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Synthesis, molecular and crystal structure of a new dicarbonylruthenium(II) complex containing a xantphos dioxide chelating ligand

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ABSTRACT

The reaction of xantphos dioxide ($\text{O}\pi\text{O}$) with the polynuclear precursor $[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$ to give the mononuclear complex $[\text{Ru}(\text{CO})_2\text{Cl}_2(\text{O}\pi\text{O})]$ (**1**) is reported together with single crystal X-ray structure analyses of both the free ligand and the ruthenium complex. The synthesized compounds have also been characterized by elemental analyses, IR and NMR (^1H , ^{31}P and ^{13}C) spectroscopy. The ligand $\text{O}\pi\text{O}$ crystallizes with lattice water molecules in a triclinic system with space group $P\bar{1}$ whereas the complex **1** crystallizes in an orthorhombic system with space group $P2_12_12_1$. Each mononuclear unit of $\text{O}\pi\text{O}$ and **1** is stabilized in their solid state through non-covalent ($\text{C}-\text{H}\cdots\text{O}$, $\text{C}-\text{H}\cdots\text{Cl}$ and $\text{C}-\text{H}\cdots\pi$) interactions to develop an extended three-dimensional network structure. The complex **1** exhibits an interesting intramolecular $\text{O}\cdots\text{O}$ interaction between one of the $\text{P}=\text{O}$ groups and backbone O leading to different electron donicity of two $\text{P}=\text{O}$ groups to the metal centre.

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1. Introduction

The assembly of mononuclear and polynuclear organometallic complexes in the solid state is governed by a balance between non-directional close packing forces and directional interactions between metal atoms or charged groups [1]. The intermolecular interactions acting among the molecules in their solid state plays an important role to understand the solid state properties of organometallic compounds as both physical (conductivity, diffusion, magnetic susceptibility, reorientation and second harmonic generation) and chemical (solid state reactivity, racemisation, resolution) properties of a compound depend on the ways in which the molecules are organized in the crystal and on the forces which hold them together [2]. The two major types of interactions employed by chemists to engineer supramolecules with predefined dimensions (1D, 2D and 3D), are metal ligand bonding and hydrogen bonding. In addition to strong ($\text{O}-\text{H}\cdots\text{O}$) and weak ($\text{C}-\text{H}\cdots\text{O}$) hydrogen bonds; halogen bonds ($\text{C}-\text{X}\cdots\text{O}$) and weak ($\text{C}-\text{H}\cdots\pi$) interactions have also been well characterized and exploited to playing an important role in crystal engineering and organometallic structures [3–7]. The metallosupramolecules thus generated have attracted attention in recent years due to their applications in diverse areas such as catalysis, optoelectronics, gas storage, ion exchange, molecular recognition and magnetism [8–21]. To develop such extended network structures, an appropriate ligand should be chosen, which on complexation with metal ions forms ‘supra-

molecular glue’ resulting in polymeric network. As a part of our continuing research activity [22–26], herein we report the synthesis, structure and self-aggregation of oxygen functionalised robust backbone ditertiary phosphine like xantphos [9,9-dimethyl-4,5-bis(diphenyl phosphino)xanthene] and its ruthenium(II) carbonyl complex.

