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芳基取代茚基钌羰基化合物的合成及晶体结构

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摘要:配体 C_9H_7R(R=Ph (**1**),4-tolyl (**2**),4-chlorophenyl (**3**),4-methoxyphenyl (**4**),2-thienyl (**5**))分别与 $Ru_3(CO)_{12}$ 在甲苯或二甲苯中加热回流,得到了 5 个双核配合物[$(\eta^5$-$C_9H_6R)Ru(CO)]_2(\mu$-$CO)_2$(R=Ph (**6**),4-tolyl (**7**),4-chlorophenyl (**8**),4-methoxyphenyl (**9**),2-thienyl (**10**))。通过元素分析、红外光谱、核磁共振氢谱对配合物的结构进行了表征,并用 X-射线单晶衍射法测定了配合物 **6**,**7** 和 **10** 的结构。

关键词:茚基;羰基钌;X-射线衍射;结构

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Ruthenium Carbonyl Complexes Involving Aryl-Substituted Indenyl Ligands: Syntheses and Structures

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Abstract: Reactions of aryl-substituted indenyl ligands (R=Ph (**1**), 4-tolyl (**2**), 4-chlorophenyl (**3**), 4-methoxyphenyl (**4**), 2-thienyl (**5**)) with $Ru_3(CO)_{12}$ in refluxing toluene or xylene gave the responding dinuclear metal carbonyl complexes [$(\eta^5$-$C_9H_6R)Ru(CO)]_2(\mu$-$CO)_2$ (R=Ph (**6**), 4-tolyl (**7**), 4-chlorophenyl (**8**), 4-methoxyphenyl (**9**), 2-thienyl (**10**)), respectively. These complexes have been characterized by elemental analysis, IR, and ^{1}H NMR spectroscopy. The molecular structures of **6**, **7** and **10** were determined by X-ray diffraction analysis. CCDC: 943948, **6**; 946643, **7**; 966285, **10**.

Key words: indenyl; ruthenium carbonyl; X-ray diffraction; structure

0 Introduction

Transition metal complexes containing the indenyl ligand have received much attention due to their various metal-indenyl bonding modes, enhanced reactivity and catalytic ability as compared to the cyclopentadienyl analogues[1-10]. It was found that the steric and electronic effects of indenyl ring substituents greatly influence catalytic activity and stability of the substituted indenyl metal complexes[5]. Thus the variation of indenyl ligands is the first strategy for efficient catalysts. In our previous work we studied the

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reactions of substituted indenes with $Ru_3(CO)_{12}$ and obtained a series of dinuclear and trinuclear ruthenium carbonyl complexes involving novel intramolecular C-H activation[11-13]. Because of the special electronic and steric effect of the phenyl group[14-18], as a part of an effort to explore the relationship between the substituent with the structure (such as metal-metal bond length) and the reactivity in the bis(indenyl) dinuclear metal complexes, here we report the synthesis and structures of a series of aryl-substituted bis(indenyl) diruthenium complexes.

1 Experimental

1.1 General considerations

All manipulations of air- and moisture-sensitive complexes were performed at an argon/vacuum manifold using standard Schlenk techniques. Solvents were distilled from appropriate drying agents under an atmosphere of nitrogen prior to use. ^{1}H NMR spectra were recorded on a Bruker AV 500 instrument, while IR spectra were recorded as KBr disks on a FT IR 8900 spectrometer. X-ray measurements were made on a Bruker Smart APEX diffractometer with graphite monochromated Mo $K\alpha$ (λ=0.071 073 nm) radiation. Elemental analyses were performed on a Vario EL Ⅲ analyzer. The ligand precursors (R=Ph (**1**), 4-tolyl (**2**), 4-chlorophenyl (**3**), 4-methoxyphenyl (**4**), 2-thienyl (**5**)) were synthesized according to the literature[19]. $Ru_3(CO)_{12}$ were purchased from J&K Scientific Ltd and used without further purification, other reagents were purchased from commercial suppliers.

