

Note

Synthesis and characterization of bimetallic ruthenium complexes connected through linear (CH)₁₄ chain

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Abstract

Treatment of RuHCl(CO)(PPh₃)₃ with (3*E*,5*E*,7*E*,9*E*,11*E*)-HC≡C-(CH=CH)₅-C≡CH produces [RuCl(CO)(PPh₃)₂]₂[μ-(CH=CH)₇]. The later complex reacts with PMe₃ to give [RuCl(CO)(PMe₃)₃]₂[μ-(CH=CH)₇], the structure of which has been confirmed by X-ray diffraction. The through-space distance from one Ru to the other is 19.88 Å.

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1. Introduction

Bimetallic and polymetallic complexes with conjugated hydrocarbon ligands bridging metal centers are attracting considerable current interest [1,2]. Bimetallic complexes with polyynediyl bridges, M-(C≡C)_n-M', constitute the most fundamental class of carbon-based molecular wires and they have been proposed for construction of nanoscale electronic devices [2]. To date, C₁, C₂, C₃, C₄, C₅, C₆, C₈, C₁₀, C₁₂, C₁₆, and C₂₀, C₂₄, C₂₈ adducts have been isolated [2]. In contrast, few studies have been carried out with bimetallic complexes with polyenediyl bridges, despite the fact that many conjugated organic materials (e.g. polyacetylenes, push/pull stilbenes) have only sp² hybridized carbon in their backbones, and polyacetylenes have high electrical conductivity (up to 10⁵ Scm⁻¹) upon doping [3]. Linear monodisperse π-conjugated oligoenes R(CR'=CR'')_nR(R', R''=H or Me, R=Ar or CHO, n = 3, 5, ... up to 11) have been synthesized, and they have promising electronic and

optical properties [3]. Previously reported examples of (CH)_x-bridged bimetallic complexes are limited to a few of those with linear (CH)₂ [4], (CH)₄ [5–7], (CH)₅ [8], (CH)₆ [9,10], (CH)₈ [11] and (CH)₁₀ [12] bridges. In this report, We wish to report the synthesis, properties, and molecular structure of the first (CH)₁₄-bridged bi-ruthenium complexes, which features the longest sp² carbon chain bimetallic complexes synthesized and structurally characterized to date with the distance between the two metals 19.88 Å.