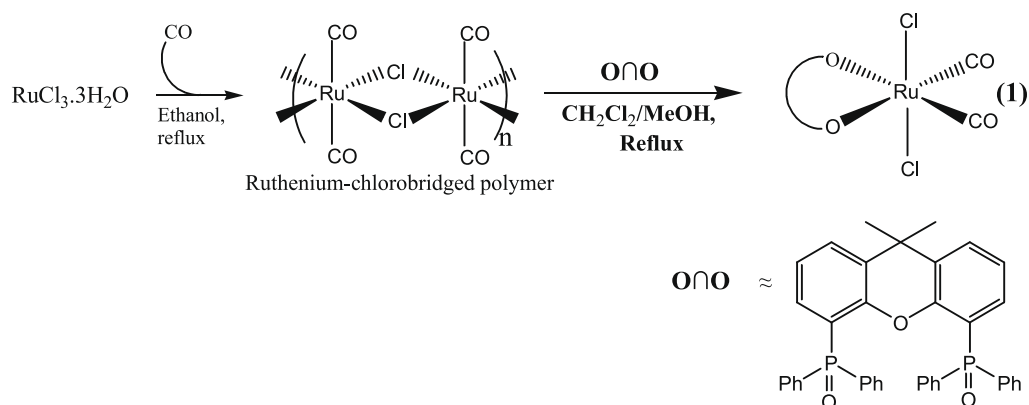
2. Results and discussion

The rigid backboned xantphos dioxide ($\text{O}\pi\text{O}$) reacts with the polymeric precursor $[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$ in 1:1 molar ratio to afford hexa-coordinated mononuclear complex $[\text{Ru}(\text{CO})_2\text{Cl}_2(\text{O}\pi\text{O})]$ (**1**) (Scheme 1). The IR spectra of **1** show two equally intense $\nu(\text{CO})$ bands at around 1973 and 2049 cm^{-1} attributing the presence of two terminal carbonyl groups only (Fig. 1). The $\nu(\text{P}-\text{O})$ band of **1** at 1187 cm^{-1} is about 6 cm^{-1} lower than that of the free ligand ($\nu(\text{P}-\text{O}) = 1193 \text{ cm}^{-1}$) revealing the coordination to metal through O-donor. The ^{13}C NMR spectrum of **1** shows only one signal for the two non-equivalent carbonyl carbons as broad singlet at around δ 195 ppm. The ^{31}P NMR spectra of **1** exhibit only one sharp signal indicating the presence of a single isomer.

The ligand $\text{O}\pi\text{O}$ and its metal complex **1** have been structurally characterized by single crystal X-ray diffraction. The crystal information for the two compounds is summarized in Table 1. The ligand $\text{O}\pi\text{O}$ crystallizes with lattice water molecules in a triclinic system with space group $P\bar{1}$. The asymmetric unit of $\text{O}\pi\text{O}$, shown in Fig. 2 consists of three xantphos dioxide moieties along with three disorder water molecules. The two $\text{P}=\text{O}$ groups of each molecule form strong hydrogen bonds with one water molecule. The

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Scheme 1. Synthesis of complex 1.

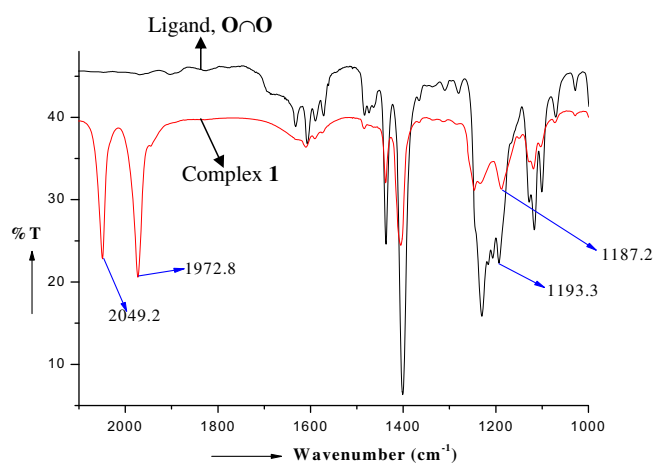


Fig. 1. IR spectra of the ligand (ONO) and complex 1.

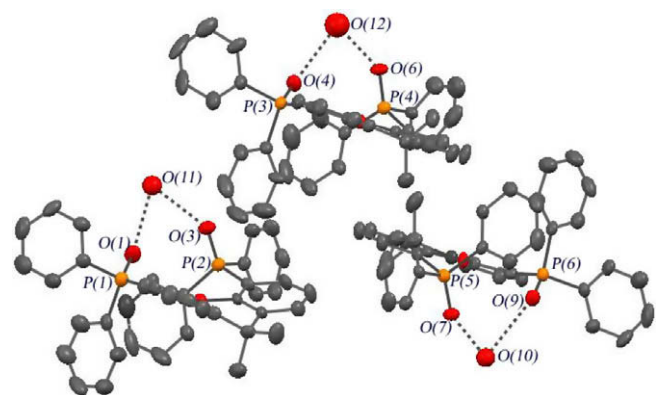


Fig. 2. Asymmetric structure of the ligand xantphos dioxide (ONO). Hydrogen atoms are omitted for clarity.

Table 1
Crystallographic data for ONO and 1.

