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New dinuclear arene ruthenium complexes with P,S- or P,O-chelating ligands

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Abstract

Two equivalents of 2-diphenylphosphinobenzoic acid react with 1,2-ethanedithiol and 1,8-diaminonaphthalene under peptidic coupling conditions to give the new ligands 1,2-bis-*S*-[2′-(diphenylphosphino)benzoyl]dithioethane (dppte) (**1**) and 1,2-bis-*N*-[2′-(diphenylphosphino)benzoyl]diaminonaphthalene (dppan) (**2**), respectively. **1** and **2** have been characterised by mass spectrometry, elemental analysis, NMR, IR spectroscopy, and by single-crystal X-ray structure analysis. **2** is easily oxidised by air to give the monophosphine oxide derivatives (**3**). Single-crystal X-ray structure analysis of **3** shows an intramolecular hydrogen bond between an amido and the phosphoryl oxygen atom. Compounds **1** and **2** react with $[RuCl_2(\eta^6\text{-}p\text{-cymene})]_2$ to give the dinuclear complexes $[RuCl(\eta^6\text{-}p\text{-cymene})(dppte)RuCl(\eta^6\text{-}p\text{-cymene})]^{2+}$ (**4**) and $[RuCl(\eta^6\text{-}p\text{-cymene})(dppan)RuCl(\eta^6\text{-}p\text{-cymene})]^{2+}$ (**5**). As determined by single-crystal X-ray structure analysis, **4** and **5** adopt different coordination modes to the ruthenium atoms. In **4** the symmetric dppte ligand is P,S coordinated to the ruthenium atom, whereas in **5** the dppan ligand prefers a P,O coordination mode.

Keywords: Diphosphine ligand; Dinuclear complex; Ruthenium

1. Introduction

Phosphine ligands have been intensively used in coordination chemistry because of their electron-donating power [1]. Diphosphine ligands have received particular attention, because in general they form more stable complexes than their non-chelating phosphine analogues under the harsh reaction conditions required for catalysis. Diphosphine ligands have been used in particular for the synthesis of catalysts for allylic alkylation [2] and for methanol carbonylation [3]. Tetradentate C_2-symmetric ligands, possessing two phosphine groups and two other heteroatoms which increase the electron density at the metal centre, have been intensively studied [3–7]. Ligands of this type have been prepared to optimise the catalytic potential of their transition metal complexes [3]. Butts et al. [8] have shown that these ligands can form dinuclear complexes with an excess of metal precursor. Variations at the donor sites as well as in the backbone of the ligand may modify the coordination properties.

Herein we present the synthesis of two new C_2-symmetric ligands possessing mixed donor sites, and their coordination to arene ruthenium units.

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2. Experimental

2.1. General remarks

Dichloromethane was dried and distilled under nitrogen prior to use. All reactions were carried out under nitrogen, using standard Schlenk techniques. All other reagents were purchased (Aldrich, Fluka) and used as received. Nuclear magnetic resonance spectra were recorded using a Varian Gemini 200BB instrument and referenced to the signals of the residual protons in the deuterated solvents. Electro-spray mass spectra were obtained in positive-ion mode with an LCQ Finnigan

mass spectrometer. IR spectra were recorded with a Perkin–Elmer Spectrum One FTIR spectrometer. Microanalyses were carried out by the Laboratory of Pharmaceutical Chemistry, University of Geneva, Switzerland. The starting dinuclear dichloro complex $[RuCl_2(\eta^6\text{-}p\text{-cymene})]_2$ [9] was prepared according to published method.

2.2. Preparation of dppte (*1*)

A solution of 2-diphenylphosphinobenzoic acid (1.0 g, 3.26 mmol), *N*,*N*-dicyclohexylcarbodiimide (2.7 g, 13.05 mmol), 4-(dimethylamino)pyridine (100 mg, 0.82 mmol), 4-pyrrolidinopyridine (100 mg, 0.68 mmol), and 1,2-ethanedithiol (152 mg, 1.62 mmol) in CH_2Cl_2 (40 mL) was stirred at room temperature under nitrogen, until the esterification was complete, as monitored by TLC. The resulting solution was concentrated and filtered three times to remove excess *N*,*N*-dicyclohexyl urea. The filtrate was concentrated under reduced pressure. A chromatogram of the residue was recorded on a silica gel column, eluting with dichloromethane/ethanol (50/1). The product was isolated from the first fraction, giving **1** as a yellow solid (640 mg, 0.94 mmol; 58%).

