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Communication

Chlorocarbonyl ruthenium(II) complexes of tripodal triphos $\{MeC(CH_2PPh_2)_3\}$: Synthesis, characterization and catalytic applications in transfer hydrogenation of carbonyl compounds

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ABSTRACT

The complex $[Ru(CO)_2(triphos\text{-}\kappa^2P)Cl_2]$ (**1**) underwent decarbonylation in dichloromethane solution under air over a period of about two weeks to afford the chelated monocarbonyl complex $[Ru(CO)(triphos\text{-}\kappa^3P)Cl_2]$ (**2**). The Single Crystal X-ray structure of **2** showed a slightly distorted metal centred complex. The catalytic activity of one of the complexes $[Ru(CO)(triphos\text{-}\kappa^3P)Cl_2]$ (**2**) was examined in the transfer hydrogenation of aromatic carbonyl compounds and was found to be efficient with conversion up to 100% in the presence of isopropanol/NaOH.

1. Introduction

Tridentate tripodal phosphines are recognized as one of the most important classes of ligands having widespread applications in coordination chemistry [1–10]. Such ligands provide an important advantage over monodentate phosphines with respect to greater control of the coordination number, stoichiometry and stereochemistry of their complexes [11]. Moreover, polyphosphine complexes usually have two or more chelate rings which minimize the unwanted isomers, and therefore, expected to show better catalytic activities. Among these ligands, the C_3-symmetric 1,1,1-tris(diphenylphosphinomethyl)ethane(triphos)$\{MeC(CH_2PPh_2)_3\}$ and its derivatives are the most extensively investigated ones forming a large verity of transition metal complexes which are found to have applications in catalysis [12–22]. Bianchini and coworkers in particular, pioneering the use of this ligand in transition metal catalysis using platinum group metals for a number of processes including hydrogenation, hydroformylation, oxidation, etc. [23–30]. One preliminary report for synthesis of complexes such as $[Ru(CO)_2\{MeC(CH_2PPh_2)_3\}Cl_2\}]$ was found in literature [31]. As a part of our continuing research activities [19,32–39], we report here the synthesis, detailed spectroscopic characterization of ruthenium(II) carbonyl complexes of the type $[Ru(CO)_2(triphos\text{-}\kappa^2P)Cl_2]$(**1**) and $[Ru(CO)(triphos\text{-}\kappa^3P)Cl_2]$ (**2**). The molecular structure of **2** established by Single Crystal X-ray diffraction as well as its catalytic transfer hydrogenation activities are also reported.

2. Results and discussion

The polymeric complex $[Ru(CO)_2Cl_2]_n$ reacts with equimolar quantity of the triphos ligand by cleavage of the chloro bridge to afford **1** in good yield as a yellow crystalline solid (Scheme 1). The molecular composition of the complex is well supported by elemental analyses data. The probable molecular structure of **1** (Fig. 1) was assigned by elemental analyses, IR, NMR and mass spectrometry, where the tripodal triphos ligand attached in a bidentate manner. The bidentate coordination mode of the tripodal triphos ligand in **1** is confirmed by both the IR and ^{31}P NMR spectral data. The IR spectra of **1** show two equally intense $\nu(CO)$ bands at 2054 and 1988 cm^{-1} attributing the two terminal carbonyl groups *cis* to one another. The ^1H NMR spectra of **1** show characteristics reresonance at $\delta = 2.58–2.67$ ppm ($–CH_2–$) and $\delta = 0.98$ ppm ($–CH_3–$), respectively, along with their phenylic protons in the range 6.61–8.05 ppm. The ^{31}P NMR spectra for **1** at room temperature exhibit resonances at $\delta = 34.07$ ppm for the coordinated phosphorus atoms and $\delta = -23.51$ ppm due to the free dangling phosphorus atom. The free ligand, $\{MeC(CH_2PPh_2)_3\}$, has a single resonance at $\delta = -20.32$ ppm in $CDCl_3$ at 25 °C is close to the free phosphorus in **1**. Both ^1H and ^{31}P NMR spectra are in accordance with stereochemical rigidity of **1** in $CDCl_3$ solution at 25 °C. However, attempts to develop suitable single crystal of **1** for