	ONO	1
Empirical formula	C ₁₁₇ H ₉₆ O ₁₂ P ₆	C ₄₁ H ₃₂ Cl ₂ O ₅ P ₂ Ru
Formula weight	1879.76	838.64
T (K)	293	293
λ (Å)	0.71073	0.71075
Cryst. Syst.	triclinic	orthorhombic
Space group	P1	P2 ₁ 2 ₁ 2 ₁
Z	2	8
a (Å)	15.4027(5)	9.7437(19)
b (Å)	17.4463(6)	19.395(4)
c (Å)	20.3994(7)	20.072(4)
α (°)	71.715(2)	90
β (°)	70.483(2)	90
γ (°)	80.705(2)	90
μ(Mo Kα) mm ^{−1}	0.174	1.377
Reflections collected	6675	7773
Flack parameter		0.01(3)
R1 (observed data)	0.0575(4211)	0.0461(7368)
wR ₂ (all data)	0.1645(6675)	0.1399(7773)

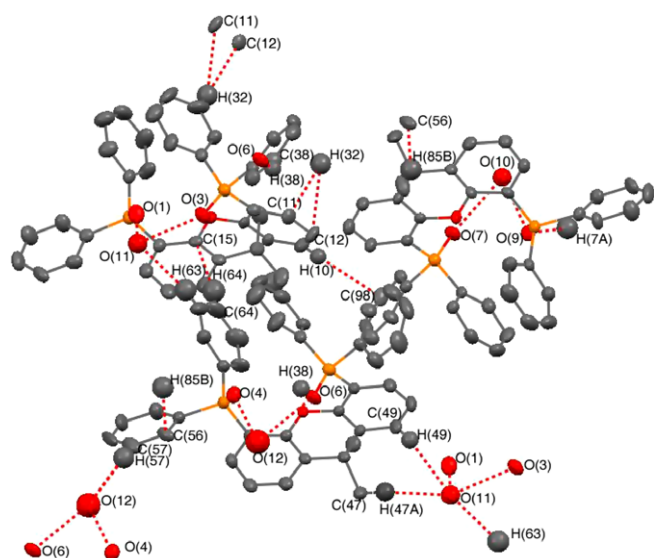
average value of P=O bond length is found to be 1.48 Å (Table 2). The compound ONO forms a three-dimensional network structure by extending infinitely via strong intermolecular C–H...O and C–H...π interactions as shown in Fig. 3. As indicated in both Figs. 3 and 4, water molecules are trapped into the network by the ligand ONO through intermolecular hydrogen bonding by bridging each molecule of xantphos dioxide generating solvent stabilized

three-dimensional network [O(3)...O(11) 2.722(3); O(1)...O(11) 2.776(2); H(49)...O(11) 2.676(1); H(63)...O(11) 2.708(2); H(47A)...O(11) 2.508(3) etc.] (Table 3). The compound loses water molecule on activating the sample at 100 °C for 1 h and this solvent loss is accompanied by the loss of transparency of the crystals. An X-ray powder diffraction analysis revealed that the solvent loss is accompanied by the loss of crystallinity, and indeed, no diffraction peaks were observed for the bulk sample.

The complex 1 crystallizes in an orthorhombic system with space group P2₁2₁2₁ where the Ru atom is situated at the centre of an octahedral environment formed by two O donors, two CO and two Cl atoms. The selected bond lengths and bond angles are presented in Table 2. The X-ray crystal structure (Fig. 5) reveals that the two Cl atoms are *trans* to each other, whereas the CO groups which are *cis* to each other are situated at the positions *trans* to two O donors. The two CO groups, two O donors and Ru metal are located in the same plane. The ligand ONO forms a chelate (bite angle O2–Ru1–O1, 85.39°) through O, O donor and forms a ten member distorted ring structure. The O1–Ru1–O2 plane is inclined by an angle 102° to the plane of the backbone of xantphos ligand. As a result of this folded structure, there appears a close intramolecular (O(1)...O(5) [2.905(4) Å, Fig. 5] interaction between the O atom of ligand backbone and one of the O atoms of P=O group of the chelating ligand. This type of O...O interaction is not commonly observed though the O–O short contact between two carbonyl groups is documented in literature [3]. The effect of the O...O interaction in 1 is reflected in CO bond order which is consistent with their corresponding Ru–C bond lengths (Table 2). The influence of O(1) *trans* to C(41) at the metal centre is less than that

Table 2Selected bond distances (Å) and angles (°) for compounds $O\pi O$ and **1**.

