

Syntheses and Characterization of Half-Sandwich Zirconium Complexes with Dichalcogenolate *o*-Carborane Ligands

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Metallocene complex $\text{Cp}_2^{\mu}\text{ZrCl}_2$ ($\text{Cp}^{\mu} = \eta^5\text{-}1,3\text{-}'\text{Bu}_2\text{C}_5\text{H}_3$) (**1**) has been prepared from the reaction of LiCp^{μ} with ZrCl_4 in good yield. Reactions of **1** with dilithium dichalcogenolate *o*-carboranes afforded new type of half-sandwich compounds with dichalcogenolate *o*-carboranyl ligands, $[\text{Li}(\text{THF})_4][\text{Cp}^{\mu}\text{Zr}(\text{E}_2\text{C}_2\text{B}_{10}\text{H}_{10})_2]$ ($\text{E} = \text{S}$, **2a**; $\text{E} = \text{Se}$, **2b**) in which only one cyclopentadienyl ring ligand existed. Complexes **1** and **2a** were structurally characterized by X-ray analyses. In complex **2a**, the Zr(IV) ion is η^5 -bound to one 1,3-di-*tert*-cyclopentadienyl ring and σ -bound to four μ_2 -sulfur atoms of two dithio-carboranes. The zirconium atom and four sulfur atoms form a distorted pyramid. The coordination sphere around the zirconium atom resembles in a piano stool structure with four legs of sulfur atoms and the fulcrum at the zirconium atom.

Keywords half-sandwich zirconium complex, metallocene, dichalcogenolate *o*-carborane, crystal structure

Introduction

During the past decade considerable attention has been devoted to the metal complexes with dichalcogenolate *o*-carboranyl ligands, to take the advantage of its unique molecular structure. Up to date intensive studies on half-sandwich transition metal complexes of Fe,¹ Ru,¹ Co,² Rh,^{3,4} and Ir²⁻⁶ containing a chelating 1,2-dicarba-closo-dodecaborane-1,2-dichalcogenolate ligands have illustrated that these complexes serve as excellent precursors to study the addition reaction of acetylene and the substitution of boron atom in carboranes.⁶⁻⁸ However, the group IV transition metal complexes with dichalcogenolate *o*-carboranyl ligands are so far unknown and the reactions of metallocene complexes with chalcogenolate carboranyl lithium salt are still limited studied. These findings prompted us to investigate the reactions of metallocene complexes with dilithium dichalcogenolate *o*-carborane.

As highly effective catalysts for olefin polymerization, many metallocene complexes Cp_2MCl_2 ($\text{Cp} = \eta^5\text{-C}_5\text{H}_5$, $\text{M} =$

Ti, Zr, Hf) have been reported during past two decades.⁹ Usually the substitutes on the cyclopentadienyl rings would make some differences in catalytic properties and have more or less influences on the structures and properties of the polymer products. The use of the cyclopentadienyl ligand with two *tert*-butyl substitutes is postulated to generate a fairly considerable steric effect on the complex structure and therefore, the catalytic activity for the ethylene polymerization.^{10,11} Yet studies on metallocene complexes with di-*tert*-butyl-cyclopentadienyl ligands were only few.¹²

In this paper the metallocene complex of $\text{Cp}_2^{\mu}\text{ZrCl}_2$ ($\text{Cp}^{\mu} = \eta^5\text{-}1,3\text{-}'\text{Bu}_2\text{C}_5\text{H}_3$) (**1**) was synthesized and its reaction with dilithium dichalcogenolate *o*-carborane was studied. The first half-sandwich group IV transition metal complexes containing dichalcogenolate carboranyl ligands (Scheme 1), $[\text{Li}(\text{THF})_4][\text{Cp}^{\mu}\text{Zr}(\text{E}_2\text{C}_2\text{B}_{10}\text{H}_{10})_2]$ ($\text{E} = \text{S}$, **2a**; $\text{E} = \text{Se}$, **2b**), were synthesized. Complexes **1** and **2a** were crystallographically studied.

