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Antiferromagnetic complexes with metal–metal bonds

XXII *. Synthesis, molecular structure and magnetic properties of the salt $[Cp_3Cr_3(\mu_3\text{-}O)(\mu\text{-}OCMe_3)_3]^+[CpMo(CO)_3]^-$ with a trinuclear cyclopentadienyl-oxo-t-butoxide cluster cation

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Abstract

The reaction of $Cp_2Cr_2(\mu\text{-}OR)_2$ (I, R = CMe_3) with $[CpMo(CO)_3]_2$ has been studied. It has been shown that in the first stage an extremely unstable adduct of I with a $CpMo(CO)_3$ group (isocarbonyl-type coordination) is formed. This adduct is easily oxidized by atmospheric oxygen, forming a 45ē antiferromagnetic trinuclear cation $[Cp_3Cr_3(\mu_3\text{-}O)(\mu\text{-}OCMe_3)_3]^+$ and $[CpMo(CO)_3]^-$ anion (V, $-2J$(Cr–Cr) = 60 cm^{-1}). According to the results of an X-ray diffraction study (space group $P2_1/c$, a 21.762(5); b 11.395(3); c 29.886(8) Å; β 93.07(2)°, $Z = 8$, V 7400.4 $Å^3$), the metal core of cation V represents an almost ideal triangle (Cr–Cr 2.920(6)–2.956(6) Å), whose edges are bridged by OR groups (Cr–O 1.99(2)–2.03(1) Å) located under the Cr_3 plane. On the other hand, μ_3-bridging oxygen atoms (Cr–O 1.89(2)–1.91(1) Å) and the centres of the C_5H_5 rings bonded to each Cr atom are located over the Cr_3 plane.

Introduction

Earlier we have seen [1] that the antiferromagnetic ($-2J$ 246 cm^{-1}) complex $Cp_2Cr_2(\mu\text{-}OCMe_3)_2$ (I) with a short Cr–Cr bond (2.635 Å) and a non-linear

* For part XXI see ref. 6.

