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New arene ruthenium complexes with planar chirality

Bruno Therrien *, Georg Süss-Fink

Institut de Chimie, Universitéde Neuchâtel, Case postale 2, CH-2007 Neuchâtel, Switzerland

Abstract

1,2,4-Trimethyl-cyclohexadiene reacts with $RuCl_3 \cdot nH_2O$ in refluxing ethanol to afford quantitatively $[RuCl_2(1,2,4\text{-}C_6H_3Me_3)]_2$ (**1**), the coordination of 1,2,4-trimethylbenzene to the ruthenium atom introducing planar chirality at the η^6-arene ligand. The dinuclear complex **1** reacts with two equivalents of triphenylphosphine (PPh_3) to give quantitatively, as a racemic mixture of enantiomers, $[RuCl_2(1,2,4\text{-}C_6H_3Me_3)(PPh_3)]$ (**2**), the structure of which has been determined by a single-crystal X-ray structure analysis of (*rac*)-**2**. Similarly, **1** reacts with two equivalents of the enantiopure phosphine (1*S*,2*S*,5*R*)-(+)-neomenthyldiphenylphosphine (nmdpp) to afford in good yield $[RuCl_2(1,2,4\text{-}C_6H_3Me_3)(\text{nmdpp})]$ (**3**) as a mixture of diastereoisomers, from which the isomer **3a** was isolated by crystallisation. A single-crystal X-ray structure analysis of **3a** allowed the determination of the absolute configuration at the planar chiral η^6-arene moiety. Finally, complex **1** reacts with one equivalent of the diphosphine ligand 1,1′-bis(diphenylphosphino)ferrocene (dppfc) to give the heteronuclear complex $[RuCl_2(1,2,4\text{-}C_6H_3Me_3)\,(\text{dppfc})RuCl_2(1,2,4\text{-}C_6H_3Me_3)]$ (**4**). All complexes were fully characterised by elemental analysis, mass spectrometry, NMR and IR spectroscopies.

Keywords: Ferrocene; Phosphine ligand; Planar chirality; Ruthenium

1. Introduction

It is known that an arene ligand bearing two non-equivalent substituents in 1,2 or 1,3 positions relative to each other, coordinated in an η^6-fashion to a transition metal, generates planar chirality at the metal with respect to the face of the coordinated arene [1]. Arene–ruthenium complexes with planar chirality have been studied first by Bennett and co-workers [2]. They are of great interest because of their potential as chiral catalysts for asymmetric synthesis [3–5].

Herein we report the synthesis of a dinuclear arene ruthenium complex with planar chirality, $[RuCl_2(1,2,4\text{-}C_6H_3Me_3)]_2$, in which the presence of three methyl groups in positions 1,2 and 4 of the η^6-arene rings is responsible for the planar chirality. The reaction of this dimer with phosphine ligands, such as triphenylphosphine, (1*S*,2*S*,5*R*)-(+)-neomenthyldiphenylphosphine, or 1,1′-bis(diphenylphosphino)ferrocene gives rise to the formation of the corresponding phosphine complexes which exist as mixtures of enantiomers or diastereomers, respectively.

2. Experimental

2.1. General remarks

Ethanol and methanol were dried and distilled under nitrogen prior to use. Except for $RuCl_3 \cdot nH_2O$ (Johnson Matthey), all reagents were purchased either from Aldrich or Fluka and used as received. Nuclear magnetic resonance spectra were recorded using a Varian Gemini 200 BB or a Bruker 400 MHz spectrometer 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,

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* Corresponding author. Tel.: +41-32-718-2499; fax: +41-32-718-2511.
E-mail addresses: bruno.therrien@unine.ch (B. Therrien), georg.suess-fink@unine.ch (G. Süss-Fink).

Switzerland. 1,2,4-Trimethyl-cyclohexadiene was prepared according to published method [6].