Experimental

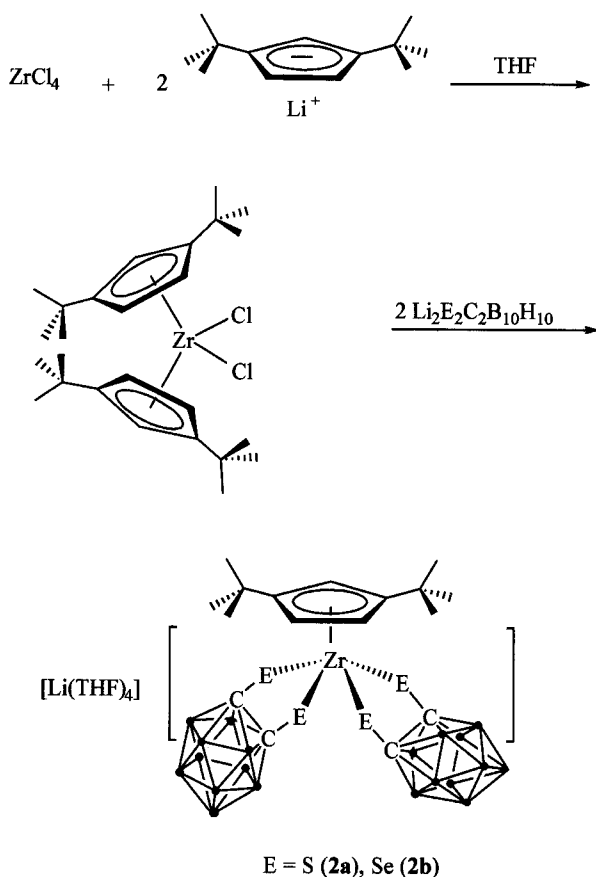
General

All the reactions and the manipulations were routinely carried out under pure argon atmosphere using the standard Schlenk technique. The use of dry and oxygen-free solvents is necessary. $[(\text{THF})_3\text{LiE}_2\text{C}_2\text{B}_{10}\text{H}_{10}\text{Li}(\text{THF})_2]$ ($\text{E} = \text{S}$, Se) were synthesized according to the procedures described in another paper.¹³ Infrared spectra were measured on a Bio-Rad FTS135 spectrometer as KBr pellets. ¹H NMR spectra were measured on the Unity-400 spectrometer in dry and oxygen-free deuterated solvents. Elemental analyses were carried out by the Analytic Center of Changchun Institute, Chinese Academy of Sciences.

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Scheme 1 Synthesis of half-sandwich zirconium bis-dichalcogenolato carborane complexes**Preparation of $\text{Cp}^{\#}\text{ZrCl}_2$ ($\text{Cp}^{\#} = \eta^5\text{-}1,3\text{-}^t\text{Bu}_2\text{C}_5\text{H}_3$) (**1**)**

$\text{Cp}^{\#}\text{ZrCl}_2$ was synthesized from the reactions of ZrCl_4 (1.07 g, 4.6 mmol) with two equivalents of $\text{LiCp}^{\#}$ in 80 mL of THF under pure argon atmosphere according to the literature¹² and recrystallized from the mixed solution of toluene/hexane at $-30\text{ }^{\circ}\text{C}$. Over one week, light yellow crystals were obtained from the solution. Yield 1.74 g (73.3%). $^1\text{H NMR}$ (400 MHz, C_6D_6) δ : 6.55 (t, $J = 1.4$ Hz, 1H), 6.03 (d, $J = 2.8$ Hz, 2H), 1.30 (s, 18H); IR (KBr) ν : 3091 (m), 2955 (s), 2873 (m), 1494 (m), 1461 (m), 1393 (m), 1368 (m), 1251 (s), 1166 (m), 1058 (m), 841 (s), 821 (m), 664 (m) cm^{-1} . EI-MS m/z (%): 514 (M^+ , 10), 499 ($\text{M}^+ - \text{CH}_3$, 4), 337 ($\text{M}^+ - ^t\text{Bu}_2\text{C}_5\text{H}_3$, 48).