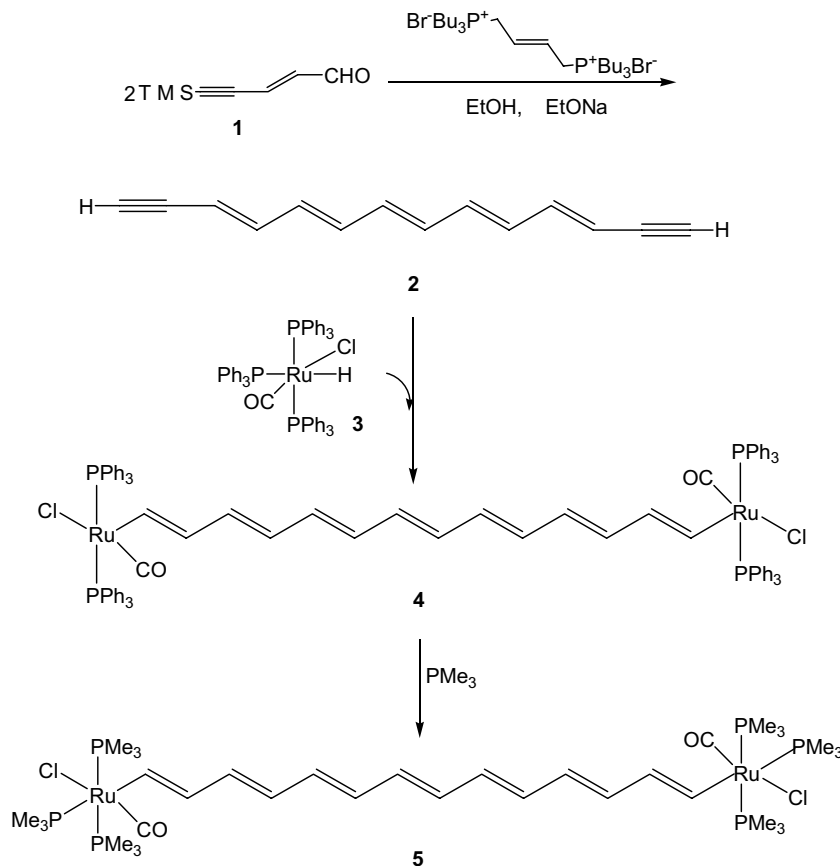
2. Results and discussion

2.1. Synthesis of metal complexes

As showed in Scheme 1, reaction of (*E*)-Me₃SiC≡CCH=CHCHO (**1**) [11] with BrBu₃PCH₂CH=CHCH₂PBu₃Br [13] in the presence of EtONa as the base produced the precursor compound **2**. The Me₃Si was hydrolysed in situ. We chose the tributylphosphonium bromide but not the triphenylphosphonium bromide to reduce the proportion of Z-isomer in the product during Wittig reaction [13]. The ¹H NMR spectrum of compound **2** (in CDCl₃) showed one

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$\equiv\text{CH}$ signal at 3.15 ppm and five $=\text{CH}$ signals at 5.61, 6.10, 6.37, 6.74, and 7.16 ppm.

Treatment of **2** with the ruthenium hydride complex $\text{RuHCl}(\text{CO})(\text{PPh}_3)_3$ (**3**) in dichloromethane produced the insertion product $[\text{RuCl}(\text{CO})(\text{PPh}_3)_2]_2[\mu-(\text{CH}=\text{CH})_7]$ (**4**), which is sensitive to air. The ^{31}P NMR spectrum (in CD_2Cl_2) showed a singlet at 30.94 ppm, the chemical shift of which is typical for $\text{RuCl}(\text{CH}=\text{CHR})(\text{CO})(\text{PPh}_3)_2$ [14]. Treatment of **4** with PMe_3 produced the six-coordinated complex $[\text{RuCl}(\text{CO})(\text{PMe}_3)_3]_2[\mu-(\text{CH}=\text{CH})_7]$ (**5**), which is isolated through silica gel chromatography. The PMe_3 ligands in **5** are meridionally coordinated to ruthenium as indicated by the AM_2 pattern $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum. In the ^1H NMR spectrum (in CD_2Cl_2), the vinyl proton signals were showed at 7.66 (Ru-CH), 6.32 (β -CH), 6.14 (γ , δ , ϵ , ζ -CH) and 5.83 (η -CH) ppm. In the ^{13}C NMR spectrum (in CD_2Cl_2), the CH signals were observed at 122.49 ($-\eta$ -CH), 129.82 (ζ -CH), 131.54 (ϵ -CH), 134.15 (δ -CH), 137.88 (γ -CH), 138.95 (β -CH) and 178.79 (Ru-CH) ppm.

2.2. Crystal structures of complex **5**

Crystal structure of **5** was confirmed by X-ray diffraction study. The molecular structure of complex **5** is depicted in Fig. 1. As shown in Fig. 1, complex **5** contains two ruthenium centers linked by a linear $(\text{CH})_{14}$ bridge,

The plane of the $(\text{CH})_{14}$ chain is slightly distorted with a S-shaped conformation, and the distance between the two metal centers is 19.88 Å. The linear $(\text{CH})_{14}$ bridge shows a single/double carbon-carbon bond alternation. All the olefinic double bonds are in trans geometry. Their double bonds have an average bond distance of 1.344 Å and the single bonds have an average bond distance of 1.435 Å. The difference in the average single and double bond distances is at 0.091 Å, which is similar to those in $\text{PhCH}=\text{CH}(\text{CH}=\text{CH})_2\text{CH}=\text{CHPh}$ (0.092 Å) [15]. The vinyl group of complex **5** is essentially co-planar with Cl-Ru-CO. The atoms Cl(1), Ru(1), C(10), O(10), C(1) and C(2) are in a plane with maximum deviation from the least-square plane of 0.037 Å for Ru(1). The coplanarity of vinyl group and CO is expected because stabilization due to interaction of CO and vinyl through metal centers is maximized in such a conformation [16].

