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Crystal structures of $(\eta^5\text{-}C_5Me_5)(\eta^5\text{-}2,4\text{-}C_7H_{11})Cr$ and $(\eta^5\text{-}C_5Me_5)(\eta^5\text{-}2,4\text{-}C_7H_{11})CrCO$. An example of 'S' 2,4-dimethylpentadienyl ligand geometry

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Abstract

The half-open chromocene complex $(\eta^5\text{-}C_5Me_5)(\eta^5\text{-}2,4\text{-}C_7H_{11})Cr$ (**1**) has been characterized by X-ray diffraction. It crystallizes in space group $P\bar{1}$ with $a=7.592(2)$, $b=8.665(6)$, $c=12.452(14)$ Å, $\alpha=78.50(8)$, $\beta=79.41(6)$ and $\gamma=86.72(4)$°, $Z=2$, $V=787(15)$ Å^3 and $D_{calc}=1.19$ g/cm^3. The structure of this paramagnetic complex reveals a normal U-shaped pentadienyl moiety. Complex **1** is carbonylated in hexane solution under 40 psi CO at room temperature to yield the diamagnetic monocarbonyl adduct $(\eta^5\text{-}C_5Me_5)(\eta^5\text{-}2,4\text{-}C_7H_{11})CrCO$ (**2**). The X-ray structure of **2** has also been determined; the complex crystallizes in space group $P\bar{1}$ with $a=8.2810(14)$, $b=8.8096(17)$, $c=12.0825(24)$ Å, $\alpha=97.147(15)$, $\beta=91.712(15)$ and $\gamma=116.128(14)$°, $Z=2$, $V=781.7(3)$ Å^3 and $D_{calc}=1.319$ g/cm^3. The structure of **2** reveals an unusual S-pentadienyl ligand geometry.

Introduction

Chromocene serves as a precursor to heterogeneous olefin polymerization catalysts (Union Carbide-type catalysts). The active catalyst species is believed to result from protolytic cleavage of one cyclopentadienyl ring from chromium by a surface hydroxyl group of silica to afford a surface-bound chromium monocyclopentadienyl moiety [1]. Much effort has centered on preparation of homogeneous analogs, however complexes of the type $CpCrOSiR_3$ ($Cp=\eta^5\text{-}C_5H_5$; R = Me, Ph) were found to exist as unreactive dimers [2]. We embarked on a synthetic pathway aimed at preparation of more sterically encumbered congeners of this type with the goal of isolating presumably reactive monomers $Cp^*CrL_2(OSiR_3)$ ($Cp^*=\eta^5\text{-}C_5Me_5$; L = CO, PMe_3, C_5H_5N; R = CMe_3, Ph) [3]. We found Cp^*_2Cr to be unreactive toward the silanols of interest and turned our attention to preparation of the half-open chromocene Cp*(DMPD)Cr (**1**) ($DMPD=\eta^5\text{-}2,4\text{-}C_7H_{11}$). This choice was based on the observation that an analog of **1**, Cp(PD)Cr ($PD=\eta^5\text{-}C_5H_7$), affords a Union Carbide-type catalyst by presumed loss of a more labile pentadienyl ligand [4]. Although we have yet to obtain a siloxide complex of the structure desired, we have found that **1** reacts with CO under mild conditions to afford Cp*(DMPD)CrCO (**2**) which possesses a 2,4-dimethylpentadienyl ligand in the unusual S (sickle-shaped) geometry. During the course of our work Ernst and co-workers reported the preparation of **1** and **2** including X-ray structure determinations of the related half-open chromocene species Cp*(PD)Cr and an isonitrile complex $Cp(PD)CrCN[2,6\text{-}(CH_3)_2C_6H_3]$, the latter of which exhibits an S-pentadienyl arrangement [5]. Herein we report the X-ray crystal structures of **1** and **2**.