^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 8.01$–6.97 (m, 28H; ArH), $\delta = 2.99$ ppm (s, 4H; CH_2); $^{13}C\{^1H\}$ NMR (50 MHz, $CDCl_3$): δ = 168.12, 143.45, 141.20, 141.00, 140.50, 137.62, 137.39, 134.62, 133.90, 130.84 129.68, 128.94, 128.80, 128.65 ppm; $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); $\delta = -3.81$ ppm; IR (KBr): 1652 s (C═O) cm^{-1}; ESI-MS: *m*/*z*: 671 $[M + H^+]$; elemental *Anal*. Calc. (%) for $C_{40}H_{32}O_2P_2S_2$ (670.13): C, 71.6; H, 4.8; Found: C, 71.2; H, 4.6%.

2.3. Preparation of dppan (*2*)

A solution of 2-diphenylphosphinobenzoic acid (1.0 g, 3.26 mmol), *N*,*N*-dicyclohexylcarbodiimide (2.7 g, 13.05 mmol), 4-(dimethylamino)pyridine (100 mg, 0.82 mmol), 4-pyrrolidinopyridine (100 mg, 0.68 mmol), and 1,8-diaminonaphthalene (253 mg, 1.62 mmol) in CH_2Cl_2 (40 mL) was stirred at room temperature under nitrogen, until esterification was complete, as monitored by TLC. The resulting solution was concentrated and filtered three times to remove excess *N*,*N*-dicyclohexyl urea. The filtrate was concentrated under reduced pressure. A chromatogram of the residue was recorded on a silica gel column, eluting with hexane/acetone (3/1). The product was isolated from the fourth fraction, giving **2** as a white solid (300 mg, 0.44 mmol; 27%).

^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 9.30$ (sl, 2H; NH), 7.8–6.9 ppm (m, 34H; ArH); $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); $\delta = -11.03$ ppm; IR (KBr): 3369 m (N–H amide), 1643 s (C═O amide) cm^{-1}; ESI-MS: *m*/*z*: 735 $[M + H^+]$; elemental *Anal.* Calc. (%) for $C_{48}H_{36}N_2O_2P_2$ (734.23): C, 78.5; H, 4.9; N, 3.8; Found: C, 77.9; H, 4.8; N, 4.0%.

2.4. Preparation of dppte-oxide (*3*)

A solution of dppte (**1**) in dichloromethane was stirred 48 h under air. Crystals of **3** were obtained by slow evaporation of the solvent.

$^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); $\delta = 40.5$ (P═O), −3.8 ppm (non-oxidised P).

2.5. Preparation of $[RuCl(\eta^6\text{-}p\text{-cymene})(dppte)RuCl(\eta^6\text{-}p\text{-cymene})]^{2+}$ (*4*)

To a solution of dppte (**1**) (10 mg, 0.015 mmol) in $CDCl_3$ (1 ml) were added one equivalent of $[RuCl_2(\eta^6\text{-}p\text{-cymene})]_2$ (4.6 mg) and 1.2 equivalents of $NaBF_4$ (2 mg). The solution was left at room temperature. After 3 days, crystals of **4** were obtained by slow evaporation of the chloroform solution.

^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 8.1$–7.1 ppm (m, 28H; ArH), $\delta = 6.12$ (d, 2H, $J = 6.0$ Hz; ArH), $\delta = 5.78$ (d, 2H, $J = 6.0$ Hz; ArH), $\delta = 3.93$ (s, 4H), $\delta = 3.30$ (m, 2H), $\delta = 2.19$ (s, 6H), $\delta = 1.22$ (d, 12H, $J = 7.0$ Hz); $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); $\delta = -11.03$ ppm; $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); $\delta = 36.5$ ppm.

2.6. Preparation of $[RuCl(\eta^6\text{-}p\text{-cymene})(dppan)RuCl(\eta^6\text{-}p\text{-cymene})]^{2+}$ (*5*)

To a solution of dppan (**2**) (10 mg, 0.014 mmol) in $CDCl_3$ (1 ml) was added one equivalent of $[RuCl_2(\eta^6\text{-}p\text{-cymene})]_2$ (4.2 mg). The solution was stirred at room temperature. After 3 days, **5** was obtained by slow evaporation of the chloroform solution.