Preparation of $[\text{Li}(\text{THF})_4][\text{Cp}^{\#}\text{Zr}(\text{S}_2\text{C}_2\text{B}_{10}\text{H}_{10})_2]$ (2a**)**

A solution containing $\text{Cp}^{\#}\text{ZrCl}_2$ (0.129 g, 0.25 mmol) and $[(\text{THF})_3\text{LiS}_2\text{C}_2\text{B}_{10}\text{H}_{10}\text{Li}(\text{THF})_2]$ (0.254 g, 0.25 mmol) in 20 mL of THF was kept stirring at room temperature for 24 h. After the removal of solvent under vacuum, the residue was extracted by 20 mL of toluene and then the white precipitate of LiCl was removed by centrifugation. Removal of

the solvent gave the yellow solid. Recrystallization from the mixed solution of toluene/hexane at $15\text{ }^{\circ}\text{C}$ gave the yellow crystals of **2a**. Yield 0.138 g (56.7%). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 6.47 (t, $J = 1.4$ Hz, 1H), 5.94 (d, $J = 2.8$ Hz, 2H), 1.24 (s, 18H); IR (KBr) ν : 3101 (w), 3003 (w), 2955 (s), 2902 (m), 2876 (m), 2586 (s), 2570 (s), 2170 (w), 1493 (m), 1461 (s), 1448 (m), 1393 (w), 1366 (s), 1359 (s), 1293 (w), 1251 (s), 1200 (m), 1167 (m), 1045 (s), 970 (w), 921 (w), 886 (s), 842 (s), 819 (m), 724 (m), 664 (m), 419 (w) cm^{-1} . Anal. calcd for $\text{C}_{33}\text{H}_{73}\text{B}_{20}\text{LiO}_4\text{S}_4\text{Zr}$: C 40.59, H 7.48; found C 40.88, H 7.87.

Preparation of $[\text{Li}(\text{THF})_4][\text{Cp}^{\#}\text{Zr}(\text{Se}_2\text{C}_2\text{B}_{10}\text{H}_{10})_2]$ (2b**)**

The complex **2b** was prepared as yellow precipitate from $\text{Cp}^{\#}\text{ZrCl}_2$ (0.129 g, 0.25 mmol) and $[(\text{THF})_3\text{LiSe}_2\text{C}_2\text{B}_{10}\text{H}_{10}\text{Li}(\text{THF})_2]$ (0.301 g, 0.25 mmol) in 20 mL of THF by using procedures similar to those used in the synthesis of **2a**. Yield 0.205 g (70.4%). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 6.48 (t, $J = 1.4$ Hz, 1H), 6.05 (d, $J = 2.8$ Hz, 2H), 1.33 (s, 18H); IR (KBr) ν : 3101 (w), 3003 (w), 2955 (s), 2902 (m), 2876 (m), 2586 (s), 2570 (s), 2170 (w), 1493 (m), 1461 (s), 1448 (m), 1393 (w), 1366 (s), 1359 (s), 1293 (w), 1251 (s), 1200 (m), 1167 (m), 1045 (s), 970 (w), 921 (w), 886 (s), 842 (s), 819 (m), 724 (m), 664 (m), 419 (w) cm^{-1} . Anal. calcd for $\text{C}_{33}\text{H}_{73}\text{B}_{20}\text{LiO}_4\text{Se}_4\text{Zr}$: C 34.04, H 6.27; found C 33.82, H 6.74.

