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Combining two coordinatively unsaturated diruthenium cores by the tetradentate ligand $(Ph_2P)_2NCH_2C_6H_4CH_2N(PPh_2)_2$

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Dedicated to Dr. Bernhard Walther on the occasion of his 75th birthday.

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ABSTRACT

The tetradentate *N*,*N*-bis(diphenylphosphanyl)amine ligand (Ph₂P)₂NCH₂C₆H₄CH₂N(PPh₂)₂ (**1**, xdppa) reacted with in situ prepared [Ru₂(μ -H)(μ -PBu^t₂)(PBu^t₂H)₂(CO)₄] (**2**) in a molar ratio of 1:2 affording the novel twofold coordinatively unsaturated tetranuclear metal complex [Ru₂(μ -H)(μ -PBu^t₂)(CO)₄(μ -xdppa) Ru₂(μ -H)(μ -PBu^t₂)(CO)₄] (**3**). Compound **3** was protonated by tetrafluoroboric acid at each side of the unsaturated diruthenium frameworks affording the oxidative addition product [Ru₂(μ -H)₂(μ -PBu^t₂)(-CO)₄(μ -xdppa)Ru₂(μ -H)₂(μ -PBu^t₂)(CO)₄](BF₄)₂ (**4**). All new compounds were characterized by spectroscopic means and the molecular structures of compounds **1** and **4** were determined by X-ray diffraction. © 2012 Elsevier B.V. All rights reserved.

1. Introduction

Recently we reported the synthesis and X-ray crystal structures of some new coordinatively unsaturated complexes with the general formula $[Ru_2(CO)_4(\mu-H)(\mu-PBu_2^t)(\mu-P^P)]$ (P^P = diphos phanes or N,N-substituted bis(diphenylphosphanyl)amines) [1]. Because of the unsaturated character of the central $Ru_2(\mu-H)$ moiety including the Ru-Ru double bond, these complexes exhibited an enhanced reactivity towards a great variety of small molecules under mild conditions [2–10]. The most important prerequisite for the successful synthesis of all these unsaturated species is only given by the presence of the bulky μ -PBu^t₂ group. Obviously this ligand supports optimal sterical and electronical conditions to realize coordinative unsaturation. All efforts to prepare similar constituted diruthenium complexes with other substituents at the bridging phosphanido group failed. The important starting complex in the synthesis of these unsaturated species in this mind is represented by the compound $[Ru_2(\mu-H)(\mu-H)]$ $PBu_{2}^{t})(PBu_{2}^{t}H)_{2}(CO)_{4}$ [11]. Multidentate ligands offer the opportunity of connecting several small metal frameworks together, and in this direction we were interested in such a kind of synthetic strategies. In this paper we describe the synthesis, spectroscopic characterization, and the X-ray crystal structure of a novel tetraruthenium complex supported by the tetradentate bridging coordination modus of the ligand N,N-bis(diphenylphosphanyl)-pxylylene-diamine, (Ph₂P)₂NCH₂C₆H₄CH₂N(PPh₂)₂ (**1**), between two coordinatively unsaturated diruthenium frameworks.

2. Experimental

2.1. General considerations

All synthetic operations were performed under anaerobic conditions using conventional Schlenk techniques. Solvents were dried over sodium-benzophenone ketyl or molecular sieves and were distilled under argon prior to use. Compound **1** was prepared as reported in the literature [12] however with some modifications as described below. Chemicals were purchased commercially from Aldrich. IR spectra were recorded as solid with a JASCO FT/IR-460 plus spectrometer. NMR spectra were obtained using Jeol Eclipse 270 and 400 instruments operating at 270 and 400 (¹H), 69 (¹³C), and at 109 MHz (³¹P), respectively. Chemical shifts are given in ppm from SiMe₄ (¹H, ¹³C{¹H}), and 85% H₃PO₄ (³¹P{¹H}). Microanalyses (C, H, N) were performed by the Microanalytical Laboratory of the Department of Chemistry, LMU Munich, using a Heraeus Elementar Vario El instrument.

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2.2. Synthesis of $(Ph_2P)_2NCH_2C_6H_4CH_2N(PPh_2)_2$ (1)

A solution of *p*-xylylenediamine (1.16 g, 8.50 mmol) in dichloromethane (100 mL) was treated with triethylamine (20 mL) by stirring at 0 °C. To this solution chlorodiphenylphosphane (3.66 mL, 19.07 mmol) was added and stirred for 30 min. Then chlorodiphenylphosphane (3.66 mL, 19.07 mmol) was added again and the mixture was stirred for 18 h at room temperature. The solvent was evaporated to dryness under reduced pressure, and the remaining residue was treated with water (50 mL) by stirring. The resulting white precipitate was filtered off and washed with water (50 mL), twice with ethanol (40 mL), and twice with hexane (50 mL), and dried in vacuo. The product was recrystallized from dichloromethane/acetonitrile (1:2) affording colorless crystal plates, m.p. 195 °C.