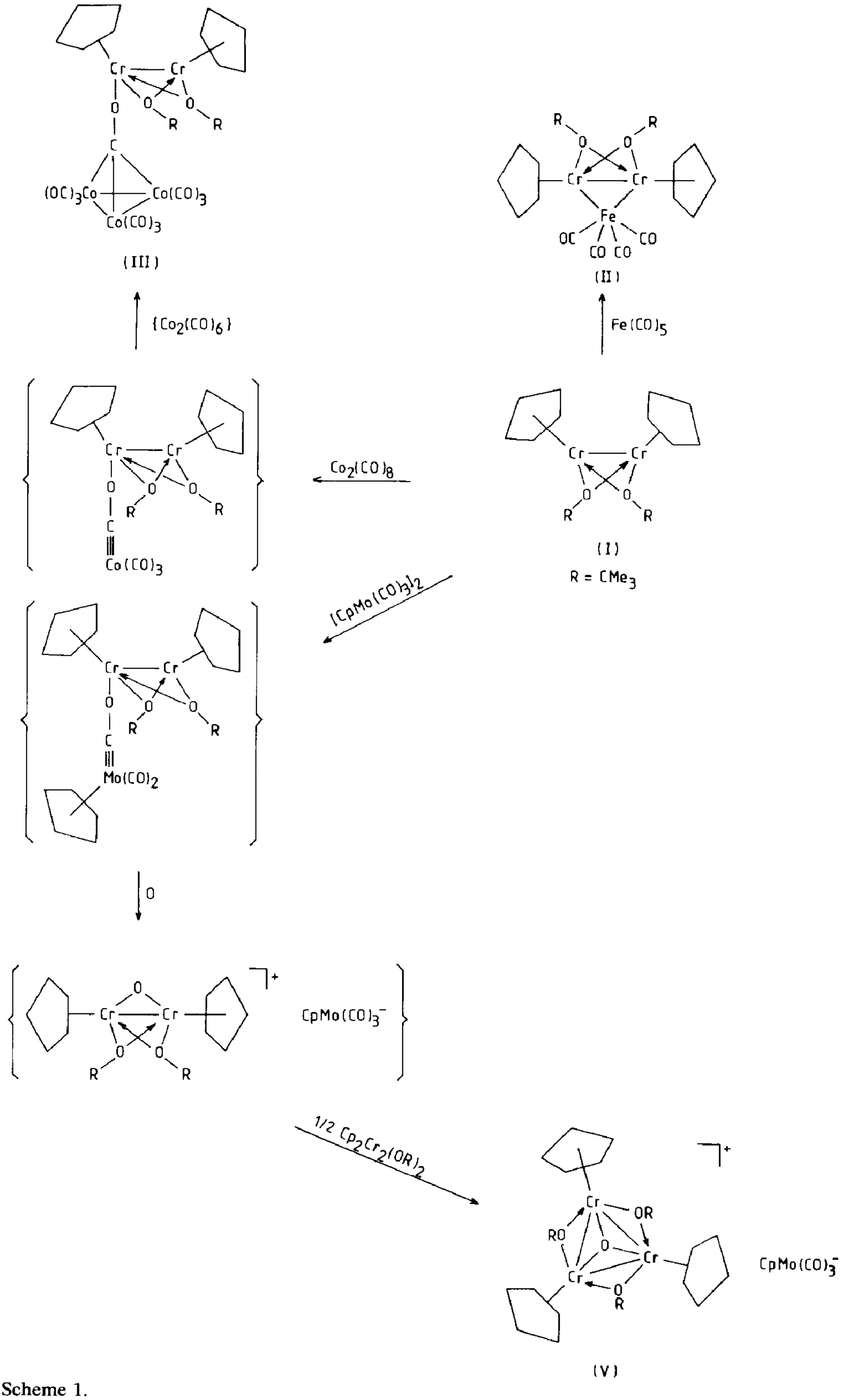
CpCrCrCp fragment (CpCrCr angle 143.9; 146.3°) [2] reacts in different ways with $Fe(CO)_5$ and $Co_2(CO)_8$. In the first case, a triangular $Cp_2Cr_2(\mu\text{-}OCMe_3)_2Fe(CO)_4$ cluster (II) is formed, in which a carbenoid fragment, $Fe(CO)_4$, is attached by two direct Fe–Cr bonds (2.7 Å); the CpCrCrCp fragment becomes linear and the interaction of the d_{z^2}-orbitals and consequently the exchange antiferromagnetic interactions in the dichromium system ($-2J$ 304 cm^{-1}) becomes stronger [1].

On the other hand, $Co_2(CO)_8$ is readily reduced to form $Co(CO)_4^-$ and in reaction with I gives a $Cp_2Cr_2(\mu\text{-}OCMe_3)_2\text{-}[OCCo_3(CO)_9]$ cluster (III) [3], in which a tricobaltdecacarbonyl fragment is attached to one of the chromium atoms via the oxygen atom of the tridentate CO group. III is probably formed by the addition of a $Co_2(CO)_6$ fragment to the carbyne intermediate $Cp_2Cr_2(\mu\text{-}OCMe_3)_2$ $OCCo(CO)_3$. The chromium atoms in III are in different oxidation states, while the cobalt-containing group behaves like a usual terminal alkoxide ligand. As a result, the non-linear distortion of the CpCrCrCp fragment is more pronounced (CpCrCr 117.2° and 155.7°); the overlap of the d_{z^2}-orbitals is hindered; and the Cr–Cr bond (2.766 Å) and consequently the antiferromagnetic interactions ($-2J$ 180 cm^{-1}) are weakened.

In this work the interaction of I with the dimer $[CpMo(CO)_3]_2$ was studied; the latter readily generates $CpMo(CO)_3^-$, which is capable of isocarbonyl-type coordination, e.g. in the complex $Cp_2Ti(thf)OCMo(CO)_2Cp$ [4].