^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 8.1$–7.1 ppm (m, 34H; ArH), $\delta = 6.15$ (d, 2H, $J = 6.0$ Hz; ArH), $\delta = 5.80$ (d, 2H, $J = 6.0$ Hz; ArH), $\delta = 3.31$ (m, 2H), $\delta = 2.19$ (s, 6H), $\delta = 1.21$ (d, 12H, $J = 7.0$ Hz); $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); $\delta = 35.2$ ppm.

3. X-ray crystallography

Crystals of **1**, **2**, **3**, $[\mathbf{4}][BF_4]_2$, and $[\mathbf{5}][Cl]_2$ were mounted on a Stoe Image Plate Diffraction System equipped with a ϕ circle goniometer, using Mo Kα graphite monochromated radiation ($\lambda = 0.71073$ Å with ϕ range 0–200°, increment of 1.0–2.3°, D_{max}–D_{min} = 12.45–0.81 Å. The structures were solved by direct methods using the program SHELXS-97 [10]. The refinement and all further calculations were carried out using SHELXL-97 [11]. The hydrogen atoms have been included in calculated positions and treated as riding atoms using the SHELXL default parameters. All non-H atoms were refined anisotropically, using weighted full-

Table 1
Crystallographic and selected experimental data of **1**, **2**, **3**, [**4**][BF_4]$_2$ and [**5**][Cl]$_2$

	1	**2**	**3**	[**4**][BF_4]$_2$ · 4 $CHCl_3$	[**5**][Cl]$_2$ · 3 $CHCl_3$
Chemical formula	$C_{40}H_{32}O_2P_2S_2$	$C_{48}H_{36}N_2O_2P_2$	$C_{50}H_{38}Cl_6N_2O_{2.50}P_2$	$C_{64}H_{64}B_2Cl_{14}F_8O_2P_2Ru_2S_2$	$C_{71}H_{77}Cl_{11}N_2O_2P_2Ru_2$
Formula weight	670.72	734.73	981.46	1863.36	1644.48
Crystal system	triclinic	monoclinic	triclinic	monoclinic	triclinic
Space group	$P\bar{1}$	$P\ 21/c$	$P\bar{1}$	$P\ 21/n$	$P\bar{1}$
Crystal colour and shape	yellow, plate	colourless, rod	colourless, block	orange, block	orange, block
Crystal size	$0.25 \times 0.25 \times 0.10$	$0.40 \times 0.10 \times 0.10$	$0.20 \times 0.20 \times 0.10$	$0.45 \times 0.30 \times 0.25$	$0.18 \times 0.15 \times 0.10$
a (Å)	9.8123(11)	10.415(3)	12.8894(13)	14.3348(15)	17.7127(19)
b (Å)	12.3529(13)	12.859(2)	14.5075(14)	18.072(3)	23.2473(18)
c (Å)	14.5803(19)	28.170(8)	14.7427(15)	15.5726(17)	25.3669(18)
α (°)	92.163(14)	90	111.857(11)	90	65.431(8)
β (°)	107.335(14)	94.53(3)	112.871(11)	111.754(12)	78.223(11)
σ (°)	97.324(13)	90	90.725(12)	90	87.874(11)
V (Å$_3$)	1667.9(3)	3760.8(16)	2317.2(4)	3747.0(8)	9286.3(14)
Z	2	4	2	2	4
T (K)	153	153	153	153	153
D_c (g · cm^{-3})	1.336	1.298	1.407	1.636	1.163
μ (mm^{-1})	0.291	0.159	0.484	1.057	0.602
Scan range (°)	$2.13 < 2\theta < 25.97$	$1.96 < 2\theta < 25.90$	$2.10 < 2\theta < 26.07$	$2.25 < 2\theta < 25.99$	$2.10 < 2\theta < 26.07$
Unique reflections	6028	7251	8434	7005	33971
Reflections used $[I > 2\sigma(I)]$	4203	3446	4629	5568	8913
R_{int}	0.0461	0.1060	0.0470	0.0599	0.1934
Final R indices $[I > 2\sigma(I)]$	0.0388, wR_2 0.0896	0.0489, wR_2 0.1009	0.0383, wR_2 0.0800	0.0545, wR_2 0.1638	0.2686, wR_2 0.5659
R indices (all data)	0.0639, wR_2 0.0966	0.1185, wR_2 0.1153	0.0866, wR_2 0.0901	0.0653, wR_2 0.1730	0.4066, wR_2 0.6188
Goodness-of-fit	0.921	0.778	0.804	1.057	1.330
Max, Min $\Delta\rho$/e (Å^{-3})	0.388, −0.341	0.528, −0.315	0.692, −0.541	1.548, −2.186	6.484, −1.527

