

Reactivity of Two MeCp-Cobalt Complexes Containing 1,2-Dicarba-*closo*-Dodecaborane-1,2-Dichalcogenolate ($\text{S}_2\text{C}_2\text{B}_{10}\text{H}_{10}$)²⁻ Units toward Alkynes¹

H. D. Ye*, J. R. Hu, H. N. Peng, J. L. Xie, Q. Ye, Y. H. Li, and Z. S. Xing

Key Laboratory of Applied Organic Chemistry, Higher Institutions of Jiangxi Province, School of Chemistry and Chemical Engineering, Shangrao Normal University, Jiangxi, 334001 P.R. China

*e-mail: yehongde@163.com

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Abstract—Treatment of *o*-carborane, *n*-butyllithium, sulfur, and MeCpCo(CO)I₂ under argon leads to two MeCp-cobalt complexes MeCpCoS₂C₂B₁₀H₁₀ (**I**) and (MeCp)₂Co₂S₂C₂B₁₀H₁₀ (**II**). Complex **I** reacts with phenylacetylene or dimethyl acetylene dicarboxylate to afford B–H activated complex (MeCpCoS₂C₂B₁₀H₉)(Ph–C≡CH) (**III**) and addition complex (MeCpCoS₂C₂B₁₀H₁₀)(MeO₂C–C≡C–CO₂Me) (**IV**), respectively. Complex **II** reacts neither with phenylacetylene nor with dimethyl acetylene dicarboxylate. Complexes **II**–**IV** have been characterized by elemental analysis, mass, NMR spectroscopy, IR, and X-ray structural analyses (CIF files CCDC nos. 1045892 (**II**), 1045893 (**III**) and 1045894 (**IV**)).

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INTRODUCTION

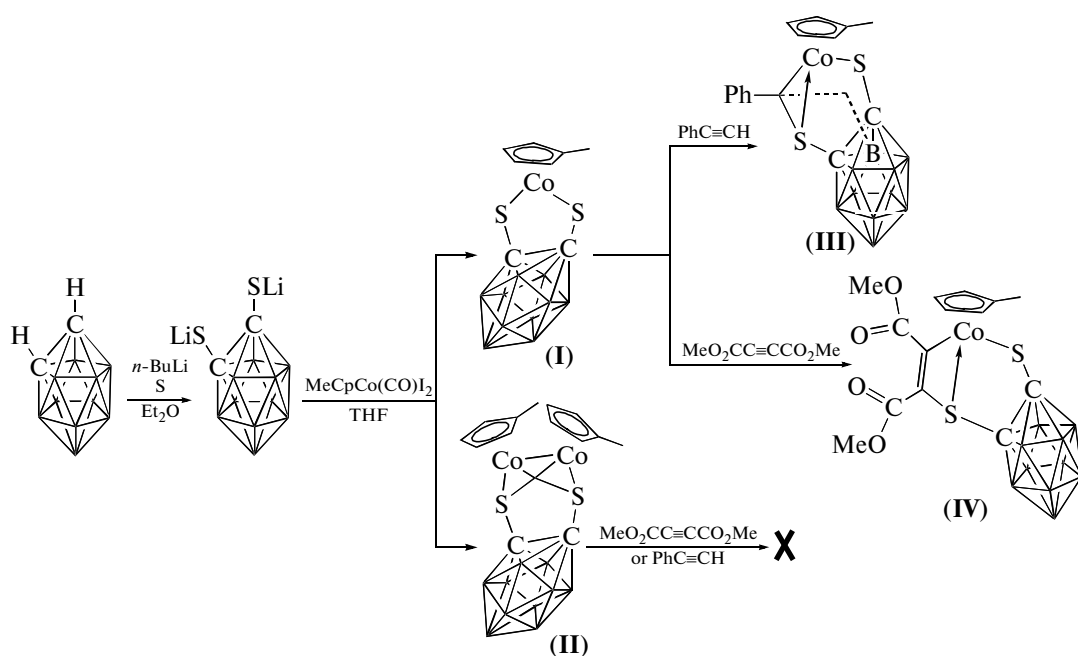
Boron cluster compounds are a kind of distinctive covalent species with a unique molecular architecture, nonconventional cluster bonding, and unusual chemistry. They show rather specific properties not encountered in other types of compounds [1]. As this field of chemistry has been developed rapidly for almost half a century, major advances in theory and experiment on the boranes and carboranes have been established, and they have shown many practical applications [2, 3]. Siloxane-linked polymers containing *m*-carborane icosahedral units show extraordinary chemical and thermal stability. Derivatives of carboranes have been used in the areas of boron neutron capture therapy (BNCT) for tumors [4]. Besides, carboranes and their derivatives have also been used to synthesize catalysts [5], radiopharmaceuticals [6], polymers [7], and coordination compounds [8]. Among the known polyhedral carboranes, 1,2-dicarba-*closo*-dodecaborane (*o*-carborane) has been intensively studied due to its relatively easy preparation [9].