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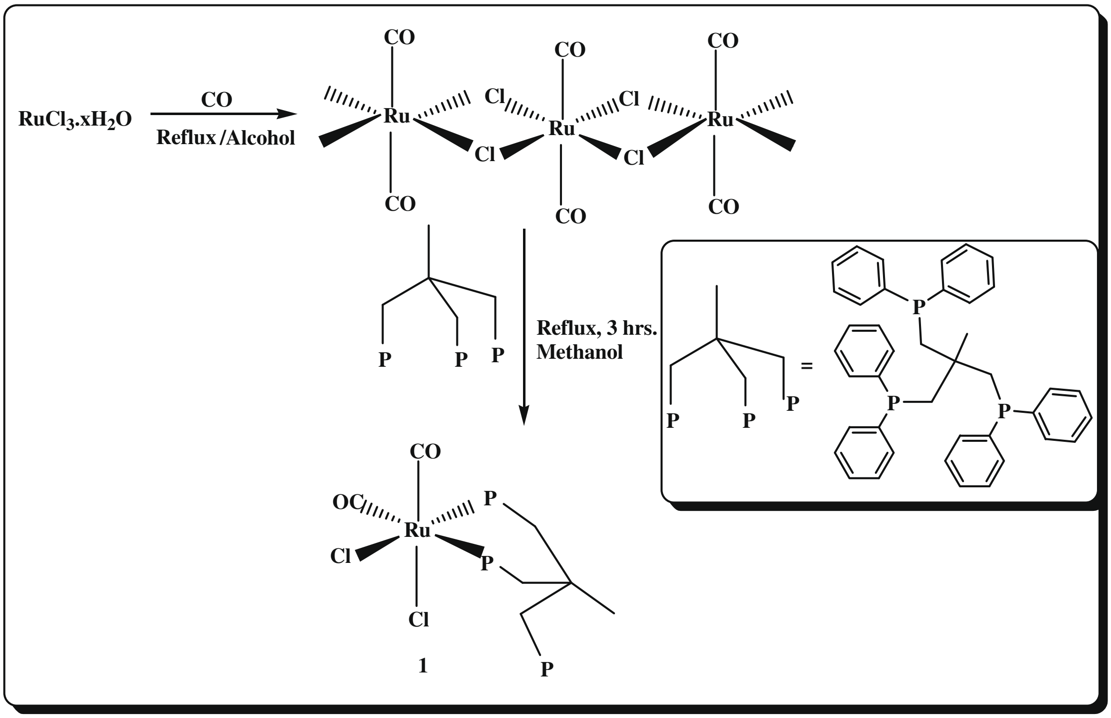


Scheme 1. Synthesis of $[Ru(CO)_2(triphos\text{-}\kappa^2P)Cl_2]$ (**1**).

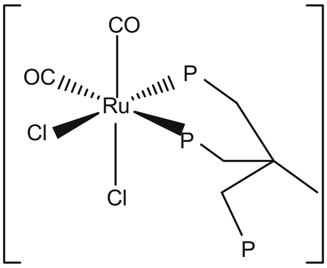
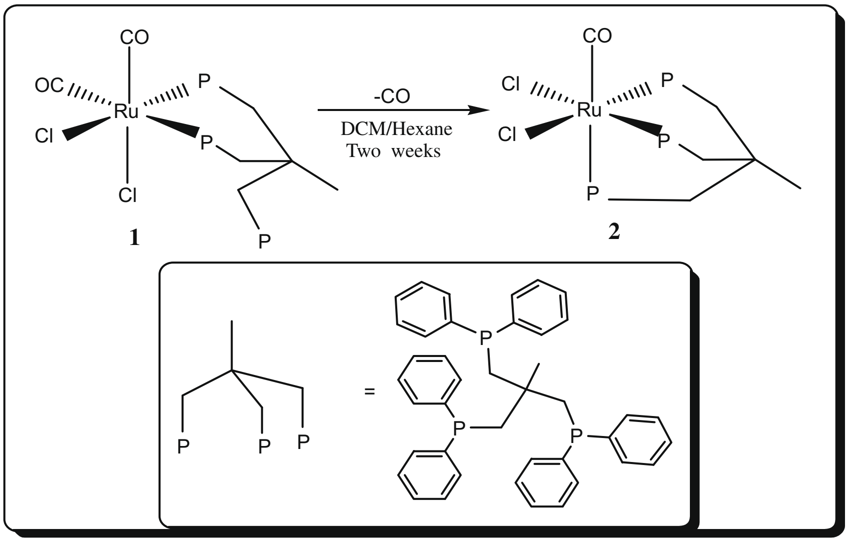


