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Synthesis, characterization, crystal structure and chemical behavior of [1,1-bis(diphenylphosphinomethyl)ethene] ruthenium(II) complex toward primary alkylamine addition

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Abstract Reaction of the ruthenium phosphine complex $[RuCl_2(PPh_3)_3]$ **1** with an unsaturated functionalized diphosphine ligand, namely 1,1-bis(diphenyl-phosphinomethyl)-ethene, $H_2C{=}C(CH_2PPh_2)_2$, (dppme) gave *trans*-$Cl_2Ru(dppme)_2$ **2.** Complex **2** served as a precursor for the synthesis of *trans*-$Cl_2Ru(dppme)(alkylamine)_2$ complexes **3–5** by reaction with RNH_2. All these complexes were characterized by spectroscopic techniques and by elemental analysis. The solid-state structure of complex **2** was determined by X-ray crystallography.

Introduction

Phosphines and diphosphines are versatile ligands, which can be readily functionalized by means of the groups bound to the phosphorus atom [1–8]. These functionalized phosphine ligands have important industrial applications such as water-soluble and asymmetric phosphines [9, 10]. Metal complexes containing phosphorus ligands have always been important due to their possible catalytic activity, and a variety of them have been reported [3–20]. Several six-coordinated complexes of ruthenium(II) of the general formula $[RuX_2(diphosphine)_2]$ have been reported in the last few years [3–8]. We are interested in the chemical properties and structures of new ruthenium(II) complexes containing an alkene group in the backbone of the diphosphine ligand, such as 1,1-bis(diphenylphosphinomethyl)ethane, dppme.

Our ongoing research interest is to synthesize supported and non-supported phosphine Ru(II) complexes and to examine their activity for catalytic hydrogenation of unsaturated carbonyl compounds in homogenous and heterogeneous phase [11–19]. In this article, we describe the synthesis and characterization of mixed ligand ruthenium(II) complexes containing substitute dppme as primary ligand and RNH_2 as secondary ligands. The single crystal X-ray structure of $[RuCl_2(dppme)_2]$ is also reported.

Experimental

All reactions were carried out in an inert atmosphere (argon) by using, standard high vacuum and Schlenk-line techniques unless otherwise mentioned. Prior to use CH_2Cl_2, *n*-hexane, and Et_2O were distilled from CaH_2, $LiAlH_4$, and from sodium/benzophenone, respectively. 1,1-bis(diphenylphosphinomethyl)ethene (dppme) [20] and $RuCl_2(PPh_3)_3$ [21] were prepared according to literature methods. 3-(trimethoxysilyl)propylamine, 3-Chloro-2-(chloromethyl)-1-propene, Ph_3P, *n*-BuLi, and $RuCl_3 \cdot 3H_2O$ were purchased from Merck and Chempur, respectively, and were used without further purification. Elemental analyses (C, H, and N) were carried out on an Elementar Vario EL analyzer. High-resolution ^1H, $^{13}C\{^1H\}$, DEPT 135, and $^{31}P\{^1H\}$ NMR spectra were recorded on a Bruker DRX 250 spectrometer at 298 K. Frequencies are as follows: ^1H NMR 250.12 MHz, $^{13}C\{^1H\}$ NMR 62.9 MHz, and $^{31}P\{^1H\}$ NMR 101.25 MHz. Chemical shifts in the ^1H and $^{13}C\{^1H\}$ NMR spectra were measured relative to partially deuterated solvent peaks which are reported relative to TMS. ^{31}P chemical shifts were measured relative to 85% H_3PO_4. FTIR data were obtained

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on a Bruker IFS 48 FT-IR spectrometer and FAB-MS, Finnigan 711A (8 kV), modified by AMD and reported as mass/charge *(m/z)*.

Synthesis of *trans*-$Cl_2Ru(dppme)_2$ **2**

A solution of dppme (0.88 g, 2.10 mmol) in dichloromethane (40 ml) was added dropwise to a stirred solution of complex **1** (1.00 g, 1.043 mmol) in dichloromethane (50 ml). The reaction mixture was stirred for approximately 50 min at room temperature during which the color changed from brown to yellow. After removal of any turbidity by filtration (P4), the volume of the solution was concentrated to about 3 ml under reduced pressure. Addition of 80 ml of diethyl ether caused the precipitation of a solid which was filtered off (P4), washed three times with 25 ml of *n*-hexane each and dried under vacuum.