1.2 Reaction of 1 with $Ru_3(CO)_{12}$ in toluene

A solution of ligand precursor **1** (0.27 g, 1.41 mmol) and $Ru_3(CO)_{12}$ (0.03 g, 0.47 mmol) in 25 mL of toluene was refluxed for 14 h. After chromatography and eluted with petroleum ether/CH_2Cl_2 (2∶1, *V*/*V*), complex **6** was obtained (0.236 g, 48.1% yield) as orange-red solid. m.p. 198 ℃ (dec.). Anal. Calcd.(%) for $C_{34}H_{22}Ru_2O_4$: C, 58.62; H, 3.18. Found (%): C, 58.35; H, 3.51. ^{1}H NMR (500 MHz, $CDCl_3$): 7.81~7.86 (m, 4H, C_6H_5), 7.73 (d, 2H, *J*=8.0 Hz, C_6H_5), 7.46 (t, 4H, *J*=7.5 Hz, C_6H_5), 7.41~7.35 (m, 6H, C_9H_6), 7.08 (d, 2H, *J*=8.0 Hz, C_9H_6), 5.81(d, 2H, *J*=3.0 Hz, C_9H_6), 5.01 (d, 2H, *J*=3.0 Hz, C_9H_6). IR (KBr, ν_{CO} / cm^{-1}): 1 986(s), 1 772(s).

1.3 Reaction of 2 with $Ru_3(CO)_{12}$ in toluene

By using a similar procedure to that described above, ligand precursor **2** reacted with $Ru_3(CO)_{12}$ in refluxing toluene for 12 h, after chromatography and eluted with petroleum ether/CH_2Cl_2 (2∶1, *V*/*V*), complex **7** was obtained (0.165 g, 32.3% yield) as orange-red solid. m.p. 255 ℃ (dec.). Anal. Calcd.(%) for $C_{36}H_{26}Ru_2O_4$: C, 59.66; H, 3.62. Found (%): C, 59.99; H, 3.45. ^{1}H NMR (500 MHz, $CDCl_3$): 7.69~7.73 (m, 4H, C_6H_4), 7.69~7.73 (m, 2H, C_9H_6), 7.38~7.40 (t, 2H, *J*=7.5 Hz, C_9H_6), 7.32~7.35 (t, 2H, *J*=7.5 Hz, C_9H_6), 7.26 (d, 4H, *J*=7.5 Hz, C_6H_4), 7.07 (d, 2H, *J*=8.5 Hz, C_9H_6), 5.76 (d, 2H, *J*=2.5 Hz, C_9H_6), 4.97 (d, 2H, *J*=2.5 Hz, C_9H_6), 2.40 (s, 6H, CH_3). IR (KBr, ν_{CO} / cm^{-1}): 1 948(s), 1 782(s).

1.4 Reaction of 3 with $Ru_3(CO)_{12}$ in toluene

By using a similar procedure to that described above, ligand precursor **3** reacted with $Ru_3(CO)_{12}$ in refluxing toluene for 10 h, after chromatography and eluted with petroleum ether/CH_2Cl_2 (2∶1, *V*/*V*), complex **8** was obtained (0.066 g, 12.3% yield) as orange-red solid. m.p. 251 ℃ (dec.). Anal. Calcd.(%) for $C_{34}H_{20}Ru_2O_4Cl_2$: C, 53.34; H, 2.63. Found (%): C, 53.69; H, 2.85. ^{1}H NMR (500 MHz, $CDCl_3$): 7.74 (d, 4H, *J*=8.5 Hz, C_6H_4), 7.67 (d, 4H, *J*=8.5 Hz, C_6H_4), 7.36~7.43 (m, 6H, C_9H_6), 7.11 (d, 2H, *J*=8.0 Hz, C_9H_6), 5.78 (d, 2H, *J*=3.0 Hz, C_9H_6), 5.06 (d, 2H, *J*=3.0 Hz, C_9H_6). IR (KBr, ν_{CO} / cm^{-1}): 1 955(s), 1 782(s).