2.3. Electrochemical study

The cyclic voltammogram of complex **5** exhibited two partially reversible oxidation waves at 0.30 and 0.50 V vs Ag/AgCl, as shown in Fig. 2 (The ferrocene/ferrocenium redox couple was located at 0.49 V under our experimental conditions). These two waves can be attributed to the formation of $[(\text{PMe}_3)_3(\text{CO})\text{ClRu}-(\text{CH}=\text{CH})_7-\text{RuCl}$

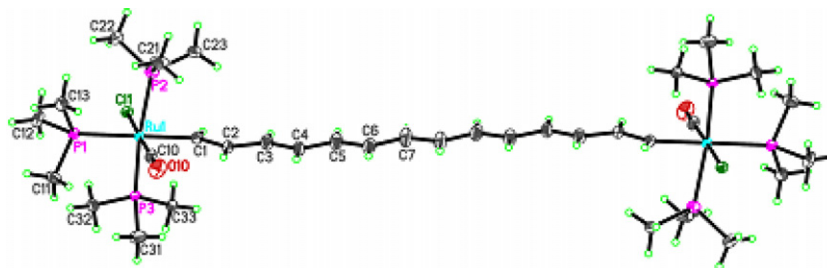


Fig. 1. The molecular structure of **5**. Important bond lengths (Å) and angles (°): Ru(1)–C(1) 2.085(5), C(1)–C(2) 1.341(7), C(2)–C(3) 1.447(8), C(3)–C(4) 1.352(8), C(4)–C(5) 1.417(8), C(5)–C(6) 1.335(8), C(6)–C(7) 1.440(9), C(7)–C(7)#1 1.351(12); C(10)–Ru(1)–C(1) 89.4(2), C(10)–Ru(1)–Cl(1) 176.53(16), C(1)–Ru(1)–Cl(1) 87.70(7), C(2)–C(1)–Ru(1) 130.7(4), C(1)–C(2)–C(3) 124.5(5), C(4)–C(3)–C(2) 127.2(6), C(3)–C(4)–C(5) 123.8(6), C(6)–C(5)–C(4) 126.9(6), C(5)–C(6)–C(7) 123.7(6), C(7)#1–C(7)–C(6) 126.3(8).

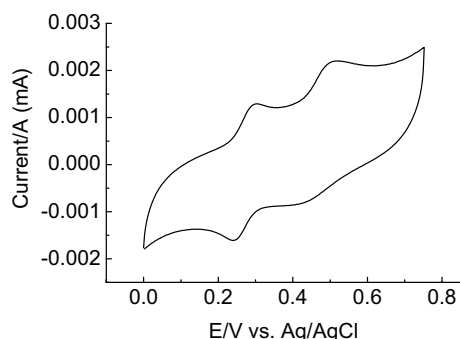


Fig. 2. Cyclic voltammogram of $[\text{RuCl}(\text{CO})(\text{PMe}_3)_2]_2[\mu-(\text{CH}=\text{CH})_7]$ (**5**).

$(\text{CO})(\text{PMe}_3)_3]^+$ and $[(\text{PMe}_3)_3(\text{CO})\text{ClRu}=\text{CH}-(\text{CH}=\text{CH})_6-\text{CH}=\text{RuCl}(\text{CO})(\text{PMe}_3)_3]^{2+}$, respectively. The peak separation of the two oxidation waves for complex **5** is at 0.20 V, which is slightly smaller than that of the bimetallic ruthenium complex $[\text{RuCl}(\text{CO})(\text{PMe}_3)_2]_2[\mu-(\text{CH}=\text{CH})_4]$ (0.24 V) [11]. The result may imply that the two metal centers in complex **5** have good electronic communications between each other.

3. Conclusion

We have prepared bimetallic $(\text{CH})_{14}$ -bridged complexes **4** and **5**. The structure of complex **5** has been confirmed by X-ray diffraction. Electrochemical study shows that the metal centers in the bimetallic complex **5** interact with each other. To date, complex **5** is the longest $(\text{CH})_x$ -bridged bimetallic complex with all the double bonds in trans geometry, which shows good electronic communication between two metals and good stability, and is a good model for molecular wire.