Complex **1** (1.00 g, 1.043 mmol) was treated with dppme (0.88 g, 2.10 mmol) to give **2.** Yield 0.98 g (92%) as yellow powder. ^1H NMR ($CDCl_3$): δ (ppm) 2.9 (m, 8H, CH_2P), 5.5 (m, 2H, CH_2C), 7.1–7.6 (m, 40H, C_6H_5). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ (ppm) −1.9. $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ (ppm) 47.5 (m, PCH_2), 121.2 (t, CH_2C), 128.3 (m, *m*-C_6H_5), 129.4 (s, *p*-C_6H_5), 133.2 (m, *o*-C_6H_5), 134.1 (m, *i*-C_6H_5). FAB-MS: *(m/z)* 1020.2 (M^+). *Anal*. Found: C, 65.5; H, 5.5; Cl, 6.5. Calc. for $C_{56}H_{52}Cl_2P_4Ru$: C, 65.9; H, 5.1; Cl, 6.9%.

General procedure for the synthesis of the *trans*-$Cl_2Ru(dppme)(alkylamine)_2$ **3–5**

A small excess (2.1 equivalents) of alkylamine was dissolved in dichloromethane (40 ml) and the solution was added dropwise to a stirred solution of complex **2** in dichloromethane (40 ml). The reaction mixture was stirred for ~10 min at room temperature. After removal of any turbidity by filtration (P4), the volume of the solution was concentrated to about 5 ml under reduced pressure. Addition of 40 ml of diethyl ether caused the precipitation of a solid which was filtered off (P4), then re-dissolved in 40 ml of dichloromethane, and concentrated again under vacuum to a volume of 5 ml. Addition of 80 ml of *n*-hexane caused the precipitation of a solid which was filtered off (P4), washed thoroughly with *n*-hexane, and dried under vacuum.

trans-$Cl_2Ru(dppme)(NH_2CH_3)_2$ **3**

Complex **2** (0.5 g, 0.49 mmol) was treated with methylamine (0.032 g, 1.0 mmol) to give **3**. Yield 0.27 g (84%) of a brown-yellow powder. ^1H NMR ($CDCl_3$): δ (ppm) 2.3 (b, 2H, NH_2), 2.5 (s, 3H, CH_3), 2.7 (m, 8H, CH_2P), 5.4 (m, 2H, CH_2C), 7.0–7.9 (m, 40H, C_6H_5). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ (ppm) 40.3. $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ (ppm) 28.5 (s, CH_3), 46.7 (m, PCH_2), 122.3 (t, CH_2C), 128.9 (m, *m*-C_6H_5), 129.5 (s, *p*-C_6H_5), 133.4 (m, *o*-C_6H_5), 134.9 (m, *i*-C_6H_5). FAB-MS: *(m/z)* 658.1 (M^+). *Anal*. Found: C, 55.0; H, 4.2; Cl, 10.4; N, 4.2. Calc. for $C_{32}H_{40}N_2P_2Ru$: C, 54.7; H, 5.5; Cl, 10.8; N, 4.2%.

trans-$Cl_2Ru(dppme)(NH_2CH_2CH_3)_2$ **4**

Complex **2** (0.5 g, 0.49 mmol) was treated with ethylamine (0.046 g, 1.0 mmol) to give **4**. Yield 0.29 g (86%) of a yellow powder. ^1H NMR ($CDCl_3$): δ (ppm) 1.2 (s, 3H, CH_3CH_2), 2.7 (m, 8H, CH_2P), 2.8 (m, 2H, CH_3CH_2), 4.5 (b, 2H, NH_2), 5.5 (m, 2H, CH_2C), 7.0–7.9 (m, 40H, C_6H_5). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ (ppm) 41.5. $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ (ppm) 19.2 (b, CH_3CH_2), 35.6 (m, CH_3CH_2), 46.7 (m, PCH_2), 122.3 (t, CH_2C), 128.9 (m, *m*-C_6H_5), 129.5 (s, *p*-C_6H_5), 133.4 (m, *o*-C_6H_5), 134.9 (m, *i*-C_6H_5). FAB-MS: *(m/z)* 686.2 (M^{+2}). *Anal*. Found: C, 55.4; H, 5.4; Cl, 10.0; N, 4.3. Calc. for $C_{32}H_{40}N_2P_2Ru$: C, 55.9; H, 5.9; Cl, 10.3; N, 4.1%.