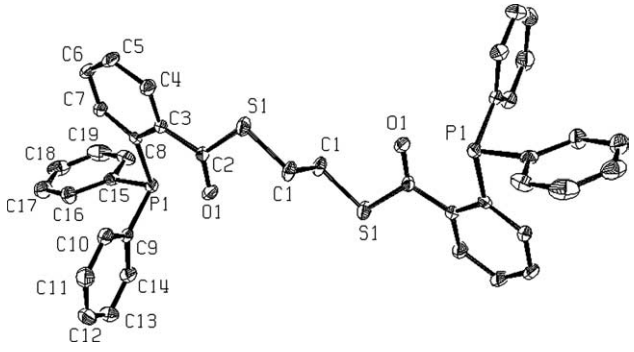


Fig. 1. ORTEP view of **1**, displacement ellipsoids are drawn at the 50% probability level, hydrogen atoms are omitted for clarity.

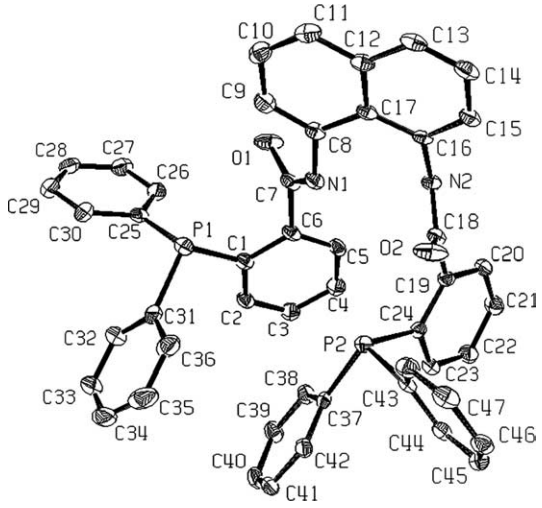


Fig. 2. ORTEP view of **2**, displacement ellipsoids are drawn at the 50% probability level, hydrogen atoms are omitted for clarity.

matrix least-square on F^2. Crystallographic details are summarised in Table 1. Figs. 1, 2, 4, 6 and 7 were drawn with ORTEP [12] and Figs. 3 and 5 with the program MERCURY [13].

4. Results and discussion

Two equivalents of 2-diphenylphosphinobenzoic acid react with 1,2-ethanedithiol and 1,8-diaminonaphthalene under standard peptidic coupling conditions [14] to give 1,2-bis-*S*-[2′-(diphenylphosphino)benzoyl]dithioethane (dppte) (**1**) and 1,2-bis-*N*-[2′(diphenylphosphino) benzoyl]diaminonaphthalene (dppan) (**2**), respectively (Scheme 1).

The diphosphine ligands **1** and **2** are symmetrical and show only one resonance in the $^{31}P\{^1H\}$ NMR spectrum

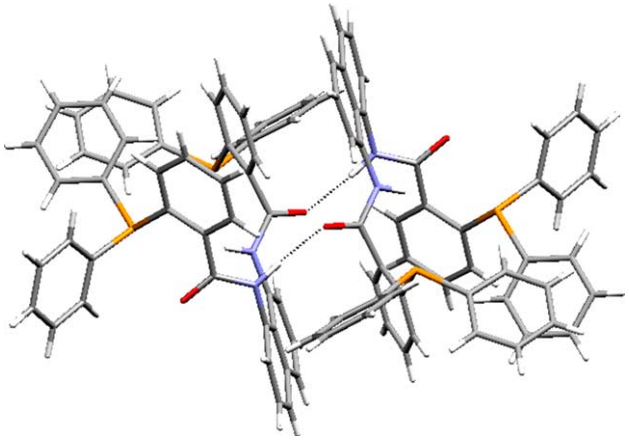


Fig. 3. Dimeric structure of **2** showing the intermolecular hydrogen bonds (dotted lines).