1: Yield: 3.90 g (53%). Anal. Calcd for C₅₆H₄₈N₂P₄ (872.91): C, 77.05; H, 5.54; N, 3.21. Found: C, 77.36; H, 5.82; N, 3.14%. ¹H NMR (270 MHz, CD₂Cl₂): δ 7.32–7.23 (m, 40H, PC₆H₅), 6.45 (s, 4H, C₆H₄), 4.37 (t, 4H, ³*J*_{PH} = 10.5 Hz, N–CH₂). ³¹P{¹H} NMR (109 MHz, CD₂Cl₂): δ 60.3 (s). ¹³C{¹H} NMR (69 MHz, CD₂Cl₂): δ 139.8, 139.0 (C₆H₄), 133.2, 129.1, 128.9 (C₆H₄), 128.4, 55.9 (t, ²*J*_{PC} = 12.9 Hz, CH₂). Suitable single crystals of **1** for X-ray diffraction were obtained from dichloromethane/acetonitrile at 4 °C overnight.

2.3. Synthesis of $[Ru_2(\mu-H)(\mu-PBu_2^t)(CO)_4$ $\{\mu-(Ph_2P)_2NCH_2C_6H_4CH_2N(PPh_2)_2\}Ru_2(\mu-H)(\mu-PBu_2^t)(CO)_4]$ (**3**)

A mixture of $[Ru_3(CO)_{12}]$ (639 mg, 1.00 mmol) and PHBu^t₂ (1.00 mL, 5.40 mmol) in THF (20 mL) was refluxed for 3 h. The solvent was evaporated to dryness under reduced pressure and the remaining residue was dissolved in acetone (20 mL). Then *p*-xyly-lene-di-bis(diphenylphosphanyl)amine (436 mg, 0.50 mmol) was added and the mixture was refluxed for 4 h. The solvent was evaporated to dryness under reduced pressure and the residue was recrystallized from diethyl ether/ethanol (1:2) affording **3** as red powder, m.p. 215–217 °C dec.

3: Yield: 807 mg (45%). Anal. Calcd for $C_{80}H_{86}N_2O_8P_6Ru_4$ (1793.69): C, 53.57; H, 4.83; N, 1.56. Found: C, 53.46; H, 4.92; N, 1.74%. IR (solid, cm⁻¹): ν (CO): 1974s, 1928s. ¹H NMR (270 MHz, CD₂Cl₂): δ 7.54–7.25 (m, 44H, Ph and C₆H₄), 4.02 (t, 4H, ³J_{PH} = 7.1 Hz, CH₂), 1.40–1.19 (m, 36H, PC₄H₉), -14.69 (m, 2H, μ -H). ³¹P{¹H} NMR (109 MHz, CD₂Cl₂): δ 279.8 (t, ²J_{PP} = 143.2 Hz, μ -*P*But₂), 111.9 (d, ²J_{PP} = 143.2 Hz, μ -xdppa).

2.4. Synthesis of $[Ru_2(\mu-H)_2(\mu-PBu^t_2)(CO)_4$ { $\mu-(Ph_2P)_2NCH_2C_6H_4CH_2N(PPh_2)_2$ } $Ru_2(\mu-H)_2(\mu-PBu^t_2)(CO)_4$](BF₄)₂ (**4**)

Compound **3** (538 mg, 0.3 mmol) was dissolved in diethyl ether (20 mL) with stirring. To this mixture a solution of HBF₄ (51%, 0.6 mmol, 86.8 μ L) in diethyl ether (10 mL) was added. Within 1 h the color of the solution changed from red to yellow and a yellow precipitate was obtained. The solid was filtered off and washed three times with diethyl ether (10 mL). The yellow powder was dried in vacuo. The product was recrystallized from dichloromethane/diethyl ether affording **4** as yellow needles suitable for X-ray diffraction, m.p. 236–238 °C dec.

