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Synthesis and Crystal Structure of the First Neutral Bis(η^5-C_2B_4-carborane)chromium(IV) Sandwich Complex

Aderemi R. Oki, Hongming Zhang, John A. Maguire, and Narayan S. Hosmane*
Department of Chemistry, Southern Methodist University, Dallas, Texas 75275

Hyekyeong Ro and William E. Hatfield
Department of Chemistry, University of North Carolina at Chapel Hill, Chapel Hill, North Carolina 27599

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Summary: Oxidation of the lithium salt of the paramagnetic chromium(III) sandwich complex, $\mathrm{Li^+(THF)_4\{1,1'}$-*commo*-$\mathrm{Cr[2,3\text{-}(SiMe_3)_2\text{-}2,3\text{-}C_2B_4H_4]_2\}^-}$ (**2**) with anhydrous $PbCl_2$ in a molar ratio 2:1 in dry hexane/THF solution produced the novel, diamagnetic and neutral chromium(IV) sandwich complex 1,1′-*commo*-$\mathrm{Cr[2,3\text{-}(SiMe_3)_2\text{-}2,3\text{-}C_2B_4H_4]_2}$ (**1**) as a dark red, air-sensitive crystalline solid in 63% yield. Complex **1** was characterized by ^{1}H, ^{11}B, and ^{13}C NMR spectra, mass and IR spectra, and also by X-ray crystallography. The crystal structure of **1** reveals that the central chromium metal, in a formal oxidation state of 4+, lies at a crystallographic center of symmetry and adopts an essentially η^5-bonding posture with respect to each of the C_2B_3 faces. However, the chromium is slipped significantly toward the cage carbons giving an average Cr–B distance of 2.321 (7) Å, which is considerably longer than the average Cr–C distance of 2.179 (5) Å, thus showing a trend opposite to those observed in the structures of main-group heterocarboranes.

Compounds containing chromium in formal oxidation states ranging from 0 to 6+ are known. In general, the 0, 2+, and 3+ are the most stable oxidation states, and a variety of compounds, including π complexes with cyclopentadienide and carboranyl ligands, has been reported.[1-3] Many of these compounds have been found to be useful as catalysts in olefin polymerization, hydrogenation, and isomerization reactions.[1,2] The chemistry of 1+, 5+, and 6+ oxidation states of chromium comprise a somewhat smaller but rapidly growing area.[1,4,5] On the other hand, our knowledge of the chemistry of chromium(IV) is quite limited, being restricted to the paramagnetic oxides, fluorides, alkyls, and alkoxides in addition to several σ-bonded porphyrin and 1,2-bis(dimethylphosphino)ethane complexes.[6] To date, there have been no reports on any of the π complexes of Cr(IV). Herein we report the synthesis and X-ray crystal structure of 2,2′,3,3′-tetrakis(trimethylsilyl)[1,1′-*commo*-bis(2,3-dicarba-1-chroma-*closo*-heptaborane)](12), 1,1′-$\mathrm{Cr[2,3\text{-}(SiMe_3)_2\text{-}2,3\text{-}C_2B_4H_4]_2}$ (1). To the best of our knowledge, compound 1 represents the first Cr(IV) sandwiched π-complex ever to be reported.

Oxidation of the lithium salt of the paramagnetic chromium(III) sandwich complex, $\mathrm{Li^+(THF)_4\{1,1'}$-*commo*-$\mathrm{Cr[2,3\text{-}(SiMe_3)_2\text{-}2,3\text{-}C_2B_4H_4]_2\}^-}$ (2)[7] with anhydrous $PbCl_2$ in a molar ratio 2:1 in dry hexane/tetrahydrofuran (THF) solution resulted in the isolation of dark red, air-sensitive crystals of 1 in 63% yield, as shown in eq 1.[8]

$$2\underset{2}{\mathrm{Li^+(THF)_4\{1,1'\text{-}}\mathit{commo}\mathrm{\text{-}Cr[2,3\text{-}(SiMe_3)_2\text{-}2,3\text{-}C_2B_4H_4]_2\}^-}} + \mathrm{PbCl_2} \xrightarrow[25\ °\mathrm{C},\ 72\ \mathrm{h}]{\text{hexane/THF}} 2\mathrm{LiCl} + 2\underset{1}{\{\mathrm{1,1'\text{-}}\mathit{commo}\mathrm{\text{-}Cr[2,3\text{-}(SiMe_3)_2\text{-}2,3\text{-}C_2B_4H_4]_2}\}} + 8\mathrm{THF} + \mathrm{Pb^0} \quad (1)$$

