ISSN 1070-3284, Russian Journal of Coordination Chemistry, 2016, Vol. 42, No. 12, pp. 783–788. © Pleiades Publishing, Ltd., 2016..

Reactivity of Dinuclear Ruthenium Complex Containing Two 1,2-Dicarba-*closo*-Dodecaborane-1,2-Dithiolate Ligands toward $HC{\equiv}CCH(OH)(CH_3)_2$[1]

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Received December 24, 2015

Abstract—The reaction of (p-cymene)$Ru_2(\mu\text{-}S_2)(S_2C_2B_{10}H_{10})_2$ (**I**) with $HC{\equiv}CCH(OH)(CH_3)_2$ in dichloromethane led to addition complexes, (p-cymene)$Ru_2(\mu\text{-}S_2)(S_2C_2B_{10}H_{10})_2(R_1C{=}CR_2)$ (R_1 = H, R_2 = $C(OH)(CH_3)_2$ (**II**); R_1 = $C(OH)(CH_3)_2$, R_2 = H (**III**)). In boiling chloroform both complexes **II** and **III** spontaneously lose water to generate two geometrical isomers (p-cymene)$Ru_2(\mu\text{-}S_2)(S_2C_2B_{10}H_{10})_2(R_1C{=}CR_2)$ (R_1 = H, R_2 = $C(CH_3){=}CH_2$ (**IV**); R_1 = $C(CH_3){=}CH_2$, R_2 = H (**V**)), respectively. Complexes **IV** and **V** could be interconverted in boiling toluene. All these complexes were characterized by elemental analysis, mass spectrometry, and NMR spectroscopy. The molecular structure of complex **IV** has been determined by X-ray crystallography (CIF file CCDC no. 1443964). Complex **IV** crystallizes in monoclinic system, space group $P2_1/c$ with a = 10.3717(9), b = 20.3982(17), c = 18.6428(13) Å, β = 111.096(4)°, $C_{19}H_{40}B_{20}Ru_2S_6$, M_r = 879.27, V = 3679.8(5) Å^3, ρ_c = 1.587 g/cm^3, Z = 4, $F(000)$ = 1752, $\mu(\text{Mo}K_\alpha)$ = 1.179 mm^{-1}, R = 0.0416 and wR = 0.0848 for 4602 observed reflections ($I > 2\sigma(I)$).

Keywords: ortho-carborane, $HC{\equiv}CCH(OH)(CH_3)_2$, ruthenium complexes, synthesis

DOI: 10.1134/S1070328416120034

INTRODUCTION

The chemistry of dicarba-*closo*-dodecaboranes is a fascinating research area owing to their unique properties and a variety of potential applications in material synthesis [1], microelectronics [2], optics and medicines [3, 4]. During the past decade, to take advantage of their unique molecular structures, the synthesis and reactivity of mononuclear 16e half-sandwich Co, Rh, Ir, Ru complexes containing an o-carborane-1,2-dichalcogenolate ligand [5–7] and unsaturated dinuclear ruthenium complexes (p-cymene)$Ru(\mu\text{-}E_2)Ru(S_2C_2B_{10}H_{10})_2$ (E = S, Se) [8], have been investigated. However, considerable attention has been devoted to the reactivity of organometallic complexes containing o-carborane-1,2-dichalcogenolate ligands with monodentate Lewis bases [9] or alkynes [10, 11], such as pyridine, methyl acetylene carboxylate, phenylacetylene, ferrocenylacetylene et al.