trans-$Cl_2Ru(dppme)(NH_2CH_2CH_2CH_2Si(OCH_2CH_3)_3)_2$ **5**

Complex **2** (0.5 g, 0.49 mmol) was treated with 3-(trimethoxysilyl)propylamine (0.22 g, 1 mmol) to give **5**. Yield 0.45 g (88%) of a yellow powder. ^1H NMR ($CDCl_3$): δ (ppm) 0.1 (m, 4H, $SiCH_2$), 1.0 (m, 18H, CH_3), 1.5 (m, 4H, $SiCH_2CH_2$) 2.2 (b, 8H, NH_2 and CH_2N), 2.8 (m, 4H, CH_2P), 5.6 (m, 4H, CH_2C), 7.10–7.9 (m, 40H, C_6H_5). $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ (ppm) 45.7. $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ (ppm) 5.5 (s, $SiCH_2$), 16.6 (s, CH_3CH_2), 24.6 (s, $SiCH_2CH_2$), 43.2 (s, CH_2N), 45.4 (m, PCH_2), 56.6 (s, OCH_2), 123.0 (m, CH_2C), 126.7 (m, *m*-C_6H_5), 127.2 (s, *p*-C_6H_5), 132.1 (m, *o*-C_6H_5), 133.9 (m, *i*-C_6H_5). 135.2 (m, CH_2C). FAB-MS: *(m/z)* 1038.2 (M^+). *Anal*. Found: C, 53.4; H, 6.7; Cl, 6.9, N, 2.8. Calc. for $C_{46}H_{72}Cl_2N_2O_6P_2Si_2Ru$: C, 53.2; H, 6.9; Cl, 6.8, N, 2.7 Found: C, 53.4; H, 6.7; Cl, 6.9, N, 2.8. Found: C, 53.4; H, 6.7; Cl, 6.9, N, 2.8%.

Crystallographic analysis of *trans*-$Cl_2Ru(dppme)_2$ **2**

Single crystals suitable for X-ray structural determination were grown by slow diffusion of diethyl ether into a solution of the complex in chloroform at room temperature in sealed Schlenk tube. The selected crystal was mounted on a P4 Siemens diffractometer using perfluorinated polyether (Riedel de Haen) as protecting agent. Graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) was used for the measurement of intensity data in the ω-scan mode. The data were corrected for polarization and Lorentz effects. The structure was solved by direct methods with SHELXS-90 [22]. Refinement was carried out with full-matrix least-squares

Table 1 Selected bond lengths [Å] and bond angles (°) of complex **2**

Bond lengths	
Ru(1)–Cl(1)	2.4158(9)
Ru(1)–Cl(1A)	2.4158(9)
Ru(1)–P(1)	2.4192(10)
Ru(1)–P(1A)	2.4192(10)
Ru(1)–P(2)	2.4118(11)
Ru(1)–P(2A)	2.4118(11)
Bond angles	
P(1)–Ru(1)–P(1A)	94.47
P(2)–Ru(1)–P(2A)	94.99
P(1)–Ru(1)–P(2)	86.17
P(1A)–Ru(1)–P(2A)	86.17
P(1)–Ru(1)–P(2A)	169.81
P(2)–Ru(1)–P(1A)	169.81
Cl(1)–Ru(1)–Cl(2)	179.99
Cl(1)–Ru(1)–P(1)	95.24
Cl(1)–Ru(1)–P(1A)	84.75
Cl(1)–Ru(1)–P(2)	85.07
Cl(1)–Ru(1)–P(2A)	94.94
Cl(1A)–Ru(1)–P(1)	84.75
Cl(1A)–Ru(1)–P(1A)	95.24
Cl(1A)–Ru(1)–P(2)	94.94
Cl(1A)–Ru(1)–P(2A)	85.07

methods based on F^2 in SHELXL-97 [23] with anisotropic thermal parameters for all non-hydrogen atoms. Hydrogen atoms were included at calculated positions using a riding model. In the difference map high peaks were found. These are included as diethyl ether and chloroform, as the crystallization was carried out in using these solvents. However, they have been omitted from the molecular structure view for simplification. Details of crystal data, data collection, and structure refinement are given in Table 1. Crystallographic data has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 694035. Copies of this information may be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk).