Ligand $O\pi O$					
P(1)–O(1)	1.476(4)	P(2)–O(3)	1.479(4)	P(5)–O(7)	1.484(4)
P(6)–O(9)	1.475(4)	P(4)–O(6)	1.478(4)	P(3)–O(4)	1.476(5)
Complex 1					
P(1)–O(1)	1.491(3)	O(1)–Ru(1)	2.118(3)	P(2)–O(2)	1.487(3)
O(2)–Ru(1)	2.118(3)	Ru(1)–Cl(1)	2.3796(17)	Ru(1)–Cl(2)	2.3739(13)
Ru(1)–C(40)	1.833(7)	C(40)–O(4)	1.129(10)	Ru(1)–C(41)	1.931(7)
C(41)–O(3)	0.973(9)				
Cl(1)–Ru(1)–Cl(2)	176.59(5)	O(1)–Ru(1)–O(2)	85.39(12)		
C(40)–Ru(1)–O(2)	176.7(2)	C(41)–Ru(1)–O(1)	172.3(2)		
C(40)–Ru(1)–C(41)	89.1(3)				

**Fig. 3.** Intermolecular short contacts in the molecule $O\pi O$. All the hydrogen atoms except those involved in hydrogen bonding have been omitted for clarity.

of O(2) trans to C(42), which may be due to O(1)···O(5) interaction. Because of the weak Ru–O interaction in the former, the electron density increases on the metal centre in the latter case resulting in more d π -electrons to the antibonding π^* orbital of the CO and hence decreases the Ru–C bond length and consequently reduces the C–O bond order (increases C–O bond length). The longer bond length of Ru1–C41 (1.931(7) Å) than Ru1–C40 (1.833(7) Å)

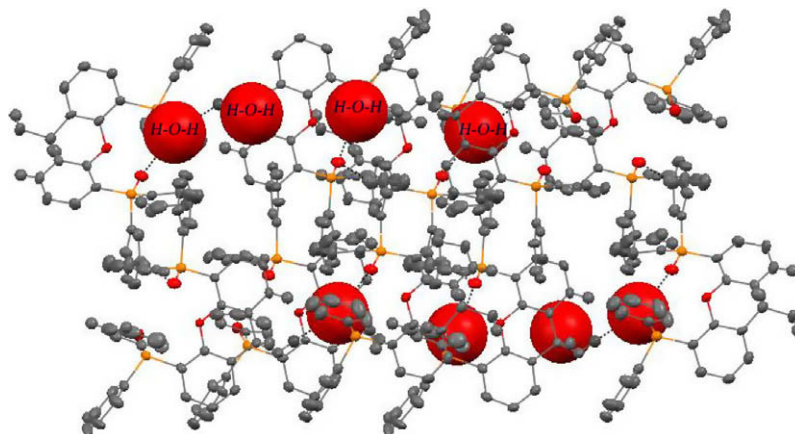
indicates a weaker interaction in the former and is therefore likely to dissociate more easily.

The complex **1** exhibit some interesting intermolecular interactions involving shorter distances than the sum of van der Waals radii (Fig. 6). The interactions between these molecules are presented by dotted lines and summarized in Table 4. The molecules are connected by weak hydrogen bondings between H atoms of the phenyl ring of xantphos ligand and O atom of CO group (C–H···OC) and Cl atom (C–H···Cl) of the adjacent molecules [C(23)–H(23)···Cl(2) 2.763(4) Å, C(36)–H(36)···Cl(2) 2.879(4) Å, C(12)–H(12)···O(4) 2.582(7) Å and C(35)–H(35)···O(3) 2.704(6) Å]. In addition to these hydrogen bondings, intermolecular C–H··· π interactions between H atoms of xantphos ligand and the phenyl ring of these molecules [C(30)–H(30)···C(33) 2.812(7) Å, C(8)–H(8)···C(24) 2.822(5) Å, and C(32)–H(32)···C(11) 2.832(8) Å] are also observed in their solid

Table 3Significant intermolecular interactions in the crystal $O\pi O$.