4: Yield: 460 mg (78%). Anal. Calcd for $C_{80}H_{88}B_2F_8N_2O_8P_6Ru_4$ (1969.32): C, 48.79; H, 4.50; N, 1.42. Found: C, 49.06; H, 4.62; N, 1.49%. IR (solid, cm⁻¹): ν (CO): 2060sh, 2040s, 1995s, 1970sh. ¹H NMR (270 MHz, CD₂Cl₂): δ 7.65–7.43 (m, 44H, Ph and C₆H₄), 4.15 (t, 4H, ³J_{PH} = 6.9 Hz, CH₂), 1.37 (d, 36H, ³J_{PH} = 15.8 Hz, PC₄H₉), -14.03 (dt, 4H, ²J_{PH} = 19.8 Hz, ²J_{PH} = 13.9 Hz, μ -H). ³¹P{¹H} NMR (109 MHz, CD₂Cl₂): δ 240.2 (t, ²J_{PP} = 160.8 Hz, μ -PBu^t₂), 106.6 (d, ²J_{PP} = 160.8 Hz, μ -xdppa).

2.5. X-ray structural determination

Suitable single crystals for X-ray diffraction of the compounds **1** and **4** were obtained as described in the Experimental section. Crystals were selected by means of a polarization microscope, mounted on the tip of a glass fiber, and investigated on a Bruker Nonius-Kappa CCD diffractometer (**1**) and on an Oxford XCalibur diffractometer (**4**) using Mo-K α radiation ($\lambda = 0.71073$ Å). The structures were solved by direct methods (SHELXS) [15] and refined by full-matrix least-squares calculations on F^2 (SHELXL-97) [16]. The positions of the bridging hydrido ligands in **4** were free refined. Thus the Ru–H distances were set to equal lengths and free refined. Details of the crystal data, data collection, structure solution, and refinement parameters of compounds **1** and **4** are summarized in Table 1.

3. Results and discussion

3.1. Preparation and characterization of new compounds

During our studies on the coordination behavior of the sterically demanding secondary phosphane PHBu^t₂ we observed a cluster degradation of [Ru₃(CO)₁₂] affording the coordinatively unsaturated complex $[Ru_2(\mu-H)(\mu-PBu_2^t)(PBu_2^tH)_2(CO)_4]$ (2) [11]. The latter species served as an useful intermediate in the synthesis of the dinuclear complex $[Ru_2(CO)_4(\mu-H)(\mu-PBu_2^t)(\mu-dppm)]$ (Ru=Ru) $(5, dppm = Ph_2PCH_2PPh_2)$ [2]. Recently we could apply these findings to a series of other bidentate organophosphorus ligands bridging the unsaturated diruthenium core [1]. In this light we were also interested to study synthetic pathways affording multinuclear complexes supported by multidentate *N*,*N*-bis(diphenylphosphanyl) amine ligands. In 2002 Gaw et al. described the ligand p-xylylenedi-bis(diphenylphosphanyl)amine, (Ph₂P)₂NCH₂C₆H₄CH₂N(PPh₂)₂ (1, xdppa). This ligand afforded in the reaction with 2 mol of $[Mo(CO)_4(\eta^4-nbd)]$ (nbd = norbornadiene) the dinuclear complex $[Mo(CO)_4[\mu-(Ph_2P)_2NCH_2C_6H_4CH_2N(PPh_2)_2]Mo(CO)_4]$ [12]. In a similar manner we studied the reaction of compound **2** with **1** in a molar ratio of 2:1 in refluxing acetone. Thus the tetranuclear

Table 1
Crystal data and structure refinement details for compounds 1 and 4

Compound	1	4 3CH ₂ Cl ₂
Empirical formula	C ₅₆ H ₄₈ N ₂ P ₄	C83H94B2Cl6F8N2O8P6Ru4
Formula weight	872.84	2224.02
Temperature (K)	173(2)	203(2)
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a (Å)	16.2534(6)	10.3648(8)
b (Å)	9.3947(3)	16.4147(15)
<i>c</i> (Å)	16.1459(6)	31.926(2)
α (°)	90	90
β(°)	112.289(2)	99.867(6)
γ (°)	90	90
Volume (Å ³)	2281.20(14)	5351.4(7)
Ζ	2	2
ρ_{calcd} (g cm ⁻³)	1.271	1.380
μ/mm^{-1}	0.206	0.853
heta range for data collection (°)	3.33-27.50	4.16-25.43
Reflections measured	18 229	24 880
R _{int}	0.1054	0.0485
Observed reflections	5232	7987
Reflections, unique	9128	9780
Parameters/restraints	280/0	575/4
$R(F_{obs})$	0.0500	0.0597
$R_{\rm w}(F^2)$	0.1209	0.1955
S	1.039	1.075
Max electron density (e ⁻³)	0.285	1.688
Min electron density (e^{-3})	-0.301	-1.125