Results and discussion

The air-stable precursor *trans*-$Cl_2Ru(dppme)_2$ (**2**) was obtained in a very good yield by substitution of $Cl_2Ru(PPh_3)_3$ (**1**) with two equivalents of dppme in dichloromethane as in Scheme 1. Complex **2** has been characterized by elemental analysis, infrared spectroscopy, mass spectrometry, ^1H, $^{13}C\{^1H\}$ and $^{31}P\{^1H\}$ NMR spectroscopy, as well as X-ray crystallography.

Complexes with general formula $RuCl_2(P_2)_2$, where P_2 is a diphosphine ligand can show only two possible geometries (*trans* and *cis*) as illustrated in Scheme 2.

The *trans*-isomer is deduced from the single $^{31}P\{^1H\}$ NMR chemical shift in $CDCl_3$ solvent which appears at approximately −1.9 ppm as a singlet; no trace of thermodynamically expected *cis*-$Cl_2Ru(dppme)_2$ isomer is visible. This should give doublet of doublets in the $^{31}P\{^1H\}$ NMR. Assignment of the *trans*-isomer in solution is consistent with the X-ray diffraction result.

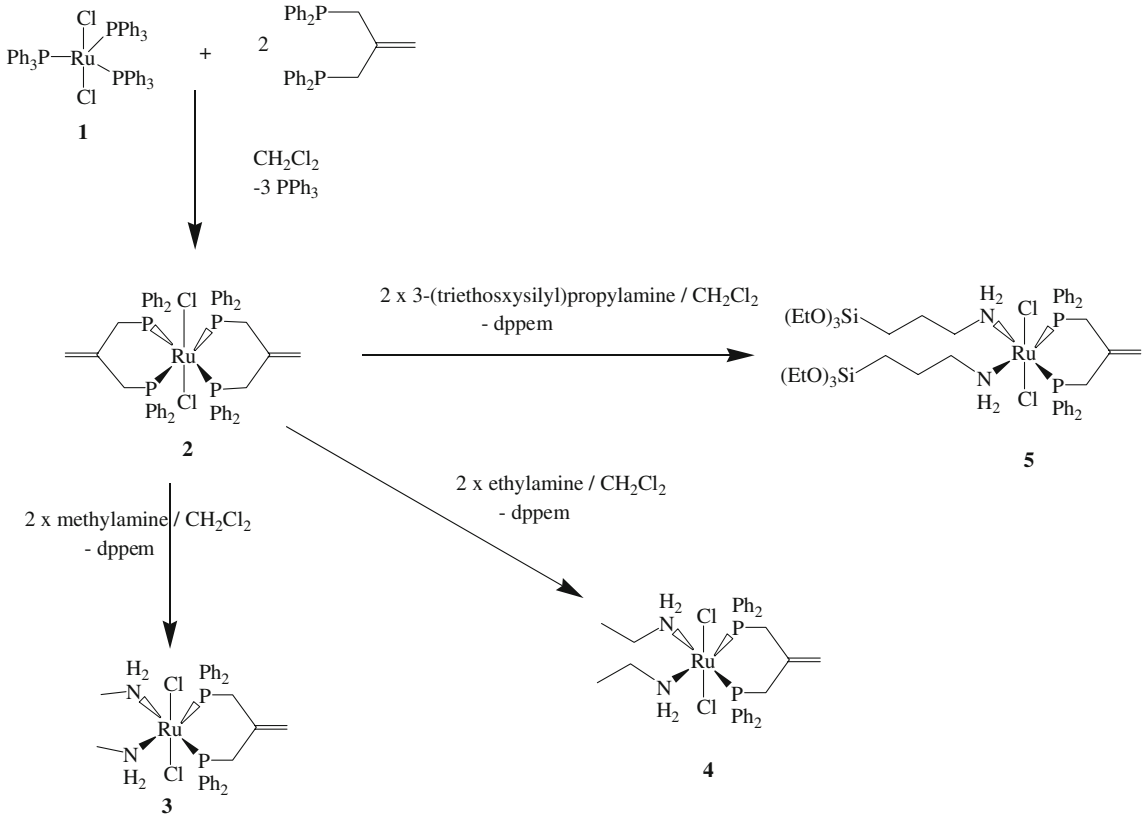
Reaction of complex **2** with two equivalents of methylamine, ethylamine, or 3-(trimethoxysilyl)propylamine gave **3**, **4,** and **5**, respectively, as *trans*-$Cl_2Ru(dppme)(NH_2R)_2$ in very good yield. The stepwise formation of complex **5** was monitored by $^{31}P\{^1H\}$ spectroscopy. In the NMR tube experiment, addition of 3-(trimethoxysilyl)propylamine to a $CDCl_3$ solution containing complex **2** generated a downfield shift of 48 ppm. One diphosphine ligand was exchanged by two molecules of alkylamine, shown by the observation that within 10 min, only traces of complex **2** at −1.9 ppm were observed, together with the free dppme at −17.2 ppm and complex **5** at 45.7 ppm (Fig. 1).