The fact that the well-resolved NMR spectra could be obtained indicates that 1 is diamagnetic, which is in agreement with the temperature-dependent magnetic susceptibility measurements on the powdered sample of 1.[9] Reproducible microanalytical data for 1 could not be obtained, as it is extremely sensitive to air and moisture.[8] The ^{1}H and ^{13}C NMR spectra of 1 were consistent with

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(7) In a procedure identical with that employed in the synthesis of a zwitterionic zirconium sandwich complex (Siriwardane, U.; Zhang, H.; Hosmane, N. S. *J. Am. Chem. Soc.* **1990**, *112*, 9637), a 6.7-mmol sample of $\mathrm{Na^+(THF)Li^+[2,3\text{-}(SiMe_3)_2\text{-}2,3\text{-}C_2B_4H_4]^{2-}}$ double salt (Siriwardane, U.; Islam, M. S.; West, T. A.; Hosmane, N. S.; Maguire, J. A.; Cowley, A. H. *J. Am. Chem. Soc.* **1987**, *109*, 4600) was allowed to react with 3.3 mmol of anhydrous $CrCl_3$ (0.523) in dry benzene (30 mL) at 0 °C for 5 h and then 24 h at room temperature with constant stirring, during which time the solution became turbid and its color turned to reddish brown. At this point, the heterogeneous product mixture was filtered through a frit in vacuo, and the residue was washed repeatedly with a solvent mixture of anhydrous benzene (95%) and THF (5%), to collect a clear red-orange filtrate. After slow removal of the solvents from the filtrate in vacuo, an orange, air-sensitive crystalline solid, identified as $\mathrm{Li^+(THF)_4\{1,1'}$-*commo*-$\mathrm{Cr[2,3\text{-}(SiMe_3)_2\text{-}2,3\text{-}C_2B_4H_4]_2\}^-}$ (2), was obtained in 77% yield (2.0 g, 2.55 mmol; soluble in polar solvents; mp 210 °C dec). The anionic sandwiched geometry for 2 was confirmed by X-ray crystallography. The magnetic moment of 3.62 μ is consistent with the high-spin d^3 system. A comprehensive study on 2 will be published as a full paper in the future (Oki, A. R.; Zhang, H.; Maguire, J. A.; Hosmane, N. S.; Ro, H.; Hatfield, W. E. To be published).

(8) A 0.257-mmol sample of $\mathrm{Li^+(THF)_4\{1,1'}$-*commo*-$\mathrm{Cr[2,3\text{-}(SiMe_3)_2\text{-}2,3\text{-}C_2B_4H_4]_2\}^-}$ (2)[7] (0.2 g) was allowed to react with 0.137 mmol of anhydrous lead(II) chloride (0.038 g) in dry, 1:1 mixture of THF/hexane (20 mL) at room temperature for 72 h, during which time the solution became turbid and its color turned to dark red. After removal of THF and hexane from the heterogeneous solution in vacuo, the brown residue was heated to 120 °C, and the dark red crystalline solid was sublimed from the reactor into a detachable U-trap that was held at 0 °C. The solid in the U-trap was later identified as neutral 1,1′-*commo*-$\mathrm{Cr[2,3\text{-}(SiMe_3)_2\text{-}2,3\text{-}C_2B_4H_4]_2}$ (1) (0.08 g, 0.162 mmol; 63% yield; mp 165 °C; *air-sensitive, turns green when exposed to air*; soluble in both polar and nonpolar organic solvents). The residue that remained in the reactor after sublimation was identified by qualitative analysis as a mixture of elemental lead (Pb^0) and LiCl, and was therefore discarded.

(9) The temperature-dependent magnetic susceptibility of a powdered sample of 1 (placed in a polycarbonate capsule in an atmosphere of N_2) using the Faraday technique in the temperature range of 77 to 294 K, as described elsewhere (Dobson, J. C.; Helms, J. H.; Doppelt, P.; Sullivan, B. P.; Hatfield, W. E.; Meyer, T. J. *Inorg. Chem.* **1989**, *28*, 2200), showed a small paramagnetic signal of $\chi = 2.0 \times 10^{-7}$ emu/g. Since a carefully purified sample gave well-resolved NMR spectra, it was concluded that the paramagnetic signal was due to an impurity that could have arisen from reaction of 1 with the sample holder.