As the CH groups of *o*-carborane are weakly acidic and can be deprotonated by strong base such as *n*-butyllithium [10], it can be used to prepare a kind of mononuclear 16e half-sandwich complexes of Co, Rh, Ir, Ru, and Os containing a chelating 1,2-dicarba-*closo*-dodecaborane-1,2-dichalcogenolate ligand [8–13]. These sterically congested, coordinatively unsaturated

compounds can be stored conveniently and used for the completion of various chemical transformations [11]. As the metal atoms of these half-sandwich complexes are coordinatively and electronically unsaturated, they can combine metal fragments to afford novel homometallic or heterometallic clusters containing metal-metal bonds [12]. They react with Lewis bases to give their corresponding 18-electron stable species [13]. Furthermore, the metal centers of these 16e compounds are electronically deficient and metal-chalcogen bonds are reactive, which render them interesting candidates for reactions with alkynes [14].

In the course of preparing the above mononuclear 16e compounds, some other binuclear [15–18], trinuclear [19], and multinuclear [20] carborane products have been successfully isolated. Furthermore, some species have shown further reactivity toward organic molecules [18, 21]. Recently, we have isolated a binuclear MeCp-cobalt carborane complex (**II**) in the course of preparing the mononuclear MeCp-cobalt carborane complex MeCpCoS₂C₂B₁₀H₁₀ (**I**). To the best of our knowledge, the studies on the two carborane complexes are relatively less explored. Herein, we report the syntheses of the above carborane compounds **I**, **II**, their further reactivity toward organic molecules and preparation of complexes **III** and **IV**. Synthesis of complexes **I**–**IV** is given in Scheme.

¹ The article is published in the original.



Scheme.

EXPERIMENTAL

Materials and methods. All experiments were carried out under an argon atmosphere using standard Schlenk techniques. Solvents were dried by refluxing over sodium (petroleum ether, ether, and THF) or calcium hydride (dichloromethane) under nitrogen and then distilled prior to use. *n*-Butyllithium (2.0 M in cyclohexane, Aldrich), *o*-carborane (Acros), phenylacetylene (Alfa Aesar), dimethyl acetylene dicarboxylate (Aldrich) and other chemicals were used as commercial products without further purification. $\text{MeCpCoS}_2\text{C}_2\text{B}_{10}\text{H}_{10}$ (I) was prepared according to literature [22]. Elemental analysis was performed in an elemental vario EL III elemental analyzer. NMR data were recorded on a Bruker DRX-500 spectrometer. ^1H NMR and ^{13}C NMR spectra were reported in ppm with respect to $\text{CHCl}_3/\text{CDCl}_3$ ($\delta^1\text{H} = 7.26$, $\delta^{13}\text{C} = 77.0$) and ^{11}B NMR spectra were reported in ppm with respect to external $\text{Et}_2\text{O}\cdot\text{BF}_3$ ($\delta^{11}\text{B} = 0$). The IR spectra were recorded on a Bruker Vector 22 spectrophotometer with KBr pellets in the $4000\text{--}400\text{ cm}^{-1}$ region. The mass spectra were recorded on Micromass GC-TOF for EI-MS (70 eV).

