1—Mo1—Cl2 | 95.78 (6) | O2—Mo1—Cl4 | 87.40 (5) |
| O2—Mo1—Cl1 | 94.06 (6) | O1—Mo1—Cl4 | 170.36 (5) |
| O1—Mo1—Cl1 | 93.64 (6) | Cl2—Mo1—Cl4 | 84.99 (2) |
| Cl2—Mo1—Cl1 | 165.71 (2) | Cl1—Mo1—Cl4 | 83.94 (2) |
| O2—Mo1—Cl3 | 169.07 (5) | Cl3—Mo1—Cl4 | 81.717 (16) |
| O1—Mo1—Cl3 | 88.75 (5) | | |

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| N1—H1$A$⋯Cl6$^{i}$ | 0.88 | 2.16 | 3.0424 (19) | 175 |
| N2—H2$A$⋯Cl5$^{ii}$ | 0.88 | 2.17 | 3.0304 (19) | 166 |
| N3—H3$A$⋯Cl4 | 0.88 | 2.45 | 3.243 (2) | 150 |
| N3—H3$A$⋯Cl3 | 0.88 | 2.69 | 3.277 (2) | 126 |

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 2005); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXTL*.

We wish to thank Dr Martyn Pillinger (CICECO, University of Aveiro) and Dr André D. Lopes (University of the Algarve) for their collaboration in the preparation of this communication. We are also grateful to Fundação para a Ciência e a Tecnologia (FCT, Portugal) for their general financial support, for the post-doctoral research grant SFRH/BPD/63736/2009 (to JAF), SFRH/BPD/25269/2005 (to SG), SFRH/BD/45116/2008 (to SF) and for specific funding toward the purchase of the single-crystal diffractometer. We also wish to thank the Associated Laboratory CICECO for a research grant to AG.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5018).

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*Acta Cryst.* (2010). E**66**, m862–m863 Fernandes *et al.* • $(C_5H_6N)_3[MoCl_4O_2]Cl$ **m863**

# supplementary materials

*Acta Cryst.* (2010). E**66**, m862-m863    [ **doi:10.1107/S1600536810023950** ]

# Tripyridinium *cis*-tetrachloridodioxidomolybdate(VI) chloride

**J. A. Fernandes, A. C. Gomes, S. Figueiredo, S. Gago, P. J. A. Ribeiro-Claro, I. S. Gonçalves and F. A. Almeida Paz**

## Comment

Our research group has been interested in the development of novel catalysts based on dioxomolybdenum complexes (Monteiro *et al.*, 2010; Gago *et al.*, 2009; Pereira *et al.*, 2007; Cunha-Silva *et al.*, 2007; Bruno *et al.*, 2007). During our recent efforts to coordinate pyridine to the molybdenum centre, we have isolated the title compound: $[C_5H_6N]_3[MoCl_4O_2]Cl$. Remarkably, a search in the literature and in the Cambridge Structural Database (Allen, 2002) reveals the existence of only one other structure with the $[MoCl_4O_2]^{2-}$ anion (Luan *et al.*, 2008).

The asymmetric unit of the title compound **I** is composed of three pyridinium (PyH) cations whose charge is balanced by the metallic dianion, $[MoCl_4O_2]^{2-}$, and by two chloride anions located at special positions (Cl5 and Cl6 on $C_2$ axes) The $Mo^{6+}$ centre is coordinated by four chloro and two oxo terminal ligands in a distorted octahedral geometry (Figure 1), $\{MoCl_4O_2\}$. The Mo—Cl distances range from 2.3750 (6) to 2.5953 (5) Å and the Mo═O distance is either 1.6988 (13) or 1.7004 (13) Å (Table 1). The *cis* and *trans* octahedral angles fall within a short range of the ideal values [81.717 (16)—103.11 (7)° and 165.71 (2)—170.36 (5)°, respectively - see Table 1].