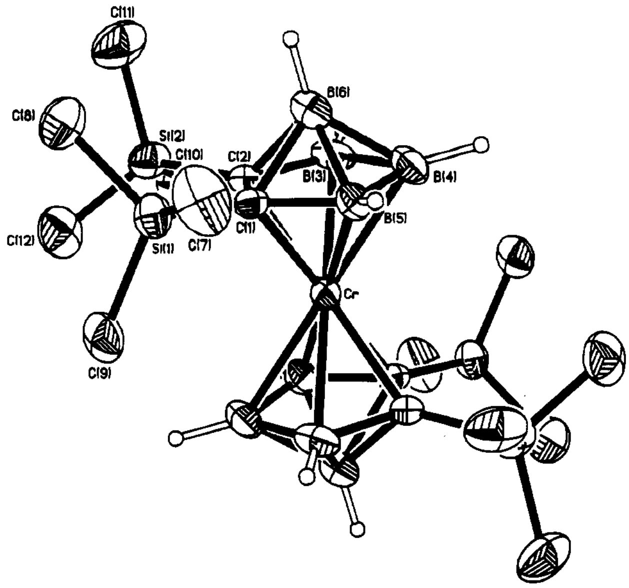

Figure 1. Perspective view of 1 with thermal ellipsoids drawn at the 40% probability level and showing the atom-numbering scheme. The central Cr atom lies at a crystallographic center of symmetry. Pertinent parameters include the following: Cr–C(1) 2.192 (5); Cr–C(2) 2.167 (5); Cr–B(3) 2.304 (7); Cr–B(4) 2.384 (7); Cr–B(5) 2.276 (6); Cr–C_2B_3 centroid 1.809 Å; C_2B_3 centroid(1)–Cr-C_2B_3 centroid(2) 180.0°.

the presence of two equivalent $SiMe_3$ groups. The ^{11}B NMR spectrum showed only a single broad doublet at −4.93 ppm [$^1J(^{11}B-^1H)$ = 141.3 Hz].[10] This could be the result of an accidental overlap of the apical and basal boron resonances, which is a commonly observed phenomenon in polyhedral borane chemistry.[3e] In an effort to obtain more information, $^{11}B(T_1)$ NMR measurements were made.[11] The $^{11}B(T_1)$ NMR spectra of 1 showed a single broad signal at −4.9 ppm, over a period of 1 μs–5 ms with an inversion recovery of 0.99 ms. Thus, T_1 measurements are not helpful in clarifying the ^{11}B NMR spectra of 1. The most interesting feature of the NMR spectra is the appearance of the cage carbon resonance as a broad singlet at −10.92 ppm in both the proton-coupled and proton-decoupled ^{13}C NMR spectra of 1. A significant upfield shift, by about 154 ppm, from that of the *nido*-carborane precursor[12] indicates that complexation with chromium results in strong shielding of these cage carbons. Such shieldings of the cage carbons are not observed in the ^{13}C NMR spectra of main-group heterocarboranes.[13] The high-resolution electron-impact (EI) mass spectrum of 1 exhibited a grouping with the major cutoff at m/z 489 (exact mass, 489.2699) corresponding to the parent ion $[^{53}Cr^{28}Si_4(^{12}CH_3)_{12}{}^{12}C_4{}^{11}B_8H_8]^+$. The IR spectrum of 1 is also consistent with the formation of a heterocarborane complex.[10] Unfortunately, none of these spectral data offers an insight into the exact mode of the bonding between chromium and carborane. Indeed, none clearly demonstrates the existence of a *commo*-chromacarborane complex. Therefore, an X-ray crystallographic study of 1 was undertaken which showed this complex to be the novel chromacarborane 1,1′-*commo*-Cr[2,3-$(SiMe_3)_2$-2,3-$C_2B_4H_4]_2$ (1), as represented in a thermal ellipsoid diagram in Figure 1.[14]