D–H···A	H···A/Å	D···A/Å	(D–H···A)/°
C(64)–H(64)···C(15) ⁱ	2.749(8)	3.670(1)	170.2(1)
C(38)–H(38)···O(6) ⁱⁱ	2.394(1)	3.242(2)	151.6(2)
C(47)–H(47A)···O(11) ^j	2.508(3)	3.463(9)	174.1(2)
C(57)–H(57)···O(12) ⁱ	2.377(9)	3.182(1)	144.6(1)
C(49)–H(49)···O(11) ⁱ	2.676(1)	3.532(7)	153.5(7)
C(63)–H(63)···O(11) ⁱⁱⁱ	2.708(2)	3.626(5)	169.6(1)
O(1)···O(11) ^{iv}		2.776(2)	
O(3)···O(11) ^{iv}		2.722(3)	
O(4)···O(12) ^v		2.692(1)	
O(6)···O(12) ^v		2.726(1)	
O(7)···O(10) ^j		2.869(6)	
O(9)···O(10) ⁱ		2.860(2)	

Symmetry operators are (i) x, y, z (ii) $1-x, 1-y, 1-z$ (iii) $1+x, y, z$ (iv) $1-x, y, z$ (v) $1-x, -y, 1-z$.

**Fig. 4.** 3-D network structure of $O\pi O$ containing water molecules inside the network when viewed down the c -axis.

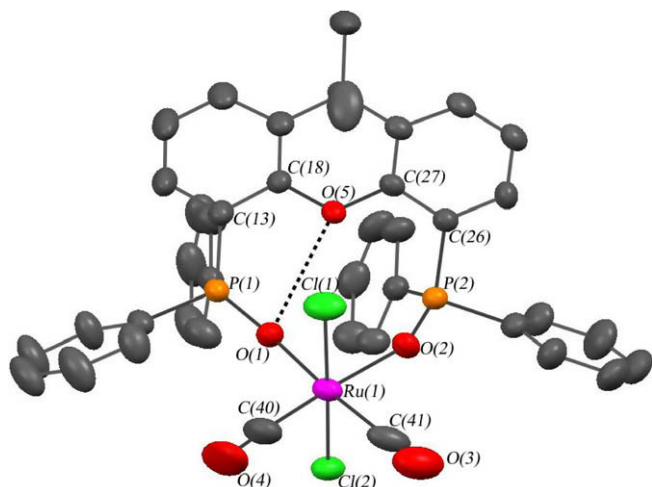


Fig. 5. X-ray crystal structure of **1**. Hydrogen atoms have been omitted for clarity.

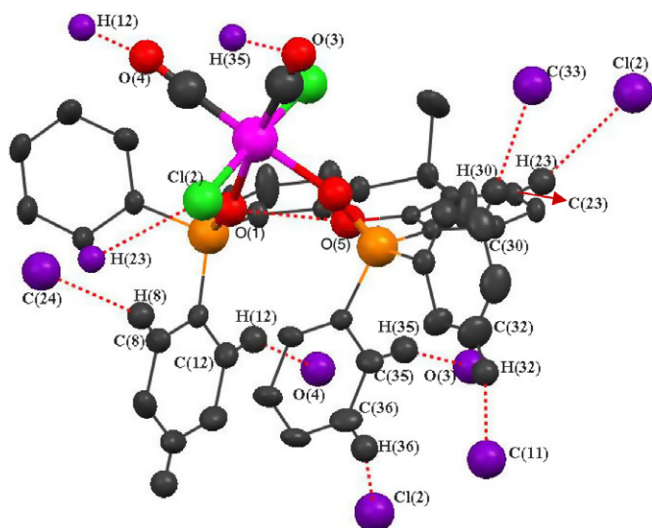


Fig. 6. Intermolecular short contacts in **1**. The atoms involved in short contact (only a few selective interactions) from surrounding molecules have been designated by dotted lines.

Table 4
Significant intermolecular interactions in the complex **1**.