Synthesis of complexes I and II. To a solution of *o*-carborane (57.7 mg, 0.4 mmol) in dry diethylether (20 mL) was added a 2.0 M solution of *n*-butyllithium (0.4 mL, 0.8 mmol). After 30 min, sulfur (26.3 mg, 0.82 mmol) was added, followed by addition of MeCpCo(CO)I_2 (163.7 mg, 0.39 mmol) in dry THF (25 mL) at 0°C . The resulting mixture was stirred for

0.5 h, and then the temperature gradually rose to ambient temperature. The solvents were evaporated under reduced pressure. The components of the residue were separated by column chromatography on silica gel. Elution with dichloromethane–petroleum ether (1 : 1 v/v) gave pure complexes brown solid I (118.8 mg, 87% yield) and green solid II (3.8 mg, 2% yield). Crystals suitable for X-ray crystallography were obtained by slow diffusion of petroleum ether into a dichloromethane solution of the corresponding compound.

For $\text{C}_{14}\text{H}_{24}\text{B}_{10}\text{Co}_2\text{S}_2$ (II)

anal. calcd., %:	C, 34.85;	H, 5.01.
Found, %:	C, 34.49;	H, 4.78.

EI-MS (m/z): calcd. for $\text{C}_{14}\text{H}_{24}\text{B}_{10}\text{Co}_2\text{S}_2$, 482.45; found, 482.1 ($[\text{M}]^+$, 100%). ^1H NMR (δ , ppm): 4.98 (s., 2H, CH), 4.73 (s., 2H, CH), 1.90 (s., 3H, CH_3). ^{13}C : 95.04 (MeCp–C); 87.33 (carborane–C); 78.75 (MeCp–CH); 75.53 (MeCp–CH); 13.94 (CH_3). $^{11}\text{B}\{^1\text{H}\}$ NMR (δ , ppm): -1.47 , -5.23 , -8.89 (2 : 3 : 5). IR (ν , cm^{-1}): 2588 (B–H).

Synthesis of complex III. To a solution of I (103 mg, 0.3 mmol) in dry CH_2Cl_2 (20 mL) was added phenylacetylene (107 mg, 1.2 mmol). The resulting mixture was stirred for 10 h at ambient temperature. After removal of the solvent, the residue was chromatographed on silica gel. Elution with petroleum ether– CH_2Cl_2 (1 : 2 v/v) gave pure compound III (64 mg, 48%) as black solid. Suitable single crystal of

III was obtained by slow diffusion of petroleum ether into its CH_2Cl_2 solution.

For $\text{C}_{16}\text{H}_{23}\text{B}_{10}\text{S}_2\text{Co}$ (**III**)

anal. calcd., %: C, 43.04; H, 5.19.
 Found, %: C, 42.69; H, 5.01.

EI-MS (m/z): calcd. for $\text{C}_{16}\text{H}_{23}\text{B}_{10}\text{S}_2\text{Co}$, 446.53; found, 446.2 ($[\text{M}]^+$, 100%). ^1H NMR (δ , ppm): 1.95 (d., $J = 16$ Hz, 1H, B- CH_2), 2.30 (d., $J = 16$ Hz, 1H, B- CH_2), 3.69 (s., 1H, MeCp-CH), 4.25 (s., 1H, MeCp-CH), 4.33 (s., 1H, MeCp-CH), 4.46 (s., 1H, MeCp-CH), 7.29 (s., 5H, Ph). ^{13}C NMR (δ , ppm): 167.70 (Co-C); 152.95 (Ph-C); 130.82 (Ph-CH); 128.97 (Ph-CH); 128.74 (Ph-CH); 126.08 (Ph-CH); 124.58 (Ph-CH); 104.06 (MeCp-C); 97.93 (carborane-C); 94.43 (carborane-C); 88.71 (MeCp-CH); 85.67 (MeCp-CH); 84.12 (MeCp-CH); 82.50 (MeCp-CH); 34.25 (B- CH_2) (br); 12.93 (CH_3). $^{11}\text{B}\{^1\text{H}\}$ NMR (δ , ppm): -4.75, -5.66, -6.22, -7.36, -11.29 (2 : 3 : 3 : 1 : 1). IR (ν , cm^{-1}): 2581 (B-H).