Fig. 1. Probable structure of $[Ru(CO)_2(triphos\text{-}\kappa^2P)Cl_2]$ (**1**).

X-ray analysis from dichloromethane solution under air led to the formation of κ^3-P coordinated complex $[Ru(CO)(triphos\text{-}\kappa^3P)Cl_2]$ (**2**) over a period of about two weeks in relatively good yield (Scheme 2). The resulting monocarbonyl complex exhibits single terminal ν(CO) band at 1959 cm^{-1}. Such decarbonylation reaction was also observed in ruthenium complexes of the type $[Ru(CO)_2(Ph_2PCH_2P(S)Ph_2)_2Cl_2]$ [36]. The dangling P atom in **1** underwent chelation which is substantiated by a single ^{31}P NMR resonance data. In order to obtain an unambiguous characterization of **2**, an X-ray diffraction study was undertaken. The arrangement of the atoms in the crystal is shown in Fig. 2. Ru(II) is situated in the centre of a slightly distorted octahedral coordination environment. Crystal data and structure refinement as well as some selected bond lengths (Å) and bond angles (°) for the complex **2** are



Scheme 2. Decarbonylation reaction of **1**.

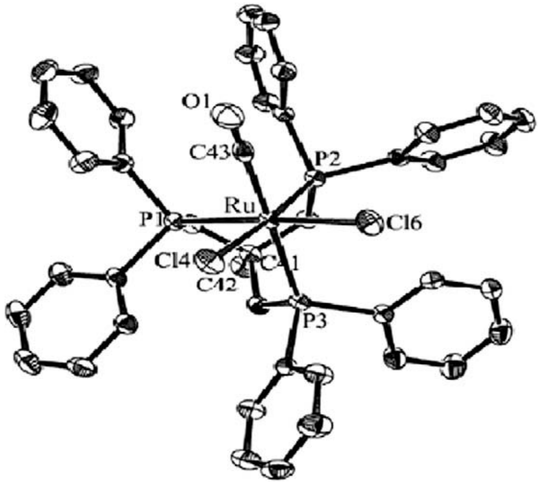


Fig. 2. Single Crystal X-ray structure of [Ru(CO)(triphos-κ^3P)Cl$_2$] (**2**) with thermal ellipsoids drawn at the 30% level. All hydrogen atoms omitted for clarity.

listed in Tables 1 and 2. The three bite angles (P–Ru–P) of triphos ligand are close to a right angle and cap a triangular face of the octahedron with *fac* stereochemistry. Two *trans* P–Ru–Cl bond angles are in the range 174.00–175.39°. The Ru–P bond lengths in **2** clearly indicate the elongation of the Ru–P(3) bond due to the strong *trans* influence of the carbonyl group. The Ru–P bond *trans* to CO is longer than those *trans* to Cl by about 0.075 Å. Thus, one of the three M–P bonds is more labile and may show hamilabile behavior by "Opening–Closing" mechanism during the course of catalytic reactions. The Ru–CO bond length (1.97 Å) falls within the range 1.81–2.08 Å observed for second and third row transition metal carbonyls [40,41].

Table 1
Crystal data and structure refinement for the complex **2**.