1.5 Reaction of 4 with $Ru_3(CO)_{12}$ in toluene

By using a similar procedure to that described above, ligand precursor **4** reacted with $Ru_3(CO)_{12}$ in refluxing toluene for 12 h, after chromatography and eluted with petroleum ether/CH_2Cl_2 (2∶1, *V*/*V*), complex **9** was obtained (0.107 g, 20.1% yield) as orange-red solid. m.p. 226 ℃. Anal. Calcd.(%) for $C_{36}H_{26}Ru_2O_6$: C, 57.14; H, 3.46. Found(%): C, 57.46; H, 3.18. ^{1}H NMR (500 MHz, $CDCl_3$): 7.70~7.77 (m, 4H, C_6H_4), 7.70~7.77 (m, 2H, C_9H_6), 7.32~7.40 (m, 6H, C_9H_6), 6.99 (d, 4H, *J*=8.0 Hz, C_6H_4), 5.74 (d, 2H, *J*=3.0 Hz, C_9H_6), 4.99 (d, 2H, *J*=3.0 Hz, C_9H_6), 3.86 (s, 6H, CH_3). IR (KBr, ν_{CO} / cm^{-1}): 1 952(s), 1 772(s).

1.6 Reaction of 5 with $Ru_3(CO)_{12}$ in xylene

By using a similar procedure to that described above, ligand precursor **5** reacted with $Ru_3(CO)_{12}$ in refluxing xylene for 12 h, after chromatography and eluted with petroleum ether/CH_2Cl_2 (2∶1, *V*/*V*), complex **10** was obtained (0.429 g, 86.0% yield) as dark-red solid. m.p. 215 ℃. Anal. Calcd.(%) for $C_{30}H_{18}O_4Ru_2S_2$: C, 50.84; H, 2.56. Found (%): C, 50.58; H, 2.81. ^{1}H NMR (500 MHz, $CDCl_3$): 7.31~7.42 (m, 8H, C_9H_6), 7.07~7.09 (m, 4H, C_4H_3S), 7.20 (d, 2H, *J*=8.0 Hz, C_4H_3S), 5.84 (d, 2H, *J*=3.0 Hz, C_9H_6), 5.24 (d, 2H, *J*=3.0 Hz, C_9H_6). IR (KBr, ν_{CO} / cm^{-1}): 1 961(s), 1 770(s).

1.7 Crystal structure determination

Crystals of the complexes **6**, **7** and **10** suitable for X-ray diffraction were isolated from the slow evaporation of hexane-dichloromethane solution. Data collection were performed on a Bruker SMART APEX Ⅱ-CCD detector with graphite monochromated Mo *Kα* (*λ*=0.071 073 nm) radiation using the *φ*-*ω* scan technique. The structures were solved by direct methods and refined by full-matrix least-squares procedures based on F^2 using the SHELX-97[20] program system. Hydrogen atoms were included in calculated positions riding on the parent atoms and refined with fixed thermal parameters. Crystallographic data and experimental details of the structure determinations are given in Table 1.

CCDC: 943948, **6**; 946643, **7**; 966285, **10**.

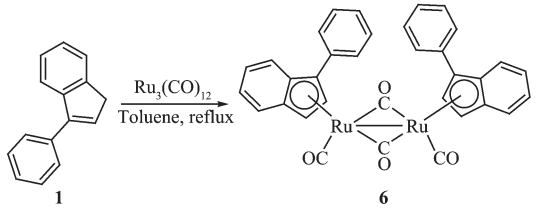
2 Results and discussion

Thermal treatment **1**~**4** with $Ru_3(CO)_{12}$ in refluxing toluene, the corresponding products **6** (48.1%), **7** (32.3%), **8** (12.3%) and **9** (20.1%) were obtained, respectively (Scheme 1 and Scheme 2). Based on their

Table 1 Crystal data and structure refinement parameters for complexes 6, 7 and 10