Results and discussion

$Cp_2Cr_2(OR)_2$ (I, R = CMe_3) reacts readily with $[CpMo(CO)_3]_2$ under reflux in benzene. The interaction is accompanied by a change in colour of the reaction mixture from red to brown-yellow. Therewith, the CO bands characteristics of the initial $[CpMo(CO)_3]_2$ disappear in the IR spectrum, giving way to two bands of approximately equal intensity at 1845 and 1765 cm^{-1}, which are characteristic of the $MOCMo(CO)_2Cp$ fragment [5]. However, this intermediate is extremely sensitive to atmospheric oxygen and is immediately oxidized, giving the ionic cluster $[Cp_3Cr_3(\mu_3\text{-}O)(\mu\text{-}OCMe_3)_3]^+[CpMo(CO)_3]^-$ (V, 20% yield), which is probably formed following Scheme 1 via the $Cp_2Cr_2(\mu\text{-}OCMe_3)_2(\mu\text{-}O)$ intermediate. The possibility of the formation of this kind of complex has recently been proved by the synthesis of the $Cp_2Cr_2(\mu\text{-}OCMe_3)_2(\mu\text{-}Se)$ complex (Cr–Cr 2.61 Å) [6]. V was isolated as green-brown crystals. In the IR spectrum of V, there are the CO stretching vibration bands at 1770, 1910 and 1945 cm^{-1} typical of the $CpMo(CO)_3^-$ anion observed [7]. According to the results of an X-ray diffraction study, the metal core of cation V(Fig. 1) represents a triangle with almost equivalent Cr–Cr distances (2.920(6), 2.943(6) and 2.956(6) Å). All the edges of the triangle are bridged by $OCMe_3$ groups (Cr–O 1.99(2)–2.05(2)Å) which are located on the opposite side of the Cr_3 plane relative to a μ_3-bridging oxygen atom equivalently bonded to all the metal atoms (Cr–O 1.89(2)–1.91(1) Å); the μ_3-O atom is displaced from the Cr_3 plane by 0.85(1) Å. The centres of the C_5H_5 rings bonded to each chromium atom are displaced in the same direction, the CpCrCrCp fragment being non-linear (CpCrCr 135°). Thus, the geometry of each binuclear fragment $Cp_2Cr_2(\mu\text{-}OR)(\mu\text{-}O)(X)_2$ (X = μ-OR) in the triangular cation $[Cp_3Cr_3(\mu_3\text{-}O)(\mu\text{-}OR)_3]^+$ is very much the same as the geometry of the antiferromagnetic ($-2J$ 70 cm^{-1}) dimer $Cp_2Cr_2(\mu\text{-}OR)_2(OR)_2$ (Cr–Cr 3.005 Å) (VI, R = CMe_3) [3], which we



(III)
{Co2(CO)6}
(II)
Fe(CO)5
Co2(CO)8
(I)
R = CMe3
[CpMo(CO)3]2
O
CpMo(CO)3−
1/2 Cp2Cr2(OR)2
CpMo(CO)3−
(V)

Scheme 1.