Experimental

Reactions were carried out under nitrogen on a Schlenk line. Solids were handled in a Vacuum Atmospheres glovebox under nitrogen atmosphere. Dry and oxygen-free solvents were distilled from sodium/benzophenone. Anhydrous $CrCl_2$ was purchased from Strem. Potassium 2,4-dimethylpentadienide [6] and so-

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dium pentamethylcyclopentadienide [7] were prepared by literature methods. NMR spectra were recorded on a Varian XL-400 instrument and FT-IR spectra on a Perkin-Elmer model 1620 spectrometer. X-ray data for **1** were collected on an Enraf-Nonius CAD4 diffractometer and for **2** on a Siemens P3 instrument.

Preparation of the complexes

(η^5-C_5Me_5)(η^5-2,4-C_7H_{11})Cr (1)

Anhydrous $CrCl_2$ (2.65 g, 21.6 mmol) was suspended in 120 ml tetrahydrofuran in a Schlenk flask. The flask was fitted with an addition funnel charged with a 120 ml solution of potassium 2,4-dimethylpcntadienide (2.9 g, 22 mmol) and sodium pentamethylcyclopentadienide (3.4 g, 22 mmol) in tetrahydrofuran. The stirred suspension was chilled in an ice bath and the reagent mixture was added dropwise. Immediately after addition the cooling bath was removed and the solvent evaporated *in vacuo* to afford a dark brown solid. The solid was extracted with three 20 ml portions of hexane and filtered. Removal of solvent gave a dark brown solid which was recrystallized twice from hexane and sublimed to afford a deep red product. Yield 4.0 g (65%). *Anal.* Calc. for $C_{17}H_{26}Cr$: C, 72.31; H, 9.28. Found: C, 72.72; H, 9.13%. ^1H NMR (C_6D_6): δ 10.15 (s,br).

(η^5-C_5Me_5)(η^5-2,4-C_7H_{11})CrCO (2)

A 750 mg (2.66 mmol) sample of **1** was dissolved in 15 ml hexane in a glass pressure reactor. The solution was allowed to stand under 40 psi of CO in the dark for 4 h after which the orange solution was transferred to a Schlenk tube and cooled slowly from room temperature to -25 °C overnight. Large orange crystals were collected and dried *in vacuo*. Yield 560 mg (68%). ^1H NMR (C_7D_8): δ 1.01 (s, 1H); 1.26 (s, 3H); 1.36 (d, 1H); 1.48 (s, 15H); 1.71 (s, 3H); 1.72 (d, 1H); 1.87 (s, 1H); 2.23 (s, 1H). ^{13}C NMR (C_7D_8): δ 10.7 (q); 18.2 (q); 21.6 (q); 45.0 (t); 52.5 (t); 82.4 (d); 93.4 (s); 97.2 (s); 109.9 (s); 257 (s). IR (CCl_4): 1888 cm^{-1} (CO).

X-ray methods and structure determination

(η^5-C_5Me_5)(η^5-2,4-C_7H_{11})Cr (1)

Collection of X-ray diffraction data for 1. A red crystal of approximate size 0.3×0.3×0.3 mm was placed in a glass capillary inside a dry box and sealed under nitrogen. The capillary was attached to a goniometer head and mounted on a Syntex $P2_1$ diffractometer for preliminary examination. Polaroid rotation photographs in combination with the Syntex autoindexing and cell refinement procedures were used to identify the crystal system and possible unit cells. The cell parameters were determined and refined from 25 reflections well dispersed in reciprocal space, using Syntex autoindexing and cell refinement procedures. The crystal system was triclinic. The space group $P\bar{1}$ was initially chosen based on intensity statistics and was confirmed as correct in subsequent structure refinement. Intensity data (2946 reflections) were collected at 295 K on an Enraf-Nonius CAD4 diffractometer using a θ–2θ scan technique with Mo Kα radiation. For the crystal, instrumental and compound stability were monitored throughout the data collection period. The crystal exhibited no significant decay during the data collection period. Measured reflections were flagged as unobserved if they were less than three standard deviations from the measured intensity. Relevant crystal parameters are $a = 7.592(2)$, $b = 8.665(6)$, $c = 12.452(14)$ Å, $\alpha = 78.50(8)$, $\beta = 79.41(6)$ and $\gamma = 86.72(4)°$, $Z = 2$, $V = 787(15)$ Å^3 and $D_{calc} =$ 1.19 g/cm^3.