Recently, we are interested in using alkyne alcohols to prepare half-sandwich saturated organometallic complexes and in studying the impact of hydroxyl group in these type of reactions [12]. The reaction system of (p-cymene)$Ru(\mu\text{-}E_2)Ru(S_2C_2B_{10}H_{10})_2$ (E = S, Se) and ($cyclo$-C_6H_{10})(OH)C≡CH has been discussed, and some interesting stable products have been isolated and characterized [13]. In our further efforts to extend the scope of the chemistry, we now have investigated the reaction of (p-cymene)$Ru_2(\mu\text{-}S_2)(S_2C_2B_{10}H_{10})_2$ (**I**) with commercially available $HC{\equiv}CCH(OH)(CH_3)_2$ leading to addition complexes, (p-cymene)$Ru_2(\mu\text{-}S_2)(S_2C_2B_{10}H_{10})_2(R_1C{=}CR_2)$ (R_1 = H, R_2 = $C(OH)(CH_3)_2$ (**II**); R_1 = $C(OH)(CH_3)_2$, R_2 = H (**III**)). In boiling chloroform complexes **II** and **III** generate two geometrical isomers (p-cymene)$Ru_2(\mu\text{-}S_2)(S_2C_2B_{10}H_{10})_2(R_1C{=}CR_2)$ (R_1 = H, R_2 = $C(CH_3){=}CH_2$ (**IV**); R_1 = $C(CH_3){=}CH_2$, R_2 = H (**V**)), respectively (scheme):

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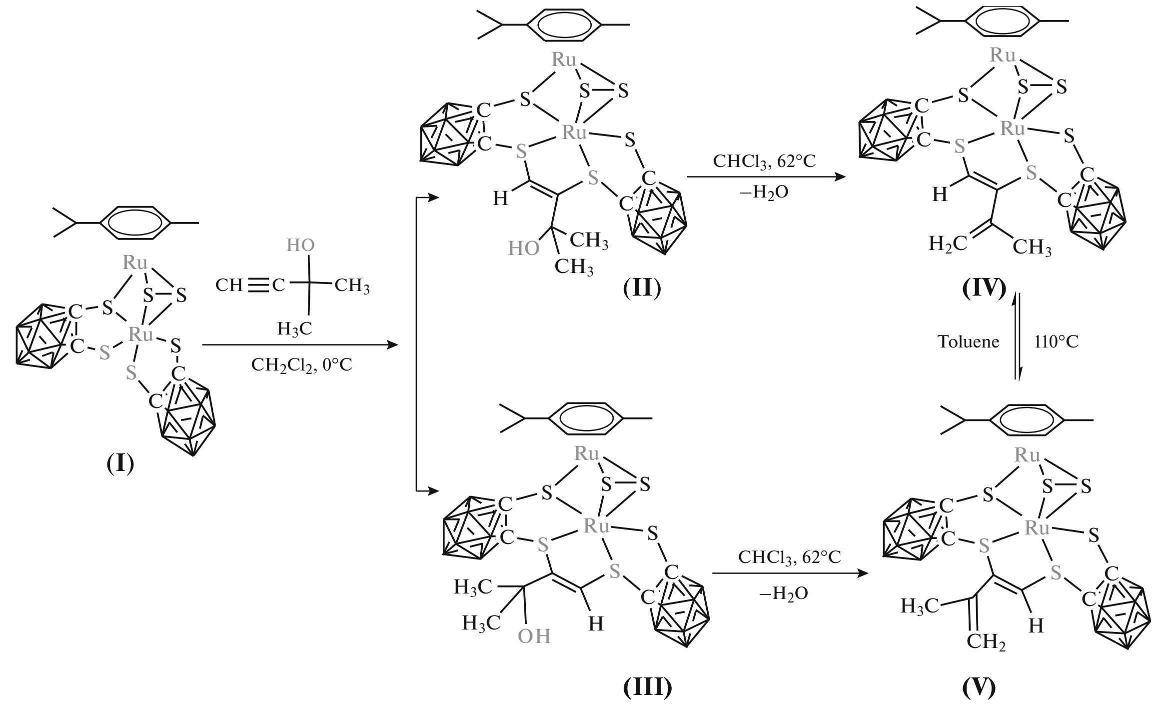