2.2. Preparation of *[$RuCl_2(1,2,4\text{-}C_6H_3Me_3)]_2$ (**1**)*

A solution of $RuCl_3 \cdot nH_2O$ (3.2 g, 12.0 mmol) and 10 equivalents of 1,2,4-trimethyl-cyclohexadiene (14.7 g, 0.12 mol) in ethanol (40 ml) was refluxed for 12 h. The volume was then reduced to 5 ml, and the solid precipitated with ether. The red, air-stable, microcrystalline material was isolated by filtration, giving **1** (550 mg, 0.81 mmol) in 50% yield. ^1H NMR (400 MHz, $CDCl_3$): $\delta = 5.34$ (d, 1H; CH_{ar}), 5.33 (d, 1H; CH_{ar}), 5.27 (d, 1H; CH_{ar}), 5.25 (d, 1H; CH_{ar}), 5.00 (s, 1H; CH_{ar}), 4.99 (s, 1H; CH_{ar}), 2.17 (s, 3H, CH_3), 2.16 (s, 3H, CH_3), 2.11 (s, 3H, CH_3), 2.10 (s, 6H, CH_3), 2.09 ppm (s, 3H, CH_3); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): $\delta = 96.97$, 96.93, 95.75, 95.67, 90.81, 90.78, 84.57, 84.52, 80.90, 80.86, 79.63, 79.53, 19.17, 17.74, 17.21 ppm; IR (KBr): 3043 m, 2918 m, 1621 w, 1531 w, 1448 s, 1376 s, 1122 w, 1031 s, 889 w, 734 w, 631 w cm^{-1}; ESI-MS: *m/z*: 606 $[M + Na^+]$; elemental *Anal.* Calc. For $C_{18}H_{24}Cl_4Ru_2$ (584.21), C, 37.0; H, 4.1. Found: C, 36.8; H, 4.2%.

2.3. Preparation of *[$RuCl_2(1,2,4\text{-}C_6H_3Me_3)(PPh_3)$] (**2**)*

A solution of **1** (290 mg, 0.5 mmol) and triphenylphosphine (270 mg, 1.0 mmol) in methanol (20 ml) was heated at 60 °C for 3 h. Then the solvent was evaporated to dryness to afford quantitatively [$RuCl_2(1,2,4\text{-}C_6H_3Me_3)(PPh_3)$] (**2**) as an orange solid. ^1H NMR (200 MHz, $CDCl_3$): $\delta = 7.80$ (m, 6H; PPh_3), 7.40 (m, 9H; PPh_3), 5.40 (s, 1H, CH_{ar}), 4.29 (m, 1H, CH_{ar}), 4.18 (m, 1H, CH_{ar}), 2.22 (s, 3H, CH_3), 2.01 (s, 3H, CH_3), 1.86 ppm (s, 3H, CH_3); $^{13}C\{^1H\}$ NMR (50 MHz, $CDCl_3$): = 134.65, 134.55, 134.36, 130.48, 128.32, 128.14, 107.57, 97.65, 96.30, 94.67, 86.11, 83.78, 17.93, 16.84, 16.20 ppm; $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); $\delta = 28.5$ ppm; IR (KBr): 1630 m, 1479 w, 1436 s, 1375 w, 1089 m, 1023 m, 753 s, 698 s, 528 s cm^{-1}; ESI-MS: *m/z*: 519 $[M - Cl^-]$; elemental *Anal.* Calc. For $C_{27}H_{27}Cl_2P_1Ru_2$ (554.46), C, 58.5; H, 4.9. Found: C, 58.6; H, 4.8%.

