

Acta Cryst. (1997). C53, 1198–1199

**(Acetonitrile-*N*)trichloro( $\eta^5$ -1,3-di-*tert*-butylcyclopentadienyl)molybdenum(IV)**MANFRED SCHEER<sup>a</sup> AND PETER G. JONES<sup>b\*</sup>

<sup>a</sup>Institut für Anorganische Chemie, Universität Karlsruhe, Engesserstrasse, Geb. 30.45, 76128 Karlsruhe, Germany, and <sup>b</sup>Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany. E-mail: jones@xray36.anchem.nat.tu-bs.de

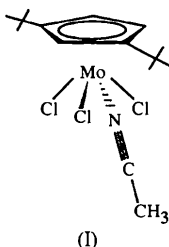
(Received 24 March 1997; accepted 7 April 1997)

**Abstract**

The title compound, [MoCl<sub>3</sub>(C<sub>13</sub>H<sub>21</sub>)(C<sub>2</sub>H<sub>3</sub>N)], displays crystallographic mirror symmetry. The coordination geometry lies between trigonal bipyramidal and square pyramidal. Bond lengths at molybdenum are Mo—N 2.182 (2), and Mo—Cl 2.3909 (6) and 2.4084 (9) Å.

**Comment**

We were recently able to show that reactions of PX<sub>5</sub> (X = Cl, Br) with cyclopentadienyl (Cp) complexes [CpM(CO)<sub>3</sub>]<sub>2</sub> (M = Cr, Mo, W) lead to cyclopentadienyl-halogeno complexes of Cr<sup>III</sup>, Mo<sup>V</sup> and W<sup>V</sup> (Scheer *et al.*, 1990). We report here the structure of the Mo<sup>IV</sup> title complex, (I), [Cp''MoCl<sub>3</sub>(CH<sub>3</sub>CN)], where Cp'' =  $\eta^5$ -1,3-*t*Bu<sub>2</sub>C<sub>5</sub>H<sub>3</sub>, which was obtained by chlorinating the triple-bond derivative [Cp''Mo(CO)<sub>2</sub>]<sub>2</sub>(Mo≡Mo) with PCl<sub>5</sub>.



The molecule (Fig. 1) possesses crystallographic mirror symmetry; the mirror plane contains the atoms C1, Cl2, Mo, N, C8 and C9. Necessarily, Cl2 is then eclipsed by C1, whereas the other Cl and N ligands are staggered with respect to the Cp'' ring. The coordination geometry is irregular, but can approximately be described either as distorted square pyramidal with 'Cent' (the centre of gravity of the five-membered ring) axial or as distorted trigonal bipyramidal with N and Cl2 axial. The distance Mo—Cent is 2.023 Å.

A search of the Cambridge Structural Database (Allen & Kennard, 1993) revealed only one compound containing chloro, acetonitrilo and cyclopentadienyl ligands

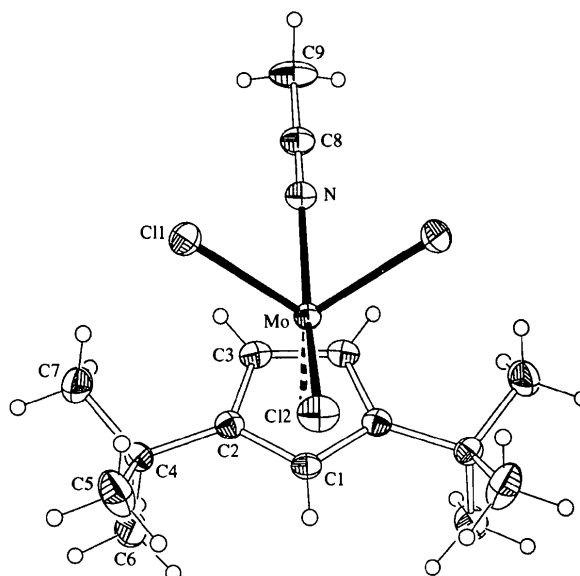


Fig. 1. Structure of the title compound in the crystal. H atoms have been omitted for clarity. Displacement ellipsoids represent 50% probability levels. Only the asymmetric unit is numbered.