The PyH cations are engaged in strong and relatively directional $N^+$—H···$Cl^-$ hydrogen bonds with the chloride anions (not shown; Table 2). We note that N3 is interacting with two spatially close chloro ligands (Cl3 and Cl4), forming a $R_1^2(4)$ graph set motif (Grell *et al.*,1999) typical of bifurcated interactions. The crystal packing is, thus, mediated by the need to effectively fill the available space (Figure 2) since no significant supramolecular contacts are present in the crystal structure (*e.g.*, C—H···O or C—H···Cl contacts are all greater than 3.18 Å).

## Experimental

Chemicals were purchased from commercial sources and were used as received without purification.

To an aqueous solution (30 ml) of HCl (3.3 mol $dm^{-3}$) containing 2.0 g (8.3 mmol) of $Na_2MoO_4\cdot 2H_2O$, a solution of pyridine (1.34 ml, 16.6 mmol) in $CH_2Cl_2$ (60 ml) was slowly added dropwise. The biphasic mixture was vigorously stirred for 3 h at ambient temperature. The aqueous phase was separated and washed three times with $CH_2Cl_2$, and then allowed to evaporate yielding a solid. Crystals of the title compound were ultimately isolated by slow diffusion of diethyl ether into a concentrated solution in acetonitrile.

Crystals of the title compound were ultimately isolated by slow diffusion of diethyl ether into a concentrated solution in acetonitrile. Yield: 65%.

$^1H$ NMR (300.13 MHz, 298 K, $CD_3CN$): δ = 8.75 (*d*, 2H), 8.56 (*t*, 1H), 8.03 (*t*, 2H) p.p.m..

Selected FT—IR (ATR, cm$^{-1}$): 923 [*vs*, $\nu_{sym}$(Mo═O)], 884 [*vs*, $\nu_{asym}$(Mo═O)], 320 [*vs*, ν(Mo—Cl)].

### Refinement

Hydrogen atoms bound to carbon and nitrogen were located at their idealized positions and were included in the final structural model in riding-motion approximation with: C—H = 0.95 Å (aromatic) and N—H = 0.88 Å. The isotropic thermal displacement parameters for these atoms were fixed at 1.2 times $U_{eq}$ of the respective parent atom.

A total of 4490 estimated Friedel pairs have not been merged and were used as independent data for the structure refinement. The Flack parameter (Flack, 1983) converged to 0.03 (4), ultimately assuring a correct absolute structure determination from the single-crystal data set.

### Figures

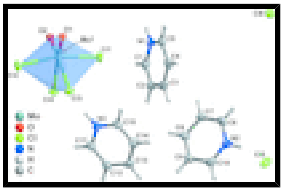

Fig. 1. Schematic representation of the asymmetric unit. The $[MoCl_4O_2]^{2-}$ anion has a highly distorted $\{MoCl_4O_2\}$ octahedral coordination environment, and the anions Cl5 and Cl6 are located on C2 axes. Thermal ellipsoids are drawn at the 50% probability level. For selected bond lengths (in Å) and angles (in degrees) see Table 1.

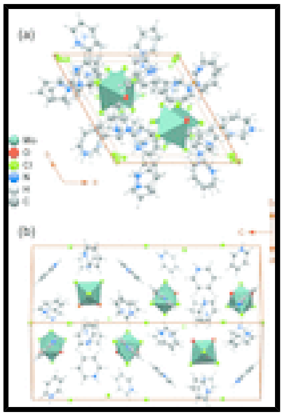

Fig. 2. Crystal packing of the title compound viewed in perspective along the ***(a)*** [001] and ***(b)*** [110] directions of the unit cell.