Solution and refinement of the crystal structure for 1. All crystallographic calculations were carried out using Enraf-Nonius SDP software. The analytical scattering factors for the neutral atoms were used throughout the analysis [8a]; both the real ($\Delta f'$) and imaginary ($i\Delta f''$) components of anomalous dispersion were included [8b]. The quantity minimized during least-squares analysis was $\Sigma w(|F_o| - |F_c|)^2$ where $w^{-1} = (\sigma_I^2 + 0.0004F^4)/4F^2$.

The structure was solved by heavy-atom methods, with the chromium atom being identified in Patterson maps and the non-H atoms by Fourier syntheses. Hydrogen atoms were not located. Refinement of positional and thermal parameters led to convergence with $R_F = 5.6\%$; $R_{wF} = 7.8\%$ and $GOF = 4.19$ for 163 variables refined against those 2192 data with $|F_o| > 3.0\sigma(|F_o|)$. A final difference-Fourier synthesis showed no significant features, $\rho_{max} = 0.44$ e Å^{-3}.

(η^5-C_5Me_5)(η^5-2,4-C_7H_{11})CrCO (2)

Collection of X-ray diffraction data for 2. An orange crystal was oil-mounted on a glass fiber and transferred to the Siemens P3 diffractometer equipped with a locally (UCI) modified LT-2 low temperature system. The determination of Laue symmetry, crystal class, unit cell parameters and the crystal's orientation matrix were carried out by methods similar to those of Churchill *et al.* [9]. Intensity data were collected at 173 K using a θ-2θ scan technique with Mo Kα radiation. All 3874 data were corrected for absorption and for Lorentz polarization effects and were placed on an approximately absolute scale. There were no systematic absences nor any diffraction symmetry other than the Friedel condition. The triclinic centrosymmetric space group $P\bar{1}$ was chosen based on intensity statistics and confirmed by successful refinement of the model. Relevant crystal parameters are $a = 8.2810(14)$, $b = 8.8096(17)$, $c = 12.0825(24)$ Å, $\alpha = 97.147(15)$, $\beta = 91.712(15)$ and $\gamma = 116.128(14)°$, $Z = 2$, $V = 781.7(3)$ Å^3 and $D_{calc} = 1.319$ g/cm^3.

Solution and refinement of the crystal structure for **2**. All crystallographic calculations were carried out using the UCI modified version of the UCLA Crystallographic computing package or the SHELXTL PLUS program set [10]. The analytical scattering factors for neutral atoms were used throughout the analysis [8a]. Both the real ($\Delta f'$) and imaginary ($i\Delta f''$) components of anomalous dispersion were included [8b]. The quantity minimized during least-squares analysis was $\Sigma w(|F_o| - |F_c|)^2$ where $w^{-1} = \sigma^2(|F_o|) + 0.0003(|F_o|)^2$. The structure was solved by an automatic Patterson routine (SHELXTL PLUS) and refined by full-matrix least-squares techniques. Hydrogen atoms were located from subsequent difference-Fourier syntheses and included with isotropic temperatures factors. Refinement of positional and thermal parameters led to convergence with $R_F = 4.2\%$; $R_{wF} = 5.6\%$ and $GOF = 2.22$ for 286 variables refined against those 3438 data with $|F_o| > 1.0\sigma(|F_o|)$; ($R_F = 3.6\%$ and $R_{wF} = 5.4\%$ for those data with $|F_o| > 6.0\sigma(|F_o|)$). A final difference-Fourier synthesis showed no significant features, $\rho_{max} = 0.42$ e Å^{-3}.