Synthesis of complex IV. To a solution of **I** (103 mg, 0.3 mmol) in dry CH_2Cl_2 (20 mL) was added dimethyl acetylene dicarboxylate (171 mg, 1.2 mmol). The resulting mixture was stirred for 10 h at ambient temperature. After removal of the solvent, the residue was chromatographed on silica gel. Elution with petroleum ether- CH_2Cl_2 (1 : 4 v/v) gave pure compound **IV** (134 mg, 92%) as black solid. Suitable single crystal of **IV** was obtained by slow diffusion of petroleum ether into its CH_2Cl_2 solution.

For $\text{C}_{14}\text{H}_{23}\text{B}_{10}\text{O}_4\text{S}_2\text{Co}$ (**IV**)

anal. calcd., %: C, 34.56; H, 4.77.
 Found, %: C, 34.28; H, 4.59.

EI-MS (m/z): calcd. for $\text{C}_{14}\text{H}_{23}\text{B}_{10}\text{O}_4\text{S}_2\text{Co}$, 486.50; found, 486.1 ($[\text{M}]^+$, 2.52%). ^1H NMR (δ , ppm): 1.72 (s., 3H, MeCp- CH_3), 3.78 (s., 3H, OCH_3), 3.94 (s., 3H, OCH_3), 4.72 (s., 1H, MeCp-CH), 5.10 (s., 1H, MeCp-CH), 5.11 (s., 1H, MeCp-CH), 5.22 (s., 1H, MeCp-CH). ^{13}C NMR (δ , ppm): 180.30 (Co-C); 171.29 (CO); 155.31 (CO); 118.48 (S-C); 105.46 (MeCp-C); 98.45 (carborane-C); 89.80 (MeCp-CH); 85.87 (MeCp-CH); 83.21 (MeCp-CH); 82.69 (MeCp-CH); 78.95 (carborane-C); 52.62 (OCH_3); 52.58 (OCH_3); 12.98 (MeCp- CH_3). $^{11}\text{B}\{^1\text{H}\}$ NMR (δ , ppm): -0.73, -1.73, -4.25, -5.88, -8.09 (1 : 3 : 2 : 1 : 3). IR (ν , cm^{-1}): 2587 (B-H); 1717 (C=O).

X-ray crystallography. Suitable single crystals of **II**, **III**, and **IV** were selected and mounted in air onto thin glass fibers. X-ray diffraction data were collected on a Bruker SMART APEX II CCD diffractometer at 291(2) K using MoK_α radiation ($\lambda = 0.71073$ Å) by multi-scan mode. The SAINT program was used for integration of the diffraction profiles. The structures

were solved by direct methods using the SHELXS-97 [23] program package and refined against F^2 by full-matrix least-squares with SHELXL-97 [24]. All non-hydrogen atoms were refined with anisotropic thermal parameters. Hydrogen atoms on carbon were set in calculated positions and refined as riding. The crystallographic data are summarized in Table 1 and selected bond lengths and angles are listed in Table 2 containing the supplementary crystallographic data for this article. Copies of the data can be obtained free of charge from the Cambridge Crystallographic Data Centre (nos. 1045892 (**II**), 1045893 (**III**) and 1045894 (**IV**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

The reactivity of 16e half-sandwich carborane compounds $\text{CpCoS}_2\text{C}_2\text{B}_{10}\text{H}_{10}$ [25] and $\text{Cp}^*\text{CoS}_2\text{C}_2\text{B}_{10}\text{H}_{10}$ [26] have been investigated. In order to study the ancillary ligands of 16e half-sandwich carborane compounds on their reactivity toward alkynes, we set out to prepare **I** and investigate the reaction of **I** with phenylacetylene. As a result, a metal-induced B-H activation product **III** was isolated. In order to investigate the structures of alkynes on these types of reactions, we set out to conduct the reaction of **I** with dimethyl acetylene dicarboxylate. As expected, we have successfully isolated and characterized interesting stable 1 : 1 adduct compound **IV** in 93% yield. It is worth noting that in the reaction of synthesizing **I**, another MeCp-cobalt complex **II** containing two cobalt atoms has been isolated. In **II**, there are two cobalt atoms containing a Co-Co bond. Owing to the formation of the Co-Co bond, two cobalt atoms of **II** are electronically saturated. **II** shows higher stability than **I** and reacts neither with phenylacetylene nor with dimethyl acetylene dicarboxylate.