EXPERIMENTAL

Materials and methods. The preparative work was carried out under an argon atmosphere using standard Schlenk techniques except mentioned. Solvents were freshly distilled under nitrogen from either sodium or calcium hydride prior to use. *n*-Butyllithium (2.0 M in cyclohexane, Aldrich), HC≡CCH(OH)$(CH_3)_2$ (Aldrich), other chemicals were used as commercial products without further purification. [(*p*-Cymene)$RuCl_2]_2$ was prepared according to literature [14]. Elemental analysis was performed in an elementar vario EL III elemental analyzer. NMR data were obtained on a Bruker DRX-500 spectrometer. Chemical shifts were given with respect to $CHCl_3/CDCl_3$ (δ ^{1}H = 7.24 ppm) and external Et_2O-BF_3 (δ ^{11}B = 0 ppm). The IR spectra were recorded on a Bruker Vector 22 spectrophotometer with KBr pellets in the 4000–400 cm^{-1} region. Matrix-assisted laser desorption/ionization (MALDI) in a linear time-of-flight mass spectrometry (MS) was recorded in a Bruker autoflex TOF/TOF equipped with an acquisition operation mode of reflector and signal averaging of 30 laser shots.

Synthesis of II and III. HC≡CCH(OH)$(CH_3)_2$ (16.8 mg, 0.2 mmol) was added to **I** (81 mg, 0.1 mmol) in CH_2Cl_2 (15 mL). The mixture was stirred for 3 h at 0°C. After removal of the solvent the residue was chromatographed on silica to give yellow **II** and **III** (petroleum ether–CH_2Cl_2 (1 : 4)).

II: yield 34 mg (38%); mp (dec.) 219°C.

For $C_{19}H_{42}B_{20}OS_6Ru_2$

anal. calcd., %:	C, 25.41;	H, 4.68.
Found, %:	C, 25.69;	H, 4.73.

MALDI-TOF MS (*m/z*): calcd. for $C_{19}H_{42}B_{20}OS_6Ru_2$, 897.27; found, 898.31 ($[M + H]^+$, 100%). ^{1}H NMR ($CDCl_3$; δ, ppm): 0.87, 1.25 (d., *J* = 7.0 Hz, 3H, $CH(C\mathit{H}_3)_2$), 1.53 (s., $C(C\mathit{H}_3)_2$), 2.15 (s., 3H, CH_3), 2.73 (sept., 1H, $C\mathit{H}(CH_3)_2$), 3.76 (s, 1H, OH), 4.98, 5.03, 6.06, 6.09 (d., *J* = 6.0 Hz, 1H, C_6H_4), 7.12 (s., 1H, HC=C). ^{13}C NMR ($CDCl_3$; δ, ppm): 18.66 ($C_6H_4-\boldsymbol{C}H_3$), 22.53, 22.87 ($CH(\boldsymbol{C}H_3)_2$), 26.43, 26.51 ($C(\boldsymbol{C}H_3)_2$), 31.45 ($\boldsymbol{C}H(CH_3)_2$), 76.01 ($\boldsymbol{C}OH$), 80.35, 81.51, 81.93, 85.78 (***C***H in *p*-cymene), 90.86, 94.97, 96.68, 99.15, 99.95, 106.88 (*o*-carborane and quaternary C in *p*-cymene), 136.87 (***C***=CH), 159.66 (H***C***=C). ^{11}B{^{1}H} NMR ($CDCl_3$; δ, ppm): −0.69, −1.12, −2.01, −5.08, −7.38 (2 : 1 : 3 : 2 : 2). IR (KBr; ν, cm^{-1}): 2584 ν(B–H).

III: yield 35 mg (39%); mp (dec.) 220°C.

For $C_{19}H_{42}B_{20}OS_6Ru_2$

anal. calcd., %:	C, 25.41;	H, 4.68.
Found, %:	C, 25.68;	H, 4.75.

