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苯基或联苯基桥连双环戊二烯基铼羰基化合物的合成、结构及催化性能

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摘要：两个单桥连的双环戊二烯$(C_5Me_4H)E(C_5Me_4H)(E=C_6H_4,(C_6H_4)_2)$分别与 $Re_2(CO)_{10}$ 在均三甲苯中加热回流,得到了 2 个双核配合物$(E)[(\eta^5\text{-}C_5Me_4)Re(CO)_3]_2(E=C_6H_4(\mathbf{1}),(C_6H_4)_2(\mathbf{2}))$。通过元素分析、红外光谱、核磁共振氢谱和碳谱对配合物 **1** 和 **2** 的结构进行了表征,用 X 射线单晶衍射分析测定了配合物的结构。同时对 2 个配合物在芳香族化合物 Friedel-Crafts 酰基化反应中的催化活性进行了研究。

关键词：合成；单桥连双环戊二烯；Friedel-Crafts 酰基化反应；铼羰基配合物；催化

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Syntheses, Structures and Catalytic Activity of *p*-Phenylene- or *p*-Biphenylene-Bridged Biscyclopentadienyl Dinuclear Rhenium Carbonyl Complexes

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Abstract: Thermal treatment of two *p*-phenylene- and *p*-biphenylene-bridged biscyclopentadienes $(C_5Me_4H)E(C_5Me_4H)$ (E=C_6H_4, $(C_6H_4)_2$) with $Re_2(CO)_{10}$ in refluxing mesitylene gave the corresponding complexes $(E)[(\eta^5\text{-}C_5Me_4)Re(CO)_3]_2$ (E = C_6H_4 (**1**), $(C_6H_4)_2$ (**2**)), which were separated by chromatography, and characterized by elemental analysis, IR, ^{1}H NMR and ^{13}C NMR spectroscopy. The molecular structures of complexes **1** and **2** were characterized by X-ray crystal diffraction analysis, showing both them are monobridged biscyclopentadienyl dinuclear rhenium carbonyl complexes. In addition, the catalytic performance of complexes **1** and **2** was also tested, and it was found that both complexes were obviously active for Friedel-Crafts acylation reactions. CCDC: 1506755, **1**; 1506754, **2**.

Keywords: synthesis; mono-bridged biscyclopentadiene; Friedel-Crafts acylation reaction; rhenium carbonyl complex; catalysis

0 Introduction

Much attention has been focused on the synthesis of a series of biscyclopentadienyl carbonyl ruthenium complexes in recent decades, which mainly include non-bridged [1-2], singly bridged [3-8] and doubly bridged[9-10] biscyclopentadienyl complexes. Bridged bis (cyclopentadienyl) ligands have been extensively studied as frameworks for dinuclear metal complexes that are resistant to fragmentation and maintain two

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metal centers in close proximity even after metal-metal bond cleavage[11-13]. Especially, rhenium carbonyl complexes have been studied as catalysts for many reactions due to their catalytic activity. When compared to mononuclear rhenium complexes, bridged dicyclopentadienyl dirhenium analogues, in which the bridging ligand binds two reactive metal centers, may promote the distinctive chemical reactivity and catalytic properties. However, only a few examples of Friedel-Crafts reactions catalyzed by rhenium carbonyl complexes have been reported up to now [14-16]. Recently, Our group have reported the synthesis and catalytic activity of three monobridged bis (cyclopentadienyl)rhenium carbonyl complexes [17], showing that these rhenium carbonyl complexes have catalytic activity for Friedel-Crafts alkylation reactions. To develop a deeper understanding of the structures and reactivity of bridged bis (cyclopentadienyl)rhenium carbonyl complexes, here in this paper we selected two bridged ligand precursors $(C_5Me_4H)E(C_5Me_4H)$ ($E=C_6H_4$, $(C_6H_4)_2$), in which both two bridging groups have planer structure, and expected to see the catalytic reactivity of both *p*-phenylene- and *p*-biphenylene-bridged bis (cyclopentadienyl)rhenium carbonyl complexes.