D–H...A	H...A/Å	D...A/Å	(D–H...A)/°
C(23)–H(23)...Cl(2) ⁱ	2.763(4)	3.519(4)	138.9(4)
C(36)–H(36)...Cl(2) ⁱ	2.879(4)	3.729(8)	152.6(2)
C(12)–H(12)...O(4) ⁱⁱ	2.582(7)	3.369(9)	142.8(1)
C(35)–H(35)...O(3) ⁱⁱⁱ	2.704(6)	3.519(9)	146.7(7)
C(30)–H(30)...C(33) ⁱⁱⁱ	2.812(7)	3.740(8)	175.6(2)
C(8)–H(8)...C(24) ^{iv}	2.822(5)	3.679(3)	153.7(1)
C(32)–H(32)...C(11) ^v	2.832(8)	3.633(9)	149.9(5)
O(1)...O(5) ^{vi}		2.905(4)	

Symmetry operators are (i) $-x, -1/2 + y, 1.5 - z$ (ii) $-1 + x, y, z$ (iii) $1/2 + x, 1/2 - y, 2 - z$ (iv) $-x, 1/2 + y, 1.5 - z$ (v) $-1/2 - x, -1 - y, 1/2 + z$ (vi) x, y, z .

state. All these results demonstrate the extended three-dimensional network structure as shown in Fig. 7 which exhibits high thermal stability ($>300^\circ\text{C}$). This result infers that the intermolecular interactions of one mononuclear unit of **1** with the adjacent members have a profound effect on overall thermal stability of the network.

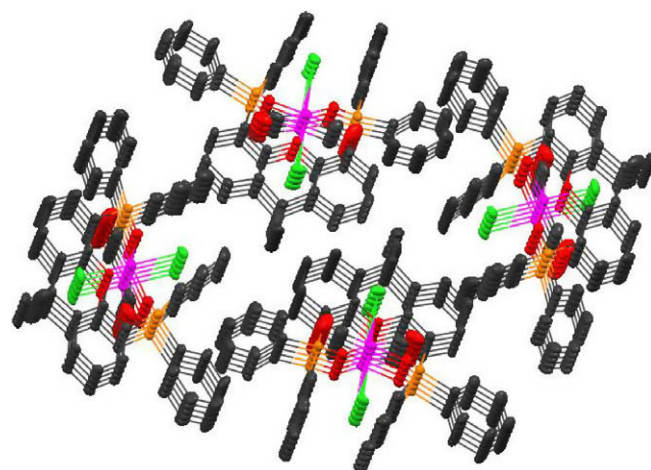


Fig. 7. 3D stacked structure of **1** stabilized by weak hydrogen bonding. Hydrogen atoms are omitted for clarity.

3. Experimental

3.1. General

All operations were carried out under nitrogen atmosphere. All solvents were distilled under N_2 prior to use. $\text{RuCl}_3 \cdot x\text{H}_2\text{O}$ was purchased from M/S Arora Matthey Ltd., Kolkata, India. The ligand xantphos was purchased from M/S Aldrich, USA and used without further purification. H_2O_2 was obtained from Ranbaxy, New Delhi, India and estimated before use.

Elemental analyses were performed on a Perkin–Elmer 2400 elemental analyzer. IR spectra ($4000\text{--}400\text{ cm}^{-1}$) were recorded on KBr discs in a Perkin–Elmer system 2000 FT-IR spectrophotometer. The ^1H , ^{13}C and ^{31}P NMR spectra were recorded in CDCl_3 solution on a Bruker DPX-300 Spectrometer and chemical shifts were reported relative to SiMe_4 and $85\% \text{H}_3\text{PO}_4$ as internal and external standards, respectively. Thermal analyses of the complexes were carried out using a thermal analyzer (TA instrument, Model STD 2960 simultaneous DTA–TGA) in presence of N_2 atmosphere with a heating rate of $10^\circ\text{C}/\text{min}$.

3.2. Synthesis of xantphos dioxide ($\text{O}^{\text{O}}\text{O}$)

The ligand xantphos dioxide was synthesized by oxidation of xantphos with H_2O_2 following the literature protocol [27].

IR (KBr, cm^{-1}): 1193 [$\nu(\text{P}=\text{O})$]. ^1H NMR (CDCl_3 , ppm): δ 6.74–7.74 (m, 30H, Ph), 2.17 (s, 6H, CH_3). ^{13}C NMR (CDCl_3 , ppm): δ 153.10–124.1 (Ar), δ 32.3, 34.9 (CH_3). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3 , ppm): δ 30.95 [s, P(v)]. Anal. Calc for $\text{C}_{39}\text{H}_{32}\text{O}_3\text{P}_2$: C, 74.40; H, 5.08. Found: C, 73.98; H, 5.09%.