### Tripyridinium *cis*-tetrachloridodioxidomolybdate(VI) chloride

*Crystal data*

$(C_5H_6N)_3[MoCl_4O_2]Cl$

$M_r$ = 545.51

Trigonal, $P3_121$

Hall symbol: P 31 2"

*a* = 11.3972 (2) Å

*c* = 29.4265 (9) Å

*V* = 3310.28 (13) Å$^3$

*Z* = 6

*F*(000) = 1632

$D_x$ = 1.642 Mg m$^{-3}$

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 6207 reflections

θ = 5.5–33.1°

μ = 1.21 mm$^{-1}$

*T* = 150 K

Block, yellow

0.18 × 0.12 × 0.10 mm

### *Data collection*

| | |
|---|---|
| Bruker X8 Kappa CCD APEXII diffractometer | 10395 independent reflections |
| Radiation source: fine-focus sealed tube | 8105 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{\text{int}} = 0.047$ |
| ω & φ scans | $\theta_{\text{max}} = 36.3°$, $\theta_{\text{min}} = 5.4°$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998) | $h = -18\rightarrow 16$ |
| $T_{\text{min}} = 0.811$, $T_{\text{max}} = 0.888$ | $k = -17\rightarrow 8$ |
| 33230 measured reflections | $l = -48\rightarrow 49$ |

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.038$ | H-atom parameters constrained |
| $wR(F^2) = 0.074$ | $w = 1/[\sigma^2(F_o^2) + (0.0224P)^2 + 0.183P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.05$ | $(\Delta/\sigma)_{\text{max}} = 0.003$ |
| 10395 reflections | $\Delta\rho_{\text{max}} = 0.46$ e Å$^{-3}$ |
| 237 parameters | $\Delta\rho_{\text{min}} = -0.68$ e Å$^{-3}$ |
| 0 restraints | Absolute structure: Flack (1983), 4490 Friedel pairs |
| Primary atom site location: structure-invariant direct methods | Flack parameter: 0.03 (4) |

### *Special details*

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

### *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{\text{iso}}$*/$U_{\text{eq}}$ |
|---|---|---|---|---|
| Mo1 | 0.663054 (16) | 0.338004 (17) | 0.915715 (4) | 0.01643 (3) |
| O1 | 0.77729 (15) | 0.39544 (18) | 0.87246 (4) | 0.0238 (3) |
| O2 | 0.76505 (15) | 0.38526 (17) | 0.96219 (4) | 0.0233 (3) |
| Cl1 | 0.64140 (7) | 0.53687 (6) | 0.916423 (17) | 0.02869 (12) |
| Cl2 | 0.62465 (7) | 0.11310 (6) | 0.914936 (17) | 0.02893 (12) |