MALDI-TOF MS (*m/z*): calcd. for $\mathrm{C_{19}H_{42}B_{20}ORu_2S_6}$, 897.27; found, 898.31 ($[\mathrm{M + H}]^+$, 100%). ^{1}H NMR ($\mathrm{CDCl_3}$; δ, ppm): 0.88, 1.26 (d., $J = 7.0$ Hz, 3H, $\mathrm{CH(C}\mathit{H}_3)_2$), 1.56 (s., $\mathrm{C(C}\mathit{H}_3)_2$), 2.19 (s., 3H, $\mathrm{CH_3}$), 2.78 (sept., 1H, $\mathrm{C}\mathit{H}\mathrm{(CH_3)_2}$), 3.78 (s., 1H, OH), 5.01, 5.07, 6.09, 6.11 (d., $J = 6.0$ Hz, 1H, $\mathrm{C_6H_4}$), 7.15 (s., 1H, HC=C). ^{13}C NMR ($\mathrm{CDCl_3}$; δ, ppm): 18.96 ($\mathrm{C_6H_4{-}}\boldsymbol{C}\mathrm{H_3}$), 22.63, 22.89 ($\mathrm{CH(}\boldsymbol{C}\mathrm{H_3)_2}$), 26.48, 26.59 ($\mathrm{C(}\boldsymbol{C}\mathrm{H_3)_2}$), 31.46 ($\boldsymbol{C}\mathrm{H(CH_3)_2}$), 76.08 ($\boldsymbol{C}$OH), 80.45, 81.58, 82.03, 85.98 ($\boldsymbol{C}$H in *p*-cymene), 90.96, 95.17, 96.88, 99.35, 100.01, 106.98 (*o*-carborane and quaternary C in *p*-cymene), 136.99 ($\boldsymbol{C}$=CH), 159.98 (H$\boldsymbol{C}$=C). $^{11}\mathrm{B\{^1H\}}$ NMR ($\mathrm{CDCl_3}$; δ, ppm): −0.71, −1.16, −2.11, −5.11, −7.68 (2 : 1 : 3 : 2 : 2). IR (KBr; ν, cm^{-1}): 2585 ν(B−H).

Synthesis of IV and V. The solution of **II** (89.7 mg, 0.1 mmol) or **III** (89.7 mg, 0.1 mmol) in chloroform (10 mL) was refluxed for 3 h to give a dark yellow solution. After removal of the solvent the residue was chromatographed on silica to give **IV** or **V** (petroleum ether/$\mathrm{CH_2Cl_2}$ (2 : 1)). Recrystallization from $\mathrm{CH_2Cl_2}$ afforded air-stable yellow crystals of **IV**.

IV: yield 69.5 mg (79%); mp (dec.) 223°C.

For $\mathrm{C_{19}H_{40}B_{20}S_6Ru_2}$

anal. calcd., %:	C, 25.93;	H, 4.55.
Found, %:	C, 26.12;	H, 4.63.

MALDI-TOF MS (*m/z*): calcd for $\mathrm{C_{19}H_{40}B_{20}S_6Ru_2}$, 879.25; found, 880.31 ($[\mathrm{M + H}]^+$, 100%). ^{1}H NMR ($\mathrm{CDCl_3}$; δ, ppm): 0.89, 1.23 (d., $J = 7.0$ Hz, 3H, $\mathrm{CH(C}\mathit{H}_3)_2$), 2.17 (s., 3H, $\mathrm{C{-}C}\mathit{H}_3$), 2.26 (s., 3H, $\mathrm{C_6H_4{-}C}\mathit{H}_3$), 2.71 (sept., 1H, $\mathrm{C}\mathit{H}\mathrm{(CH_3)_2}$), 4.93 (s, 2H, $\mathrm{C{=}C}\mathit{H}_2$), 5.03, 5.11, 6.13, 6.26 (d, $J = 6.0$ Hz, 1H, $\mathrm{C_6H_4}$), 6.87 (s., 1H, *H*C=C). ^{13}C NMR ($\mathrm{CDCl_3}$; δ, ppm): 19.40 ($\mathrm{C_6H_4{-}}\boldsymbol{C}\mathrm{H_3}$), 22.52, 22.82 ($\mathrm{CH(}\boldsymbol{C}\mathrm{H_3)_2}$), 27.07 ($\mathrm{C{-}}\boldsymbol{C}\mathrm{H_3}$), 31.91 ($\boldsymbol{C}\mathrm{H(CH_3)_2}$), 80.79, 82.08, 84.81, 85.48 ($\boldsymbol{C}$H in *p*-cymene), 90.03, 91.07, 93.51, 99.85, 100.61, 106.90 (*o*-carborane and quaternary C in *p*-cymene), 126.10 ($\boldsymbol{C}\mathrm{H_2{=}C}$), 134.74 ($\boldsymbol{C}\mathrm{{=}CH_2}$), 136.45 ($\boldsymbol{C}$=CH), 152.81 (H$\boldsymbol{C}$=C). $^{11}\mathrm{B\{^1H\}}$ NMR ($\mathrm{CDCl_3}$; δ, ppm): −0.67, −1.12, −1.93, −5.14, −7.18 (2 : 1 : 3 : 2 : 2). IR (KBr; ν, cm^{-1}): 2580 ν(B−H).