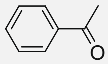
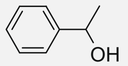
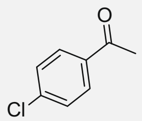
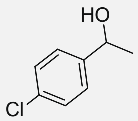
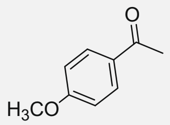
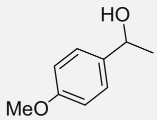
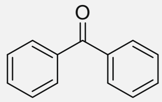
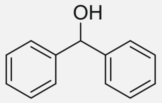
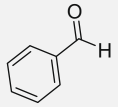
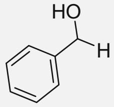
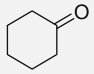
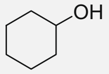
Empirical formula	$C_{42}H_{39}Cl_2OP_3Ru$
Formula weight	824.61
Temperature (K)	296(2)
Wavelength (Å)	0.71073
Crystal system	monoclinic
Space group	*Cc*
Unit cell dimensions	
a (Å)	21.8609(4)
b (Å)	10.3959(2)
c (Å)	18.5067(4)
α (°)	90
β (°)	117.467(2)
γ (°)	90
Volume (Å^3)	3731.80(13)
Z	4
Density (calculated) (Mg/m^3)	1.468
Absorption coefficient (mm^{-1})	0.725
F(0 0 0)	1688
Crystal size (mm^3)	0.40 × 0.30 × 0.16
Theta range for data collection (°)	2.95–28.24
Index ranges	$-28 \leqslant h \leqslant 29$, $-13 \leqslant k \leqslant 13$, $-24 \leqslant l \leqslant 24$
Reflections collected	19538
Independent reflections	8690 [$R_{(int)} = 0.0337$]
Completeness to theta = 28.24°	99.1%
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	8690/2/443
Goodness-of-fit on F^2	1.016
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0314$, $wR_2 = 0.0667$
R indices (all data)	$R_1 = 0.0360$, $wR_2 = 0.0692$
Absolute structure parameter	−0.016(19)
Largest diff. peak and hole (e Å^{-3})	0.661 and −0.409

Table 2
Selected bond lengths [Å] and angles [°] for the complex **2**.

Bond lengths	
Ru(1)–C(43)	1.968(4)
Ru(1)–P(2)	2.3197(7)
Ru(1)–P(1)	2.3209(7)
Ru(1)–P(3)	2.3951(7)
Ru(1)–Cl(4)	2.4648(9)
Ru(1)–Cl(6)	2.4720(8)
Bond angles	
C(43)–Ru(1)–P(2)	92.58(9)
C(43)–Ru(1)–P(1)	97.27(9)
P(2)–Ru(1)–P(1)	87.61(3)
C(43)–Ru(1)–P(3)	175.92(10)
P(2)–Ru(1)–P(3)	89.14(3)
P(1)–Ru(1)–P(3)	86.49(3)
C(43)–Ru(1)–Cl(4)	86.64(10)
P(2)–Ru(1)–Cl(4)	174.00(3)
P(1)–Ru(1)–Cl(4)	98.38(3)
P(3)–Ru(1)–Cl(4)	91.27(3)
C(43)–Ru(1)–Cl(6)	78.96(9)
P(2)–Ru(1)–Cl(6)	89.89(3)
P(1)–Ru(1)–Cl(6)	175.39(3)
P(3)–Ru(1)–Cl(6)	97.35(3)
Cl(4)–Ru(1)–Cl(6)	84.12(3)

Ruthenium mediated transfer hydrogenation reactions are found to be effective catalytic systems in which hydrogen is transferred from one organic molecule to another and this made us to carry out this type of reactions. One of the complexes [Ru(CO)(triphos-κ^3P)Cl$_2$] (**2**) is taken as model catalyst and the catalytic activity in the transfer hydrogenation of various aldehydes and ketones in the presence of isopropanol and NaOH as promoter has been explored, while **1** due to its instability does not show any catalytic properties. Complex **2** catalyzes the reduction of carbonyl compounds to corresponding alcohols with high yield and the results are shown in Table 3. The ruthenium complex catalyzed transfer hydrogenation reaction proposed to follow the classical mecha-