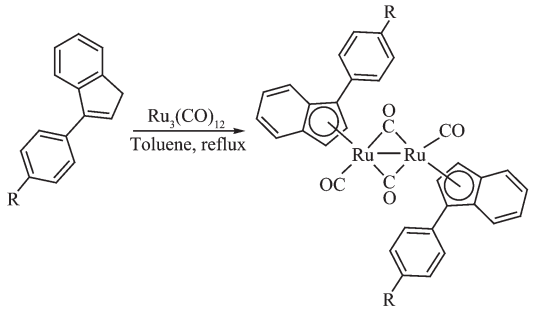
Complex	**6**	**7**	**10**
Empirical formula	$C_{34}H_{22}O_4Ru_2$	$C_{36}H_{26}O_4Ru_2$	$C_{30}H_{18}O_4Ru_2S_2$
Formula weight	696.66	724.71	708.70
Temperature / K	298(2)	298(2)	298(2)
Crystal system	Triclinic	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P\bar{1}$	$P2_1/n$
a / nm	0.999 5(4)	0.811 5(3)	0.941 5(3)
b / nm	1.097 6(4)	0.893 5(3)	0.685 7(3)
c / nm	1.497 1(5)	1.019 3(3)	2.079 1(8)
α / (°)	94.625(4)	98.455(4)	90
β / (°)	106.481(4)	97.947(4)	102.003(5)
γ / (°)	116.326(4)	96.654(4)	90
V / nm^3	1.370 3(8)	0.717 0(4)	1.312 9(8)
Z	2	1	2
F(000)	692	362	700
D_c / ($g\cdot cm^{-3}$)	1.688	1.678	1.793
Crystal dimensions / mm	0.35×0.27×0.04	0.32×0.22×0.20	0.43×0.31×0.20
θ range / (°)	1.46~25.50	2.05~25.48	2.61~25.50
Reflections collected	7 134	3 694	6 628
Independent reflections	4 981	2 571	2 433
R_{int}	0.029 7	0.037 6	0.030 3
Parameters	361	192	209
Goodness of fit on F^2	1.097	1.132	1.119
R_1, wR_2[a] ($I>2\sigma(I)$)	0.040 2, 0.108 8	0.044 3, 0.112 3	0.037 4, 0.098 5
R_1, wR_2[a] (all data)	0.046 5, 0.121 4	0.045 7, 0.114 9	0.040 1, 0.101 1

[a] For complex **6**: $w=1/[\sigma^2(F_o^2)+(0.072\ 8p)^2+1.274\ 9p]$, where $p=(F_o^2+2F_c^2)/3$; for complex **7**: $w=1/[\sigma^2(F_o^2)+(0.087\ 0p)^2+0.180\ 7p]$, where $p=(F_o^2+2F_c^2)/3$; for complex **10**: $w=1/[\sigma^2(F_o^2)+(0.061\ 5p)^2+0.971\ 2p]$, where $p=(F_o^2+2F_c^2)/3$



1 6

Scheme 1 Synthesis of complex **6**

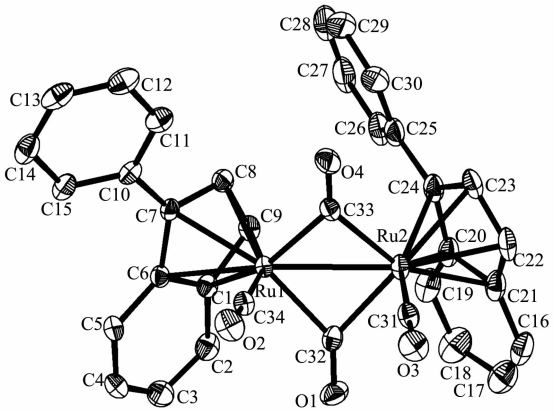


R=CH_3 (**7**); R=Cl (**8**); R=OCH_3 (**9**)

Scheme 2 Syntheses of complexes **7~9**

^{1}H NMR and IR spectra, **6~9** were assigned as the normal Ru-Ru single bonded dinuclear complexes. The IR spectra of **6~9** all exhibited a strong terminal carbonyl absorption at 1 948~1 986 cm^{-1} and a strong bridging carbonyl absorption at 1 772~1 782 cm^{-1}, which are comparable to other metal-metal bond spectra found in other single substituted indenyl ruthenium dimers. The ^{1}H NMR spectra of **6~9** are similar, and they all show peaks at 6.99~7.86 for the C6-ring protons and phenyl protons of the indenyl ligands and two doublets at 4.97~5.81 for the C5-ring protons of the indenyl ligands.

The crystal structures of the complexes **6** and **7** were determined by X-ray diffraction analysis. The molecular structures of **6** and **7** are illustrated in Fig.1 and 2, respectively. Selected bond lengths and angles are given in Table 2.