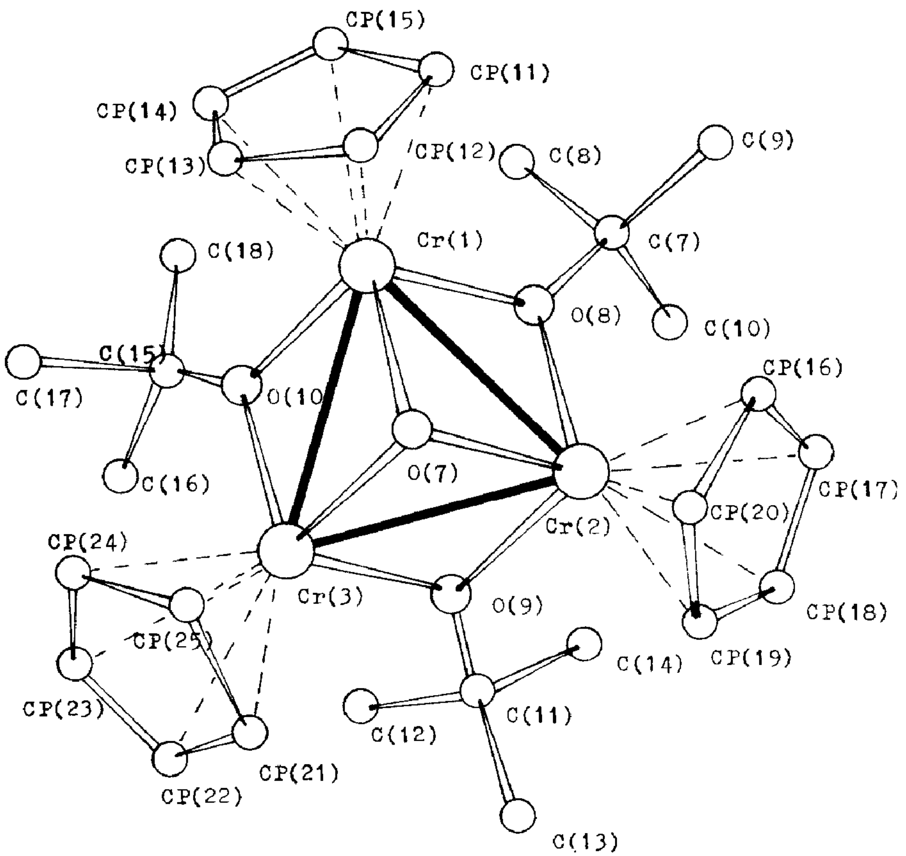


Fig. 1. Molecular structure of the cluster cation $Cp_3Cr_3(\mu_3\text{-}O)(\mu\text{-}OR)_3^+$.

have recently described. The only difference is the planarity of the Cp(centroid)CrCrCp(centroid) system in molecule V (the corresponding torsion angle in VI is equal to 27°). In such a situation, the Cr–Cr bonds in the cluster cation, as well as those in VI, are considerably weakened in comparison with I because of the increase in non-linearity of the CpCrCrCp fragment. Therefore, in the electron-deficient (45ē) complex V there is an additional π-interaction of the μ-OR and μ_3-O lone electron pairs with the half-filled Cr^{III} orbitals, which should strengthen the Cr–O bonding and indirect antiferromagnetic exchange. However, the significant decrease in the direct exchange due to the weakening of the Cr–Cr bonds seems more important. The effective magnetic moment of V decreases from 2.96 to 2.37 BM in the temperature range 77–296 K, which corresponds to the Heisenberg–Dirac–Van Vleck model [8] for an ideal triangular trimer with exchange parameter $-2J$(Cr–Cr) 60 cm^{-1} and spin values $S = 3/2$.

It is noteworthy that complex III is the first example of a trinuclear cluster of chromium(III) with alkoxide bridges. It is formally analogous to the well-known oxocarboxylate clusters of the $L_3Cr_3(\mu_3\text{-}O)(\mu\text{-}OOCR)_6^+$ type with monodentate ligands L = Py, THF, etc. [9]. The ligand L and two O atoms of the carboxylate bridges occupy three coordination positions at the Cr atom, as well as the C_5H_5 ligand. However, in the carboxylate clusters the Cr...Cr distances are elongated to 3.3 Å, and the exchange parameter consequently decreases to 10–20 cm^{-1} [9]. On the other hand, the geometry of V resembles that of the 42-electron diamagnetic "crown-like" sulphide cluster Cp_3Mo_3 (μ_3-S)(μ-S)$_3^+$ [10] and the nitrene cluster $Cp_3Cr_3(\mu_3\text{-NPh})(\mu\text{-NPh})_3^+OH^-$ [11] with short Mo–Mo (2.81 Å) and Cr–Cr (2.530

Table 1

Atomic coordinates of the cluster $Cp_3Cr_3(\mu_3\text{-O})(\mu\text{-OCMe}_3)_3{}^+$ $CpMo(CO)_3{}^-$ (V) (for Mo, Cr and O×10^4, for C×10^3).