complex [Ru₂(μ -H)(μ -PBu^t₂)(CO)₄(μ -xdppa)Ru₂(μ -H)(μ -PBu^t₂)(CO)₄] (**3**) was obtained in good yields (Scheme 1). The new twofold coordinatively unsaturated complex **3** was characterized by analytical and spectroscopic methods (see Experimental section). The ³¹P{¹H} NMR spectrum of **3** is comparable to that of **5**. Thus a pattern of two signals was found, namely a triplet corresponding to the μ -PBu^t₂ group at down field and a doublet at higher field indicating the bridging ligand xdppa. The found coupling constant ²*J*_{PP} of **3** is comparable to the observed one of **5** (143.2 Hz vs. 134.5 Hz). This is an indication of a similar structural ligand environment in both complexes. Even the ¹H NMR data of **3** exhibited a signal corresponding to hydrido ligands in the high field region at –14.69 ppm as a multiplet (compare for **5**: –14.30 ppm; CD₂Cl₂).

Despite of intense efforts we were unable to obtain suitable crystals of **3** for an X-ray crystallographic study to confirm its molecular structure. Therefore we searched for another possibility to bring more insight in its structural elucidation. In this context we found that compound **5** could be protonated by HBF₄ in an oxidative addition reaction affording the complex salt [Ru₂(CO)₄(μ -H)₂(μ -PBu^t₂)(μ -dppm)]BF₄ (**6**) [5]. Thus we investigated the reaction of **3** with tetrafluoroboric acid under similar conditions. Indeed, treatment of **3** with HBF₄ in diethyl ether (molar ratio 1:2) resulted in an analogous oxidative addition reaction indicated by a color change from deep red to yellow. After work up by crystallization the corresponding addition product between **3** and 2 mol HBF₄ could be isolated in good yields (Scheme 2). The novel complex salt [Ru₂(μ -H)₂(μ -PBu^t₂)(CO)₄(BF₄)₂ (**4**) was characterized by analytical and

spectroscopic methods (see Experimental section). Moreover we obtained suitable crystals for an X-ray diffraction study and we could confirm the composition and the structure of **3** indirectly by the formation of its derivative **4**.



Fig. 1. Molecular structure of **1** in the crystal. Thermal ellipsoids were drawn at the 50% probability level. The asymmetric unit contains a half molecule of **1**. Symmetry operation: i - x, -y, 1 - z. Selected bond lengths (Å) and angles (°): P1–N, 1.718(2); P2–N, 1.7225(19); N–C1, 1.489(3), P1–C5, 1.832(2); P1–C11, 1.837(2); P2–C17, 1.820(3); P2–C23, 1.832(2). P1–N–P2, 120.55(12), P1–N–C1, 119.25(15); P2–N–C1, 114.45(16). Maximal deviation from the least square plane: P1–P2–N–C1, 0.171(2) Å (N).



Fig. 2. Molecular structure of the dication of **4** in the crystal. Thermal ellipsoids were drawn at the 50% probability level. Hydrogen atoms and the solvate molecules are omitted for clarity (except hydrido ligands). Symmetry operation: ${}^{i}2 - x$, 1 - y - z. Selected bond lengths (Å) and angles (°): Ru1–Ru2, 2.6148(7); Ru1–H1, 1.85(7); Ru1–H2, 1.75(6); Ru2–H1, 1.74(6); Ru2–H2, 1.70(6); Ru1–P1, 2.3906(14); Ru2–P1, 2.3587(15); Ru1–P2, 2.3899(14); Ru2–P3, 2.3601(14); P2–N, 1.705(5); P3–N, 1.719(4); N–C5, 1.515(7). Ru1–H1–Ru2, 93.4(39), Ru1–H2–Ru2, 98(4); Ru1–P1–Ru2, 66.81(4); P2–N–P3, 121.9(2). Maximal deviation from the least square plane: P2–P3–N–C5, 0.120(5) Å (N).