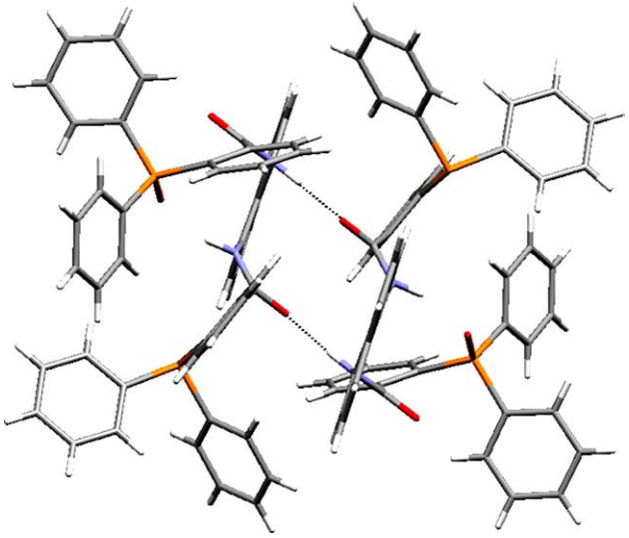


Fig. 5. Dimeric structure of **3** showing the intermolecular hydrogen bonds (dotted lines).

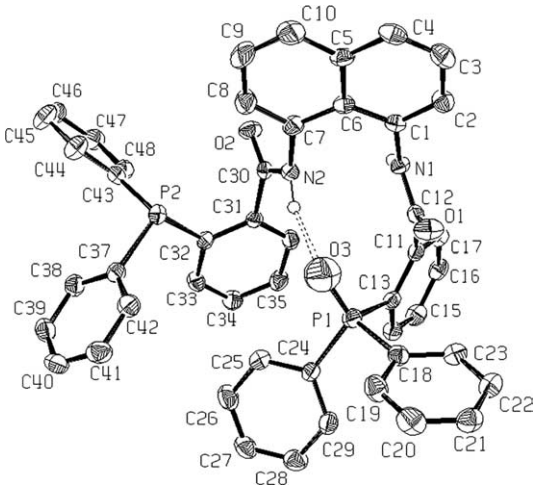


Fig. 4. ORTEP view of **3** showing the intramolecular hydrogen bond, displacement ellipsoids are drawn at the 50% probability level, hydrogen atoms are omitted for clarity.

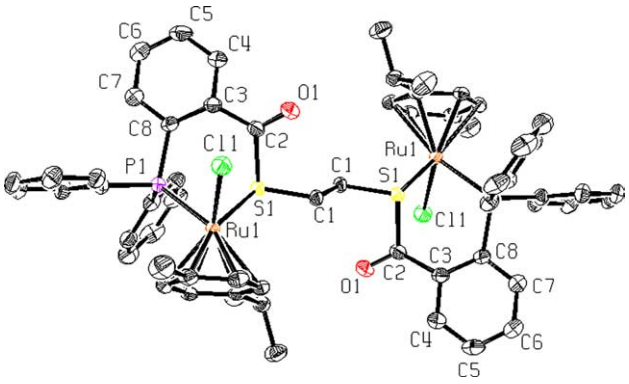


Fig. 6. ORTEP view of **4**, displacement ellipsoids are drawn at the 50% probability level, hydrogen atoms, anions and chloroform molecules are omitted for clarity.

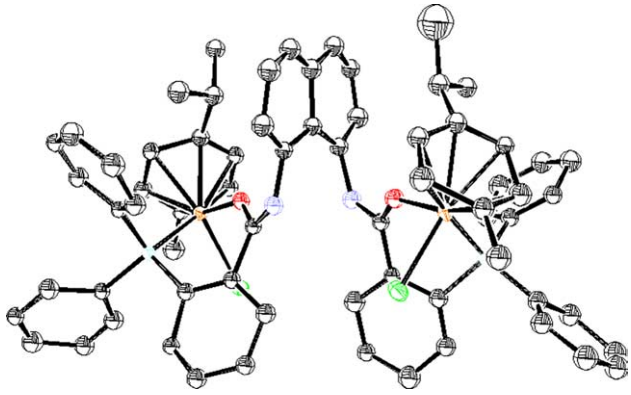
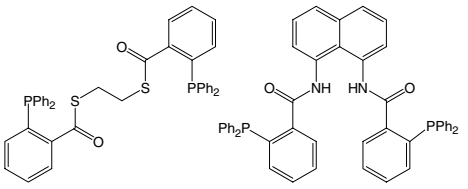