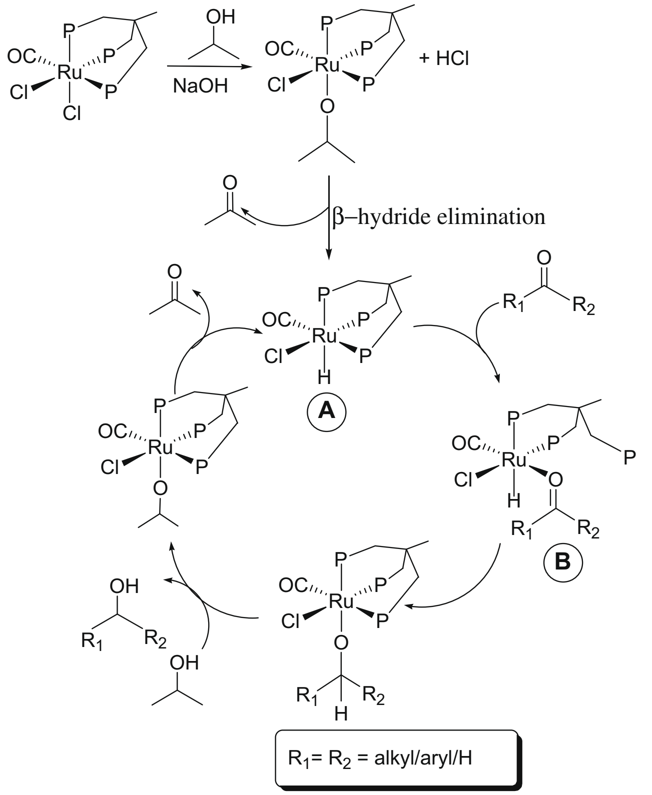
Table 3
Transfer hydrogenation of carbonyl compounds catalyzed by **2** using 2-propanol and NaOH[a].

Entry	Substrate	Product	Conversion (%)
1			85
2			91
3			75
4			94
5			98
6			100

[a] Conditions: reactions were carried out at 82 °C using 5 mmol of substrate for 24 h. substrate/Ru/NaOH ratio: 100/1/24. Yield determined by GC analysis.

nism through the formation of metal hydrides as shown in Scheme 3 [42–44]. The base facilitates the formation of a ruthenium alkoxide by abstracting the proton of the alcohol (Scheme 3) followed by β-elimination to yield a monohydride ruthenium species (**A**), which is the active catalyst. The proposed mechanism for this process involves κ^3–κ^2 dissociation of triphos ligand which is well reported in literature [17]. It is believed that during the catalytic cycle one phosphine ligating site of triphos ligand of **2** is dissociated to create vacant coordination site for the incoming substrate. The most labile phosphine site would be the one *trans* to CO. X-ray crystal structure of **2** also clearly reveals the elongation of one of the three Ru–P bonds due to the strong trans influence of CO.

Therefore, it is reasonable to assume that the phosphine ligating site of triphos ligand *trans* to CO is dissociated affording metal-alkoxide intermadiate (**B**). The complex efficiently catalyzes the reduction of cyclohexanone to the corresponding alcohol with 100% conversion. The conversion in case of acetophenone is 85%, while benzophenone is converted to its corresponding alcohol with 94% yield. The presence of electron withdrawing (Cl) and electron donating (OCH_3) substituents on the substrates (entries 2 and 3, Table 3) plays a significant role in the conversion of ketones to alcohols, which is clearly reflected for higher conversion of 4-chloroacetophenone (91%) over 4-methoxyacetophenone (75%). The above process involves reduction i.e. gaining of electrons, and is facilitated by electron deficient centers caused by the substituents [45]. Interestingly, the complex also efficiently catalyzes the reduction of benzaldehyde to benzyl alcohol with 98% yield. It was observed that the catalytic activity of **2** in terms of conversion is comparable to that of $[RuCl_2(PPh_3)_3]$ [46], but the former takes more time to achieve the required conversion. This is due to the high stability of **2** which results the slow formation of ruthenium-hydride species responsible for hydrogen-transferring process.



Scheme 3. Proposed mechanism for the transfer hydrogenation of carbonyl compounds by chlorocarbonyl Ru(II) complexes of tripodal phosphine ligand $\{MeC(CH_2PPh_2)_3\}$.