Structure solution and refinement for complexes **1 and **2a****

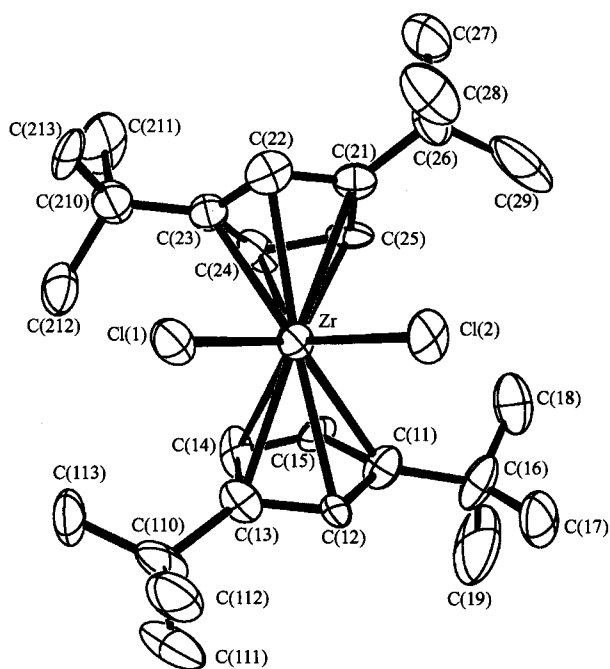
For complexes **1** and **2a**, a single crystal of each suitable for X-ray determination was sealed into a glass capillary respectively, and mounted on a Siemens P4 diffractometer. All the determinations of unit cell and intensity data were performed with graphite monochromated $\text{Mo K}\alpha$ radiation ($\lambda = 0.071073$ nm). All the data were collected at room temperature using the ω -scan technique. The structure was solved by the direct methods, expanded using Fourier techniques and refined on F^2 by a full-matrix least-squares method. The non-hydrogen atoms were refined anisotropically and hydrogen atoms were included but not refined. All the calculations were carried out with Siemens SHELXTL PLUS program. Details of crystal data are summarized in Table 1.

Results and discussion

The reaction between ZrCl_4 and $\text{LiCp}^{\#}$, which was prepared from the reaction of $\text{Cp}^{\#}\text{H}$ with $^n\text{BuLi}$, afforded good. $\text{Cp}^{\#}\text{ZrCl}_2$ was isolated as light yellow crystals in moderate yield. The molecular structure of complex $\text{Cp}^{\#}\text{ZrCl}_2$ is presented in Fig. 1, together with the atomic label.

Table 1 Crystallographic data for complexes **1** and **2a**

	Complex 1	Complex 2a
Color	Light yellow	Yellow
Habit	Block	Block
Formula	C ₂₆ H ₄₂ Cl ₂ Zr	C ₃₃ H ₇₃ B ₂₀ LiO ₄ S ₄ Zr
Formula weight	516.72	976.51
Cryst size (mm)	0.46 × 0.32 × 0.08	0.52 × 0.36 × 0.28
Cryst system	Monoclinic	Monoclinic
Space group	<i>Pn</i>	<i>P2₁/c</i>
<i>a</i> (nm)	1.2874(5)	1.1241(2)
<i>b</i> (nm)	0.6687(2)	2.7581(4)
<i>c</i> (nm)	1.6137(4)	1.7445(3)
β (°)	97.09(3)	103.83(2)
<i>V</i> (nm ³)	1.3785(8)	5.252(2)
<i>Z</i>	2	4
<i>D</i> _{calcd} (Mg/m ³)	1.245	1.235
θ range (°)	2.16—25.06	1.87—25.00
μ (mm ⁻¹)	0.602	0.403
Reflections collected	3381	11552
Independent reflections	2763 (<i>R</i> _{int} = 0.0368)	9220 (<i>R</i> _{int} = 0.0443)
<i>R</i> [<i>I</i> > 2 σ (<i>I</i>)]	0.0538	0.0752
<i>R</i> _w [<i>I</i> > 2 σ (<i>I</i>)]	0.1484	0.1816
No. of parameters	274	415
GOF on <i>F</i> ²	1.137	1.205

**Fig. 1** Crystal structure of complex **1** (the hydrogen atoms are omitted for clarity).

There are two independent Cp^zZrCl₂ molecules in the unit cell. In this complex, the zirconium atom is coordinated to two Cp^z ligands and two chloride atoms. The two *tert*-butyl substitutes on the Cp^z rings are opposite to each other to decrease the bulky steric effect. Table 2 exhibits the selected

bond distances and angles for complex **1**. The averaged C—C bond distances for the two Cp^z rings are 0.142 and 0.140 nm, respectively, close to the C—C distance of 0.140 nm found in analogous metallocene complexes, (t-BuC₅H₄)₂TiCl₂, and (t-BuC₅H₄)₂Ti(NCS)₂.^{14–16} The respective averaged Zr—C bond distances [C(11)—C(15) ring and C(21)—C(25) ring] are 0.255 and 0.254 nm, longer than the Ti—C length of 0.2355 nm in corresponding titanium complexes with Cp^z ligand after the subtraction of the difference between ionic radii.¹⁶ The distances between the zirconium atom and the two centers of the Cp^z rings are 0.2246 and 0.2239 nm, respectively. The bond distances of Zr—Cl(1) and Zr—Cl(2) [0.2465(5) and 0.2428(4) nm] are not beyond the range for normal zirconium complexes.¹⁷ The two centers of Cp^z rings and the two chlorides form a distorted tetrahedral sphere with the Cp^z(1)—Zr—Cp^z(2) angle of 129.6°. The chloride atoms are located at the *cis*-position around the zirconium atom with the nearly perpendicular angle of 93.60(8)°.