4. Experimental

All manipulations were carried out at room temperature under a nitrogen atmosphere using standard Schlenk techniques, unless otherwise stated. Solvents were distilled under nitrogen from sodium-benzophenone (hexane, diethyl ether, THF, benzene) or calcium hydride (dichloromethane, EtOH). The starting materials RuHCl

$(\text{CO})(\text{PPh}_3)_3$ [17], $(E)\text{-Me}_3\text{SiC}\equiv\text{CCH}=\text{CHCHO}$ [11] and $(E)\text{-Br}^-\text{Bu}_3\text{P}^+\text{CH}_2\text{CH}=\text{CHCH}_2\text{P}^+\text{Bu}_3\text{Br}^{-13}$ were prepared according to literature methods. Elemental analyses (C, H) were performed by the Microanalytical Services, College of Chemistry, CCNU. ^1H , ^{13}C and ^{31}P NMR spectra were collected on an American VARIAN MERCURY Plus 400 spectrometer (400 MHz), except for ^{13}C NMR spectra of complex **5** which was collected on a Bruker ARX-300 spectrometer (300 MHz). ^1H and ^{13}C NMR chemical shifts are relative to TMS, and ^{31}P NMR chemical shifts are relative to 85% H_3PO_4 .

The electrochemical measurements were performed on Autolab PGSTAT 30. A three-component electrochemical cell was used with a glassy-carbon electrode as the working electrode, a platinum wire as the counter electrode, and a Ag/AgCl electrode as the reference electrode. The cyclic voltammograms were collected with a scan of 50 mV/s in CH_2Cl_2 containing 0.10 M $n\text{-Bu}_4\text{NClO}_4$ as the supporting electrolyte. The peak potentials reported were referenced to Ag/AgCl. The ferrocene/ferrocenium redox couple was located at 0.49 V under our experimental conditions.

4.1. Synthesis of $(3E,5E,7E,9E,11E)\text{-HCC}-(\text{CH}=\text{CH})_5\text{-CCH}$ (**2**)

To a solution of (E) -but-2-ene-1,4-diylbis(tributylphosphonium) dibromide (0.4 g, 0.96 mmol) and $\text{TMSC}\equiv\text{CCH}=\text{CHCHO}$ (0.36 g, 2.37 mmol) in absolute ethanol (22 mL). A solution of sodium ethoxide in ethanol (1 M, 3.2 mL, 0.0032 mol) was added dropwise with stirring. After the addition was complete, the mixture was stirred 2 days. Saturated aqueous solution of sodium chloride (50 mL) was added to the mixture. The solution was extracted with hexane (4×30 mL). The organic layer were separated, and dried over MgSO_4 , filtered, and then concentrated. The residue was purified by column chromatography (silica gel, hexane) to give a yellow solid. Yield: 0.12 g, 67%. Anal. Calc. for $\text{C}_{14}\text{H}_{12}$: C, 93.29; H, 6.71. Found: C, 93.56; H, 6.83%. ^1H NMR (400.13 MHz, CDCl_3): δ 3.15 (s, 2H, $\equiv\text{CH}$), 5.61 (m, 2H, $=\text{CH}$), 6.10 (m, 2H, $=\text{CH}$), 6.37 (m, 2H, $=\text{CH}$), 6.74 (m, 2H, $=\text{CH}$), 7.16 (m, 2H, $=\text{CH}$).

4.2. Synthesis of $[RuCl(CO)(PMe_3)_3]_2[\mu-(CH=CH)_7]$ (**5**)

To a suspension of $RuHCl(CO)(PPh_3)_3$ (1.00 g, 1.05 mmol) in CH_2Cl_2 (20 mL) was added dropwisely a solution of (3*E*,5*E*,7*E*,9*E*,11*E*)- $HC\equiv C-(CH=CH)_5-C\equiv CH$ (0.10 g, 0.56 mmol) in CH_2Cl_2 (10 mL). The reaction mixture was stirred for 30 min to give a red solution. The mixture was filtered through a column of Celite. The volume of the filtrate was reduced to ca. 10 mL under vacuum. Addition of hexane (50 mL) to the residue produced a purple solid, which was collected by filtration, washed with hexane, and dried under vacuum to give complex **4** as a red solid (0.80 g). ^{31}P NMR (160 MHz, CD_2Cl_2): δ 30.94 (s).