| | | | | |
|---|---|---|---|---|
| Cl3 | 0.47182 (5) | 0.25297 (6) | 0.856354 (14) | 0.02601 (11) |
| Cl4 | 0.45883 (5) | 0.24295 (6) | 0.971200 (14) | 0.02623 (11) |
| Cl5 | 1.00836 (8) | 0.0000 | 0.8333 | 0.0421 (2) |
| Cl6 | 0.94835 (8) | 0.94835 (8) | 0.0000 | 0.0352 (2) |
| N1 | 0.6973 (2) | 0.8428 (2) | 0.94054 (6) | 0.0266 (4) |
| H1A | 0.7726 | 0.8765 | 0.9564 | 0.032* |
| N2 | 0.1156 (2) | 0.8720 (2) | 0.89938 (5) | 0.0306 (4) |
| H2A | 0.0989 | 0.9161 | 0.8782 | 0.037* |
| N3 | 0.2586 (2) | 0.3111 (2) | 0.91300 (7) | 0.0333 (5) |
| H3A | 0.3299 | 0.3029 | 0.9186 | 0.040* |
| C1 | 0.5793 (3) | 0.7649 (3) | 0.96154 (7) | 0.0289 (5) |
| H1 | 0.5782 | 0.7449 | 0.9929 | 0.035* |
| C2 | 0.4601 (3) | 0.7139 (3) | 0.93800 (7) | 0.0304 (5) |
| H2 | 0.3759 | 0.6572 | 0.9526 | 0.037* |
| C3 | 0.4649 (3) | 0.7467 (3) | 0.89223 (7) | 0.0321 (5) |
| H3 | 0.3832 | 0.7154 | 0.8756 | 0.039* |
| C4 | 0.5876 (3) | 0.8244 (3) | 0.87119 (7) | 0.0313 (6) |
| H4 | 0.5916 | 0.8454 | 0.8398 | 0.038* |
| C5 | 0.7049 (3) | 0.8714 (3) | 0.89623 (7) | 0.0295 (5) |
| H5 | 0.7907 | 0.9239 | 0.8821 | 0.035* |
| C6 | 0.2380 (3) | 0.9313 (3) | 0.91843 (8) | 0.0335 (6) |
| H6 | 0.3064 | 1.0183 | 0.9085 | 0.040* |
| C7 | 0.2645 (3) | 0.8657 (3) | 0.95249 (8) | 0.0327 (6) |
| H7 | 0.3501 | 0.9082 | 0.9673 | 0.039* |
| C8 | 0.1656 (3) | 0.7374 (3) | 0.96502 (8) | 0.0345 (6) |
| H8 | 0.1833 | 0.6902 | 0.9882 | 0.041* |
| C9 | 0.0411 (3) | 0.6777 (3) | 0.94404 (8) | 0.0367 (6) |
| H9 | −0.0272 | 0.5887 | 0.9522 | 0.044* |
| C10 | 0.0168 (3) | 0.7481 (3) | 0.91116 (8) | 0.0345 (6) |
| H10 | −0.0693 | 0.7094 | 0.8969 | 0.041* |
| C11 | 0.1483 (3) | 0.2387 (3) | 0.93686 (7) | 0.0339 (6) |
| H11 | 0.1464 | 0.1775 | 0.9592 | 0.041* |
| C12 | 0.0362 (3) | 0.2502 (3) | 0.92996 (8) | 0.0361 (6) |
| H12 | −0.0434 | 0.1989 | 0.9475 | 0.043* |
| C13 | 0.0413 (3) | 0.3393 (3) | 0.89645 (9) | 0.0395 (6) |
| H13 | −0.0356 | 0.3487 | 0.8908 | 0.047* |
| C14 | 0.1574 (3) | 0.4132 (3) | 0.87173 (9) | 0.0389 (7) |
| H14 | 0.1618 | 0.4743 | 0.8490 | 0.047* |
| C15 | 0.2673 (3) | 0.3979 (3) | 0.88024 (8) | 0.0351 (6) |
| H15 | 0.3485 | 0.4478 | 0.8633 | 0.042* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Mo1 | 0.01527 (8) | 0.01530 (8) | 0.01882 (5) | 0.00773 (7) | 0.00036 (5) | 0.00003 (5) |
| O1 | 0.0216 (7) | 0.0270 (9) | 0.0249 (5) | 0.0137 (7) | 0.0046 (5) | 0.0054 (6) |
| O2 | 0.0215 (7) | 0.0220 (9) | 0.0248 (5) | 0.0096 (7) | −0.0042 (5) | −0.0012 (6) |
| Cl1 | 0.0361 (3) | 0.0210 (3) | 0.0348 (2) | 0.0187 (3) | −0.0008 (2) | −0.0004 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| Cl2 | 0.0381 (4) | 0.0186 (3) | 0.0334 (2) | 0.0167 (3) | 0.0018 (2) | −0.0006 (2) |
| Cl3 | 0.0213 (2) | 0.0335 (3) | 0.02208 (16) | 0.0128 (2) | −0.00321 (16) | −0.00294 (19) |
| Cl4 | 0.0216 (2) | 0.0311 (3) | 0.02295 (17) | 0.0109 (2) | 0.00463 (16) | 0.00273 (19) |