2.4. Preparation of *[$RuCl_2(1,2,4\text{-}C_6H_3Me_3)(nmdpp)$] (**3**)*

A solution of (1*S*,2*S*,5*R*)-(+)- neomenthyldiphenylphosphine (110 mg, 0.34 mmol) and **1** (100 mg, 0.17 mmol) in ethanol (20 ml) was refluxed for 2 h. The solution was evaporated under vacuum to give **3** [$RuCl_2(1,2,4\text{-}C_6H_3Me_3)(nmdpp)$] (182 mg, 0.29 mmol). Yield: 87%. ^1H NMR (200 MHz, $CDCl_3$): $\delta = 7.90$ (m, 8H, PPh_2), 7.55 (m, 4H, PPh_2), 7.25 (m, 8H, PPh_2), 5.16 (s, 1H, CH_{ar}), 5.06 (s, 1H, CH_{ar}), 4.34 (m, 2H, CH_{ar}), 3.53 (m, 4H), 2.95 (m, 2H), 2.27 (m, 2H), 2.05 (m, 2H), 1.97 (s, 3H), 1.92 (s, 6H), 1.76 (m, 2H), 1.71 (s, 3H), 1.50 (s, 3H), 1.39 (s, 3H), 1.25 (s, 3H), 1.06 (m, 2H), 0.95 (m, 2H), 0.83 (s, 3H), 0.55 (s, 3H), 0.04 (s, 3H), 0.01 ppm (s, 3H); $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); $\delta = 24.6$ and 24.7 ppm; IR (KBr): 2954 s, 1632 w, 1438 s, 1374 m, 1094 m, 1032 m, 883 w, 748 s, 698 s, 519 s cm^{-1}; ESI-MS: *m/z*: 581 $[M - Cl^-]$; elemental *Anal.* Calc. For $C_{31}H_{41}Cl_2PRu$ (616.6), C, 60.4; H, 6.7. Found: C, 60.6; H, 6.7%. The less soluble diastereomer **3a** crystallised in a pure form from a methanol solution of **3** at room temperature.

2.5. Preparation of *[$RuCl_2(1,2,4\text{-}C_6H_3Me_3)$ (dppfc) $RuCl_2(1,2,4\text{-}C_6H_3Me_3)$] (**4**)*

A solution of **1** (58 mg, 0.1 mmol) and 1,1′-bis(diphenylphosphino)ferrocene (55 mg, 0.1 mmol) in methanol (10 ml) was heated at 60 °C for 4 h. The solvent was reduced to 2 ml, and the product purified on silica gel plates (CH_2Cl_2/acetone, 15:1) to afford [$RuCl_2(1,2,4\text{-}C_6H_3Me_3)(dppfc)RuCl_2(1,2,4\text{-}C_6H_3Me_3)$] **4** as an orange solid (35 mg, 0.03 mmol). Yield: 31%. ^1H NMR (200 MHz, $CDCl_3$): $\delta = 7.67$ (m, 8H, PPh_2), 7.36 (m, 12H, PPh_2), 5.37 (s, 2H, CH_{ar}), 4.14 (m, 2H, CH_{ar}), 4.11 (m, 2H, CH_{ar}), 4.03 (m, 4H, CH_{Fc}), 3.50 (s, 4H, CH_{Fc}), 2.15 (s, 6H, CH_3), 1.86 (s, 6H, CH_3), 1.61 ppm (s, 6H, CH_3); $^{31}P\{^1H\}$ NMR (81 MHz, $CDCl_3$); $\delta = 20.7$ ppm; IR (KBr): 1630 w, 1434 s, 1161 m, 1095 m, 1026 m, 752 s, 698 s, 545 m cm^{-1}; ESI-MS: *m/z*: 1161 $[M + Na^+]$; elemental *Anal.* Calc. For $C_{52}H_{52}Cl_4FeP_2$ Ru_2(1138.74), C, 54.8; H, 4.6. Found: C, 55.0; H, 4.7%.

2.6. *X-ray crystallographic study*

Crystals of (*rac*)-**2** and **3a** 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°–100°, increment of 0.9° and 0.7°, respectively, 2θ range from 2.0° to 26°, D_{max}–$D_{min} = 12.45$–0.81 Å. The structures were solved by direct methods using the programme SHELXS-97 [7]. The refinement and all further calculations were carried out using SHELXL-97 [8]. The H-atoms were included in calculated positions and treated as riding atoms using the SHELXL default parameters. The non-H atoms were refined anisotropically, using weighted full-matrix least-square on F^2. Crystallographic details are summarised in Table 1. Figures were drawn with the ORTEP program [9].

