known to be longer than the ones in cyclohexane.

The C_2GeC_6 valence angle in GCH (102.5 (35)°) is smaller than tetrahedral, as the CSiC angle in silacyclohexane (104.2 (14)°), while the CCC angle in cyclohexane is 111.4 (19)°. This variation is expected in order for the long Ge–C and Si–C bonds to close the ring. Both GCH and DMGCH exist in a flattened chair conformation. The chair at the germanium end is less puckered than the carbon end of the molecule. Similar puckering angles were observed in silacyclohexane.

Molecular mechanics (MM2) calculations using a force field reported by Ouelette have been carried out by Takeuchi and co-workers.[1] The calculated Ge–C bond length is shorter than the experimental value. The Ge–C_m bond length is found to be 0.002 Å longer than the Ge–C_2 bond in the ring. The two different types of C–C bonds have a calculated difference of 0.005 Å and an average value of 1.540 Å, which are in fairly good agreement with the experimental results. The calculated valence angles and torsional angles in general agreed very well with the observed values. Calculations using the MM3[12] force field

showed that the Ge–C_m bonds in DMGCH are 0.02 Å shorter than the Ge–C_2 bonds. The experimental average Ge–C bond lengths for GCH (1.956 (4) Å) and DMGCH (1.957 (2) Å) suggest that the Ge–C bonds in DMGCH should be very similar in length. MM3 calculations showed that for dimethylsilacyclohexane, the two types of Si–C bonds are virtually identical.

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Synthesis and Structure of the Trimolybdenum Cluster [(CpMoCl)₃(μ-Cl)₄(μ₃-O)]

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Summary: The title compound is obtained from CpMoCl₃ in warm THF. The source of the oxygen atom is the solvent, the byproduct being 1,4-dichlorobutane. The structure shows a triangular array of molybdenum atoms without direct metal–metal bonds. Each atom bears one cyclopentadienyl group and a terminal Cl ligand, and four additional Cl atoms edge-bridge the triangular unit, two on one edge and the others one each on the two remaining edges. The oxygen atom is bonded to all three Mo atoms to complete a pseudooctahedral geometry for two metals and a four-legged piano stool geometry for the other metal. Crystal data: monoclinic, $P2_1/m$, $a = 7.950$ (2) Å, $b = 13.442$ (4) Å, $c = 10.049$ (4) Å, $\beta = 97.87$ (3)°, $V = 1063.7$ (6) Å³, $Z = 2$, and $R = 2.98\%$. The solid-state structure is consistent with the magnetic susceptibility and with the ¹H-NMR spectrum of the compound. These indicate that two unpaired electrons are localized on the electronically unsaturated unique metal.

Recent investigations in our laboratory have focused on the chemistry of cyclopentadienylmolybdenum(III) complexes.[2] The most convenient starting material for these

complexes is $CpMoCl_2$ ($Cp = \eta^5\text{-}C_5H_5$), which can be prepared by reduction of $CpMoCl_4$.[3] We have previously reported the kinetics and mechanism of the halide exchange on the $CpMoX_2(PMe_3)_2$ (X = Cl, I) class of compounds.[4] During these studies it was discovered that these substitutions are catalyzed by oxidation leading to the production of complexes with the metal in the +IV oxidation state.[5] This observation led to our interest in synthesizing cyclopentadienylmolybdenum(IV) complexes in order to probe their role as catalytic entities in the above substitution reactions. An attractive entry into this chemistry is $CpMoCl_3$, a material that was not reported before we began our investigations.[6] We have recently[7] described the preparation and the physical properties of this compound, but because of its practically complete insolubility in organic solvents, its molecular structure could not be determined. To date only a relatively few examples of (ring)MX₃ compounds have been characterized structurally.[6] However, we noted that the material dissolves in warm THF. The compound obtained by this treatment, which results from oxygen abstraction from the

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Table I. Crystallographic Data for [(CpMoCl)(μ-Cl)$_4$O]