The crystal structure reveals that the chromium atom adopts an essentially η^5-bonding posture with respect to each of the C_2B_3 faces with the Cr–C_2B_3(centroid) distance of 1.809 Å and yielding a $Cr(C_2B_4)_2$ cage symmetry of C_{2h} for 1. However, it should be noted that a close examination of the metal to cage distances shows that the chromium is slipped significantly toward the cage carbons. Each chromium–boron(unique) distance [Cr–B(4)] is longer than the Cr–B(3,5) distance by about 0.1 Å, and the average Cr–B distance [2.321 (7) Å] is considerably longer than the average Cr–C distance [2.179 (5) Å]. This slippage also explains the strong shielding of the cage carbon resonance observed in the ^{13}C NMR spectra of 1. It is of interest to note that the slippage of the metal in the present case is exactly opposite to that observed in the main-group heterocarborane system.[15]

Although the geometry of 1 resembles that of a chromocene derivative,[16] the two systems are quite dissimilar. The most important difference to be noted is that of charge; each carborane ligand bears a 2− charge while the cyclopentadienide ligands are monoanions. The chromium metal in the formal oxidation state of 3+ would result in an anionic sandwich complex with the carboranyl ligands, while Cr(IV) would yield the neutral sandwich complex. The crystal structure clearly shows the complex to be neutral. Therefore, the carboranyl ligands seem to stabilize unusual oxidation states of the transition metals more effectively than do their organic counterparts.

Room-temperature magnetic susceptibility measurements on 2[7] and the icosahedral analogue $Cs[Cr(\eta^5\text{-}3,1,2\text{-}C_2B_9H_{11})_2]$[3a] show that these complexes are of the high-spin d^3 system. Since the sandwich geometry of each of these anionic Cr(III) complexes is almost similar to that of neutral Cr(IV) derivative 1, the diamagnetism exhibited by this complex is somewhat surprising. The paramagnetism, observed for the anionic Cr(III) sandwich complexes, indicates that the d electrons of the Cr metal occupy a set of MO's with very similar energies; preliminary molecular orbital calculations[17] show this to be the case. The chromium's d electrons can be assigned to a set of very weakly bonding and nonbonding MO's composed of the metal d orbitals that are oriented parallel to the C_2B_3 face of the carborane ligands. The differences in the energies of these MO's are very sensitive to the charge on the chromium and increase as the metal becomes more posi-

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[10] Spectroscopic data for 1: ^1H NMR (C_6D_6, relative to external Me_4Si) δ 3.24 [q(br), 8 H, basal and apical H_t, $^1J(^1H-^{11}B)$ = 142 Hz], 0.17 [s, 36 H, $SiMe_3$]; ^{11}B NMR (C_6D_6, relative to external $BF_3{\cdot}OEt_2$) δ −4.93 [d(br), 8 B, basal and apical BH, $^1J(^{11}B-^1H)$ = 141.3 Hz]; ^{13}C NMR ($CDCl_3$, relative to external Me_4Si) δ −1.08 [q, 12 C, $SiMe_3$, $^1J(^{13}C-^1H)$ = 119.4 Hz], −10.92 [s (br), 4 C, cage carbons (SiCB)]. IR (cm^{-1}; C_6D_6 vs C_6D_6): 3230 (m), 2960 (vs), 2900 (m, s), 2850 (m) [ν(C–H)], 2580 (vvs), 2520 (vvs) [ν(B–H)], 2270 (vs), 1615 (m), 1450 (m, s), 1405 (m, s) [$\delta(CH)_{asym}$], 1330 (s), 1250 (vs) [$\delta(CH)_{sym}$], 1200 (vs), 1130 (s), 1095 (s), 1030 (w), 1020 (w), 985 (m, s), 970 (m), 955 (m), 935 (m, s), 900–800 (vvs, br) [ρ(CH)], 750 (s), 690 (s), 660 (m), 622 (m), 585 (m), 500 (vs, br), 345 (s).

[11] The 115-MHz NMR data of ^{11}B T_1 measurements on the $CDCl_3$ solution of 1 were obtained from the Spectral Data Services, Inc., Champaign, IL.

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[14] A data set was collected at 230 K on a dark red rectangular crystal of monoclinic space group $P2_1/n$ with the following unit cell parameters: a = 9.851 (3) Å, b = 13.813 (5) Å, c = 11.195 (5) Å, β = 95.66 (3)°, V = 1519.9 (10) $Å^3$, Z = 4, and D_{calcd} = 1.068 g/cm^3. Full-matrix least-squares refinements of 1 converged at R = 0.047, and R_w = 0.068 for 1350 observed ($I > 3.0\sigma(I)$) reflections. The structure was solved by the heavy-atom methods stored in the program package SHELXTL-PLUS (Sheldrick, G. M. *Structure Determination Software Programs*; Siemens Analytical X-ray Instruments, Inc.: Madison, WI, 1990).