CCDC-212744 (*rac*)-**2** and 212743 **3a** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge

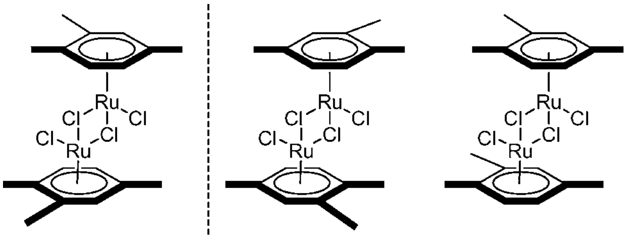
Table 1
Crystallographic and selected experimental data of (*rac*)-**2** and **3a**

	(*rac*)-**2** · $2CHCl_3$	**3a**
Chemical formula	$C_{29}H_{29}Cl_8PRu$	$C_{31}H_{41}Cl_2PRu$
Formula weight	793.16	616.58
Crystal system	orthorhombic	orthorhombic
Space group	*P bca*	$P\,2_12_12_1$
Crystal colour and shape	orange plate	orange block
Crystal size	0.30 × 0.20 × 0.05	0.40 × 0.30 × 0.15
a (Å)	18.885 (2)	9.2724(5)
b (Å)	13.7506(6)	9.6036(4)
c (Å)	25.1922(12)	32.447(2)
V (Å^3)	6541.8(7)	2889.3(3)
Z	8	4
T (K)	153(2)	153(2)
D_c (g cm^{-1})	1.611	1.417
μ (mm^{-1})	1.202	0.801
Scan range (°)	$3.88 < 2\theta < 51.92$	$4.42 < 2\theta < 51.76$
Unique reflections	6355	4884
Reflections used [$I > 2\sigma(I)$]	3001	4023
R_{int}	0.0861	0.0534
Final R indices [$I > 2\sigma(I)$]	0.0484, wR_2 0.1012	0.0425, wR_2 0.1028
R indices (all data)	0.1193, wR_2 0.1160	0.0538, wR_2 0.1060
Goodness-of-fit	0.814	0.981
Max., Min. $\Delta\rho$/e (Å^{-3})	0.920, −0.806	1.059, −0.668

Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (internat.) +44-1223/336-033; e-mail: deposit@ccdc.cam.ac.uk].

3. Results and discussion

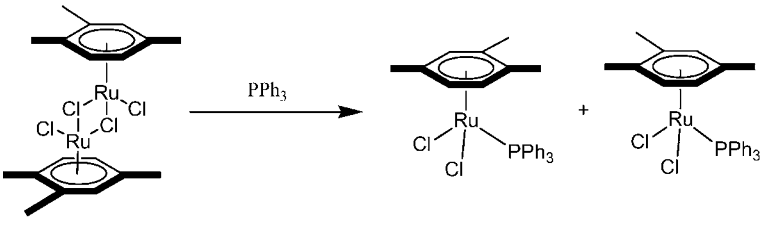
1,2,4-Trimethyl-cyclohexadiene, accessible by standard *Birch* reduction [10], reacts with $RuCl_3 \cdot nH_2O$ in refluxing ethanol to afford quantitatively $[RuCl_2(1,2,4\text{-}C_6H_3Me_3)]_2$ (**1**). Coordination of 1,2,4-trimethylbenzene to the ruthenium atom introduces a planar chirality at the η^6-arene ligand. The dinuclear complex **1** is present as a mixture of three species, as shown in Scheme 1, in which **1a** and **1b** are enantiomers and **1c** the *meso* form. The $^{13}C\{^1H\}$ NMR spectrum of **1** shows in the arene–ruthenium carbons region two sets of signals due to the two diastereoisomers that are present in equal amounts.



1a **1b** **1c**

Scheme 1.