Reactions of Cp^zZrCl₂ with an equiv. molar amount of [(THF)₃LiE₂C₂B₁₀H₁₀Li(THF)]₂ in THF at room temperature gave the half-sandwich compounds of formula [Li(THF)₄][Cp^zZr(E₂C₂B₁₀H₁₀)₂] (E = S, **2a**; E = Se, **2b**) in which one cyclopentadienyl ring was omitted (Scheme 1). The chlorine atoms in the metallocene complex can be easily replaced by the dichalcogenolate carboranyl ligands via metathesis reaction. Complexes **2a** and **2b** are air

and moisture sensitive crystals and soluble in polar organic solvents such as THF and toluene but insoluble in nonpolar solvents such as hexane. Their IR spectra show a typical strong and broad characteristic B—H absorption at about 2570 cm^{-1} , which is consistent with the previous reported complexes with carboranyl ligands.¹⁻⁶ $[\text{Li}(\text{THF})_4][\text{Cp}^*\text{Zr}(\text{E}_2\text{C}_2\text{B}_{10}\text{H}_{10})_2]$ can also be obtained from the reaction of

half-sandwich complex Cp^*ZrCl_3 with $[(\text{THF})_3\text{LiE}_2\text{C}_2\text{B}_{10}\text{H}_{10}\text{Li}(\text{THF})_2]$.

Recrystallization of **2a** from a THF/hexane solution led to yellow single crystals which is suitable for X-ray crystallographical analysis. The anionic moiety of complex **2a** is shown in Fig. 2, together with the atomic labeling scheme.

Table 2 Selected bond lengths ($\times 10^{-1}\text{ nm}$) and angles ($^\circ$) for complex **1**

Zr—Cl(1)	2.465(5)	Zr—Cl(2)	2.428(4)
Zr—C(11)	2.58(2)	Zr—C(12)	2.63(2)
Zr—C(13)	2.56(2)	Zr—C(14)	2.48(2)
Zr—C(15)	2.50(2)	Zr—C(21)	2.64(2)
Zr—C(22)	2.56(2)	Zr—C(23)	2.56(2)
Zr—C(24)	2.46(2)	Zr—C(25)	2.46(2)
C(11)—C(12)	1.40(3)	C(11)—C(15)	1.44(2)
C(12)—C(13)	1.42(3)	C(13)—C(14)	1.47(3)
C(14)—C(15)	1.36(3)	C(21)—C(22)	1.38(3)
C(21)—C(25)	1.34(3)	C(22)—C(23)	1.43(3)
C(23)—C(24)	1.36(3)	C(24)—C(25)	1.48(3)
Cl(1)—Zr—Cl(2)	93.60(8)	Cl(1)—Zr—C(13)	80.0(5)
Cl(1)—Zr—C(11)	129.8(5)	Cl(1)—Zr—C(14)	99.9(4)
Cl(1)—Zr—C(12)	98.8(4)	Cl(1)—Zr—C(15)	131.2(5)
Cl(1)—Zr—C(21)	111.7(5)	Cl(1)—Zr—C(23)	84.1(5)
Cl(1)—Zr—C(25)	134.8(4)	Cl(1)—Zr—C(24)	113.2(5)
C(11)—Zr—Cl(2)	83.8(5)	Cl(1)—Zr—C(22)	83.4(5)
C(12)—Zr—Cl(2)	81.7(4)	Cl(2)—Zr—C(13)	108.3(5)
C(21)—Zr—Cl(2)	80.9(5)	Cl(2)—Zr—C(14)	133.6(5)
C(25)—Zr—Cl(2)	98.3(5)	Cl(2)—Zr—C(15)	115.4(4)
Cl(2)—Zr—C(23)	127.1(5)	Cl(2)—Zr—C(22)	94.6(4)
Cl(2)—Zr—C(24)	132.2(4)		