bonded to Mo, namely [CpMoCl(NO)(NCCH<sub>3</sub>)<sub>2</sub>]BF<sub>4</sub> (Chin, Legzdins, Trotter & Yee, 1992), with bond lengths Mo—N 2.141 and 2.160 (3), and Mo—Cl 2.416 (1) Å; these are similar to those in the title compound, Mo—N 2.182 (2), and Mo—Cl 2.3909 (6) and 2.4084 (9) Å. A further comparison for Mo—Cl bond lengths is [PhMe<sub>2</sub>PCl<sub>2</sub>]<sub>2</sub>[Mo<sup>IV</sup>Cl<sub>6</sub>] (Cotton & Vidyasagar, 1995) with values of 2.358, 2.374 and 2.398 (1) Å, a large range for chemically equivalent bonds.

**Experimental**

To a solution of 1.05 g (1.6 mmol) [Cp''Mo(CO)<sub>2</sub>]<sub>2</sub> in 60 ml CH<sub>3</sub>CN was added 1.46 g (7 mmol) PCl<sub>5</sub> in small portions at room temperature. After CO evolution was complete (24 h), the solution was filtered. From the filtrate, half of the solvent was removed *in vacuo*, diethyl ether added and the solution stored at 278 K. Dark-brown crystals formed, which were filtered off and washed with diethyl ether [yield 0.48 g (71%)]. Analysis: C<sub>15</sub>H<sub>24</sub>Cl<sub>3</sub>MoN (420.66): calculated C 42.83, H 5.75, N 3.33; found C 42.61, H 5.55, N 3.02%.

**Crystal data**

[MoCl<sub>3</sub>(C<sub>13</sub>H<sub>21</sub>)(C<sub>2</sub>H<sub>3</sub>N)]  
*M<sub>r</sub>* = 420.64  
 Orthorhombic  
*Cmc*2<sub>1</sub>  
*a* = 16.840 (4) Å  
*b* = 10.859 (3) Å  
*c* = 10.039 (2) Å  
*V* = 1835.8 (8) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.522 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

Mo *K*α radiation  
 λ = 0.71073 Å  
 Cell parameters from 50 reflections  
 θ = 10.0–11.5°  
 μ = 1.142 mm<sup>-1</sup>  
*T* = 178 (2) K  
 Equidimensional  
 0.45 × 0.40 × 0.30 mm  
 Red–brown

**Data collection**

Nicolet R3 diffractometer	$R_{\text{int}} = 0.016$
$\omega$ scans	$\theta_{\text{max}} = 27.56^\circ$
Absorption correction:	$h = -21 \rightarrow 21$
$\psi$ scans (XEMP; Nicolet, 1987)	$k = 0 \rightarrow 14$
$T_{\text{min}} = 0.608$ , $T_{\text{max}} = 0.710$	$l = -13 \rightarrow 13$
3627 measured reflections	3 standard reflections
2185 independent reflections	every 147 reflections
2156 reflections with $I > 2\sigma(I)$	intensity decay: none