Results and discussion

An ORTEP diagram which illustrates the sandwich structure of the half-open paramagnetic chromocene **1** is shown in Fig. 1 and fractional coordinates, bond distances and angles are given in Tables 1 and 2. The Cp* ligand is flat, with C(1)–C(5) lying in a least-squares plane within experimental error. The mean Cr–C distance about the Cp* ring is 2.195(3) Å compared with 2.194(3) Å in Cp*(PD)Cr [5]. The DMPD ligand of **1** is U-shaped and nearly flat with all delocalized (C(11)–C(15)) carbons lying within 0.04 Å of a least-squares plane; deviations from this plane are as follows: C(11), −0.010(5); C(12), 0.029(5); C(13), −0.037(5); C(14), 0.025(5); C(15), −0.006(5) Å with negative values indicating a distortion toward the metal. The angle between the Cp*(C(1)–C(5)) plane and the DMPD

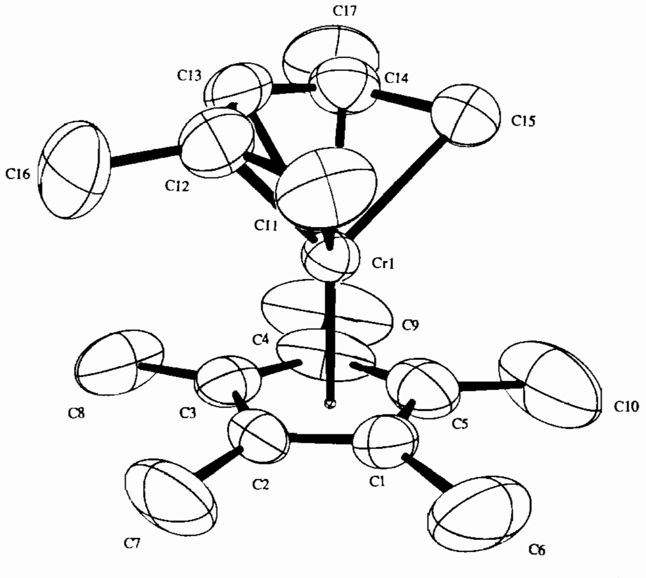

Fig. 1. ORTEP drawing and atomic labelling scheme for (η^5-C_5Me_5)(η^5-2,4-C_7H_{11})Cr (**1**).

TABLE 1. Fractional coordinates ($\times 10^4$) and isotropic temperature factors for the non-hydrogen atoms in (η^5-C_5Me_5)(η^5-2,4-C_7H_{11})Cr (**1**)

	x	y	z	B (Å^2)
Cr(1)	2445.4(8)	2671.3(6)	3057.8(9)	3.18(1)
C(1)	764(6)	3756(4)	1589(6)	4.3(1)
C(2)	921(5)	2684(4)	615(6)	3.8(1)
C(3)	2508(6)	2312(5)	81(6)	5.0(1)
C(4)	3309(6)	3205(5)	764(7)	6.4(1)
C(5)	2212(7)	4095(4)	1699(7)	5.5(1)
C(6)	−795(8)	4474(6)	2329(9)	8.2(2)
C(7)	−429(7)	2057(6)	110(10)	8.0(2)
C(8)	3190(10)	1190(6)	−999(9)	10.1(2)
C(9)	5033(8)	3214(8)	380(10)	12.4(2)
C(10)	2510(10)	5246(5)	2520(10)	11.5(2)
C(11)	964(6)	2222(5)	4805(7)	5.3(1)
C(12)	2139(6)	1272(4)	4143(7)	4.8(1)
C(13)	3784(6)	1297(4)	4202(6)	4.6(1)
C(14)	4492(6)	2241(4)	4670(6)	4.5(1)
C(15)	3644(6)	3324(4)	5392(7)	5.4(1)
C(16)	1661(9)	203(5)	313(10)	8.1(2)
C(17)	6237(6)	2111(7)	442(10)	7.7(2)

TABLE 2. Selected bond distances (Å) and angles (°) in (η^5-C_5Me_5)(η^5-2,4-C_7H_{11})Cr (**1**)