(a) Crystal Parameters

formula	$C_{15}H_{15}Cl_7OMo_3$	V, Å^3	1063.7 (6)
fw	747.24	Z	2
cryst system	monoclinic	cryst dimens, mm	0.26 × 0.38 × 0.41
space group	$P2_1/m$ (No. 11)	cryst color	black
a, Å	7.950 (2)	D(calc), g cm^3	2.333
b, Å	13.442 (4)	μ(Mo Kα), cm^{-1}	24.67
c, Å	10.049 (4)	temp, K	296
β, deg	97.866 (31)	T(max)/T(min)	1.113

(b) Data Collection

diffractometer	Nicolet R3m		
monochromator	graphite	no. of indpt rflns	3222
radiation (λ, Å)	MoKα (0.710 73)	R(merg), %	1.53
2θ scan range, deg	4–60	no. of indpt obsvd rflns $F_o \geq n\sigma(F_o)$ (n = 5)	2779
data collected (h,k,l)	±12,+19,+15	std rflns	3 std/197 rflns
no. of rflns collcd	3391	var in stds	~2

(c) Refinement

$R(F)$, %	2.98	$\Delta(\rho)$, e Å^{-3}	1.138
$R_w(F)$, %	3.53	N_o/N_v	21.7
Δ/σ(max)	0.003	GOF	1.146

solvent, is described in this note.

Experimental Section

All operations were carried out under an atmosphere of dinitrogen using standard glovebox and Schlenk line techniques. Solvents were dehydrated by conventional methods and distilled under dinitrogen prior to use. Instruments used were as follows: IR, Nicolet 5CDX (4000–400 cm^{-1}) and Perkin-Elmer 1800 (400–200 cm^{-1}); NMR, Bruker WP200; UV/visible, Milton Roy Spectronic 3000 array spectrophotometer with IBM software; magnetic susceptibility, Johnson-Matthey magnetic balance. The elemental analyses were by M-H-W Labs, Phoenix, AZ. CpMoCl$_3$ was prepared from CpMoCl$_2$ and PhI·Cl$_2$ following the literature procedure.[7]

Synthesis of [(CpMoCl)$_3$(μ-Cl)$_4$(μ_3-O)]. A slurry of CpMoCl$_3$ (0.46 g, 1.72 mmol) in 30 mL of THF was warmed to ca. 40 °C. Most of the solid went into solution within 1 h, leaving only minor amounts of a pale brown undissolved material (probably CpMoCl$_2$ that was present as a contaminant in the starting material). After filtration, the brown solution was carefully layered with 20 mL of Et$_2$O. Upon completion of the diffusion process, 174 mg of product (40.6% yield) in the form of well-formed crystals was collected by filtration and washed with Et$_2$O. One crystal from this batch was used for the X-ray analysis. NMR (CD$_2$Cl$_2$, T = 298 ± 2 K, δ): 147.1 (s, $w_{1/2}$ = 1200 Hz, 5 H), 4.6 (s, $w_{1/2}$ = 70 Hz, 10 H). μ_{eff} = 3.47 μ_B (molar diamagnetic correction: −277.85 × 10^{-6} cgsu). IR (Nujol mull, cm^{-1}): 3100 w, 1016 m, 838 s, 615 s, 578 w, 532 m, 385.3 vw, 371.1 w, 344.4 w, 325.6 vw, 305.1 m, 291.8 s, 281.8 s, 263.8 m, 246.7 m. The compound is only sparingly soluble in CH$_2$Cl$_2$ and even less soluble in other organic solvents. Once crystallized out of solution, it is practically insoluble in THF.

A similar experiment was repeated, and the final solution was investigated by gas chromatography. The formation of 1,4-dichlorobutane was confirmed by comparison with the gas chromatogram of a THF solution of an authentic sample (Aldrich).