Atom	x	y	z	Atom	x	y	z
Mo(1)	6330(1)	35(2)	1062(1)	C(25)	969(2)	669(3)	41(1)
Mo(2)	8598(1)	5360(3)	3943(1)	C(26)	1018(2)	480(3)	69(1)
Cr(1)	6439(2)	−1094(4)	3220(1)	C(27)	776(1)	555(3)	65(1)
Cr(2)	5349(2)	−275(4)	3676(1)	C(28)	771(3)	658(4)	40(1)
Cr(3)	6145(2)	1424(4)	3260(1)	C(29)	727(2)	471(5)	66(1)
Cr(4)	8133(2)	4763(4)	1657(1)	C(30)	820(2)	482(6)	37(1)
Cr(5)	9471(2)	4922(4)	1724(1)	Cp(1)	594(2)	−16(3)	29(1)
Cr(6)	8747(2)	6845(4)	1300(2)	Cp(2)	583(2)	98(3)	44(1)
O(1)	6454(14)	−2487(20)	1434(10)	Cp(3)	638(2)	160(3)	53(1)
O(2)	5280(10)	323(23)	1736(7)	Cp(4)	686(2)	83(3)	45(1)
O(3)	7281(10)	698(18)	1860(6)	Cp(5)	661(2)	−29(3)	31(1)
O(4)	8994(12)	3461(20)	3249(7)	Cp(6)	892(3)	460(4)	467(1)
O(5)	7289(10)	4774(20)	3585(8)	Cp(7)	835(2)	538(5)	474(1)
O(6)	8675(13)	7131(24)	3134(9)	Cp(8)	857(2)	646(4)	462(1)
O(7)	5768(7)	−45(15)	3137(5)	Cp(9)	919(2)	645(4)	451(1)
O(8)	6051(7)	−1381(14)	3821(5)	Cp(10)	937(2)	539(4)	455(1)
O(9)	5763(8)	1205(14)	3859(5)	Cp(11)	673(2)	−294(2)	302(1)
O(10)	6887(8)	388(15)	3377(5)	Cp(12)	714(2)	−214(3)	284(1)
O(11)	8768(8)	5850(14)	1820(5)	Cp(13)	679(2)	−144(3)	254(1)
O(12)	8842(8)	3741(14)	1519(6)	Cp(14)	615(2)	−180(3)	252(1)
O(13)	9478(8)	5889(14)	1156(5)	Cp(15)	613(2)	−272(3)	283(1)
O(14)	8105(8)	5704(14)	1079(6)	Cp(16)	438(1)	28(3)	384(1)
C(1)	642(1)	−157(3)	128(1)	Cp(17)	441(1)	27(3)	340(1)
C(2)	565(1)	23(2)	151(1)	Cp(18)	450(1)	−78(3)	323(1)
C(3)	695(1)	48(2)	158(1)	Cp(19)	458(1)	−157(3)	360(1)
C(4)	888(2)	413(3)	349(1)	Cp(20)	449(1)	−83(4)	399(1)
C(5)	780(1)	500(3)	372(1)	Cp(21)	651(1)	298(3)	287(1)
C(6)	866(2)	653(3)	345(1)	Cp(22)	606(1)	340(3)	311(1)
C(7)	616(1)	−231(2)	416(1)	Cp(23)	549(1)	284(2)	298(1)
C(8)	586(2)	−344(2)	402(1)	Cp(24)	561(2)	204(2)	263(1)
C(9)	688(1)	−242(3)	424(1)	Cp(25)	626(2)	212(2)	257(1)
C(10)	591(2)	−181(3)	460(1)	Cp(26)	789(1)	418(3)	235(1)
C(11)	580(1)	192(2)	426(1)	Cp(27)	768(1)	534(3)	228(1)
C(12)	554(2)	138(3)	464(1)	Cp(28)	723(1)	535(3)	193(1)
C(13)	630(2)	270(4)	431(1)	Cp(29)	714(1)	423(3)	177(1)
C(14)	533(3)	298(4)	416(1)	Cp(30)	753(1)	347(3)	203(1)
C(15)	756(1)	67(3)	344(1)	Cp(31)	1016(2)	375(3)	213(1)
C(16)	782(1)	79(3)	297(1)	Cp(32)	1047(1)	469(3)	196(1)
C(17)	762(1)	183(3)	373(1)	Cp(33)	1025(1)	581(3)	211(1)
C(18)	785(1)	−35(3)	369(1)	Cp(34)	980(1)	545(4)	242(1)
C(19)	888(1)	243(2)	143(1)	Cp(35)	976(1)	421(3)	241(1)
C(20)	890(2)	181(3)	190(1)	Cp(36)	817(1)	846(3)	127(1)
C(21)	829(2)	209(3)	113(1)	Cp(37)	868(2)	863(2)	101(1)
C(22)	947(2)	222(3)	115(1)	Cp(38)	922(2)	863(2)	132(1)
C(23)	993(2)	597(3)	79(1)	Cp(39)	900(2)	843(3)	171(1)
C(24)	1051(2)	674(4)	102(1)	Cp(40)	839(2)	833(3)	174(2)

Å) bonds, respectively. The character of the bridging ligands is obviously the main factor determining the geometrical and magnetic properties of triangular clusters of the $Cp_3M_3X_4$ type.