Cr(1)–C(1)	2.185(6)	C(2)–C(1)–C(5)	109.3(6)
Cr(1)–C(2)	2.209(5)	C(2)–C(1)–C(6)	124.8(6)
Cr(1)–C(3)	2.216(5)	C(5)–C(1)–C(6)	125.8(5)
Cr(1)–C(4)	2.188(7)	C(1)–C(2)–C(3)	108.7(5)
Cr(1)–C(5)	2.179(6)	C(1)–C(2)–C(7)	124.8(6)
Cr(1)–C(11)	2.112(6)	C(3)–C(2)–C(7)	126.3(6)
Cr(1)–C(12)	2.125(6)	C(2)–C(3)–C(4)	106.3(6)
Cr(1)–C(13)	2.161(5)	C(2)–C(3)–C(8)	126.1(7)
Cr(1)–C(14)	2.128(6)	C(4)–C(3)–C(8)	127.6(7)
Cr(1)–C(15)	2.108(6)	C(3)–C(4)–C(5)	108.2(5)
C(1)–C(2)	1.386(8)	C(3)–C(4)–C(9)	125(1)
C(1)–C(5)	1.392(9)	C(5)–C(4)–C(9)	127(1)
C(1)–C(6)	1.537(8)	C(1)–C(5)–C(4)	107.4(5)
C(2)–C(3)	1.418(8)	C(1)–C(5)–C(10)	125.8(8)
C(2)–C(7)	1.523(7)	C(4)–C(5)–C(10)	126.5(8)
C(3)–C(4)	1.43(1)	C(11)–C(12)–C(13)	123.0(6)
C(3)–C(8)	1.509(8)	C(11)–C(12)–C(16)	120.5(6)
C(4)–C(5)	1.42(1)	C(13)–C(12)–C(16)	116.1(7)
C(4)–C(9)	1.539(8)	C(12)–C(13)–C(14)	127.1(5)
C(5)–C(10)	1.518(8)	C(13)–C(14)–C(15)	123.4(5)
C(11)–C(12)	1.43(1)	C(13)–C(14)–C(17)	117.8(6)
C(12)–C(13)	1.428(9)	C(15)–C(14)–C(17)	118.5(6)
C(12)–C(16)	1.526(9)		
C(13)–C(14)	1.410(9)		
C(14)–C(15)	1.429(9)		
C(14)–C(17)	1.538(8)		

(C(11)–C(15)) plane is 11.1(6)°, less than the 19.4° reported for $(DMPD)_2Cr$ [11]. This distortion places the terminal DMPD carbons (C(11) and C(15)) slightly closer to the metal than the internal carbon atoms (C(12)–C(14)). The mean Cr–C distance for the DMPD ligand of **1** is 2.127(3) Å, shorter than the 2.163(3) Å value for $(DMPD)_2Cr$; correspondingly the mean C–C distance for the delocalized carbons of the DMPD ligand of **1** is longer (1.424(5) Å) than that found in $(DMPD)_2Cr$ (1.386(5) Å). These data may indicate stronger bonding of the dimethylpentadienyl ligand in **1** than in $(DMPD)_2Cr$. Note that in **1** the mean metal–carbon distance for the DMPD ligand is less than that for Cp* as is generally observed for such half-open complexes [5, 12]. As is typical [13], the bond angles at the methylated carbons of the pentadienyl fragment are less than at non-methylated positions; thus the average C(11)–C(12)–C(13) and C(13)–C(14)–C(15) angle is 123.2(5)° compared with a C(12)–C(13)–C(14) angle of 127.1(5)°.

TABLE 3. Fractional coordinates ($\times 10^4$) and isotropic temperature factors ($Å^2 \times 10^3$) for the non-hydrogen atoms in $(\eta^5\text{-}C_5Me_5)(\eta^5\text{-}2,4\text{-}C_7H_{11})CrCO$ (**2**)

	x	y	z	U_{eq}[a]
Cr(1)	10837.2(5)	3090.8(4)	2608.8(3)	145(1)
O(1)	13083(3)	1378(3)	1816(2)	333(8)
C(1)	9001(3)	3141(3)	1219(2)	207(9)
C(2)	9278(3)	4516(3)	2082(2)	201(9)
C(3)	11152(3)	5642(3)	2246(2)	207(9)
C(4)	12044(3)	4963(3)	1492(2)	213(9)
C(5)	10696(3)	3424(3)	841(2)	211(9)
C(6)	7234(4)	1734(4)	668(2)	309(11)
C(7)	7847(4)	4858(4)	2642(2)	278(11)
C(8)	11985(4)	7370(3)	2950(2)	287(10)
C(9)	14023(4)	5812(4)	1328(3)	317(11)
C(10)	10991(5)	2441(4)	−170(2)	307(12)
C(11)	9134(3)	334(3)	2705(2)	231(9)
C(12)	8619(3)	1350(3)	3481(2)	206(9)
C(13)	9944(3)	2709(3)	4217(2)	215(9)
C(14)	11811(3)	3009(3)	4222(2)	230(9)
C(15)	12948(4)	4648(3)	3983(2)	263(10)
C(16)	12491(4)	1791(4)	4567(2)	283(11)
C(17)	6687(4)	1049(4)	3475(3)	311(11)
C(18)	12225(3)	2044(3)	2144(2)	216(9)