3. Experimental

3.1. General methods

All the solvents used were distilled under N_2 prior to use. Elemental analyses were done on a Perkin–Elmer 2400 elemental analyzer. IR spectra (4000–400 cm^{-1}) were recorded in KBr discs and $CHCl_3$ solution on a Perkin–Elmer system 2000 FTIR spectrophotometer. NMR spectra were recorded in $CDCl_3$ solution on a Bruker DPX-300 MHz spectrometer. $RuCl_3\cdot 3H_2O$ was purchased from M/S Arrora Matthey Ltd., Kolkata. $\{MeC(CH_2PPh_2)_3\}$ (triphos) was purchased from Aldrich, USA and used as received. The starting complex $[Ru(CO)_2Cl_2]_n$ was prepared by following literature method [47–50].

3.2. Synthesis of metal complexes

3.2.1. $[Ru(CO)_2(triphos\text{-}\kappa^2P)Cl_2]$ (**1**)

$[Ru(CO)_2Cl_2]_n$ (40 mg, 0.175 mmol) was dissolved in 10 cm^3 methanol and to this 0.0877 mmol of triphos $\{MeC(CH_2PPh_2)_3\}$ in 10 cm^3 dichloromethane was added. The reaction mixture was refluxed for about 3 h and the solvent was evaporated under vacuum to produce a yellow compound. The compound so obtained was washed with diethyl ether and recrystallized from dichloromethane solution.

*3.2.1.1. Analytical data for the complex **1**.* Yield: 92%; *Anal.* Calc. for $C_{43}H_{39}Cl_2O_2P_3Ru$: C, 60.37; H, 4.50. Found: C, 60.47; H 4.57%. Selected IR data: 1988, 2054 [ν(CO) cm^{-1}], ^{1}H NMR data: $\delta = 0.98$ ppm (3H, s), 2.58–2.67 ppm (6H, d), 6.61–8.05 ppm (30 H, m), ^{31}P NMR data $\delta = -23.51$ ppm (1P, s), 34.07 ppm (2P, s), Mass Spectral data: $m/z^+ = 853.4$.

3.2.2. $[Ru(CO)(triphos\text{-}\kappa^3P)Cl_2]$ (**2**)

Forty milligrams of the complex **1** was dissolved in dichloromethane the solution was kept aside for about two weeks. The solvent was evaporated under reduced pressure to generate yellow-red colored compounds (**2**), which was washed with diethyl ether and stored over silica gel in a desiccator.

*3.2.2.1. Analytical data for the complex **2**.* Yield: 65%; *Anal.* Calc. for $C_{42}H_{39}Cl_2OP_3Ru$: C, 61.08; H, 4.69. Found: C 61.11; H 4.73. Selected IR data: 1959 [ν(CO) cm^{-1}], ^{1}H NMR data: $\delta = 0.96$ ppm (3H, s), 2.48–2.70 ppm (6H, d), 6.95–7.95 ppm (30 H, m), ^{31}P NMR data $\delta = 16.05$ ppm (3P, s).

3.3. General procedure for catalytic transfer hydrogenation

Under inert atmosphere, the carbonyl compound (5 mmol), the ruthenium catalyst precursor (0.05 mmol) and 45 mL of propan-2-ol were introduced into a Schlenk tube fitted with a condenser and heated at 82 °C for 15 min. Then NaOH was added (5 mL of a 0.01 M solution in propan-2-ol, 9.6 mol%), and the reaction was monitored by gas chromatography. The corresponding alcohol and acetone were the only products detected in all cases. The identity of the alcohols was assessed by comparison with commercially available pure samples.

3.4. X-ray crystallographic study

The X-ray data were collected at 296 K with Mo Kα radiation ($\lambda = 0.71073$ Å) using a Bruker Nonius SMART CCD diffractometer equipped with graphite monochromator. The SMART software was used for data collection and also for indexing the reflections and determining the unit cell parameters; the collected data were integrated using SAINT software. The structures were solved by direct methods and refined by full-matrix least-squares calculations using the SHELXTL software. All the non-H atoms were refined in the anisotropic approximation against F^2 of all reflections.

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Appendix A. Supplementary material

CCDC 668233 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2010.01.011.

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