X-ray Crystallography for [(CpMoCl)$_3$(μ-Cl)$_4$(μ_3-O)]. A black crystal, suitable for data collection, was mounted on a fine glass fiber with epoxy cement. The unit cell parameters were obtained from the least-squares fit of 25 reflections (20° ≤ 2θ ≤ 25°). Preliminary photographic characterization showed 2/m Laue symmetry, and the systematic absences in the diffraction data established the space group as $P2_1$ (No. 4) or $P2_1/m$ (No. 11). E-statistics suggested the centrosymmetric alternative, and the chemically sensible results of refinement proved $P2_1/m$ to be the correct space group. No absorption correction was applied to the data set (low μ, T_{max}/T_{min} = 1.113).

The structure was solved by direct methods which located the Mo atoms. The remaining non-hydrogen atoms were located through subsequent difference Fourier and least-squares syntheses. The hydrogen atoms were included as idealized isotropic contributions (d(CH) = 0.960 Å, U = 1.2U for attached C). All non-hydrogen atoms were refined with anisotropic thermal parameters. The molecule sits on the mirror plane which contains

Table II. Atomic Coordinates (×10^4) and Isotropic Thermal Parameters (Å^2 × 10^3) for [(CpMoCl)$_3$(μ-Cl)$_4$O]

	x	y	z	U^a
Mo(1)	2409.9 (3)	8714.7 (2)	7657.1 (3)	23.0 (1)
Mo(2)	3146.5 (5)	7500	4718.7 (4)	22.4 (1)
Cl(1)	−6 (1)	7500	7071 (1)	33 (1)
Cl(2)	3430 (2)	7500	9464 (1)	33 (1)
Cl(3)	1936 (1)	9141 (1)	5199 (1)	33 (1)
Cl(4)	5379 (1)	9258 (1)	7696 (1)	38 (1)
Cl(5)	1041 (2)	7500	2797 (2)	44 (1)
O(1)	3285 (4)	7500	6729 (3)	20 (1)
C(1)	587 (5)	9163 (3)	9030 (4)	42 (1)
C(2)	2137 (7)	9585 (4)	9641 (5)	50 (1)
C(3)	2581 (6)	10304 (3)	8785 (5)	50 (1)
C(4)	1401 (6)	10349 (3)	7637 (5)	45 (1)
C(5)	122 (5)	9665 (3)	7767 (4)	39 (1)
C(6)	6095 (7)	7500	5272 (6)	45 (2)
C(7)	5660 (5)	6659 (3)	4486 (5)	50 (2)
C(8)	4925 (6)	6982 (4)	3213 (5)	55 (2)

^{a} Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Table III. Relevant Bond Distances and Bond Angles for [(CpClMo)$_3$(μ-Cl)$_4$O]

Bond Distances			
Mo(1)–Cl(1)	2.528 (1)	Mo(1)–Cl(2)	2.494 (1)
Mo(1)–Cl(3)	2.514 (1)	Mo(1)–Cl(4)	2.466 (1)
Mo(1)–O(1)	2.049 (2)	Mo(2)–Cl(3)	2.480 (1)
Mo(2)–Cl(5)	2.376 (1)	Mo(2)–O(1)	2.009 (3)
Mo(1)–CNT(1)^a	1.981 (4)	Mo(2)–CNT(2)^a	2.000 (4)