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

The half-open chromocenes are more reactive toward coordination of donor ligands than are either the open or closed chromocenes, a fact which may correspond with the ability of the half-open species to adopt the unusual S-pentadienyl geometry, affording better access to the incoming ligand [5]. Only a few other cases of such a coordination mode for pentadienyl ligands have been reported [5, 14]. Complex **2** is a diamagnetic chromium(II) carbonyl, an ORTEP plot of which is shown in Fig. 2 with fractional coordinates, bond distances and angles reported in Tables 3 and 4. Most striking is the transformation of the DMPD ligand from a U-shaped geometry in **1** to an S-type geometry in CO adduct **2**. The torsion angle of the C(12)–C(13)–C(14)–C(15) unit is 120.9°, somewhat smaller than the 123.4° observed in the analogous isonitrile complex $Cp(PD)CrCN[2,6\text{-}(CH_3)_2C_6H_3]$ [5]. This out-of-plane distortion is also illustrated by the displacements from the C(11)–C(14) plane as follows: C(11), −0.007; C(12), 0.014; C(13), −0.014; C(14), 0.007; C(15), −1.124; C(17), 1.170 Å with negative values again indicating a distortion toward the metal. The C(11)–C(14) plane is tilted by 14.6° from the plane of the pentamethylcyclopentadienyl ligand (C(1)–C(5)). The mean Cr–C distance for the DMPD ligand of **2** is 2.172(1) Å and for the Cp* ligand this value is 2.231(1) Å, both somewhat longer than in complex **1** (2.127(3) and 2.195(3) Å, respectively). The longest C–C bond of the DMPD ligand backbone is the C(13)–C(14) bond, and the two shortest Cr–C distances to this ligand are the Cr(1)–C(13) and Cr(1)–C(14) bonds, as is true for the PD ligand of the isonitrile analog. The relatively short C(12)–C(13) bond of **2** is not reflected in the isonitrile complex. The mean of the three internal angles for the DMPD ligand of **2** (117.0(2)°) is less than 120° as is the case for the PD ligand in the isonitrile analog (116.6(3)°). The Cr(1)–C(18) bond length of 1.825(3) Å is slightly shorter than in the tetramethylethylene-bridged complex [15] $(CH_3)_4C_2(C_5H_4)_2CrCO$ (1.85(1) Å), reflecting a greater degree of backbonding in complex **1**. The strong donor effect of the combined Cp* and DMPD ligand in **1** is best illustrated by the CO stretching frequency at 1888 cm^{-1}, lower than that of $(CH_3)_4C_2(C_5H_4)_2CrCO$ (1905

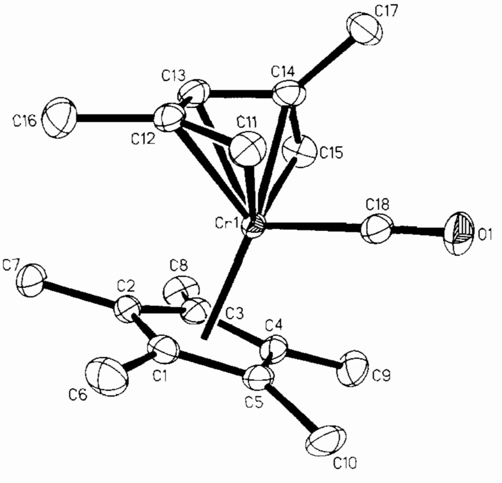

Fig. 2. ORTEP drawing and atomic labelling scheme for $(\eta^5\text{-}C_5Me_5)(\eta^5\text{-}2,4\text{-}C_7H_{11})CrCO$ (**2**).