1 Experimental

1.1 General considerations

All procedures were performed under an argon atmosphere by using standard Schlenk techniques. Solvents were distilled from appropriate drying agents under nitrogen atmosphere. Elemental analyses of C and H were performed with a Vario EL Ⅲ elemental analyzer. The IR spectra were recorded as KBr disks on a Thermo Fisher is50 spectrometer. Gas chromatograms were recorded with an Agilent 6820 gas chromatograph. ^1H and ^{13}C NMR spectra were recorded on a Bruker AV Ⅲ-500 (or 600) instrument in $CDCl_3$. The ligand precursors $(C_5Me_4H)E(C_5Me_4H)$ ($E=C_6H_4$, $(C_6H_4)_2$) were synthesized according to the literature[18-20].

1.2 Synthesis of complex 1

A solution of free ligand $(C_5Me_4H)C_6H_4(C_5Me_4H)$ (0.19 g, 0.6 mmol) and $Re_2(CO)_{10}$ (0.2 g, 0.3 mmol) in mesitylene (15 mL) was refluxed for 48 h. After removal of solvent, the residue was placed on an alumina column. Elution with petroleum ether/CH_2Cl_2 (2∶1, *V*/*V*) developed a colorless band, which was collected, and after concentration, afforded $(C_6H_4)[(\eta^5\text{-}C_5Me_4)Re(CO)_3]_2$ (**1**) as a white solid. Yield: 52.1% (0.134 g). m.p. 316 ℃; Anal. Calcd. for $C_{30}H_{28}O_6Re_2$(%): C, 42.05; H, 3.29. Found (%): C, 42.32; H, 3.13. ^1H NMR ($CDCl_3$, 500 MHz): δ 2.16 (s, 12H, C_5Me_4), 2.25 (s, 12H, C_5Me_4), 7.28 (s, 4H, C_6H_4). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 10.9, 11.4, 97.7, 102.2, 104.0, 131.8, 132.4, 197.37. IR (KBr, cm^{-1}): 1 900(s), 2 002(s).

1.3 Synthesis of complex 2

Using a procedure similar to that described above, ligand precursor $(C_5Me_4H)(C_6H_4)_2(C_5Me_4H)$ was reacted with $Re_2(CO)_{10}$ in refluxing mesitylene for 48 h. After chromatography with petroleum ether/CH_2Cl_2, $(C_6H_4)_2)[(\eta^5\text{-}C_5Me_4)Re(CO)_3]_2$ (**2**) was obtained as white crystals. Yield: 77.8% (0.217 g). m.p. 313 ℃; Anal. Calcd. for $C_{36}H_{32}O_6Re_2$ (%): C, 46.34; H, 3.46. Found (%): C, 45.98; H, 3.59. ^1H NMR ($CDCl_3$, 500 MHz): δ 2.17 (s, 12H, C_5Me_4), 2.26 (s, 12H, C_5Me_4), 7.38 (d, 4H, *J*=8.0 Hz, C_6H_4), 7.61 (d, 4H, *J*=8.5 Hz, C_6H_4). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 10.9, 11.3, 97.7, 102.1, 104.4, 127.0, 131.4, 133.0, 139.8, 197.4. IR (KBr, cm^{-1}): 1 903(s), 2 012(s).

1.4 Crystallographic analysis

Crystallographic data for complexes **1** and **2** were collected at 298 K on a Bruker AXS SMART 1000 CCD diffractometer with Mo *Kα* radiation (λ=0.071 073 nm) using the φ-ω scan technique. The crystal structures were solved by direct method and refined on F^2 by full-matrix least-squares technique using the SHELXL-97 program package [21]. All non-hydrogen atoms were found from the Fourier difference maps refined anisotropically, hydrogen atoms were included in calculated positions riding on the parent atoms and refined with fixed thermal parameters. Crystallographic data and experimental details for structural analysis of the complexes are summarized in Table 1.

CCDC: 1506755, **1**; 1506754, **2**.