The solid-state structure of **II** (figure, a) shows that it is a symmetric dinuclear molecular structure. The analogous structures $(\text{CpCo})_2\text{S}_2\text{C}_2\text{B}_{10}\text{H}_{10}$ and $(\text{CpCo})_2\text{Se}_2\text{C}_2\text{B}_{10}\text{H}_{10}$ have been reported by Kang's [22] and Yan's [15] groups. However, in the rhodium and iridium series with a large pentamethylcyclopentadienyl (Cp^*) unit such a species has never been isolated [15]. The main reason may be that the large ancillary ligand Cp^* blocks the generation of dinuclear products $(\text{Cp}^*\text{M})_2\text{S}_2\text{C}_2\text{B}_{10}\text{H}_{10}$ ($\text{M} = \text{Rh}$ and Ir) as well as $(\text{Cp}^*\text{Co})_2\text{S}_2\text{C}_2\text{B}_{10}\text{H}_{10}$. The addition of a MeCpCo moiety to the S-Co-S unit in **I** leads to the formation of **II**. Each cobalt atom is coordinated by one MeCp ligand, single bonded to the other cobalt atom, and bridged by two μ_2 -S atoms from the $\text{S}_2\text{C}_2\text{B}_{10}\text{H}_{10}$ moiety. Two Co(II) centers take three-legged piano-stool arrangements with an 18-electron configuration. Two Co-S bond lengths are 2.1945(14) and 2.2093(13) Å, respectively. The Co-Co distance is 2.3786(9) Å and falls in the normal single Co-Co bond range [22]. The

Table 1. Crystallographic data and structural refinement details of complexes **II–IV**

Parameter	Value		
	II	III	IV
Crystal size, mm	0.26 × 0.24 × 0.22	0.28 × 0.24 × 0.22	0.28 × 0.24 × 0.22
Formula weight	482.41	446.49	486.47
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	$P2_1/c$	$P2_1/c$	$P\bar{1}$
a , Å	9.5788(13)	9.989(2)	12.0894(18)
b , Å	15.2301(17)	22.2572(15)	14.7176(11)
c , Å	14.9080(12)	9.6305(12)	15.3516(17)
α , deg	90	90	63.876(13)
β , deg	98.750(12)	100.952(3)	73.955(11)
γ , deg	90.00	90.00	82.889(12)
V , Å ³	2149.6(4)	2102.2(5)	2356.9(5)
Z	4	4	4
ρ_{calcd} , g cm ^{−3}	1.491	1.411	1.371
μ , mm ^{−1}	1.735	1.016	0.924
$F(000)$	976	912	992
θ Range, deg	1.92–26.00	2.08–26.00	2.33–28.00
Reflections collected	13 267	14 221	17 145
Independent reflections (R_{int})	4182 (0.0194)	4034 (0.0262)	7971 (0.0473)
Reflections observed ($I > 2\sigma(I)$)	3038	3436	6095
Data/restraints/parameters	4182/0/255	4034/0/254	7971/0/565
GOOF	1.052	1.085	1.032
R_1 , $wR_2(I > 2\sigma(I))$	0.0535/0.1489	0.0399/0.1396	0.0457/0.1382
R_1 , wR_2 (all data)	0.0712/0.1562	0.0483/0.1462	0.0557/0.1399
Largest difference peak/hole, $e \text{ \AA}^{-3}$	1.062/−1.164	0.423/−0.544	0.383/−0.316