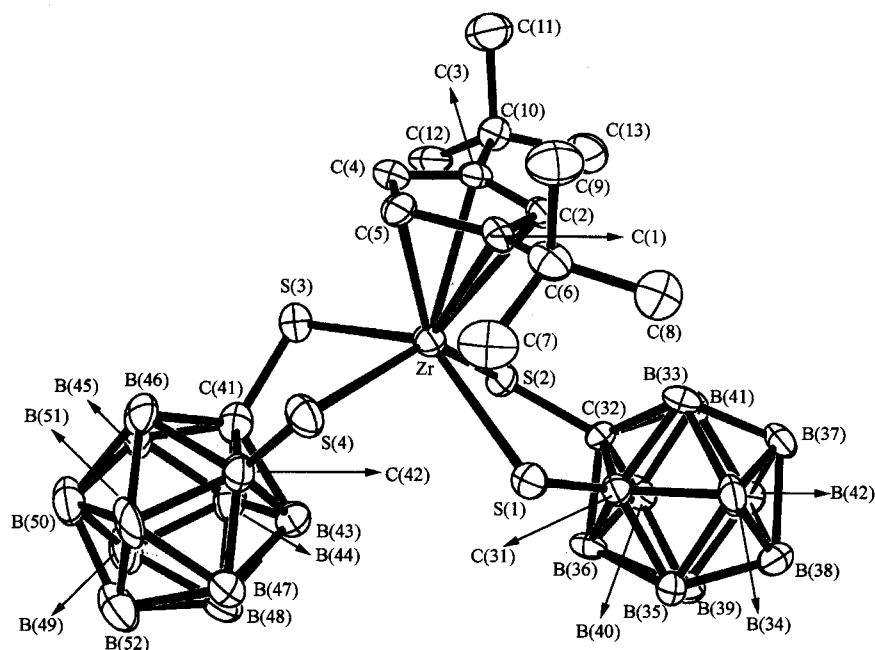


Fig. 2 Anionic moiety of complex **2a** (the hydrogen atoms are omitted for clarity).

The molecular structure of **2a** consists of one well-separated, alternating layers of the discrete tetrahedral cations $[\text{Li}(\text{THF})_4]^+$ and the complex anion $[\text{Cp}^*\text{Zr}(\text{S}_2\text{C}_2\text{B}_{10}\text{H}_{10})_2]^-$. Lithium atom is coordinated to four oxygen atoms of THF molecules to afford the cation of the complex. In the complex anion, the Zr^{4+} ion is η^5 -bound to one 1,3-di-*tert*-cyclopentadienyl rings and σ -bound to four μ_2 -sulfur atoms of two dithio-carboranes. The zirconium atom and four sulfur atoms form a distorted pyramid. The coordination sphere around the zirconium atom resembles in a piano stool structure with four legs of sulfur atoms and the fulcrum at the zirconium atom. Thus piano stool structure is also found in analogous half-sandwich zirconium complex, $[\text{Li}(\text{THF})_4][\text{Cp}^*\text{Zr}(\text{S}_2\text{C}_2\text{B}_{10}\text{H}_{10})_2]$.^{1b} The two *tert*-butyl substitutes on the Cp^* lig-

and are located in the vacancy between the two bulky dithio-carborane ligands to avoid the crowded steric effect.

The selected bond distances and bond angles are presented in Table 3. The average C—C bond distance for the Cp^* ring is 0.1414 nm, among those (0.141 nm) found in analogous complex **1**. The average Zr—C bond distance [C(1)—C(5) ring] is 0.2530 nm, shorter than the corresponding one in complex **1**. The bond distances of Zr—S are in the range from 0.2548(3) to 0.2585(2) nm and the average Zr—S length is 0.2567 nm, consistent with the reported ones.¹⁸ The S—C bond length distribution is between 0.1774(9) and 0.1803(10) nm and the average S—C distance is 0.1784 nm. The average S-Zr-S angle between the two neighboring sulfur atoms and the zirconium atom is 81.71°. Fig. 3 is the stacking effect picture of complex **2a**.