| Cl5 | 0.0355 (3) | 0.0628 (7) | 0.0371 (3) | 0.0314 (3) | 0.01181 (18) | 0.0236 (4) |
| Cl6 | 0.0300 (3) | 0.0300 (3) | 0.0359 (3) | 0.0077 (4) | −0.00797 (16) | 0.00797 (16) |
| N1 | 0.0273 (10) | 0.0259 (11) | 0.0312 (7) | 0.0169 (10) | −0.0071 (7) | −0.0074 (8) |
| N2 | 0.0337 (13) | 0.0350 (13) | 0.0280 (7) | 0.0208 (10) | −0.0009 (8) | 0.0038 (8) |
| N3 | 0.0245 (12) | 0.0311 (13) | 0.0480 (11) | 0.0166 (10) | 0.0007 (9) | 0.0044 (9) |
| C1 | 0.0393 (14) | 0.0348 (14) | 0.0213 (7) | 0.0250 (12) | 0.0004 (8) | −0.0004 (8) |
| C2 | 0.0297 (12) | 0.0334 (14) | 0.0304 (9) | 0.0174 (11) | 0.0053 (8) | −0.0008 (9) |
| C3 | 0.0338 (13) | 0.0349 (15) | 0.0303 (9) | 0.0192 (12) | −0.0104 (8) | −0.0087 (9) |
| C4 | 0.0473 (16) | 0.0272 (13) | 0.0220 (8) | 0.0206 (12) | −0.0018 (9) | −0.0011 (8) |
| C5 | 0.0323 (13) | 0.0228 (13) | 0.0340 (9) | 0.0141 (11) | 0.0068 (9) | 0.0018 (9) |
| C6 | 0.0337 (15) | 0.0233 (13) | 0.0389 (11) | 0.0108 (12) | 0.0003 (10) | 0.0010 (10) |
| C7 | 0.0234 (13) | 0.0319 (14) | 0.0363 (10) | 0.0091 (11) | −0.0074 (9) | −0.0007 (10) |
| C8 | 0.0315 (14) | 0.0366 (16) | 0.0336 (10) | 0.0157 (13) | 0.0005 (10) | 0.0110 (10) |
| C9 | 0.0247 (13) | 0.0299 (14) | 0.0458 (12) | 0.0064 (11) | 0.0035 (10) | 0.0090 (11) |
| C10 | 0.0224 (13) | 0.0401 (16) | 0.0370 (11) | 0.0126 (12) | −0.0048 (10) | −0.0032 (10) |
| C11 | 0.0409 (16) | 0.0288 (13) | 0.0285 (9) | 0.0148 (12) | 0.0000 (9) | 0.0074 (9) |
| C12 | 0.0300 (14) | 0.0321 (15) | 0.0416 (11) | 0.0122 (12) | 0.0131 (11) | 0.0031 (10) |
| C13 | 0.0250 (14) | 0.0436 (18) | 0.0508 (13) | 0.0178 (14) | −0.0058 (11) | −0.0029 (12) |
| C14 | 0.0353 (15) | 0.0410 (18) | 0.0414 (12) | 0.0200 (14) | −0.0006 (11) | 0.0158 (12) |
| C15 | 0.0325 (15) | 0.0338 (16) | 0.0395 (11) | 0.0169 (13) | 0.0130 (10) | 0.0128 (10) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Mo1—O2 | 1.6988 (13) | C4—C5 | 1.379 (4) |
| Mo1—O1 | 1.7004 (13) | C4—H4 | 0.9500 |
| Mo1—Cl2 | 2.3750 (6) | C5—H5 | 0.9500 |
| Mo1—Cl1 | 2.3995 (6) | C6—C7 | 1.372 (4) |
| Mo1—Cl3 | 2.5746 (5) | C6—H6 | 0.9500 |
| Mo1—Cl4 | 2.5953 (5) | C7—C8 | 1.377 (4) |
| N1—C1 | 1.335 (3) | C7—H7 | 0.9500 |
| N1—C5 | 1.336 (3) | C8—C9 | 1.375 (4) |
| N1—H1A | 0.8800 | C8—H8 | 0.9500 |
| N2—C6 | 1.332 (4) | C9—C10 | 1.371 (4) |
| N2—C10 | 1.338 (4) | C9—H9 | 0.9500 |
| N2—H2A | 0.8800 | C10—H10 | 0.9500 |
| N3—C11 | 1.310 (3) | C11—C12 | 1.363 (4) |
| N3—C15 | 1.349 (3) | C11—H11 | 0.9500 |
| N3—H3A | 0.8800 | C12—C13 | 1.395 (4) |
| C1—C2 | 1.369 (3) | C12—H12 | 0.9500 |
| C1—H1 | 0.9500 | C13—C14 | 1.369 (4) |
| C2—C3 | 1.392 (3) | C13—H13 | 0.9500 |
| C2—H2 | 0.9500 | C14—C15 | 1.370 (4) |
| C3—C4 | 1.373 (4) | C14—H14 | 0.9500 |
| C3—H3 | 0.9500 | C15—H15 | 0.9500 |
| | | | |
| O2—Mo1—O1 | 102.11 (7) | C5—C4—H4 | 120.4 |
| O2—Mo1—Cl2 | 94.45 (6) | N1—C5—C4 | 119.6 (2) |