Hydrogen atoms are omitted for clarity; Displacement ellipsoids are drawn at the 30% probability level

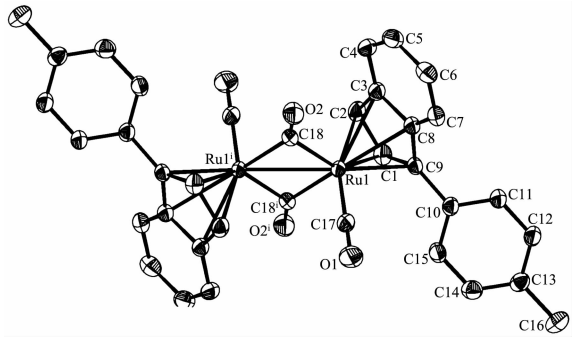
Fig.1 Molecular structure of complex **6**

Complex **6** is a *cis* diruthenium complex, in which two indenyl ligands coordinate with two

Table 2 Selected bond distances (nm) and angles (°) for complexes 6, 7 and 10

			6		
Ru(1)-Ru(2)	0.273 92(9)	C(6)-Ru(1)	0.231 3(4)	C(8)-Ru(1)	0.226 4(4)
C(1)-Ru(1)	0.235 8(4)	C(7)-Ru(1)	0.225 4(4)	C(21)-Ru(2)	0.236 2(5)
C(6)-C(1)-Ru(1)	70.5(2)	C(7)-Ru(1)-Ru(2)	147.90(11)	C(9)-Ru(1)-Ru(2)	100.49(12)
C(1)-C(9)-Ru(1)	75.7(3)	C(8)-Ru(1)-Ru(2)	113.30(12)	C(20)-Ru(2)-Ru(1)	110.62(12)
			7		
Ru(1)-Ru(1^i)	0.275 41(9)	C(2)-Ru(1)	0.227 7(4)	C(8)-Ru(1)	0.234 9(4)
C(1)-Ru(1)	0.226 0(4)	C(3)-Ru(1)	0.240 2(4)	C(17)-Ru(1)	0.187 5(4)
C(1)-C(2)-Ru(1)	71.1(2)	O(1)-C(17)-Ru(1)	177.4(4)	C(2)-Ru(1)-Ru(1^i)	105.90(10)
C(2)-C(3)-Ru(1)	67.4(2)	C(1)-Ru(1)-Ru(1^i)	126.62(10)	C(8)-Ru(1)-Ru(1^i)	148.80(9)
			10		
Ru(1)-Ru(1^i)	0.277 10(8)	C(2)-Ru(1)	0.220 2(4)	C(8)-Ru(1)	0.244 0(3)
C(1)-Ru(1)	0.226 7(4)	C(3)-Ru(1)	0.234 3(4)	C(9)-Ru(1)	0.235 6(4)
C(1)-C(2)-Ru(1)	74.3(2)	O(1)-C(14)-Ru(1)	140.0(3)	C(2)-Ru(1)-Ru(1^i)	166.77(13)
C(3)-C(8)-Ru(1)	68.73(18)	C(1)-Ru(1)-Ru(1^i)	130.27(12)	C(8)-Ru(1)-Ru(1^i)	117.92(9)

Symmetry codes: for **7**: ^{i}2–*x*, 2–*y*, 2–*z*; for **10**: ^{i}1–*x*, 1–*y*, –*z*

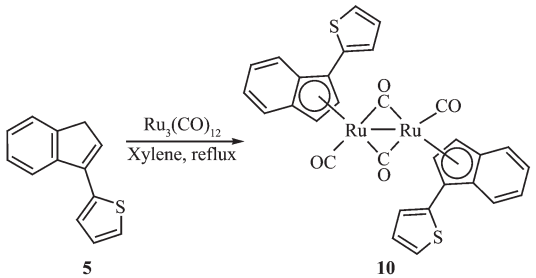


Hydrogen atoms are omitted for clarity; Displacement ellipsoids are drawn at the 30% probability level; Symmetry code: i 2−*x*, 2−*y*, 2−*z*