**Refinement**

Refinement on $F^2$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.017$	$\Delta\rho_{\text{max}} = 0.303 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.044$	$\Delta\rho_{\text{min}} = -0.339 \text{ e } \text{\AA}^{-3}$
$S = 1.123$	Extinction correction: none
2185 reflections	Scattering factors from
103 parameters	<i>International Tables for</i>
Methyl H atoms rigid, other	<i>Crystallography</i> (Vol. C)
H atoms riding	Absolute structure:
$w = 1/[\sigma^2(F_o^2) + (0.0217P)^2 + 1.2752P]$	Flack (1983), number of
where $P = (F_o^2 + 2F_c^2)/3$	Friedel pairs = 1086
	Flack parameter = $-0.04$ (3)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	$x$	$y$	$z$	$U_{\text{eq}}$
Mo	1/2	0.68595 (2)	0.50001 (2)	0.01559 (6)
Cl1	0.61002 (3)	0.63596 (5)	0.64051 (5)	0.02650 (11)
Cl2	1/2	0.87882 (6)	0.61846 (7)	0.02695 (15)
C1	1/2	0.8393 (3)	0.3298 (3)	0.0193 (5)
C2	0.56984 (11)	0.76648 (18)	0.31125 (18)	0.0196 (4)
C3	0.54265 (12)	0.64616 (18)	0.28640 (18)	0.0208 (4)
C4	0.65525 (12)	0.81059 (18)	0.3039 (2)	0.0238 (4)
C5	0.67372 (16)	0.9056 (2)	0.4109 (3)	0.0411 (6)
C6	0.66522 (14)	0.8704 (3)	0.1658 (2)	0.0402 (6)
C7	0.71309 (14)	0.7026 (2)	0.3160 (3)	0.0355 (5)
C8	1/2	0.3833 (3)	0.4639 (3)	0.0242 (6)
C9	1/2	0.2508 (3)	0.4382 (3)	0.0374 (8)
N	1/2	0.4857 (2)	0.4827 (3)	0.0230 (5)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Mo—N	2.182 (2)	Mo—Cent	2.023
Mo—Cl1	2.3909 (6)	C8—N	1.127 (4)
Mo—Cl2	2.4084 (9)	C8—C9	1.463 (5)
N—Mo—Cl1	79.67 (4)	Cl2—Mo—Cent	105.0
Cl1—Mo—Cl1'	101.59 (3)	N—Mo—Cent	100.0
N—Mo—Cl2	154.99 (7)	N—C8—C9	179.5 (3)
Cl1—Mo—Cl2	84.62 (2)	C8—N—Mo	174.9 (2)
Cl1—Mo—Cent	128.9		

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

The origin was fixed according to the method of Flack & Schwarzenbach (1988).

Data collection: *P3 Software* (Nicolet, 1987). Cell refinement: *P3 Software*. Data reduction: *XDISK* (Nicolet, 1987). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL93*.

We thank the Fonds der Chemischen Industrie and the Deutsche Forschungsgemeinschaft for financial support and Mr A. Weinkauff for technical assistance.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1323). Services for accessing these data are described at the back of the journal.

**References**

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
- Chin, T. T., Legzdins, P., Trotter, J. & Yee, V. C. (1992). *Organometallics*, **11**, 913–922.
- Cotton, F. A. & Vidyasagar, K. (1995). *Polyhedron*, **14**, 3077–3085.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Flack, H. D. & Schwarzenbach, D. (1988). *Acta Cryst.* **A44**, 499–506.
- Nicolet (1987). *P3 Program System*. Nicolet Analytical X-ray Instruments, Madison, Wisconsin, USA.
- Scheer, M., Nam, T. T., Schenzel, K., Herrmann, E., Jones, P. G., Fedin, V. P., Ikorski, V. N. & Fedorov, V. E. (1990). *Z. Anorg. Allg. Chem.* **591**, 221–229.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Siemens (1994). *XP. Interactive Molecular Graphics Program*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

*Acta Cryst.* (1997). **C53**, 1199–1201

**(C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>3</sub>)<sub>2</sub>[CdCl<sub>4</sub>]**

MONIKA GROH,<sup>a</sup> ROLAND SPENGLER,<sup>a</sup> HANS BURZLAFF,<sup>a</sup>  
FATMA ZOUARI<sup>b</sup> AND ABDELHAMID BEN SALAH<sup>b</sup>

<sup>a</sup>*Institut für Angewandte Physik, Lehrstuhl für Kristallographie, Universität Erlangen-Nürnberg, Bismarckstraße 10, D-91054 Erlangen, Germany, and* <sup>b</sup>*Faculté des Sciences, Département de Chimie, Université de Sfax, Rte. de Soukra, 3038 Sfax, Tunisia. E-mail: groh@krist.uni-erlangen.de*

(Received 27 January 1997; accepted 14 April 1997)

**Abstract**

The title compound, bis( $\beta$ -phenylethylammonium) tetrachlorocadmate, (C<sub>8</sub>H<sub>12</sub>N)<sub>2</sub>[CdCl<sub>4</sub>], crystallizes as a layer structure; the heavy atoms form a two-dimensional network of corner-connected octahedra, the organic cation being connected to the network through hydrogen bonding.

**Comment**

This crystal, (I), was investigated as part of a series of heavy-atom halides and organic cations (Zouari, Ben Salah, Daoud, Rothammel & Burzloff, 1993) to obtain