| | | | |
|---|---|---|---|
| O1—Mo1—Cl2 | 95.78 (6) | N1—C5—H5 | 120.2 |
| O2—Mo1—Cl1 | 94.06 (6) | C4—C5—H5 | 120.2 |
| O1—Mo1—Cl1 | 93.64 (6) | N2—C6—C7 | 119.4 (3) |
| Cl2—Mo1—Cl1 | 165.71 (2) | N2—C6—H6 | 120.3 |
| O2—Mo1—Cl3 | 169.07 (5) | C7—C6—H6 | 120.3 |
| O1—Mo1—Cl3 | 88.75 (5) | C6—C7—C8 | 119.2 (3) |
| Cl2—Mo1—Cl3 | 85.57 (2) | C6—C7—H7 | 120.4 |
| Cl1—Mo1—Cl3 | 83.91 (2) | C8—C7—H7 | 120.4 |
| O2—Mo1—Cl4 | 87.40 (5) | C9—C8—C7 | 120.0 (2) |
| O1—Mo1—Cl4 | 170.36 (5) | C9—C8—H8 | 120.0 |
| Cl2—Mo1—Cl4 | 84.99 (2) | C7—C8—H8 | 120.0 |
| Cl1—Mo1—Cl4 | 83.94 (2) | C10—C9—C8 | 119.2 (3) |
| Cl3—Mo1—Cl4 | 81.717 (16) | C10—C9—H9 | 120.4 |
| C1—N1—C5 | 122.4 (2) | C8—C9—H9 | 120.4 |
| C1—N1—H1A | 118.8 | N2—C10—C9 | 119.4 (2) |
| C5—N1—H1A | 118.8 | N2—C10—H10 | 120.3 |
| C6—N2—C10 | 122.8 (2) | C9—C10—H10 | 120.3 |
| C6—N2—H2A | 118.6 | N3—C11—C12 | 120.6 (2) |
| C10—N2—H2A | 118.6 | N3—C11—H11 | 119.7 |
| C11—N3—C15 | 122.7 (2) | C12—C11—H11 | 119.7 |
| C11—N3—H3A | 118.6 | C11—C12—C13 | 118.4 (3) |
| C15—N3—H3A | 118.6 | C11—C12—H12 | 120.8 |
| N1—C1—C2 | 120.24 (19) | C13—C12—H12 | 120.8 |
| N1—C1—H1 | 119.9 | C14—C13—C12 | 119.9 (3) |
| C2—C1—H1 | 119.9 | C14—C13—H13 | 120.0 |
| C1—C2—C3 | 118.6 (2) | C12—C13—H13 | 120.0 |
| C1—C2—H2 | 120.7 | C13—C14—C15 | 119.2 (3) |
| C3—C2—H2 | 120.7 | C13—C14—H14 | 120.4 |
| C4—C3—C2 | 120.0 (2) | C15—C14—H14 | 120.4 |
| C4—C3—H3 | 120.0 | N3—C15—C14 | 119.1 (3) |
| C2—C3—H3 | 120.0 | N3—C15—H15 | 120.4 |
| C3—C4—C5 | 119.14 (19) | C14—C15—H15 | 120.4 |
| C3—C4—H4 | 120.4 | | |