Some remarks on the spectroscopic investigation during the transformation from **3** to **4** in comparison with the analogous conversion from 5 to its corresponding derivative 6 should be given. During the protonation of the Ru–Ru bond on going from 5 to 6, a shortening of this bond was observed: 2.6974(4) to 2.6486(6) Å. This was in accordance with a small decrease in the angle Ru–(μ -P)–Ru: 70.46 to 68.19(3)°. In contrast to this the value $\delta(^{31}P)$ of the bridging phosphanido group changed from δ 292.3 to 254.2 ppm. In the literature a smaller angle M–(μ -P)–M was often discussed in connection with shifting of these resonance signals to down field [13]. However in the present case, one should take into account that different oxidation states have to be considered. In this light we assume similar structural changes during the conversion of 3 to 4. Thus the Ru–Ru bond in 3 should be longer than 2.6148(7) Å (see data of **4**). Following the trend of the $\delta(^{31}P)$ values for **5** and **6**, for which a difference of about 38 ppm was found, also for **3** and **4** a difference of about 39 ppm was observed. Moreover, a similar comparison of the CO stretching frequencies can be made. In going from **5** to **6**, an increase of the $\nu(CO)$ bands was observed (in the average from 1955 cm^{-1} for **5**–2035 cm^{-1} for **6**). The same trend was found for the change from 3 to 4 (in the average from 1951 cm⁻¹–2015 cm⁻¹). These findings are in accordance with the assumption of a change in the oxidation state from Ru¹ in the coordinatively unsaturated species to Ru^{II} in the product of the oxidative addition reaction. Using these aspects, the shortening of the M-M bonds upon "protonation" is in agreement with the fact that the radii of the central atoms should decrease with increasing the oxidation state, and therefore they come closer to one another.

3.2. Molecular structures of compounds 1 and 4

Compound **1** crystallizes from dichloromethane/acetonitrile in the monoclinic space group $P2_1/c$ with two formula units in the unit cell. The asymmetric unit contains a half molecule xdppa. A selected representation of molecule **1** is shown in Fig. 1, selected bond lengths and angles are summarized in the caption. As found for other similar constituted *N*,*N*-bis(diphenylphosphanyl)amines [14], also in **1** the diphenylphosphanyl groups are staggered relative to the PNP backbone. The angles around the central N atom (see caption in Fig. 1) are comparable e.g. with those in *N*,*N*-bis(diphenylphosphanyl)benzylamine: P1–N1–P2, 120.57(12); P2–N1–C25, 120.84(16) and P1–N1–C25, 113.29(16)° [14a]. These bonding characteristics indicate that these molecules exhibit a distorted trigonal-pyramidal geometry, whereas the N atom is surrounded

nearly trigonal-planar from the carbon and the both phosphorus atoms.

Compound **4** crystallizes from dichloromethane/diethyl ether in the monoclinic space group $P_{2_1/c}$ with two formulas in the unit cell. The asymmetric unit contains a half molecule of the cationic complex $[Ru_2(\mu-H)_2(\mu-PBu^t_2)(CO)_4(\mu-xdppa)Ru_2(\mu-H)_2(\mu-PBu^t_2)(CO)_4]^{2+}$, one tetrafluoroborate ion, and one and a half solvate molecule CH_2Cl_2 . A selected representation of the dication of compound **4** is shown in Fig. 2, selected bond lengths and angles are summarized in the caption.

The cationic complex of compound **4** exhibits two diruthenium tetracarbonyl cores bridged by two hydrido ligands and a phosphanido group. These dimetal frameworks are connected through the bridging ligand xdppa. The overall structural features of the complex ion are comparable to those of the known compound $[Mo(CO)_4{\mu-(Ph_2P)_2NCH_2C_6H_4CH_2N(PPh_2)_2}Mo(CO)_4]$ [12]. As observed for the dimolybdenum species, the complex cation of **4** possesses an inversion center in the middle of the central C_6H_4 ring, too. Because of the formation of five-membered chelate rings in the complex cation of **4**, a greater stability in contrast to the dimolybdenum complex should be assumed.

4. Conclusions

In this paper we described the synthesis and structural characterization of two new tetranuclear ruthenium complexes containing the tetradentate *N*,*N*-bis(diphenylphosphanyl)amine ligand, (Ph₂P)₂NCH₂C₆H₄CH₂N(PPh₂)₂ (**1**, xdppa). As described for coordinatively unsaturated diruthenium complexes of the type [Ru₂(CO)₄(μ -H)(μ -PBu^t₂)(μ -P^P)] (P^P = diphosphanes), the new twofold coordinatively unsaturated complex [Ru₂(μ -H)(μ -PBu^t₂)(-CO)₄(μ -xdppa)Ru₂(μ -H)(μ -PBu^t₂)(CO)₄] (**3**) showed a similar reactivity towards HBF₄ and afforded an oxidative addition pattern of reactivity simultaneously at both unsaturated diruthenium cores. In the future we want to look at new reactivity patterns to functionalize the tetranuclear framework further, especially at the end of the dinuclear cores.

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Appendix A. Supplementary material

CCDC-877530 (1) and CCDC-877531 (4) contain 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.

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