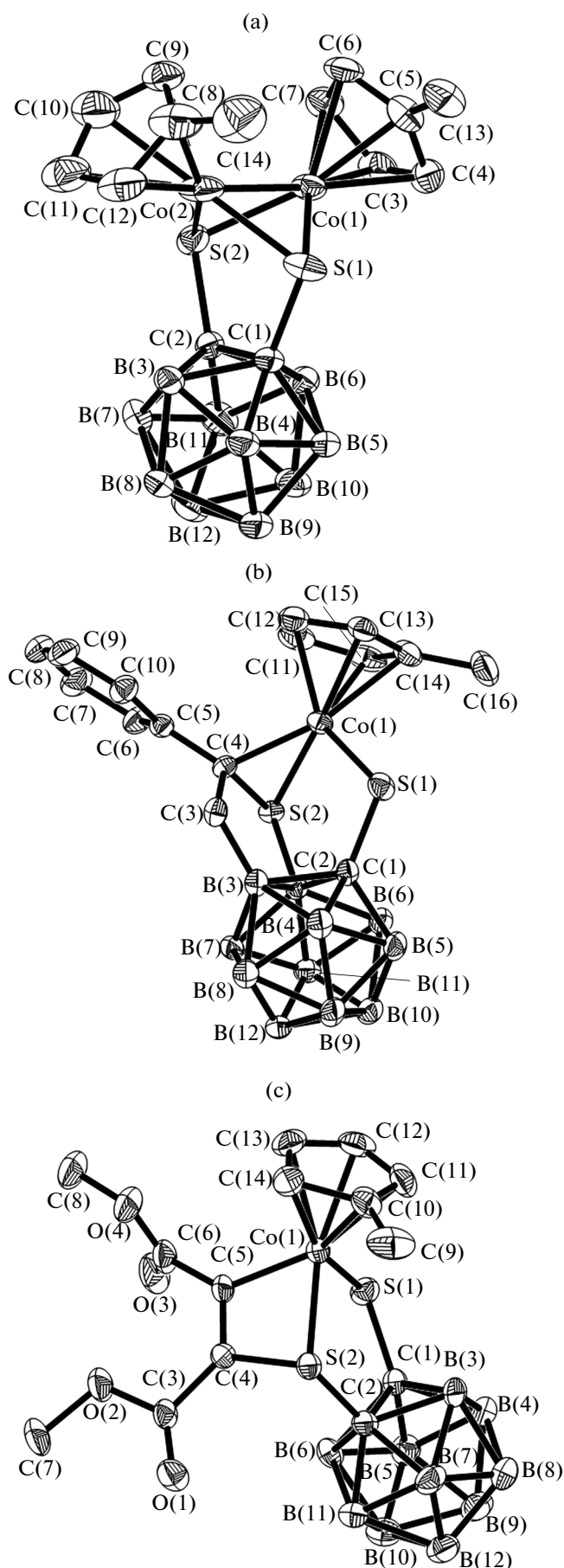
dihedral angle between the planes Co(1)Co(2)S(1) and Co(1)Co(2)S(2) is 76.5°. Compared with that in **I**, the two CoS₂C₂ rings in **II** are no longer planar. The dihedral angles of two CoS₂C₂ rings at the S(1)⋯S(2) vector are 133.3° (Co(1)–S(1)–C(1)–C(2)–S(2)) and 132.7° (Co(2)–S(1)–C(1)–C(2)–S(2)). Owing to the symmetrical structure, the chemical environments of the corresponding carbon atoms and hydrogen atoms of the two MeCp units and two carbon atoms in the carborane cage are identical. Thus, only one set of NMR signals are given. For example, the ¹H NMR signal of two CH₃ units is at 1.90 ppm while the ¹³C NMR signal of two carbon atoms in carborane cage is at 87.33 ppm.

The solid-state structure of **III** (figure, b) shows the presence of a B–CH₂ unit and a C–B bond. One hydrogen atom at B(3)/B(6) of the carborane cage has been transferred to the terminal carbon of the alkyne to generate the B–CH₂ unit. As a consequence, the C≡C bond of the alkyne is reduced to a C–C single

bond (1.537 Å). The generated five-membered ring S(2)C(2)B(3)C(3)C(4) is fused with the five-membered ring Co(1)S(1)C(1)C(2)S(2) at the S(2)–C(2) bond with a dihedral angle of 67.4°. The five-membered ring Co(1)S(1)C(1)C(2)S(2) is not planar, becoming slightly bent at the S(1)⋯S(2) vector with an angle of 171.4°. Note that the coordinative bond S(2) → Co(1) (2.1645 Å) is significantly shorter than the covalent bond S(1)–Co(1) (2.2213 Å). The shorter coordinative bond may help reduce the strain of three-membered ring Co(1)S(2)C(4). The NMR data are in agreement with the solid-state structure. The two doublets at 1.95 and 2.30 ppm with a large coupling constant value (16 Hz) are assigned to the newly generated alkyl group of the B–CH₂ unit. In the ¹³C NMR data, the low, broad signal at 34.25 ppm shows that the CH₂ group is linked to a boron atom. Complex **III** is a B–H activated product. Analogous structures have been reported [27] and mechanistic studies can be referred to the relevant literature [15].