Table 3 Selected bond lengths ($\times 10^{-1}$ nm) and angles ($^\circ$) for complex **2a**

Zr—C(1)	2.548(7)	Zr—C(2)	2.528(7)
Zr—C(3)	2.565(8)	Zr—C(4)	2.504(9)
Zr—C(5)	2.505(8)	Zr—S(1)	2.577(2)
Zr—S(2)	2.585(2)	Zr—S(3)	2.548(3)
Zr—S(4)	2.556(2)	S(1)—C(31)	1.776(8)
S(2)—C(32)	1.782(8)	S(3)—C(41)	1.803(10)
S(4)—C(42)	1.774(9)		
S(1)—Zr—S(2)	79.65(8)	S(1)—Zr—S(3)	134.15(8)
S(1)—Zr—S(4)	83.15(8)	S(2)—Zr—S(3)	82.29(8)
S(2)—Zr—S(4)	136.50(8)	S(3)—Zr—S(4)	81.73(9)
S(1)—Zr—C(1)	92.2(2)	S(1)—Zr—C(5)	118.6(2)
S(2)—Zr—C(1)	127.5(2)	S(2)—Zr—C(5)	145.1(2)
S(3)—Zr—C(1)	131.4(2)	S(3)—Zr—C(5)	99.8(2)
S(4)—Zr—C(1)	92.7(2)	S(4)—Zr—C(5)	77.6(2)

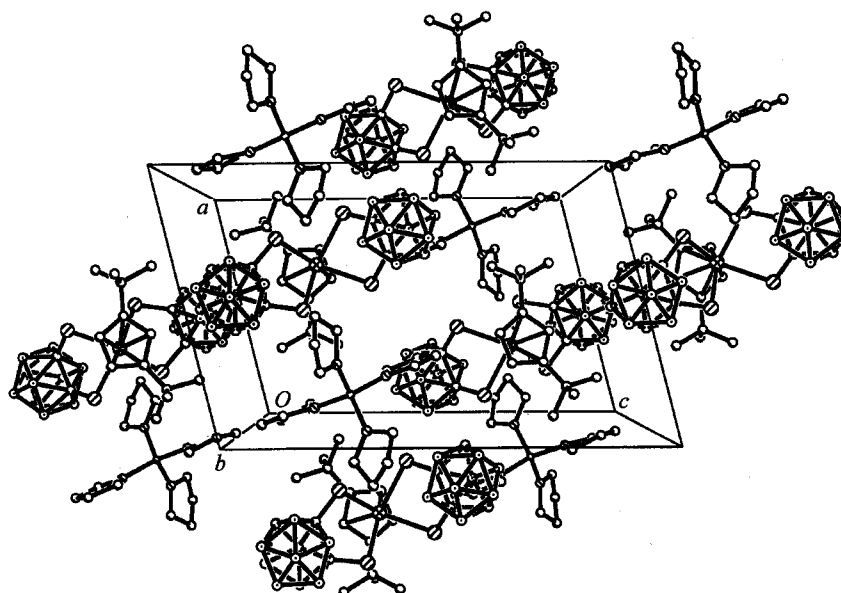


Fig. 3 Stacking effect picture of complex **2a**.

Conclusion

Reactions of metallocene complex $\text{Cp}_2^{\text{M}}\text{ZrCl}_2$ with dilithium dichalcogenolate *o*-carboranes afforded half-sandwich compounds with dichalcogenolate *o*-carboranyl ligands, $[\text{Li}(\text{THF})_4][\text{Cp}^{\text{M}}\text{Zr}(\text{E}_2\text{C}_2\text{B}_{10}\text{H}_{10})_2]$. Complexes $\text{Cp}_2^{\text{M}}\text{ZrCl}_2$ and $[\text{Li}(\text{THF})_4][\text{Cp}^{\text{M}}\text{Zr}(\text{S}_2\text{C}_2\text{B}_{10}\text{H}_{10})_2]$ were structurally characterized. As far as we know, this is the first crystal structure of group IV complexes with dichalcogenolate *o*-carboranyl ligands.

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