Bond Angles (deg)			
Cl(1)–Mo(1)–Cl(2)	84.7 (1)	Cl(1)–Mo(1)–Cl(3)	84.9 (1)
Cl(1)–Mo(1)–Cl(4)	152.9 (1)	Cl(2)–Mo(1)–Cl(3)	148.8 (1)
Cl(2)–Mo(1)–Cl(4)	88.1 (1)	Cl(3)–Mo(1)–Cl(4)	87.8 (1)
Cl(1)–Mo(1)–O(1)	71.0 (1)	Cl(2)–Mo(1)–O(1)	73.3 (1)
Cl(3)–Mo(1)–O(1)	75.5 (1)	Cl(4)–Mo(1)–O(1)	81.9 (1)
Cl(1)–Mo(1)–CNT(1)	103.7 (1)	Cl(2)–Mo(1)–CNT(1)	105.4 (1)
Cl(3)–Mo(1)–CNT(1)	105.7 (1)	Cl(4)–Mo(1)–CNT(1)	103.5 (1)
O(1)–Mo(1)–CNT(1)	174.5 (4)	Mo(1)–O(1)–Mo(2)	118.8 (1)
Mo(1)–O(1)–Mo(1A)	105.7 (1)	Mo(1)–Cl(1)–Mo(1A)	80.5 (1)
Mo(1)–Cl(2)–Mo(1A)	81.8 (1)	Mo(1)–Cl(3)–Mo(2)	88.8 (1)
Cl(3)–Mo(2)–Cl(3A)	125.6 (1)	Cl(3)–Mo(2)–Cl(5)	84.8 (1)
Cl(3)–Mo(2)–O(1)	76.9 (1)	Cl(5)–Mo(2)–O(1)	138.9 (1)
Cl(3)–Mo(2)–CNT(2)	116.9 (1)	Cl(5)–Mo(2)–CNT(2)	109.5 (1)
O(1)–Mo(2)–CNT(2)	111.7 (1)		

^{a} CNT(1) = centroid of atoms C(1)–C(5); CNT(2) = centroid of atoms C(6), C(7), C(8), C(7A), and C(8A).

Mo(2), O(1), Cl(1), Cl(2), Cl(5), and C(6). Table I contains crystal and data collection parameters, and Table II contains the positional and equivalent isotropic thermal parameters, while relevant bond distances and bond angles are listed in Table III.
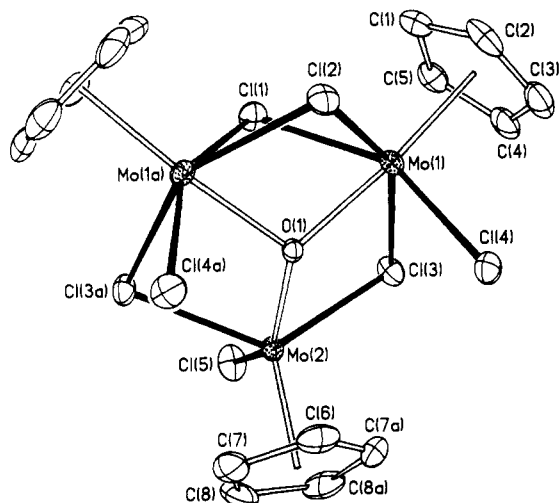
Figure 1. ORTEP drawing and labeling scheme for [(CpMoCl)₃(μ-Cl)₄(μ₃-O)]. Ellipsoids are at 35% probability. Hydrogen atoms have been omitted for clarity.

All computer programs and the sources of the scattering factors are contained in the SHELXTL program library.[8] Additional crystallographic data are available as supplementary material.

Results and Discussion

When the sparingly soluble CpMoCl₃ material was gently warmed (40 °C) in THF, dissolution occurred and the trinuclear oxo-capped cluster Cp₃Mo₃Cl₇O was formed. The stoichiometry of eq 1, which indicates THF as the source of the oxygen atom, is suggested by the gas-chromatographic identification of the 1,4-dichlorobutane by-product.

$$3CpMoCl_3 + OC_4H_8 \rightarrow Cp_3Mo_3Cl_7O + Cl(CH_2)_4Cl \quad (1)$$

The compound has also been obtained by direct reduction of CpMoCl₄ with Zn (0.5 equiv) in THF (eq 2).

$$6CpMoCl_4 + 3Zn + 2OC_4H_8 \rightarrow$$
$$2Cp_3Mo_3Cl_7O + 3ZnCl_2 + 2Cl(CH_2)_4Cl \quad (2)$$
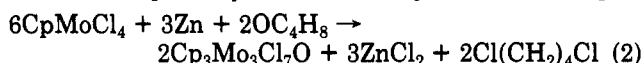
When 1 equiv of Zn is utilized, the reduction proceeds to the insoluble Mo(III) compound CpMoCl₂, which can be recovered in good yields.[3] Since it is known that the conproportionation of CpMoCl₂ and CpMoCl₄ in CH₂Cl₂ affords CpMoCl₃,[7] the use of 0.5 equiv of Zn is supposed to reduce CpMoCl₄ to CpMoCl₃. However, since the latter compound is able to deoxygenate THF quite readily according to eq 1, and since the Zn reduction is slow, the title compound is obtained directly from this procedure. Separation of the cluster from the THF-soluble ZnCl₂ by-product, however, made this procedure less desirable than that described in eq 1.