V: yield, 68.6 mg (78%); mp (dec.) 224°C.

For $\mathrm{C_{19}H_{40}B_{20}S_6Ru_2}$

anal. calcd., %:	C, 25.93;	H, 4.55.
Found, %:	C, 26.14;	H, 4.65.

MALDI-TOF MS (*m/z*): calcd. for $\mathrm{C_{19}H_{40}B_{20}S_6Ru_2}$, 879.25; found, 880.31 ($[\mathrm{M + H}]^+$, 100%). ^{1}H NMR ($\mathrm{CDCl_3}$; δ, ppm): 0.88, 1.22 (d., $J = 7.0$ Hz, 3H, $\mathrm{CH(C}\mathit{H}_3)_2$), 2.16 (s., 3H, $\mathrm{C{-}C}\mathit{H}_3$), 2.25 (s., 3H, $\mathrm{C_6H_4{-}C}\mathit{H}_3$), 2.70 (sept., 1H, $\mathrm{C}\mathit{H}\mathrm{(CH_3)_2}$), 4.91 (s., 2H, $\mathrm{C{=}C}\mathit{H}_2$), 5.02, 5.11, 6.14, 6.27 (d., $J = 6.0$ Hz, 1H, $\mathrm{C_6H_4}$), 6.86 (s., 1H, HC=C). ^{13}C NMR ($\mathrm{CDCl_3}$; δ, ppm): 19.38 ($\mathrm{C_6H_4{-}}\boldsymbol{C}\mathrm{H_3}$), 22.32, 22.79 ($\mathrm{CH(}\boldsymbol{C}\mathrm{H_3)_2}$), 27.10 ($\mathrm{C{-}}\boldsymbol{C}\mathrm{H_3}$), 31.91 ($\boldsymbol{C}\mathrm{H(CH_3)_2}$), 80.81, 82.09, 84.79, 85.37 ($\boldsymbol{C}$H in *p*-cymene), 90.13, 91.12, 93.48, 99.97, 100.89, 106.96 (*o*-carborane and quaternary C in *p*-cymene), 126.81 ($\boldsymbol{C}\mathrm{H_2{=}C}$), 134.97 ($\boldsymbol{C}\mathrm{{=}CH_2}$), 136.52 ($\boldsymbol{C}$=CH), 152.93 (H$\boldsymbol{C}$=C). $^{11}\mathrm{B\{^1H\}}$ NMR ($\mathrm{CDCl_3}$; δ, ppm): −0.69, −1.21, −1.73, −5.64, −7.38 (2 : 1 : 3 : 2 : 2). IR (KBr; ν, cm^{-1}): 2579 ν(B−H).

X-ray crystallography. Diffraction data for **IV** were collected on a Bruker SMART Apex II CCD diffractometer using graphite-monochromated MoK_α ($\lambda = 0.71073$ Å) radiation. The SAINT program was used for integration of the diffraction profiles. The structures were solved by direct methods using the SHELXS-97 [15] program package and refined against F^2 by full-matrix least-squares with SHELXL-97 [16]. 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 the selected bond lengths and bond angles are listed in Table 2.