Fig.2　Molecular structure of complex **7**

ruthenium atoms through their indenyl ring in η^5-bonding. The complex **6** has two forms of coordinated carbonyl ligands. Two carbonyls are bridged and two carbonyls are terminal. Both the two indenyl rings and terminal COs are *cis* configuration. The Ru-Ru bond distance (0.273 92(9) nm for **6**) is close to that of analogous complex $[\eta^5\text{-}(C_5H_4N)CH_2(C_9H_6)Ru(CO)]_2(\mu\text{-}CO)_2$ (0.273 89(15) nm)[21]. Different from complex 6, complex **7** is a normal *trans* diruthenium complex, and two indenyl ligands are coordinated two ruthenium atoms through their indenyl ring in η^5 mode. Two carbonyls are bridged and two carbonyls are terminal. Both the two indenyl ring and terminal CO are *trans* configuration. The two indenyl ring planes are parallel. The Ru-Ru bond distance (0.275 41(9) nm for **7**) is close to that of analogous complexes $[(\eta^5\text{-}C_9H_7)Ru(CO)_2]_2$ (0.274 12(5) nm)[22], $[\eta^5\text{-}(MeC_5H_3N)CH_2CMe_2(C_9H_6)Ru(CO)]_2(\mu\text{-}CO)_2$ (0.274 43(13) nm)[21] and $(\eta^5\text{-}C_9H_6{}^n\text{Bu})_2Ru_2(CO)_4$ (0.275 5(2) nm)[11].

Thermal treatment **5** with $Ru_3(CO)_{12}$ in refluxing xylene, the corresponding product **10** (86.0%) was obtained (Scheme 3). Based on its ^{1}H NMR and IR spectra, **10** was assigned as the normal Ru-Ru single bonded dinuclear complex. The IR spectra of complex

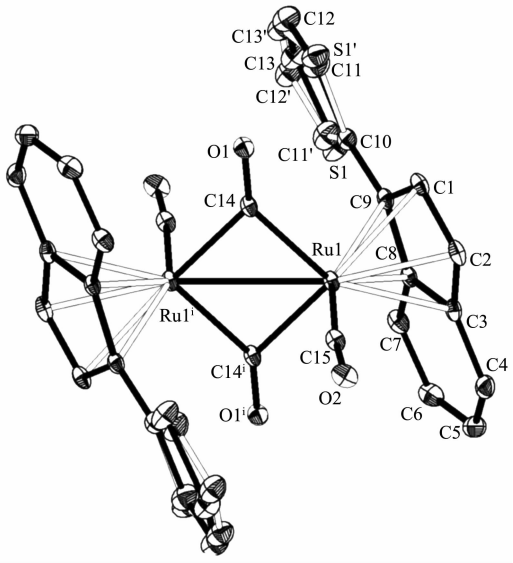


5　　**10**

Scheme 3　Synthesis of complex **10**

10 exhibited a strong terminal carbonyl absorption at 1 961 cm^{-1} and a strong bridging carbonyl absorption at 1 770 cm^{-1}, which is comparable to other metal-metal bond spectra found in other single substituted indenyl ruthenium dimers. The ^{1}H NMR spectrum of **10** shows one multiplet at 7.31~7.42 for the C6-ring protons of the indenyl ligands, two doublets at 5.24~5.84 for the C5-ring protons of the indenyl ligands and two groups peaks at 7.07~7.09 and 7.20 for the thienyl protons.

The crystal structure of the complex **10** was determined by X-ray diffraction analysis. The molecular structure of **10** is presented in Fig.3. Similar to complex **7**, complex **10** is a normal *trans* η^5-indenyl diruthenium complex. The thienyl groups only act as substituents and the S atom of thiophene does not coordinate to Ru, consistent with the weak donor properties of sulfur. The Ru-Ru bond distance is 0.277 10(8) nm, which is slight longer than those found in analogous complexes $[(\eta^5\text{-}C_5Me_4C_4H_3S)Ru(CO)_2]_2$ (0.275 11(8) nm), $[(\eta^5\text{-}C_5H_4)C(CH_3)_2(C_4H_3S)Ru(CO)_2]_2$ (0.274 29(10) nm) and $[(\eta^5\text{-}C_5H_4)C(CH_2)_5(C_4H_3S)Ru(CO)_2]_2$ (0.273 98(9) nm)[23].