Complex **1** reacts with two equivalents of triphenylphosphine (PPh_3) to give quantitatively the mononuclear complex $[RuCl_2(1,2,4\text{-}C_6H_3Me_3)(PPh_3)]$ (**2**), as a racemic mixture of enantiomers **2a** and **2b**, see Scheme 2. The $^{31}P\{^1H\}$ NMR of **2** shows a singlet at 28.5 ppm, the chemical shift being comparable to those observed for the analogous η^6-arene ruthenium complexes $[RuCl_2(C_6Et_6)(PPh_3)]$ [11], and $[RuCl_2(C_6H_5fc)(PPh_3)]$ [12] which show signals at 24.0 and 28.6 ppm, respectively. As expected for enantiomers, only one set of signals is observed in the ^1H and $^{13}C\{^1H\}$ NMR spectra. All



1 **2a** **2b**

Scheme 2.

attempts to separate the enantiomers **2a** and **2b** were unsucessful.

Crystals of the racemate have been obtained by slow evaporation of a chloroform solution containing **2**. A single-crystal X-ray analysis of a (*rac*)-**2** was performed. The molecular structure is presented in Fig. 1, and related bond lengths and angles are listed in Table 2. Complex **2** crystallises in the centrosymmetric space group *P bca*.

The ruthenium atom in **2** possesses a pseudo-octahedral geometry, and the metrical parameters around the metallic core compare well with those of similar three-legged piano-stool [$RuCl_2(\eta^6$-arene$)(PPh_3)$] complexes [11–17], the P–Ru–Cl angles being P(1)–Ru(1)–Cl(1) 85.82(5)° and P(1)–Ru(1)–Cl(2) 91.46(5)°, respectively. A distortion at the arene ligand is present, the Ru–C bond distances *trans* to the phosphorous atom, Ru(1)–C(2) 2.255(6) and Ru(1)–C(3) 2.248(7) Å, are elongated as compared to the other Ru–C bonds [ranging between 2.203(6) and 2.234(7) Å]. Similar observations have been made for the complexes [$RuCl_2(C_{10}H_{14})(PMePh_2)$], [$RuCl_2(C_6H_6)(PMePh_2)$] [13] and [$RuCl_2(C_{16}H_{16})(PPh_3)$] [14], where the distortion was attributed to the *trans* lengthening bond effect of tertiary phosphine ligands. No meaningful interactions

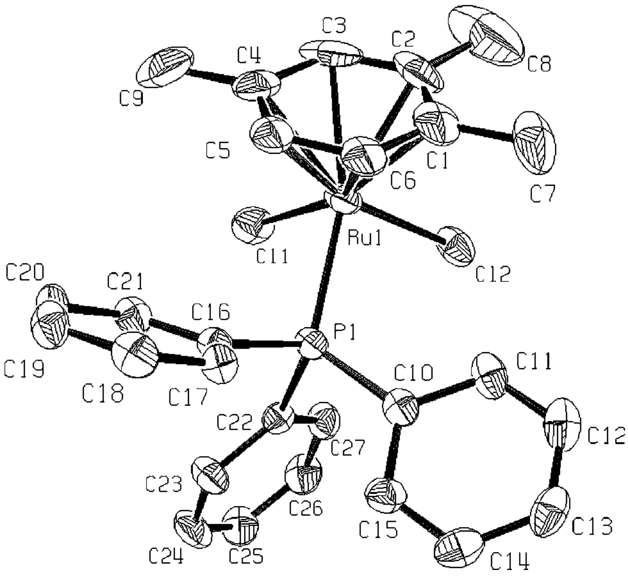


Fig. 1. ORTEP drawing of (*rac*)-**2** showing the numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms and chloroform molecules are omitted for clarity.