TABLE 4. Selected bond distances (Å) and angles (°) in $(\eta^5\text{-}C_5Me_5)(\eta^5\text{-}2,4\text{-}C_7H_{11})CrCO$ (**2**)

Cr(1)–C(1)	2.250(3)	C(1)–Cr(1)–C(18)	113.6(1)
Cr(1)–C(2)	2.286(3)	C(2)–Cr(1)–C(18)	145.3(1)
Cr(1)–C(3)	2.247(3)	C(3)–Cr(1)–C(18)	126.4(1)
Cr(1)–C(4)	2.173(3)	C(4)–Cr(1)–C(18)	90.3(1)
Cr(1)–C(5)	2.199(2)	C(5)–Cr(1)–C(18)	83.7(1)
Cr(1)–C(11)	2.228(2)	C(11)–Cr(1)–C(18)	75.4(1)
Cr(1)–C(12)	2.207(2)	C(12)–Cr(1)–C(18)	109.0(1)
Cr(1)–C(13)	2.114(2)	C(13)–Cr(1)–C(18)	115.2(1)
Cr(1)–C(14)	2.109(3)	C(14)–Cr(1)–C(18)	83.6(1)
Cr(1)–C(15)	2.200(2)	C(15)–Cr(1)–C(18)	88.8(1)
Cr(1)–C(18)	1.825(3)	Cr(1)–C(18)–O(1)	177.8(2)
O(1)–C(18)	1.154(4)	C(2)–C(1)–C(5)	108.2(2)
C(1)–C(2)	1.426(3)	C(2)–C(1)–C(6)	127.8(3)
C(1)–C(5)	1.414(4)	C(5)–C(1)–C(6)	123.5(2)
C(1)–C(6)	1.503(3)	C(1)–C(2)–C(3)	108.1(2)
C(2)–C(3)	1.418(3)	C(1)–C(2)–C(7)	127.0(2)
C(2)–C(7)	1.505(5)	C(3)–C(2)–C(7)	124.6(2)
C(3)–C(4)	1.423(4)	C(2)–C(3)–C(4)	108.0(2)
C(3)–C(8)	1.495(3)	C(2)–C(3)–C(8)	125.2(3)
C(4)–C(5)	1.433(3)	C(4)–C(3)–C(8)	126.1(2)
C(4)–C(9)	1.503(4)	C(3)–C(4)–C(5)	107.8(2)
C(5)–C(10)	1.498(4)	C(3)–C(4)–C(9)	125.8(2)
C(11)–C(12)	1.418(4)	C(5)–C(4)–C(9)	126.1(3)
C(12)–C(13)	1.396(3)	C(1)–C(5)–C(4)	107.8(2)
C(12)–C(16)	1.502(4)	C(1)–C(5)–C(10)	125.7(2)
C(13)–C(14)	1.449(4)	C(4)–C(5)–C(10)	125.9(2)
C(14)–C(15)	1.407(3)	C(11)–C(12)–C(13)	119.4(2)
C(14)–C(17)	1.507(5)	C(11)–C(12)–C(16)	120.9(2)
		C(13)–C(12)–C(16)	119.5(3)
		C(12)–C(13)–C(14)	119.7(3)
		C(13)–C(14)–C(15)	112.0(3)
		C(13)–C(14)–C(17)	124.1(2)
		C(15)–C(14)–C(17)	123.6(3)

cm^{-1}), Cp(DMPD)CrCO (1913 cm^{-1}) and Cp*(PD)-CrCO (1903 cm^{-1}).

Initial studies of **1** indicate that this complex reacts with triphenylsilanol in the presence of potential donors such as PMe_3 with some loss of Cp* in addition to the desired removal of the DMPD ligand; chromium-containing products in these reactions have not yet been characterized [3]. Attempts to prepare the target complexes $Cp^*CrL_2(OSiR_3)$ continue.

Supplementary material

Supplementary structural data are available from the authors upon request. These include anisotropic displacement coefficients of non-hydrogen atoms for both complexes and hydrogen atom coordinates with isotropic displacement coefficients for Cp*(DMPD)CrCO (**2**).

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