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Carbonyl(η^4-1,3-cyclohexadiene)[(η^5,κN)-2-methylaminoethylcyclopentadienyl]-molybdenum(II) Hexafluorophosphate

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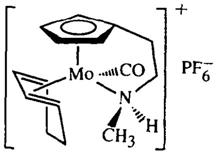
Abstract

The pseudotetrahedral structure of [Mo($C_8H_{12}N$)(C_6H_8)(CO)]PF_6 has a *transoid* CH_3—N—Mo—CO arrangement. The amino group coordinates to the Mo atom intramolecularly, causing the side chain to bend away from the cyclopentadienyl plane by about 9.3°.

Comment

The title compound, carbonyl(η^4-1,3-cyclohexadiene)[(η^5,κN)-2-methylaminoethylcyclopentadienyl]molybdenum(II) hexafluorophosphate, (I), was obtained in about 10% yield during the preparation of the corresponding *N*,*N*-dimethyl complex (Wang & Wen, 1992). The structure determination was undertaken in order to verify that demethylation had occurred and also to establish the relative stereochemistry of the methyl and carbonyl groups.



(I)

As shown in Fig. 1, the *N*-methyl group points away from the carbonyl group to minimize steric interaction. The torsion angles C7—C2—C3—C4 of −170.6 (10) and C7—C2—C6—C5 of 170.7 (9)° suggest that the C7—C2 bond is bent away from the cyclopentadienyl plane by about 9.3°. The cyclohexadiene ring has a boat conformation with two planes, C11—C12—C13—C14 and C14—C15—C16—C11, inclined at an angle of about 45° to each other. The linear Mo—C1—O1 moiety [173.8 (5)°] resides on the top of the *exo*-η^4-cyclohexadiene group. The Mo—N bond length [2.282 (5) Å] is slightly shorter (0.1 Å) than those observed in similar complexes (Wang & Wen, 1992; Wang, Lee, Chou & Ong, 1992).

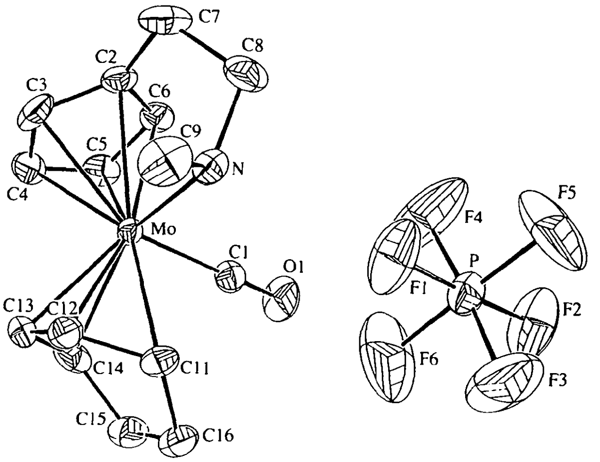


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of [Mo($C_8H_{12}N$)(C_6H_8)(CO)]PF_6. Displacement ellipsoids are shown at the 30% probability level.

Experimental

Crystals of carbonyl(η^4-1,3-cyclohexadiene)[(η^5,κN)-2-methylaminoethylcyclopentadienyl]molybdenum(II) hexafluorophosphate were obtained from acetone/dichloromethane solution.

Crystal data

[Mo($C_8H_{12}N$)(C_6H_8)(CO)]-PF_6
$M_r = 471.23$
Monoclinic
$P2_1/n$
$a = 9.5234 (20)$ Å
$b = 14.528 (5)$ Å
$c = 12.913 (3)$ Å
$\beta = 99.58 (2)°$
$V = 1761.6 (8)$ Å^3
$Z = 4$
$D_x = 1.777$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 7.43$–$17.54°$
$\mu = 0.898$ mm^{-1}
$T = 295$ K
Prism
0.39 × 0.38 × 0.34 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$2\theta/\theta$ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.968$, $T_{\max} = 0.998$
2449 measured reflections
2290 independent reflections

2026 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.022$
$\theta_{\max} = 22.5°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 15$
$l = -13 \rightarrow 13$
3 standard reflections
frequency: 60 min
intensity decay: 1%