Fig. 7. ORTEP view of **5**, displacement ellipsoids are drawn at the 50% probability level, hydrogen atoms, anions and chloroform molecules are omitted for clarity.

at −3.81 and −11.03 ppm, respectively. In the ^{1}H NMR spectrum, **1** gives rise to a characteristic singlet at 2.99 ppm ($CDCl_3$) corresponding to the CH_2 protons, whereas **2** gives rise to a singlet at 9.30 ppm ($CDCl_3$) corresponding to the amido protons. In **2** the amido function is also well identified in the IR spectrum by two



Scheme 1.

absorptions at 3369 cm^{-1} (ν_{NH}) and 1643 cm^{-1} (ν_{CO}). The molecular structure of **1** has been determined by single-crystal X-ray structure analysis, see Fig. 1.

Compound **1** crystallises in the centrosymmetric space group $P\bar{1}$, and possesses an inversion centre situated halfway between the CH_2. The intramolecular distance between the two phosphorus atoms is 10.880(2) Å. The oxygen atom of the carbonyl moiety takes an axial orientation to the phosphorus atom, P–O distance being 2.742(2) Å. A similar axial orientation is observed in 2′-(diphenylphosphino)propiophenone in which the P–O intramoleular distance is 2.669(3) Å [15]. No meaningful interactions between neighbouring molecules of **1** were observed. For structural comparison the molecular structure of **2** has been determined by single-crystal X-ray structure analysis, see Fig. 2.

Unlike **1**, the phosphorus atoms are only separated by 5.393(2) Å, the molecule of **2** prefers to fold with the diphenylphosphine units on the same side of the naphthalene spacer. The nitrogen atoms N(1) and N(2) are, respectively, −0.165(4) and +0.136(4) Å out of the naphthalene plane. The crystal structure analysis reveals **2** to exist as a dimer in the solid state, thanks to hydrogen bonding between a NH and a CO function, see Fig. 3. The N–O distance of the hydrogen bond [N(2)–H···O(1)] is 2.850(3) Å, with a N–H···O angle of 173.6°.

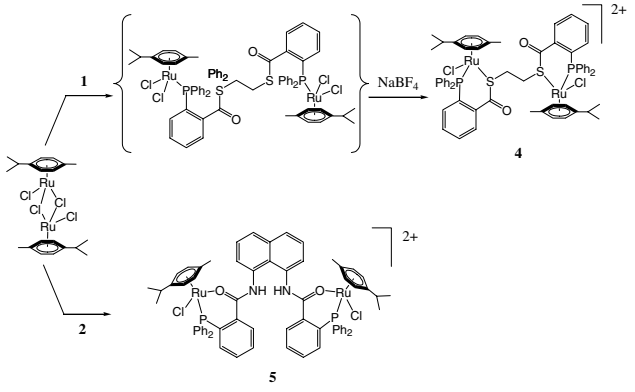
In air, compound **1** is oxidised to form the mono phosphine oxide derivative, see Scheme 2. In the $^{31}P\{^1H\}$ NMR spectrum, **3** gives rise to two singlets at −3.8 and 40.5 ppm corresponding, respectively, to the phosphine and to the phosphine oxide.

The molecular structure of **3** has been determined by single-crystal X-ray structure analysis, see Fig. 4. In the solid state, the presence of an additional oxygen slightly affects the internal folding of the molecule, but the dimeric form remains intact. An intramolecular hydrogen bond is formed between a NH and the phosphoryl oxygen atom. The N–O distance is 2.909(7) Å with a N–H···O angle of 163.9°. The intramolecular distance between the two phosphorus atoms is 5.742(1) Å.

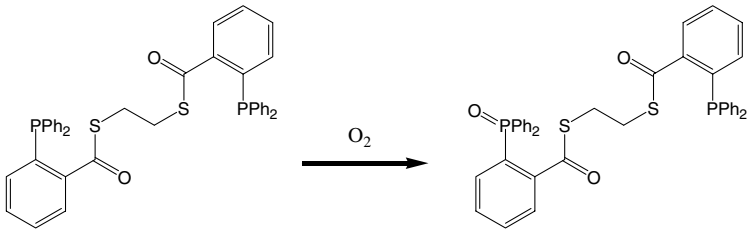
The crystal structure analysis reveals **3** to exist as a dimer in the solid state, see Fig. 5. The N–O distance of the hydrogen bond [N(1)–H···O(2)] is 2.846(3) Å, with a N–H···O angle of 175.6°, which is identical to the one observed in **2**. The chloroform molecules participate in the hydrogen bonding network. The C–H of the chloroform molecules interact with the oxygen of a carbonyl group, the C–O distances being 3.131(4) and 3.131(4) Å with C–H···O angles of 155.5° and 151.5°, respectively.