Supplementary material for **IV** has been deposited with the Cambridge Crystallographic Data Centre (CCDC no. 1443964; deposit@ccdc.cam.ac.uk or http:// www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

Studying the reaction of **I** with $\mathrm{HC{\equiv}CCH(OH)(CH_3)_2}$, we found that **I** could undergo alkyne addition to generate two geometrical isomers **II** and **III** in a ratio of approximately 1 : 1, which are converted to the corresponding complexes **IV** and **V** by loss of a water molecule in chloroform at 62°C. In boiling toluene complexes **IV** and **V** could be interconverted, which are well known in the analogous reaction chemistries [17].

The molecular structures of **II** and **III** have not been characterized by X-ray diffraction owing to lack of good quality single crystals. However, in reference to the reaction of (*p*-cymene)Ru(μ-$\mathrm{S_2}$)Ru($\mathrm{S_2C_2B_{10}H_{10}}$)$_2$ with 1-ethynyl-1-cyclohexanol previously reported [18], the two isomers **II** and **III** could be suggested as shown in the Scheme by spectroscopic data. Indeed, in the ^{1}H NMR spectra signals assigned to olefinic proton were observed at 7.12 ppm

Table 1. Crystallographic data and structural refinement details of complex **IV**

Parameter	Value
Crystal size, mm	0.12 × 0.10 × 0.10
Formula weight	879.27
Temperature, K	296(2)
Crystal system	Monoclinic
Space group	$P2_1/c$
a, Å	10.3717(9)
b, Å	20.3982(17)
c, Å	18.6428(13)
β, deg	111.096(4)
V, Å^3	3679.8(5)
Z	4
ρ_{calcd}, g cm^{-3}	1.587
μ, mm^{-1}	1.179
F(000)	1752
θ Range, deg	2.00–25.00
Reflections collected	17451
Independent reflections (R_{int})	6477 (0.0594)
Reflections with $I > 2\sigma(I)$	4602
Data/restraints/parameters	6477/88/449
GOOF	1.086
R_1, wR_2 ($I > 2\sigma(I)$)	0.0416, 0.0848
R_1, wR_2 (all data)	0.0627, 0.0895
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.904/–0.540

(HC=C) in **II**, and 7.15 ppm (HC=C) in **III**. The OH groups appear at 3.76 ppm in **II** and 3.78 ppm in **III**. The ^{13}C NMR spectrum showed additional signals for olefinic carbons at 136.87 (***C***=CH), 159.66 ppm (H***C***=C) in **II**, and 136.99 (***C***=CH), 159.98 ppm (H***C***=C) in **III**. The MALDI-TOF MS show the same molecular ion peaks $[M + H]^+$ for both **II** and **III**.

The structure of complex **IV** has been confirmed by X-ray diffraction, as depicted in figure. The alkyne addition selectively occurs at S(1) and S(3) sites from two individual $(S_2C_2B_{10}H_{10})^{2-}$ ligands to generate a perfectly planar RuSCCS unit. The maximum deviation from the least-squares plane of the whole five-membered RuSCCS ring is 0.0522 Å. As expected, the C(5)–C(6), C(6)–C(7) and C(7)–C(8) distances are 1.317(4), 1.522(4) and 1.387(5) Å, respectively, similar values described for double and single bonds [19], which clearly indicate the dissociation of OH$^-$ from **II** is occurred and a weak delocalization of the π-electron density along the C(5)–C(6)–C(7)–C(8) chain. The Ru–S bond lengths, varying from 2.2666(1) to 2.4053(9) Å, are comparable to those in (p-cymene)Ru(μ-S_2)Ru$(S_2C_2B_{10}H_{10})_2$(HC=CCO_2Me) (2.2519(16) to 2.4211(15) Å) [20]. Noticeably, the bond lengths of Ru(1)–S(1) (2.2666(1) Å) and Ru(1)–S(3) (2.2684(8) Å) are a little longer than those (2.1997(2) and 2.1919(1) Å) in complex **I**, indicating mixed-valence Ru(II)/Ru(IV) (18e/16e) in **I** are changed to the two Ru(II)/Ru(II) (18e/18e) centers in **IV** [20]. The NMR spectroscopic data of **IV** are consistent with the solid-state structure. In particular, the ^{1}H NMR spectrum has signals for the olefinic proton at 4.93 (C=CH_2), 6.87 ppm (HC=C), and the OH signals disappeared. The ^{13}C NMR spectrum shows the characteristic signals at 126.10 (***C***H_2=C), 134.74 (***C***=CH_2), 136.45 (***C***=CH), 152.81 ppm (H***C***=C).