Refinement

Refinement on F
$R = 0.035$
$wR = 0.045$
$S = 2.73$
2026 reflections
306 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{max} = 0.101$
$\Delta\rho_{max} = 0.680$ e Å^{-3}
$\Delta\rho_{min} = -0.600$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Mo	0.20106 (4)	0.18106 (3)	0.19109 (3)	2.51 (2)
P	0.69192 (16)	−0.00763 (11)	0.23347 (13)	4.33 (7)
N	0.4223 (5)	0.2016 (4)	0.1489 (4)	3.7 (2)
O1	0.2150 (5)	−0.0314 (3)	0.1908 (4)	6.4 (3)
F1	0.7065 (5)	0.1008 (3)	0.2395 (4)	8.5 (3)
F2	0.6767 (5)	−0.1154 (3)	0.2236 (5)	9.3 (3)
F3	0.8005 (7)	−0.0170 (5)	0.3350 (4)	12.1 (4)
F4	0.5751 (8)	0.0032 (4)	0.1382 (6)	16.4 (5)
F5	0.8138 (7)	−0.0122 (4)	0.1730 (6)	13.8 (5)
F6	0.5699 (7)	−0.0044 (5)	0.2983 (7)	16.0 (6)
C1	0.2179 (6)	0.0475 (4)	0.1933 (4)	3.7 (3)
C2	0.1754 (6)	0.2449 (4)	0.0203 (4)	3.9 (3)
C3	0.1053 (8)	0.3040 (4)	0.0825 (5)	4.8 (3)
C4	−0.0054 (7)	0.2545 (5)	0.1153 (5)	4.9 (3)
C5	−0.0063 (7)	0.1655 (5)	0.0756 (5)	4.5 (3)
C6	0.1037 (7)	0.1597 (5)	0.0159 (4)	4.0 (3)
C7	0.3092 (8)	0.2628 (7)	−0.0206 (6)	6.0 (4)
C8	0.4188 (8)	0.1951 (6)	0.0325 (6)	5.5 (4)
C9	0.5030 (9)	0.2844 (6)	0.1922 (8)	5.9 (4)
C11	0.3465 (6)	0.1565 (4)	0.3525 (4)	3.5 (2)
C12	0.2795 (7)	0.2440 (4)	0.3482 (4)	4.0 (3)
C13	0.1309 (7)	0.2395 (4)	0.3354 (4)	4.3 (3)
C14	0.0716 (7)	0.1512 (5)	0.3273 (5)	4.3 (5)
C15	0.1347 (8)	0.0756 (5)	0.4004 (5)	5.0 (3)
C16	0.2969 (7)	0.0805 (5)	0.4178 (5)	4.4 (3)

Table 2. *Selected geometric parameters (Å, °)*

Mo—N	2.282 (5)	C2—C3	1.417 (9)
Mo—C1	1.947 (6)	C2—C6	1.410 (9)
Mo—C2	2.368 (5)	C2—C7	1.481 (9)
Mo—C3	2.359 (6)	C3—C4	1.399 (11)
Mo—C4	2.306 (6)	C4—C5	1.390 (11)
Mo—C5	2.279 (6)	C5—C6	1.403 (9)
Mo—C6	2.318 (5)	C7—C8	1.513 (11)
Mo—C11	2.330 (5)	C11—C12	1.419 (8)
Mo—C12	2.237 (5)	C11—C16	1.512 (8)
Mo—C13	2.248 (5)	C12—C13	1.399 (10)
Mo—C14	2.351 (6)	C13—C14	1.399 (10)
N—C8	1.500 (9)	C14—C15	1.505 (9)
N—C9	1.487 (9)	C15—C16	1.525 (10)
O1—C1	1.147 (7)		
N—Mo—C1	93.3 (2)	C6—C2—C7	125.5 (6)
C1—Mo—C11	78.4 (2)	C11—C12—C13	113.7 (6)
C1—Mo—C12	112.3 (2)	C11—C16—C15	110.8 (5)
C1—Mo—C13	113.5 (2)	C12—C11—C16	119.8 (6)
C1—Mo—C14	81.8 (2)	C12—C13—C14	116.1 (6)
C2—C3—C4	108.1 (6)	C13—C14—C15	120.5 (6)
C2—C6—C5	109.1 (6)	C14—C15—C16	110.3 (5)
C2—C7—C8	107.0 (5)	Mo—N—C8	111.3 (4)
C3—C4—C5	109.0 (6)	Mo—N—C9	117.1 (5)
C3—C2—C6	106.5 (5)	Mo—C1—O1	173.8 (5)
C3—C2—C7	127.6 (7)	C8—N—C9	110.6 (6)
C4—C5—C6	107.4 (6)	N—C8—C7	108.2 (6)
C5—Mo—N—C8	−4.9 (4)	C11—C12—C13—C14	−0.1 (4)
C4—Mo—C1—O1	−3.9 (4)	C12—C13—C14—C15	44.5 (5)
C7—C2—C3—C4	−170.6 (10)	C14—C15—C16—C11	−3.2 (4)
C7—C2—C6—C5	170.7 (9)	C16—C11—C12—C13	−45.0 (5)
C2—C3—C4—C5	−0.5 (4)		

All the data processing was carried out on a MicroVAX 3600 computer using the *NRCVAX* program system (Gabe, Le Page, White & Lee, 1987).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1051). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Cadmium(II) Chloride Adduct with an Olefinic Double Betaine: *catena*-Poly[tetraaquadiethanoltris[dichlorocadmium(II)] *cis*-bis(4-dimethylamino-1-pyridinio)butenedioate hydrate]

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Abstract

The structure of a cadmium(II) chloride adduct with an olefinic double betaine {systematic name: *catena*-poly[[(diaquacadmium)-di-μ-chloro-(diethanolcadmium)-di-μ-chloro-(diaquacadmium)-di-μ-chloro] *cis*-bis(4-dimethylamino-1-pyridinio)butenedioate monohydrate]}, $[(CdCl_2)_3(C_2H_6O)_2(H_2O)_4]_n.n(C_{18}H_{20}N_4O_4).nH_2O$, is based on an infinite zigzag chain consisting of a series of corner-sharing $CdCl_2$ quadrilaterals with aqua or ethanol ligands around each Cd^{II} atom, forming a $CdCl_4O_2$ octahedral coordination polyhedron. The olefinic double betaine has no association with the metal atoms, being bound to the aqua and ethanol ligands in the chain and to the lattice water molecule by hydrogen bonding.