Table 1　Crystal data and structure refinement parameters for complexes 1 and 2

Complex	**1**	**2**
Empirical formula	$C_{30}H_{28}O_6Re_2$	$C_{36}H_{32}O_6Re_2$
Formula weight	856.92	933.02
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a / nm	0.700 82(7)	0.822 60(7)
b / nm	0.789 24(8)	0.857 01(8)
c / nm	1.375 14(11)	1.190 09(9)
α / (°)	81.907(2)	77.607 0(10)
β / (°)	84.410(2)	83.990(2)
γ / (°)	73.555 0(10)	75.732 0(10)
V / nm^3	0.720 90(12)	0.792 97(12)
Z	1	1
D_c / (g·cm^{-3})	1.974	1.954
μ / mm^{-1}	8.428	7.671
F(000)	406	446
Crystal size / mm	0.38×0.15×0.10	0.40×0.39×0.37
θ range / (°)	2.71~25.01	2.50~25.01
Reflection collected, unique	3 607, 2 457	4 054, 2 758
R_{int}	0.061 4	0.031 7
Completeness to θ	96.50%	98.50%
Absorption correction	Semi-empirical from equivalents	Multi-sacn
Max. and min. transmission	0.486 2 and 0.142 0	0.163 6 and 0.149 3
Data, restraint, parameter	2 457, 0, 176	2 758, 0, 203
R_1, $wR_2[I>2\sigma(I)]$	0.087 0, 0.230 5	0.035 6, 0.092 2
R_1, wR_2(all data)	0.092 7, 0.237 1	0.038 7, 0.093 6
$(\Delta\rho)_{max}$, $(\Delta\rho)_{min}$ / (e·nm^{-3})	5 767, −3 695	1 205, −2 382

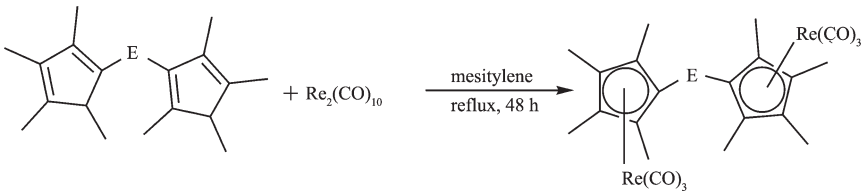
1.5　General procedure for catalytic tests

The catalytic reactions were carried out under an argon atmosphere with magnetic stirring. The required complexes (0.02 mmol) was mixed with 1, 2-dichloroethane (3.5 mL) in a 25 mL round-bottom flask at room temperature. Aromatic compounds and acylation reagents were added by syringe. The reaction mixture was heated at 80 ℃ for 24 h. After cooling to room temperature, the solid catalyst was separated from the reaction mixture by filtration. The solvent was removed by rotary evaporation, and the residue was purified by Al_2O_3 column chromatography, eluting with petroleum ether and ethyl acetate to give a white solid.

2　Results and discussion

2.1　Preparation of complexes 1 and 2

Reactions of ligand precursors $(C_5Me_4H)E(C_5Me_4H)$ (E=C_6H_4, $(C_6H_4)_2$) with $Re_2(CO)_{10}$ in refluxing mesitylene for 48 h afforded the corresponding complexes (E)[(η^5-C_5Me_4)Re(CO)$_3$]$_2$ (E=C_6H_4 (**1**), $(C_6H_4)_2$ (**2**)) in yield of 52% and 78%, respectively (Scheme 1). The IR spectra of complexes **1** and **2** all exhibited only terminal carbonyl bands (**1**: 1 900, 2 002 cm^{-1}; **2**: 1 903, 2 012 cm^{-1}). The ^{1}H NMR spectra of **1** and **2** all displayed two groups of singlets for the four methyl protons, indicating the symmetrical structure in solution, in addition, complex **1** showed a singlet at δ



E=C_6H_4(**1**), $(C_6H_4)_2$(**2**)

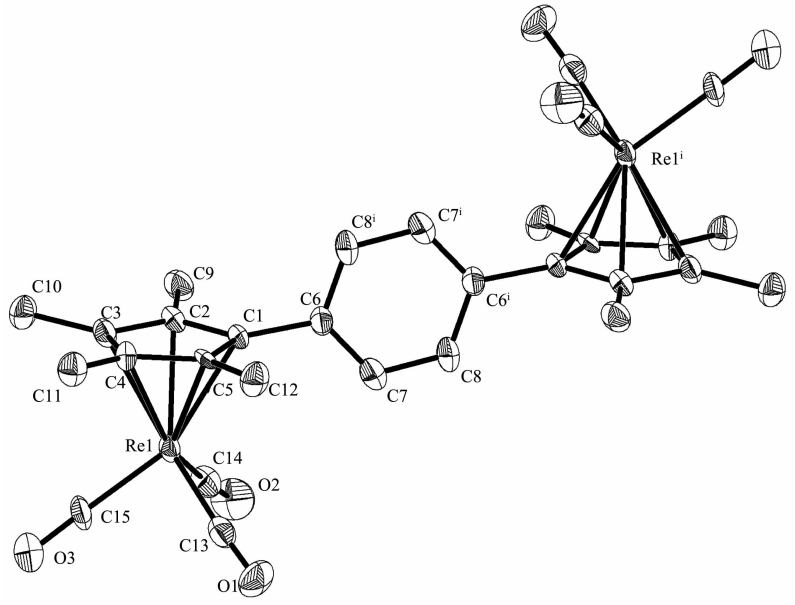
Scheme 1 Synthesis of complexes **1** and **2**