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tive. Therefore, the Cr(III) sandwiched complexes show three unpaired electrons, while the Cr(IV) derivative is diamagnetic.[17] A detailed theoretical explanation will be reported in the near future.

Investigations of the reaction chemistry of the anionic and neutral chromacarboranes and other related sandwich complexes are currently underway in our laboratories.

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Supplementary Material Available: Tables of positional and thermal parameters, selected bond distances, bond angles, and torsion angles (5 pages); a listing of observed and calculated structure factors (5 pages). Ordering information is given on any current masthead page.

C_2-Symmetric 2,2′-Dimethyl-1,1′-binaphthyl-Bridged *ansa*-Bis(1-indenyl)metal Complexes

Mark J. Burk

Central Research and Development Department, E. I. du Pont de Nemours & Company, Experimental Station, Wilmington, Delaware 19880-0328

Steven L. Colletti and Ronald L. Halterman*

Department of Chemistry, Boston University, Boston, Massachusetts 02215

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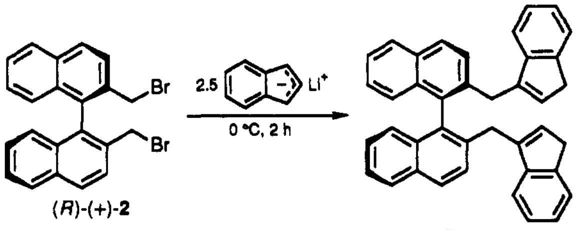
Summary: The use of a C_2-symmetric 2,2′-dimethyl-1,1′-binaphthyl bridge between two 1-indenyl ligands, readily obtained from known, enantiomerically pure 2,2′-bis(bromomethyl)-1,1′-binaphthyl, suppresses the formation of an undesired meso orientation of *ansa*-bis(indenyl)metal complexes of titanium and zirconium dichlorides **3** and favors the desired isolation of one of the possible two C_2-symmetric isomers in enantiomerically enriched form, avoiding the need for resolution of the metal complexes.

C_2-symmetric ethylene-bridged *ansa*-bis(indenyl)titanium and -zirconium dichlorides are well-known to behave as homogeneous catalysts for stereoregular propylene polymerizations[1] and enantioselective alkene hydrogenations[2] and as reagents for allyl additions to aldehydes.[3] Despite these successful applications and the implied great potential of these compounds in other enantioselective transformations, no practical direct synthetic route to enantiomerically pure bis(indenyl) ligands and/or their corresponding complexes has yet been developed. Current strategies involve the initial formation of an isomeric mixture of racemic (C_2-symmetric) and meso (achiral) bis(indenyl)metal complexes, which first must be separated by crystallization, followed by optical resolution of the C_2-symmetric enantiomers (via diastereomeric derivatives) and finally conversion to the enantiomerically pure dichlorides.[4,5] The introduction of a C_2-symmetric chiral bridge between the two indene moieties could potentially suppress formation of the undesired meso complex and favor the formation of one of the two possible C_2-symmetric bis(indenyl)metal diastereomers. We report here the preparation of the new chiral ligand (*R*)-(+)-(1,1′-binaphthyldiyl-2,2′-dimethylene)bis(1-indene) (**1**), which is shown to form a single C_2-symmetric stereoisomer upon coordination to titanium and zirconium.[6]

We recently have reported the conversion of (*R*)-(+)-2,2′-bis(bromomethyl)-1,1′-binaphthyl (**2**) to a binaphthyl-annulated C_2-symmetric chiral cyclopentadiene.[7] We now find that addition of dibromide (*R*)-(+)-**2** to 2.5 equiv of indenyllithium in THF at 0 °C and warming to room temperature affords bis(indene) (*R*)-(+)-**1** (mp 195 °C, $[\alpha]_D^{23}$ +18°)[8] as a single thermodynamically favored double-bond isomer in 71% yield after chromatography



(*R*)-(+)-**2**

(*R*)-(+)-**1**

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* To whom correspondence should be addressed at the Department of Chemistry, University of Oklahoma, 620 Parrington Oval, Norman, OK 73019-0370.

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