Our intent in carrying out reaction 1 was to recrystallize the starting compound CpMoCl₃ either as such or as a THF adduct. THF adducts of other CpMX₃ molecules are known, e.g. CpZrCl₃(THF)₂,[9] and it has been established that other neutral donors form adducts with CpMoCl₃ [e.g. dmpe, dppe, and P(OCH₂)₃CEt].[7] However, the NMR spectroscopic properties of the product (vide infra) were inconsistent with either formulation and did not allow an unequivocal structural assignment. Therefore, an X-ray structural analysis was undertaken.

Figure 1 shows an ORTEP view of the molecule. Crystallographic and data collection parameters are listed in

Table I, and selected bond distances and angles are assembled in Table II. The molecule can be best viewed as composed of three distinct fragments, two pseudooctahedral (considering the Cp ligand as occupying a single coordination site) CpMoCl₄O fragments and one four-legged piano stool CpMoCl₃O fragment, which are held together by the capping oxygen and the bridging chlorine atoms. Each Mo center bears a terminal Cl ligand, and the additional four Cl ligand occupy bridging positions. The two pseudooctahedral centers Mo(1) and Mo(1a) are bridged by two Cl ligands, whereas the other two edges of the Mo₃ triangle are bridged by a single Cl ligand. The oxygen atom is elevated 0.48 Å from the Mo(1), Mo(2), Mo(1a) plane toward Cl(2), Cl(4) and Cl(4a). There are no metal-metal bonds in the molecule (Mo–Mo separations are 3.266 Å for the unique distance and 3.494 Å for the two symmetry-related distances), nor are any expected on the basis of simple electronic considerations (vide infra). The molecule is in all respects structurally equivalent to the zirconium trimer Cp*₃Zr₃Cl₇O, where the metal–metal separations are in the 3.478–3.562-Å range.[10] The shorter metal–metal separations in the Mo trimer probably reflect the smaller size of the Mo⁴⁺ ion as compared with the Zr⁴⁺ ion. In analogy to the zirconium structure, the shorter Mo–O and Mo–Cl distances are those involving the electronically unsaturated unique metal, indicating Mo–O and Mo–Cl π bonding. The group 5 Cp*₃M₃(μ₂-O)₃(μ₃-O)(μ₂-Cl)Cl₃ (M = Nb, Ta) compounds also have a structure identical to that of the title compound, although in those cases three bridging chlorine atoms have been replaced by as many oxygen atoms.[11]

The average oxidation state for the metal is IV, and the most logical assignment of oxidation states around the molecule is IV for each of the three metal centers. According to this model, the two equivalent Mo(1) and Mo(1a) centers reach a 18-electron configuration, whereas Mo(2) has a 16-electron configuration. Four-legged piano stool, mononuclear Mo(IV) (d²) complexes often have a high-spin configuration (S = 1),[2a,5] due to the difference in orbital energy between the d_{xy} and d_{z^2} frontier orbitals[12] being smaller than the pairing energy. Two unpaired electrons are therefore predicted for the trinuclear cluster, and these should be localized on the unique metal center. The magnetic and ¹H-NMR properties of the compound are consistent with this view.