7.28 for the phenylene protons and complex **2** showed two doublets at δ 7.38 and 7.61 for the biphenylene protons.

2.2 Crystal structures of complexes 1 and 2

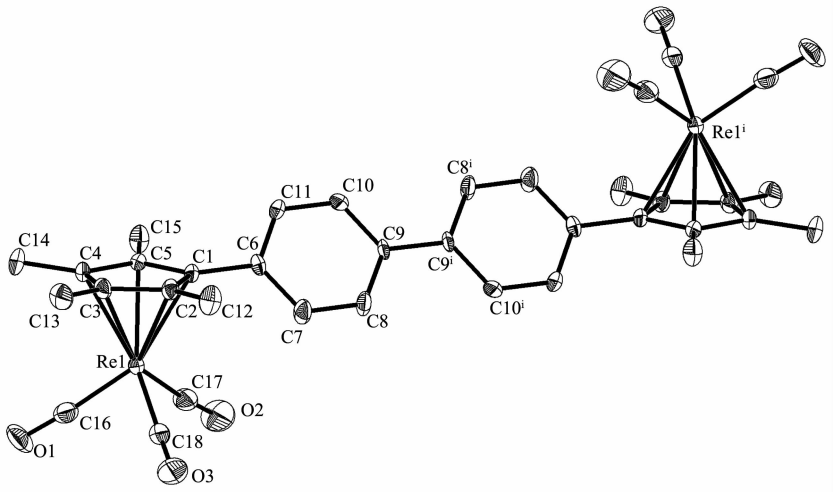
Slow evaporation of the solvent from the complexes in hexane-CH_2Cl_2 solution gave single crystals **1** and **2** suitable for X-ray diffraction. Selected bond parameters are presented in Table 2 and their structures are depicted in Fig.1 and 2, respectively.

Single-crystal X-ray diffraction analysis reveals that **1** and **2** crystallize in the triclinic space group $P\bar{1}$ Complexes **1** and **2** are monobridged bis (cyclopentadienyl)rhenium carbonyl complexes and similar to each other. Both structures show that the molecule consists of two $[(\eta^5\text{-}C_5Me_4)Re(CO)_3]$ moieties linked by a single bridge, with each rhenium atom is η^5-coordinated to the cyclopentadienyl ring and three terminal CO ligands. Two $Re(CO)_3$ units are located on the opposite site of the monobridged ligand and anti to each other, the Re atoms exhibit a three-legged piano-stool geometry. Re-Re bonds are not observed in both complexes. Complex **1** has a symmetric structure, the molecule consists of two $[(\eta^5\text{-}C_5Me_4)Re(CO)_3]$ moieties linked by a *p*-phenylene-bridge. Complex **2** also has similar structure, consisting of two $[(\eta^5\text{-}C_5Me_4)Re(CO)_3]$ moieties linked by a *p*-biphenylene-bridge. The dihedral angle between the two Cp ring planes is 0° and the torsion angle Re1-Cp(centroid) ⋯ Cp(centroid)-Re1^{i} is −180°, indicating two Cp rings are parallel to each other. For complexes **1** and **2**, the distances of Re1-CEN and Re1^{i}-CEN (CEN means the centroid of the cyclopentadienyl ring) are equal (Table 2). The Re-CEN distance in **1** is 0.195 6 nm and the Re-CEN distance



Ellipsoids correspond to 30% probability; Hydrogen atoms are omitted for clarity; Symmetry codes: ^{i} 1−*x*, 1−*y*, 1−*z*