Experimental

All operations connected with the synthesis of the initial compounds or new complexes were carried out under pure argon in absolute solvents. The initial compounds Cp_2Cr and I were prepared by techniques described previously [2,12]. IR spectra were recorded with Specord IR-75 spectrometer; spectra of the benzene solutions of IV were recorded in KBr cells, while spectra of V were recorded in KBr pellets. Magnetic susceptibility was measured according to the Faraday method with an instrument designed in the Institute of General and Inorganic Chemistry [13]. X-ray diffraction data were obtained with an automatic Hilger & Watts diffractometer (λ(Mo-K_α), θ–2θ scan, $2\theta \leqslant 60°$, T 20 °C, 4674 reflections). Crystals of V are monoclinic, space group $P2_1/c$, a 21.762(6), b 11.395(3), c 29.866 (8) Å, β 93.07(2)°, $Z = 8$ *, V 7400.4 Å^3. The structure of the ionic complex was solved by the direct method using a modified MULTAN program of the INEXTL program package [14]. All non-hydrogen atoms were refined by means of the least-squares technique in anisotropic block-diagonal approximation to R_1 7.70%, R_w 10.20% (Table 1). Relevant bond lengths and bond angles are listed in Tables 2 and 3.

$[Cp_2Cr_2(\mu_3\text{-}O)(\mu\text{-}OCMe_3)_3]^+[CpMo(CO)_3]^-$ (V)

A solution of 0.4 g (0.8 mmol) of $[(C_5H_5)Mo(CO)_3]_2$ in 10 ml of benzene was added to 15 ml of a red-brown solution of $Cp_2Cr_2(\mu\text{-}OCMe_3)_2$ (obtained from 0.6 g (3.8 mmol) of chromocene and $HOCMe_3$) in benzene. The reaction mixture was refluxed for 0.5 h, and then air was bubbled through the resulting brown solution

Table 2

Bond lengths d(Å) of the cluster $Cp_3Cr_3(\mu_3\text{-}O)(\mu\text{-}OCMe_3)_3{}^+ CpMo(CO)_3{}^-$ (V)

Bond	d (Å)	Bond	d (Å)
Cr(1)–Cr(2)	2.950(6)	Cr(5)–Cr(6)	2.946(7)
Cr(1)–Cr(3)	2.943(6)	Cr(4)–O(11)	1.90(2)
Cr(2)–Cr(3)	2.920(6)	Cr(4)–O(12)	1.99(2)
Cr(1)–O(7)	1.91(1)	Cr(4)–O(14)	2.03(2)
Cr(1)–O(8)	2.05(2)	Cr(5)–O(11)	1.89(2)
Cr(1)–O(10)	1.99(2)	Cr(5)–O(12)	1.99(2)
Cr(2)–O(7)	1.91(1)	Cr(5)–O(13)	2.03(2)
Cr(2)–O(8)	2.01(2)	Cr(6)–O(11)	1.92(2)
Cr(2)–O(9)	1.98(2)	Cr(6)–O(13)	1.99(2)
Cr(3)–O(7)	1.89(2)	Cr(6)–O(14)	2.00(2)
Cr(3)–O(9)	2.03(2)		
Cr(3)–O(10)	2.01(2)		
Cr(4)–Cr(5)	2.913(6)		
Cr(4)–Cr(6)	2.951(6)		

* Two independent molecules in the cell.

Table 3

Bond angles of the cluster $Cp_3Cr_3(\mu_3\text{-}O)(\mu\text{-}OCMe_3)_3^+$ $CpMo(CO)_3^-$ (V)