Table 2. Selected bond lengths (Å) and bond angles (deg) for **II–IV**

Bond	<i>d</i> , Å	Angle	w, deg
II			
C(1)–C(2)	1.608(6)	S(1)Co(1)S(2)	84.96(5)
C(1)–S(1)	1.826(4)	S(1)Co(1)Co(2)	56.76(4)
C(2)–S(2)	1.822(4)	S(2)Co(1)Co(2)	58.09(4)
S(1)–Co(1)	2.2093(13)	S(1)Co(2)S(2)	84.76(5)
S(1)–Co(2)	2.1859(17)	S(2)Co(2)Co(1)	56.81(4)
S(2)–Co(1)	2.1945(14)	S(1)Co(2)Co(1)	57.71(4)
S(2)–Co(2)	2.2262(16)	C(1)S(1)Co(1)	102.38(14)
Co(1)–Co(2)	2.3786(9)	C(2)S(2)Co(2)	102.30(15)
III			
C(1)–C(2)	1.667(4)	S(1)Co(1)S(2)	95.72(3)
C(1)–S(1)	1.756(3)	S(1)Co(1)C(4)	97.36(9)
S(1)–Co(1)	2.2213(10)	S(2)Co(1)C(4)	51.14(8)
S(2)–Co(1)	2.1645(9)	C(1)S(1)Co(1)	103.91(10)
C(2)–S(2)	1.787(3)	C(2)S(2)Co(1)	106.12(10)
C(1)–B(3)	1.741(5)	C(2)S(2)C(4)	97.36(14)
C(2)–B(3)	1.732(4)	C(4)S(2)Co(1)	60.02(10)
C(3)–B(3)	1.547(5)	B(3)C(3)C(4)	110.9(2)
C(3)–C(4)	1.537(4)	C(3)C(4)Co(1)	117.9(2)
C(4)–S(2)	1.807(3)	C(3)C(4)S(2)	113.8(2)
C(4)–Co(1)	2.010(3)	S(2)C(4)Co(1)	68.84(10)
IV			
C(1)–C(2)	1.688(6)	S(1)Co(1)S(2)	93.46(4)
C(1)–S(1)	1.792(4)	S(1)Co(1)C(5)	91.84(13)
C(2)–S(2)	1.830(4)	S(2)Co(1)C(5)	71.73(12)
S(1)–Co(1)	2.2701(13)	C(1)S(1)Co(1)	104.12(13)
S(2)–Co(1)	2.2992(11)	C(2)S(2)Co(1)	105.09(13)
S(2)–C(4)	1.792(3)	C(4)S(2)Co(1)	77.83(13)
C(4)–C(5)	1.320(5)	S(2)C(4)C(5)	105.7(3)
C(5)–Co(1)	1.933(4)	C(4)C(5)Co(1)	104.6(2)

Molecular structures of **II** (a), **III** (b) and **IV** (c).

Complex **I** reacts with dimethyl acetylene dicarboxylate at ambient temperature to give the sole product **IV**. The solid-state structure (figure, c) shows that **IV** is a 1 : 1 adduct with a newly generated four-membered ring Co(1)S(2)C(4)C(5). However, the four-membered ring is rather stable, since there is no other product and no further reaction takes place even in the presence of excess alkyne. This may be that two methyl carboxylate groups of the alkyne block other kinds of conversions and reactions. Actually, in the most cases, this alkyne reacts with other mononuclear 16e half-sandwich complexes to give the sole products analogous to **IV** [26]. The generated four-membered ring Co(1)S(2)C(4)C(5) is fused with the five-membered ring Co(1)S(1)C(1)C(2)S(2) at the Co(1)–S(2) bond with a dihedral angle of 97.5°. Owing to the insert of the alkyne, the five-membered ring Co(1)S(1)C(1)C(2)S(2) is no longer planar, becoming slightly bent at the S(1)···S(2) vector with an angle of 162.4°. The coordinative bond S(2) → Co(1) (2.2992 Å) is slightly longer than that of the covalent bond S(1)–Co(1) (2.2701 Å).

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