| | | | |
|---|---|---|---|
| C5—N1—C1—C2 | −1.7 (4) | C7—C8—C9—C10 | −0.9 (4) |
| N1—C1—C2—C3 | −1.0 (4) | C6—N2—C10—C9 | −0.3 (4) |
| C1—C2—C3—C4 | 2.5 (4) | C8—C9—C10—N2 | 1.6 (4) |
| C2—C3—C4—C5 | −1.5 (4) | C15—N3—C11—C12 | −1.3 (4) |
| C1—N1—C5—C4 | 2.8 (4) | N3—C11—C12—C13 | 1.0 (4) |
| C3—C4—C5—N1 | −1.1 (4) | C11—C12—C13—C14 | −0.5 (4) |
| C10—N2—C6—C7 | −1.9 (4) | C12—C13—C14—C15 | 0.2 (5) |
| N2—C6—C7—C8 | 2.6 (4) | C11—N3—C15—C14 | 1.0 (4) |
| C6—C7—C8—C9 | −1.2 (4) | C13—C14—C15—N3 | −0.5 (5) |

### *Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1A···Cl6$^{i}$ | 0.88 | 2.16 | 3.0424 (19) | 175 |
| N2—H2A···Cl5$^{ii}$ | 0.88 | 2.17 | 3.0304 (19) | 166 |

|  |  |  |  |  |
|---|---|---|---|---|
| N3—H3A···Cl4 | 0.88 | 2.45 | 3.243 (2) | 150 |
| N3—H3A···Cl3 | 0.88 | 2.69 | 3.277 (2) | 126 |

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

Fig. 1

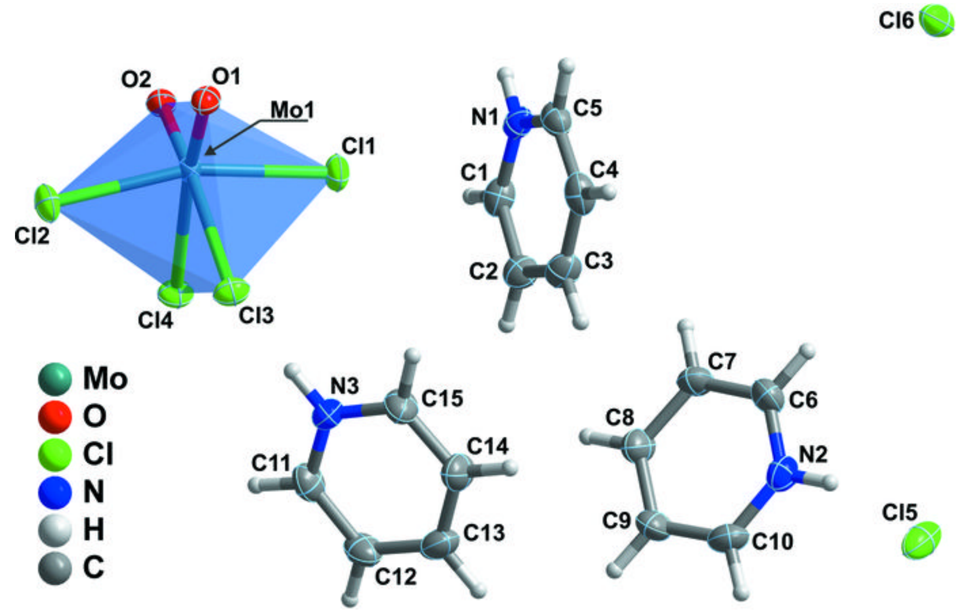
Cl6
O2
O1
Mo1
N1
C5
Cl1
C1
C4
Cl2
C2
C3
Cl4
Cl3
Mo
O
Cl
N
H
C
N3
C15
C7
C6
C8
C14
N2
C11
C13
C9
C10
Cl5
C12

**Fig. 2**