3.3. Synthesis of the starting complex $[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$

The starting complex $[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$ was prepared by passing CO through a refluxing solution of $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$ in ethanol for about 24 h [28–31].

3.4. Synthesis of $[\text{Ru}(\text{CO})_2\text{Cl}_2(\text{O}^{\text{O}}\text{O})]$ (**1**)

$[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$ (0.877 mmol, 200 mg) was dissolved in methanol (10 cm^3) and xantphos dioxide (0.877 mmol, 536 mg) was dissolved in dichloromethane (10 cm^3). Both the solutions were mixed together and refluxed for 4 h. The solvent was removed and washed with diethyl ether. The resulting reddish yellow

compound was recrystallised from dichloromethane/*n*-hexane to give the complex **1** (640 mg, 87%)

IR (KBr, cm^{-1}): 2049, 1973 [$\nu(\text{CO})$], 1187 [$\nu(\text{P}=\text{O})$]. ^1H NMR (CDCl_3 , ppm): δ 6.53–7.92 (m, 30H, Ph), 1.74 (s, 3H, CH_3), 1.82 (s, 3H, CH_3). ^{13}C NMR (CDCl_3 , ppm): δ 152.78–122.13 (Ar), δ 33.59, 34.20 (CH_3), 195.64 (CO). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3 , ppm): δ 41.96 [s, P(v)]. Anal. Calc for $\text{C}_{41}\text{H}_{32}\text{Cl}_2\text{O}_5\text{P}_2\text{Ru}$: C, 57.40; H, 3.73. Found: C, 56.85; H, 3.65%.

3.5. X-ray structural analysis

Single crystals of $\text{O}=\text{O}$ and **1** were grown by layering a CH_2Cl_2 solution of $\text{O}=\text{O}$ and **1** with *n*-hexane. The intensity data of $\text{O}=\text{O}$ and **1** were collected on Bruker Smart-CCD and Rigaku Saturn CCD, respectively with Mo $\text{K}\alpha$ radiation ($\lambda = 0.71073 \text{ \AA}$). The structures were solved with SHELXS-97 [32] and refined by full-matrix least squares on F^2 using SHELXL-97 computer program [33]. Hydrogen atoms were idealized by using the riding models.

Powder XRD spectra of the compound $\text{O}=\text{O}$ were recorded on a Bruker AXS D8 ADVANCE from 2 to 80°C at 4°min^{-1} with a step size of 0.02° using Cu $\text{K}\alpha$ source ($\lambda = 1.54 \text{ \AA}$). Samples were prepared as front-coated packed powders in aluminium sample holders.

4. Conclusion

In this paper, the synthesis of ruthenium(II) carbonyl complex (**1**) containing a xantphos dioxide chelating ligand along with the structural characterization of the ligand ($\text{O}=\text{O}$) and its ruthenium complex (**1**) have been carried out. The compound $\text{O}=\text{O}$ forms a three-dimensional network structure by extending infinitely via strong intermolecular $\text{C}=\text{O} \cdots \text{O}$ and $\text{C}=\text{O} \cdots \pi$ interactions by bridging each molecule of xantphos dioxide with water molecules to generate a solvent stabilized architecture. The ligand $\text{O}=\text{O}$ reacts with $[\text{Ru}(\text{CO})_2\text{Cl}_2]_n$ to afford hexa-coordinated Ru(II) complex **1**, which generates three dimensional rigid network via intermolecular $\text{C}=\text{O} \cdots \text{O}$, $\text{C}=\text{O} \cdots \text{Cl}$ and $\text{C}=\text{O} \cdots \pi$ interactions resulting in high thermal stability ($>300^\circ\text{C}$). The complex **1** exhibit an interesting intramolecular $\text{O}=\text{O}$ interactions leading to different electron donacity of two $\text{P}=\text{O}$ groups to the metal centre.

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Appendix A. Supplementary data

CCDC 683610 and 707430 contain the supplementary crystallographic data ($\text{O}=\text{O}$) and (**1**). 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; e-mail: deposit@ccdc.cam.ac.uk.

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