Angle	ω (°)	Angle	ω (°)
Cr(2)Cr(1)Cr(3)	59.4(1)	Cr(2)Cr(3)O(10)	91.5(5)
Cr(2)Cr(1)O(7)	39.3(5)	O(7)Cr(3)O(9)	82.4(7)
Cr(2)Cr(1)O(8)	42.9(5)	O(7)Cr(3)O(10)	81.4(7)
Cr(2)Cr(1)O(10)	91.0(5)	O(9)Cr(3)O(10)	97.8(7)
O(7)Cr(1)O(8)	82.2(7)	Cr(5)Cr(4)Cr(6)	60.3(2)
O(7)Cr(1)O(10)	81.9(7)	Cr(5)Cr(4)O(11)	39.7(5)
Cr(3)Cr(1)O(7)	39.0(5)	Cr(5)Cr(4)O(12)	43.0(5)
Cr(3)Cr(1)O(8)	91.1(5)	Cr(5)Cr(4)O(14)	90.6(5)
Cr(3)Cr(1)O(10)	43.0(5)	Cr(6)Cr(4)O(11)	39.8(5)
O(8)Cr(1)O(10)	98.5(7)	Cr(6)Cr(4)O(12)	91.4(5)
Cr(1)Cr(2)Cr(3)	60.2(1)	Cr(6)Cr(4)O(14)	42.4(5)
Cr(1)Cr(2)O(7)	39.0(5)	O(11)Cr(4)O(12)	82.7(7)
Cr(1)Cr(2)O(8)	43.9(5)	O(11)Cr(4)O(14)	82.1(7)
Cr(1)Cr(2)O(9)	91.8(5)	O(12)Cr(4)O(14)	96.9(7)
Cr(3)Cr(2)O(7)	39.6(5)	Cr(4)Cr(5)Cr(6)	60.5(2)
Cr(3)Cr(2)O(8)	92.6(5)	Cr(4)Cr(5)O(11)	39.9(5)
Cr(3)Cr(2)O(9)	43.9(5)	Cr(4)Cr(5)O(12)	43.1(5)
O(7)Cr(2)O(8)	82.8(7)	Cr(4)Cr(5)O(13)	91.6(5)
O(7)Cr(2)O(9)	83.4(7)	Cr(6)Cr(5)O(11)	39.9(5)
O(8)Cr(2)O(9)	98.3(7)	Cr(6)Cr(5)O(12)	91.6(5)
Cr(1)Cr(3)Cr(2)	60.4(1)	Cr(6)Cr(5)O(13)	42.4(5)
Cr(1)Cr(3)O(7)	39.0(5)	O(11)Cr(5)O(12)	82.9(7)
Cr(1)Cr(3)O(9)	90.9(5)	O(11)Cr(5)O(13)	82.3(7)
Cr(1)Cr(3)O(10)	42.4(5)	O(12)Cr(5)O(13)	98.3(7)
Cr(2)Cr(3)O(7)	40.0(5)	Cr(4)Cr(6)Cr(5)	59.2(2)
Cr(2)Cr(3)O(9)	42.5(5)	Cr(4)Cr(6)O(11)	39.2(5)
Cr(4)Cr(6)O(13)	91.2(5)	O(11)Cr(6)O(13)	82.4(7)
Cr(4)Cr(6)O(14)	43.4(5)	O(11)Cr(6)O(14)	82.5(7)
Cr(5)Cr(6)O(11)	39.1(5)	O(13)Cr(6)O(14)	97.2(7)
Cr(5)Cr(6)O(13)	43.3(5)	Cr(1)O(7)Cr(2)	101.7(7)
Cr(5)Cr(6)O(14)	90.4(5)	Cr(1)O(7)Cr(3)	102.0(7)
Cr(2)O(7)Cr(3)	100.4(7)		
Cr(1)O(8)Cr(2)	93.2(7)		
Cr(2)O(9)Cr(3)	93.6(7)		
Cr(1)O(10)Cr(3)	94.5(7)		
Cr(4)O(11)Cr(5)	100.4(8)		
Cr(4)O(11)Cr(6)	101.0(8)		
Cr(5)O(11)Cr(6)	101.0(8)		
Cr(4)O(12)Cr(5)	93.9(7)		
Cr(5)O(13)Cr(6)	94.3(7)		
Cr(4)O(14)Cr(6)	94.2(7)		

(ν(CO), 1845 and 1765 cm^{-1}) for 1–2 min. A green-brown precipitate was immediately formed. It was isolated by decantation and then extracted by 10 ml of THF. The brown-green extract thus obtained was slowly concentrated in an argon flow at room temperature for 20 h up to half the initial volume and cooled to −18°C. In 1 day green-brown prisms precipitated, which were isolated by decantation, washed with cold (−70°C) THF and dried in an argon flow at 22°C.

Yield 0.13 g (20.2%). IR spectrum (ν, cm^{-1}): 820s, 1030m, 1175m, 1405m, 1770vs br, 1910s, 1945m, 2950m br.

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