Table 2
Selected bond lengths (Å) and angles (°) for (*rac*)-**2** and **3a**

	(*rac*)-**2**	**3a**
Interatomic distances		
Ru(1)–C(1)	2.234(7)	2.207(5)
Ru(1)–C(2)	2.255(6)	2.249(6)
Ru(1)–C(3)	2.248(7)	2.226(7)
Ru(1)–C(4)	2.230(6)	2.221(6)
Ru(1)–C(5)	2.203(6)	2.223(6)
Ru(1)–C(6)	2.208(7)	2.199(7)
Ru(1)–P(1)	2.3533(14)	2.3740(14)
Ru(1)–Cl(1)	2.4008(18)	2.3961(14)
Ru(1)–Cl(2)	2.4299(15)	2.4141(14)
Angles		
Cl(1)–Ru(1)–P(1)	85.82(5)	86.16(5)
Cl(1)–Ru(1)–Cl(2)	86.40(6)	87.52(5)
P(1)–Ru(1)–Cl(2)	91.46(5)	92.66(5)

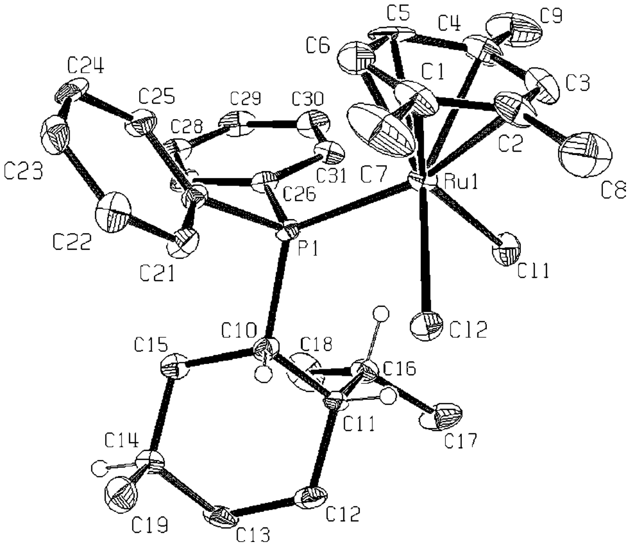
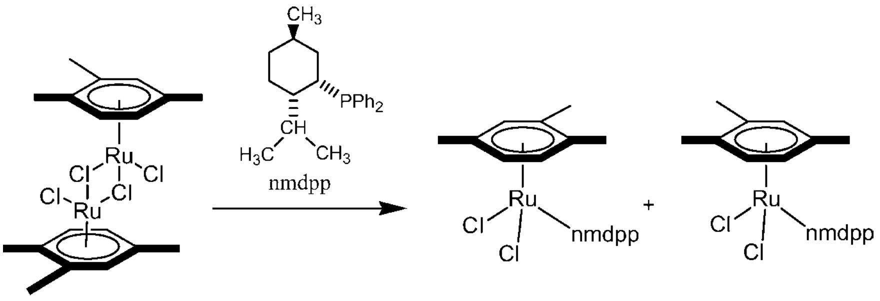
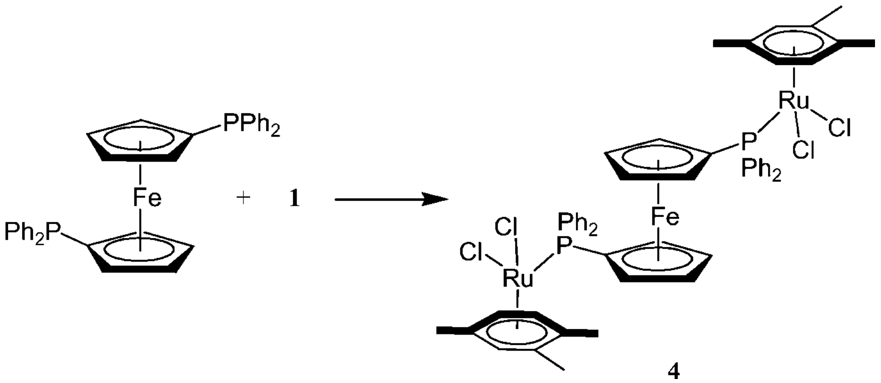


Fig. 2. ORTEP drawing of **3a** showing the numbering scheme. The hydrogen atoms are omitted for clarity. Displacement ellipsoids are drawn at the 50% probability level.