Two ¹H-NMR broad resonances are observed for the Cp protons in an approximate 2:1 ratio. The resonance corresponding to the unique Cp ligand exhibits a large paramagnetic shift at δ 147.1 ppm, consistent with the paramagnetism of one of the three metal centers. The value of the chemical shift for this paramagnetically shifted signal is comparable with the Cp proton signals of other 16-electron, mononuclear Mo(IV) compounds, for instance δ 179.5 and 181.0 for [CpMoX₂(PMe₃)₂]⁺PF₆⁻ (X = Cl and I, respectively) and δ 145.4 for CpMoCl₃(PMe₃).[5] The ¹H-NMR resonance for the two equivalent Cp ligands is found at δ 4.6, a typical value for protons of Cp ligands bonded to diagmagnetic centers. This signal is nevertheless fairly broad, although much less so than that of the unique Cp protons, probably because of the proximity to the paramagnetic, 16-electron center. The room-temper-

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ature magnetic moment is 3.47 μ_B, only slightly higher than the value expected for two unpaired electrons (2.83 μ_B). Minor amounts of mononuclear impurities might possibly be responsible for this discrepancy. A through-the-bridge magnetic interaction between the various Mo centers could only contribute to decrease the spin-only magnetic moment.

It is interesting to compare the NMR properties of our trimolybdenum complex with those of the isostructural Cp–zirconium complex mentioned above. The latter compound shows only one set of resonances for the inequivalent Cp rings both in the ^{1}H and in the ^{13}C-NMR[10] spectra. This obviously implies that a fluxional process is exchanging the three metal atoms, a likely possibility for the exchange mechanism being the rupture and reformation of M–(μ-Cl) bonds. The static nature of the trimolybdenum cluster in the ^{1}H-NMR time scale indicates that Mo(IV) is less labile than Zr(IV), an observation that may be easily rationalized on the basis of the increase in M–Cl bond covalency upon moving from left to right of the transition series. The low solubility of our Mo trimer and its paramagnetism prevented us from obtaining a ^{13}C-NMR spectrum. The compound decomposes upon warming in 1,2-dichloroethane-d_4 and is not sufficiently soluble in other high boiling point perdeuterated solvents; thus, we cannot establish the likelihood of dynamic processes at higher temperatures at this time.

Several examples of trimetallic complexes supported by a combination of Cp (or substituted Cp) groups and halide and oxo ligands are known. As a rule, these species are confined to the oxophilic transition metals in groups 4 and 5 of the periodic table, and they are generally obtained by partial hydrolysis of CpMX$_n$ precursors.[6] Examples for group 4 are [Cp*TiCl(μ-O)]$_3$,[13] [Cp*MCl]$_3$O(OH)$_3$X (M = Zr, Hf; X = Cl, OH),[14,15] and the above mentioned

[Cp*ZrCl]$_3$(μ-Cl)$_4$(μ_3-O).[10] The latter was also obtained from Cp*ZrCl$_3$ and (Me$_3$Si)$_2$O.[10] The scavenging of oxygen from THF by CpMoCl$_3$, compared to the formation of a THF adduct for the analogous CpZrCl$_3$ compound, would seem to indicate, quite surprisingly, a higher oxophilicity of Mo(IV) with respect to Zr(IV). Examples for group 5 are the above mentioned Cp*$_3$M$_3$(μ_3-O)(μ-O)$_3$(μ-Cl)Cl$_3$ (M = Nb, Ta) compounds.[11] For group 6, we are only aware of the mononuclear CpMoOX$_2$, CpMoO$_2$X (X = Cl, Br), Cp*MO$_2$Cl, and Cp*MOCl$_3$ (M = Mo, W)[16] and the dinuclear [CpMoOX]$_2$O (X = Cl, I)[17] as complexes containing a mixture of Cp, oxo, and halide ligands.[18] The compound reported here is the first trinuclear group 6 metal compound with these characteristics. During unrelated research efforts, we have obtained another cluster of this kind, [(Cp*Mo)$_3$(μ-Cl)$_5$(μ_2-O)]$^+$. This compound will be reported separately.[19]

Supplementary Material Available: For the compound (CpMoCl)$_3$(μ-Cl)$_4$(μ_3-O), full tables of bond distances and angles, anisotropic thermal parameters, and H-atom coordinates (3 pages); a table of observed and calculated structure factors (9 pages). Ordering information is given on any current masthead page.

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