To a solution of **4** (0.25 g) obtained from above procedure in CH_2Cl_2 (20 mL) was added a 2.5 mL THF solution of PMe_3 (1.0 M, 2.50 mmol). The reaction mixture was stirred for 15 h. The volatile materials were removed under vacuum. The crude product (red oil) was purified by column chromatography (silica gel, acetone: hexane = 1:4) to give a light yellow solid. Yield: 0.02 g, 11%. Anal. Calc. for $C_{34}H_{68}Cl_2O_2P_6Ru_2 \cdot C_6H_{14}$: C, 45.58; H, 7.84. Found: C, 45.32; H, 7.71%. ^{31}P NMR (160 MHz, CD_2Cl_2): δ -19.64 (t, $J(PP) = 21.6$ Hz), -8.03 (d, $J(PP) = 21.6$ Hz). 1H NMR (400 MHz, CD_2Cl_2): δ 1.27 (t, $J(PH) = 3.4$ Hz, 18H, PMe_3), 1.35 (d, $J(PH) = 6.8$ Hz, 36H, PMe_3), 5.83 (m, 2H, η -CH), 6.14 (m, 8 H, γ , δ , ϵ , ζ -CH), 6.32 (m, 2 H, β -CH), 7.66 (m, 2 H, Ru-CH). ^{13}C NMR (75 MHz, CD_2Cl_2): δ 16.45 (t, $J(PC) = 15.2$ Hz, PMe_3), 19.75 (d, $J(PC) = 20.1$ Hz, PMe_3), 122.49 (s, η -CH), 129.82 (s, ζ -CH), 131.54 (s, ϵ -CH), 134.15 (s, δ -CH), 137.88 (d, $J(PC) = 4.5$ Hz, γ -CH), 138.95 (t, $J(PC) = 4.4$ Hz, β -CH), 178.79 (dt, $J(PC) = 76.0$, 17.5 Hz, Ru-CH), 202.27 (q, $J(PC) = 12.5$ Hz, CO).

4.3. Crystallographic analysis for $[RuCl(CO)(PMe_3)_3]_2[\mu-(CH=CH)_7]$ (**5**)

Crystals suitable for X-ray diffraction were grown from a benzene solution layered with hexane. A yellow single crystal with approximate dimensions of $0.25 \times 0.08 \times 0.08$ mm³ was mounted on a glass fibre for diffraction experiment. Intensity data were collected on a Bruker Apex CCD Area Detector at 100 K and were corrected by Semi-empirical from equivalents. The structure was solved by Patterson methods, expanded by difference Fourier syntheses and refined by full matrix least-squares on F^2 using Bruker SHELXTL (Version 5.10) program package. The molecule is centro-symmetric with the inversion center at the midpoint of C7 and C7A, thus the crystallographic asymmetric unit contains half of one molecule. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were introduced at their geometric positions and refined as riding atoms. Further crystallographic details are summarized in Table 1.

Table 1
Crystal data and structure refinement for **5**

Formula	$C_{40}H_{82}Cl_2O_2P_6 Ru_2$
Formula weight	1053.92
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	$C2/c$
a (Å)	14.6611(18)
b (Å)	9.2608(11)
c (Å)	40.532(5)
β (°)	93.435(2)
Volume (Å ³)	5493.3(11)
Z	4
d_{calcd} (g cm ⁻³)	1.274
Absorption coefficient (mm ⁻¹)	0.850
$F(000)$	2200
θ Range (°)	2.78 to 25.00
Index ranges	$-17 \leq h \leq 17$, $-10 \leq k \leq 10$, $-48 \leq l \leq 39$
Number of reflections	11 948
Number of independent reflections	4026 [0.0407]
$[R_{\text{int}}]$	
Data/restraints/parameters	4026/0/245
Goodness-of-fit on F^2	1.056
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0493$, $wR_2 = 0.0996$
Largest difference in peak and hole (e Å ⁻³)	0.512 and -0.990

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Appendix A. Supplementary material

CCDC 634348 contains the supplementary crystallographic data for **5**. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.jorganchem.2007.04.040](https://doi.org/10.1016/j.jorganchem.2007.04.040).

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