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



Ellipsoids correspond to 30% probability; Hydrogen atoms are omitted for clarity; Symmetry codes: ^{i} 1−x, −y, 1−z

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

Table 2　Selected bond distances (nm) and angles (°) for complexes 1 and 2

Complex **1**					
Re1-C1	0.229 2(16)	Re1-C2	0.230 5(17)	Re1-C3	0.230 5(17)
Re1-C4	0.231 9(18)	Re1-C5	0.227 7(15)	O1-C13	0.116(3)
C1-C6	0.150(2)	C6-C7	0.134(3)	Re1-C13	0.192(2)
Re1-C14	0.188(2)	Re1-C15	0.191 5(18)	Re1-CEN	0.195 6
C1-Re1-C2	36.5(6)	C3-C2-C1	109.4(17)	C14-Re1-C13	90.6(9)
O1-C13-Re1	175.0(18)	C6-C7-C8	121.4(19)		
Complex **2**					
Re1-C1	0.228 3(7)	Re1-C3	0.229 4(7)	Re1-C4	0.229 4(7)
Re1-C5	0.229 2(7)	O1-C16	0.117 1(10)	C1-C6	0.148 5(9)
C6-C7	0.138 1(10)	Re1-C16	0.188 5(10)	Re1-C17	0.188 5(9)
Re1-C18	0.190 3(9)	Re1-CEN	0.194 6		
C1-Re1-C2	36.3(3)	C3-C2-C1	106.8(6)	C16-Re1-C17	90.6(4)
O1-C16-Re1	175.6(8)	C6-C7-C8	120.5(7)		

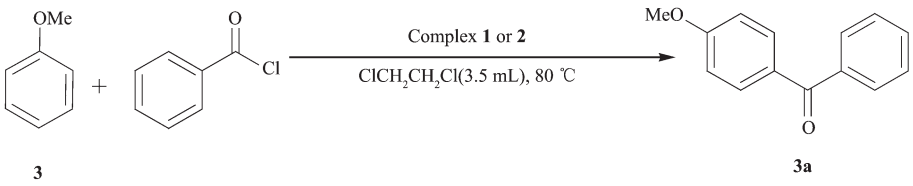
CEN: centroid of the cyclopentadienyl ring

in **2** is 0.194 6 nm, which compare very well with those values in the analogues $[(\eta^5\text{-}C_5H_4)_2C(CH_2)_5][Re(CO)_3]_2$ (0.195 7 and 0.196 0 nm) and $[(\eta^5\text{-}C_5H_4)_2Si(CH_3)_2][Re(CO)_3]_2$ (0.194 6 and 0.195 2 nm)[17]. From above data, we can conclude that the different ligand bridges in these complexes have no obvious effect on their structures.

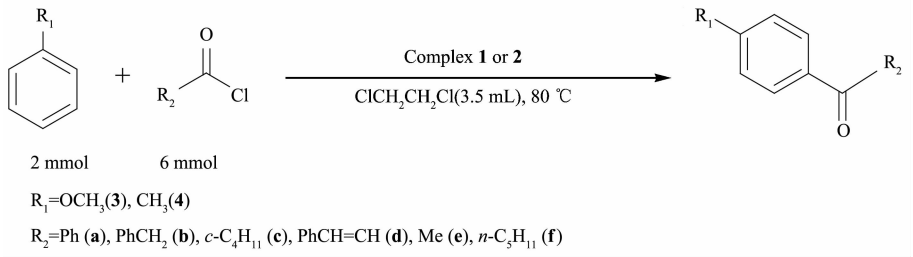
2.3　Catalysis of Friedel-Crafts acylation reactions

In order to test the catalytic capability in Friedel-Crafts acylation reactions (Scheme 2~6) catalyzed by two complexes, considering factors such as the reaction time, yield, economic considerations, the following experimental conditions were chosen for next work: reaction time 24 h; 1,2-dichloroethane as solvent; 80 ℃; the molar ratio of aromatic substrates and acylation reagents was 1∶3; the amount of catalyst was 1.0%(n/n) (substrate as reference).