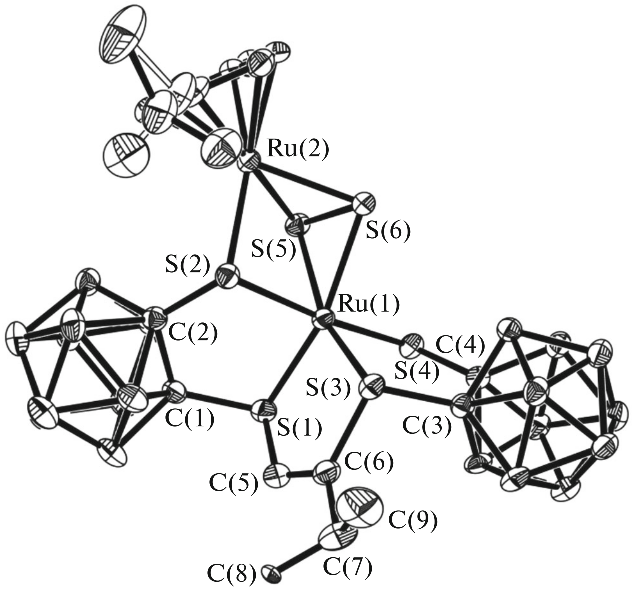
The solid-state structure of **V** can be readily assigned on the basis of its analytical and spectroscopic data. In comparison with **IV**, complex **V** has a similar structure. In **V** the terminal carbon atom of the alkyne is bonded to S(3) rather than S(1). In the ^{1}H NMR spectrum, the olefinic proton could be observed at 4.91 (C=CH_2), 6.86 ppm (HC=C). The ^{13}C NMR spectrum showed four signals of the carbons on the olefinic at 126.81 (***C***H_2=C), 134.97 (***C***=CH_2), 136.52 (***C***=CH), 152.93 ppm (H***C***=C).

ACKNOWLEDGMENTS

This work was supported by the Natural Science Foundation of Jiangxi Province (no. 20151BAB203005) and the National Natural Science Foundation of China (nos. 21261020 and 21361022).

Table 2. Selected bond lengths (Å) and bond angles (deg) of complex **IV**

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
Ru(1)–S(1)	2.2666(1)	Ru(2)–S(5)	2.3744(1)	C(1)–S(1)	1.822(3)
Ru(1)–S(2)	2.3735(1)	Ru(2)–S(6)	2.3827(9)	C(2)–S(2)	1.816(3)
Ru(1)–S(3)	2.2684(8)	C(1)–C(2)	1.698(5)	C(3)–S(3)	1.830(3)
Ru(1)–S(4)	2.3805(1)	C(3)–C(4)	1.697(4)	C(4)–S(4)	1.762(3)
Ru(1)–S(5)	2.3964(9)	C(5)–C(6)	1.317(4)	C(5)–S(1)	1.766(3)
Ru(1)–S(6)	2.3922(1)	C(6)–C(7)	1.522(4)	C(6)–S(3)	1.821(4)
Ru(2)–S(2)	2.4053(9)	C(7)–C(8)	1.387(5)	S(5)–S(6)	2.0475(1)
Angle	ω, deg	Angle	ω, deg	Angle	ω, deg
S(1)Ru(1)S(3)	86.85(3)	S(3)Ru(1)S(4)	90.87(3)	C(6)C(7)C(8)	106.7(3)
S(1)Ru(1)S(4)	92.44(4)	C(5)C(6)C(7)	120.5(3)	S(2)Ru(2)S(6)	82.68(3)
S(1)Ru(1)S(2)	91.30(3)	C(5)C(6)S(3)	116.8(3)	S(5)Ru(2)S(6)	50.98(3)



Molecular structure of complex **IV**.

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