1 **3a** **3b**

Scheme 3.



Scheme 4.

between complex **2** and the chloroform solvent molecules were observed.

As the resolution of the enantiomers of **2** was unsuccessful, we reasoned that the coordination of an enantiopure phosphine ligand to the ruthenium atom, thus giving rise to the formation of diastereoisomers, would facilitate the separation.

Thus, complex **1** reacts with two equivalents of the commercially available enantiopure (1*S*,2*S*,5*R*)-(+)-neomenthyldiphenylphosphine (nmdpp) to afford in good yield the complex [$RuCl_2(1,2,4\text{-}C_6H_3Me_3)$(nmdpp)] (**3**), as a mixture of diastereomers **3a** and **3b**, see Scheme 3. The $^{31}P\{^1H\}$ NMR spectrum of the crude product shows two singlets at 24.6 and 24.7 ppm, respectively. Crystals of the less soluble diastereoisomer **3a** were obtained from a methanol solution containing **3**.

A single-crystal X-ray structure analysis of **3a** allows us to determine the absolute configuration at the planar chiral η^6-arene moiety. The molecular structure of **3a** is presented in Fig. 2, and selected bond lengths and angles are given in Table 2.

The X-ray analysis shows that the absolute configuration of the planar chiral η^6-arene is *S*. As in the case of **2**, also in **3a** the ruthenium atom is in a slightly distorted octahedral geometry with angles closed to 90° [Cl(1)–Ru(1)–Cl(2) 87.52(5)°, P(1)–Ru(1)–Cl(1) 92.66(5)° and P(1)–Ru(1)–Cl(2) 86.16(5)°]. A similar distortion of the arene ligand is observed, the longest Ru–C distances being *trans* to the phosphorous atom, Ru(1)–C(2) 2.249(6) Å. The fact that the Ru–P bond is slightly longer [2.3740(14) Å] than that observed in **2** [2.3533(14) Å] is probably due to the greater bulk of nmdpp relative to PPh_3. The bond distances and angles in the neomenthyl fragment of **3a** are similar to those found in [$RuCl_2(\eta^6\text{-}o\text{-}MeC_6H_4CO_2Me)$(nmdpp)] [3] and [$RuCl_2(\eta^6\text{-}C_6H_5CHMeR)$(nmdpp)] (R = Et, ^{t}Bu) [4].

Finally, complex **1** reacts with 1,1′-bis(diphenylphosphino)ferrocene (dppfc) to give the heteronuclear complex [$RuCl_2(1,2,4\text{-}C_6H_3Me_3)$(dppfc)$RuCl_2(1,2,4\text{-}C_6H_3Me_3)$] **4**, see Scheme 4. The symmetrical bis-diphenylphosphine ferrocene ligand generates a trinuclear compound possessing two planar chiral η^6-arene ruthenium units per ferrocene centre. Thus, as for complex **1**, the synthesis of **4** should give rise to the formation of three different species, two enantiomers (**4a** and **4b**) and a *meso* form (**4c**).

By contrast, the $^{31}P\{^1H\}$ NMR spectrum of **4** shows only one singlet at 20.7 ppm, the chemical shift being identical to that observed for the analoguous achiral complex [$RuCl_2(1,2,3,4\text{-}C_6H_2Me_4)$(dppfc)$RuCl_2(1,2,3,4\text{-}C_6H_2Me_4)$] [18]. Accordingly, the ^1H and $^{13}C\{^1H\}$ NMR spectra of **4** show only one set of signals. This means that either only one of the diasteromers (*meso* or *rac*) is formed or that, despite the presence of both diastereomers (*meso* and *rac*), the two chiral elements are too far away from each other to differentiate between the diastereoisomers. All attempts to resolve **4** into isomers were unsuccessful.

Acknowledgements

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