One equivalent of dppte (**1**) or dppan (**2**) reacts with $[RuCl_2(\eta^6\text{-}p\text{-cymene})]_2$ to give the corresponding dinuclear complexes $[RuCl(\eta^6\text{-}p\text{-cymene})(dppte)RuCl(\eta^6\text{-}p\text{-cymene})]^{2+}$ (**4**) and $[RuCl(\eta^6\text{-}p\text{-cymene})(dppan)RuCl(\eta^6\text{-}p\text{-cymene})]^{2+}$ (**5**), see Scheme 3. The stoichiometry of the reaction, two ruthenium centres per *bis*-diphenylphosphine ligand, as well as the presence of a *p*-cymene ligand coordinated in an η^6-fashion to the ruthenium allows only the formation of dinuclear species.

The stepwise formation of **4** is best monitored by $^{31}P\{^1H\}$ spectroscopy. In a NMR tube experiment, addition of $[RuCl_2(\eta^6\text{-}p\text{-cymene})]_2$ to a $CDCl_3$ solution



Scheme 3.



Scheme 2.

containing **1** generates an immediate downfield shift of 25 ppm (as compared to the uncoordinated phosphine) corresponding to the formation of the neutral complex [$RuCl_2(\eta^6$-*p*-cymene)(dppte)$RuCl_2(\eta^6$-*p*-cymene)]. The cleavage of the dichloro bridge in [$RuCl_2(\eta^6$-*p*-cymene)]$_2$ by coordination of the phosphine units gives rise to a singlet at 21.2 ppm. By addition of $NaBF_4$ a second downfield shift of the $^{31}P\{^1H\}$ signal is observed, a singlet appears at 36.6 ppm. The second reaction step corresponds to the formation of six-membered chelate rings through coordination of the sulfur atoms to the rutheniums. The mass spectrum is in accordance with the presence of dinuclear species containing two arene-ruthenium units. The molecular structure of [**4**][BF_4]$_2$ has been confirmed by a single-crystal structure analysis, see Fig. 6.

The molecular structure of **4** shows the ruthenium atom in a distorted octahedral geometry. The diphosphine ligand adopts a *P*, *S* coordination mode, the two sulfur atoms coordinating as well to the metal. The formation of six-membered chelate rings imposes a considerable distortion around the ruthenium atom. The S–Ru–P angles [80.80(4)°] are acute due to the bidentate $PC_6H_4C(O)S$ units. In the crystal structure, there is no meaningful interaction between complex **4** and the tetrafluoroborates and the chloroform molecules. Poor quality crystals of **5** have been obtained by a slow evaporation of a $CDCl_3$ solution, and for comparison only, the molecular structure of [**5**][Cl]$_2$ is presented, see Fig. 7.

In **5**, the diphosphine ligand adopts a *P*, *O* coordination mode, the two nitrogen atoms being not coordinated to the metal. A similar six-membered chelate ring has been observed by Rasley et al. [16] in the chloro(cyclopentadienyl)phosphine ruthenium complex [$Cp(CO)Ru(\eta^2$-(*P*, *O*)–$Ph_2PC_6H_4$-*o*-$COCH_3)]^+$. As for complex **4**, the molecular structure of **5** shows the ruthenium atom in a distorted octahedral geometry. The formation of six-membered chelate rings imposes a considerable distortion around the ruthenium atom. The O-Ru-P angles [average 79.9(6)°] are acute due to the bidentate $PC_6H_4C(O)$ units. In the crystal structure, there is no meaningful interaction between complex **5** and the chlorides or chloroform molecules.

5. Supplementary material

Full tables of atomic parameters, bond lengths and angles are deposited at the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK, Deposition numbers: **1** 218531, **2** 218532, **3** 218533, [**4**][BF_4]$_2$ 218534, [**5**][Cl]$_2$ 218535.

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