Hydrogen atoms are omitted for clarity; Displacement ellipsoids are drawn at the 30% probability level; Symmetry code: i 1−*x*, 1−*y*, −*z*

Fig.3　Molecular structure of complex **10**

3　Conclusions

We have synthesized a series of dinuclear metal

carbonyl complexes by reactions of aryl-substituted indenes with $Ru_3(CO)_{12}$ in refluxing toluene or xylene. The results clearly reveal that we obtained indenyl diruthenium complexes with the η^5 coordination mode; the S atom of thiophene does not coordinate to the Ru atom because of the weak donor nature of the sulfur. The X-ray data together with NMR spectral data revealed that the substituent influenced the orientation the two indenyl ligands of the metallocenes. The two indenyl rings and terminal COs are *cis* configuration with respect to each other in **6** whereas the indenyl ligands and terminal COs in **7** and **10** are normal *trans* configuration.

References:

[1] Cadierno V, Díez J, Gamasa M P, et al. *Coord. Chem. Rev.*, **1999**,**193-195**:147-205

[2] Calhorda M J, Félix V, Veiros L F. *Coord. Chem. Rev.*, **2002**, **230**:49-64

[3] Zargarian D. *Coord. Chem. Rev.*, **2002**,**233-234**:157-176

[4] Halterman R L. *Chem. Rev.*, **1992**,**92**:965-994

[5] Möhring P C, Coville N J. *J. Organomet. Chem.*, **1994**,**479**:1-29

[6] Brintzinger H H, Fischer D, Mülhaupt R, et al. *Angew. Chem., Int. Ed. Engl.*, **1995**,**34**:1143-1170

[7] Resconi L, Cavallo L, Fait A, et al. *Chem. Rev.*, **2000**,**100**: 1253-1345

[8] Frankcom T M, Green J C, Nagy A, et al. *Organometallics*, **1993**,**12**:3688-3697

[9] Bonifaci C, Ceccon A, Santi S, et al. *Inorg. Chim. Acta*, **1995**, **240**:541-549

[10]Gamasa M P, Gimeno J, Gonzalez-bernardo C, et al. *Organometallics*, **1996**,**15**:302-308

[11]MA Zhi-Hong(马志宏), LI Fang(李放), LIU Xiao-Huan(刘晓焕), et al. *Chinese J. Org. Chem.*(有机化学), **2009**,**29**: 1294-1297

[12]GUO Kai-Ming(郭凯明), MA Zhi-Hong(马志宏), LI Su-Zhen (李素贞), et al. *Chinese J. Inorg. Chem.*(无机化学学报), **2012**,**28**:643-646

[13]Ma Z, Fan D, Li S, et al. *New. J. Chem.*, **2015**,**39**:1075-1082

[14]Coates G W, Waymouth R M. *Science*, **1995**,**267**:217-219

[15]Bruce M D, Coates G W, Hauptman E, et al. *J. Am. Chem. Soc.*, **1997**,**119**:11174-11182

[16]Kravchenko R, Masood A, Waymouth R M, et al. *J. Am. Chem. Soc.*, **1998**,**120**:2039-2046

[17]Zhang F, Mu Y, Zhao L, et al. *J. Organomet. Chem.*, **2000**, **613**:68-76

[18]Zhang F, Mu Y, Wang J, et al. *Polyhedron*, **2000**,**19**:1941-1947

[19]Grimmer N E, Coville N J, de Koning C B, et al. *J. Organomet. Chem.*, **2000**,**616**:112-127

[20]Sheldrick G M. *SHELX*-97, *Program for the Refinement of Crystal Structures*, University of Göttingen, Germany, **1997**.

[21]Chen D F, Xu S S, Song H B, et al. *Eur. J. Inorg. Chem.*, **2008**:1854-1864

[22]Sridevi V S, Leong W K. *J. Organomet. Chem.*, **2007**,**692**: 4909-4916

[23]Liu X H, Ma Z H, Tian L J, et al. *Transition Met. Chem.*, **2010**,**35**:393-397