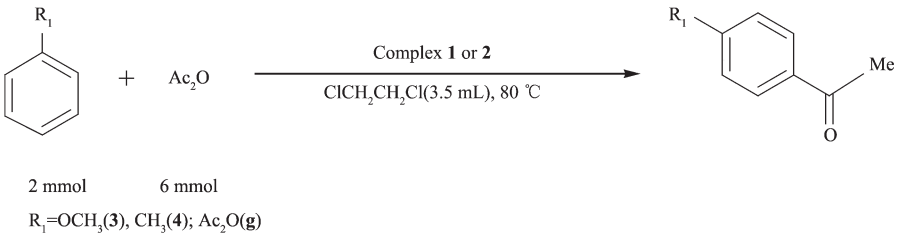
With the above studies, the yields(%) were found



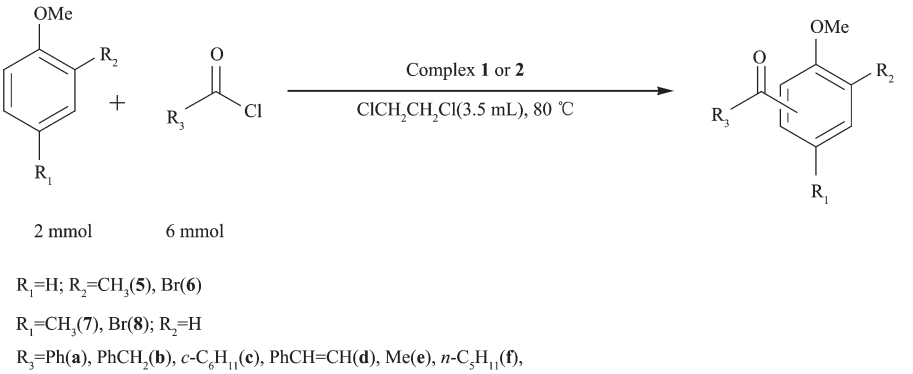
Scheme 2 Complex **1** or **2** catalyzed Friedel-Crafts acylation reaction of anisole with benzoyl chloride



Scheme 3 Complex **1** or **2** catalyzed Friedel-Crafts acylation reactions of anisole and methylbenzene with acyl chlorides

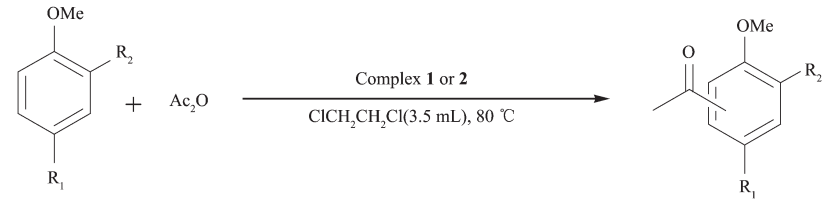


Scheme 4 Complex **1** or **2** catalyzed Friedel-Crafts acylation reactions of anisole and methylbenzene with acetic anhydride



Scheme 5 Complex **1** or **2** catalyzed Friedel-Crafts acylation reactions of acyl chlorides

to vary with aromatic substrates with different acylation reagents, the catalytic results of complex **1** and **2** are shown in Table 3. Friedel-Crafts acylation reactions are electrophilic substitution reactions, however halogen element and carbonyl formed p-π conjugation in acylating agent, thus the acylating agents were difficult to lose halogen element to form carbocation. Benzoyl chloride, phenylacetyl chloride, cinnamoyl chloride and cyclohexanecarboxylic acid chloride could be used as acylation reagents in these reactions and the corresponding products were obtained with high selectivity for the para-products without detection of di-substituted in all cases, suggesting that the catalytic reaction has high



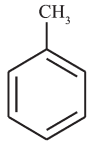
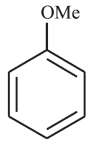
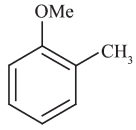
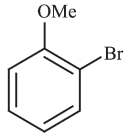
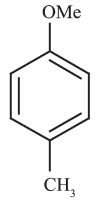
2 mmol　　6 mmol

R_1=H; R_2=CH_3(**5**), Br(**6**)

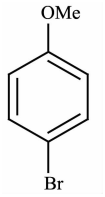
R1=CH_3(**7**), Br(**8**); R_2=H Ac_2O(**g**)

Scheme 6　Complex **1** or **2** catalyzed Friedel-Crafts acylation reactions of acetic anhydride

Table 3　Catalyzed Friedel-Crafts acylation reaction of aromatic substrates with different acylation reagents

Entry	Benzene derivative	Reagent	Yield catalyzed by **1** / %	Yield catalyzed by **2** / %	Route
1		MeCOCl	–	–	Scheme 3
2		PhCOCl	14.9	15.3	Scheme 3
3		$PhCH_2COCl$	10.6	11.9	Scheme 3
4		*c*-$C_6H_{11}COCl$	–	–	Scheme 3
5		*n*-$C5H_{11}COCl$	–	–	Scheme 3
6		PhCH=CHCOCl	–	–	Scheme 3
7		Ac_2O	–	–	Scheme 4
8		MeCOCl	11.9	13.1	Scheme 3
9		PhCOCl	32.6	34.2	Scheme 3
10		$PhCH_2COCl$	28.7	30.1	Scheme 3
11		*c*-$C_6H_{11}COCl$	13.3	15.1	Scheme 3
12		*n*-$C_5H_{11}COCl$	10.6	12.6	Scheme 3
13		PhCH=CHCOCl	15.4	19.4	Scheme 3
14		Ac_2O	4.3	4.3	Scheme 4
15		MeCOCl	12.7	13.2	Scheme 5
16		PhCOCl	32.4	36.3	Scheme 5
17		$PhCH_2COCl$	29.3	31.7	Scheme 5
18		*c*-$C_6H_{11}COCl$	14.1	20.4	Scheme 5
19		*n*-$C_5H_{11}COCl$	11.7	15.6	Scheme 5
20		PhCH=CHCOCl	16.3	24.3	Scheme 5
21		Ac_2O	5.2	4.6	Scheme 6
22		MeCOCl	5.7	6.8	Scheme 5
23		PhCOCl	24.9	27.7	Scheme 5
24		$PhCH_2COCl$	22.6	24.4	Scheme 5
25		*c*-$C_6H_{11}COCl$	11.8	13.5	Scheme 5
26		*n*-$C_5H_{11}COCl$	5.4	6.9	Scheme 5
27		PhCH=CHCOCl	12.1	13.9	Scheme 5
28		Ac_2O	3.8	–	Scheme 6
29		MeCOCl	–	–	Scheme 5
30		PhCOCl	15.6	16.3	Scheme 5
31		$PhCH_2COCl$	10.3	12.7	Scheme 5
32		*c*-$C_6H_{11}COCl$	5.1	5.9	Scheme 5
33		*n*-$C_5H_{11}COCl$	–	–	Scheme 5

Continued Table 3

34		PhCH=CHCOCl	9.7	10.6	Scheme 5
35		Ac_2O	–	–	Scheme 6
36		MeCOCl	–	–	Scheme 5
37		PhCOCl	10.4	12.6	Scheme 5
38		$PhCH_2COCl$	7.3	9.3	Scheme 5
39		c-$C_6H_{11}COCl$	–	–	Scheme 5
40		n-$C_5H_{11}COCl$	–	–	Scheme 5
41		PhCH=CHCOCl	–	–	Scheme 5
42		Ac_2O	–	–	Scheme 6

Reagents and conditions: catalyst 1%(*n*/*n*), solvent 3.5 mL, 80 ℃, 24 h

regioselectivity. The order of increasing reactivity was found to be: 4-bromoanisole <4-methyl anisole < methylbenzene <2-bromoanisole <anisole <2-methylanisole, which was consistent with the characteristics of the aromatic electrophilic substitution mechanism. Overall, both complexes gave similar results, showing that the different ligand bridges have only a small influence on the catalytic behavior.

3 Conclusions

Two new bridge d bis (cyclopentadienyl)rhenium carbonyl complexes (E) $[(\eta^5\text{-}C_5Me_4)Re(CO)_3]_2$ (E =C_6H_4 (**1**), $(C_6H_4)_2$ (**2**)) have been synthesized and structurally characterized. Friedel-Crafts reactions of aromatic substrates with acylation reagents showed that two complexes have catalytic activity. Compared with traditional catalysts, these complexes have significant practical advantages, namely lower amounts of catalyst, mild reaction conditions, and high selectivity. Further studies to elucidate the reaction mechanism and expand the synthetic utility of these catalysts are in progress.

Supporting information is available at http://www.wjhxxb.cn

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