It is worth noting that a previous study showed that the double bond of the $Ph_2P)_2C{=}CH_2$, (dppen) ligand in *trans*-$[RuCl_2(dppen)_2]$ becomes activated toward nucleophilic addition upon the addition of 3-(trimethoxysilyl)propylamine [24] as in Scheme 3, while in this study one of the $H_2C{=}C(CH_2PPh_2)_2$ ligands in complex **2** is exchanged *via* primary-alkylamine addition instead of nucleophilic addition.

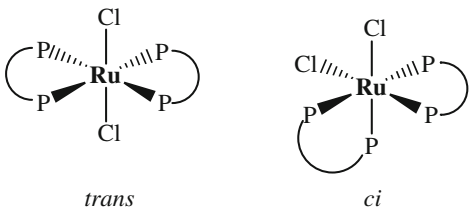
Mixed diamine(phosphine)ruthenium(II) complexes have received much attention in recent years due to their remarkable performance in selective hydrogenation [11–14] and asymmetric hydrogenation of unsaturated carbonyl compounds [25–32]. Complexes such as **5** are very important in interphase chemistry. They can be converted as primary complexes to prepare stationary phases via sol gel technique in order to transfer the system from homogenous to heterogeneous phase catalysts [33–35].

The ^1H NMR spectrum of complex **2** shows only the diphosphine ligand, whereas the ^1H NMR spectra of complexes **3–5** reveal the diphosphine and the alkylamine ligands, complex **5** assignment was supported by two-dimensional H,H-COSY experiments which establish the connectivity between protons in the amine ligand.

The $^{31}P\{^1H\}$ NMR spectra of complexes **3**, **4,** and **5** show singlets at 40.3, 41.5, and 45.7 ppm, respectively**,** indicating that the phosphine groups are chemically equivalent in solution which is compatible with the C_{2v} symmetry of these complexes. The phosphorus chemical shifts [11–16] suggest that the diphosphine ligands are *trans* to the amine



Scheme 1 Synthesis of the ruthenium(II) complexes



Scheme 2 The *trans* and *cis* possible geometrical isomers of $RuCl_2(P_2)_2$ complex

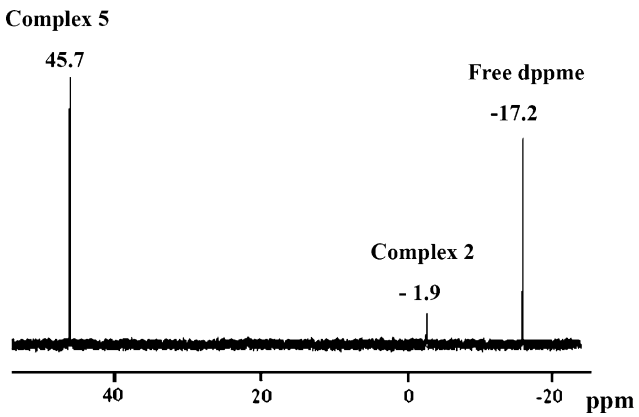


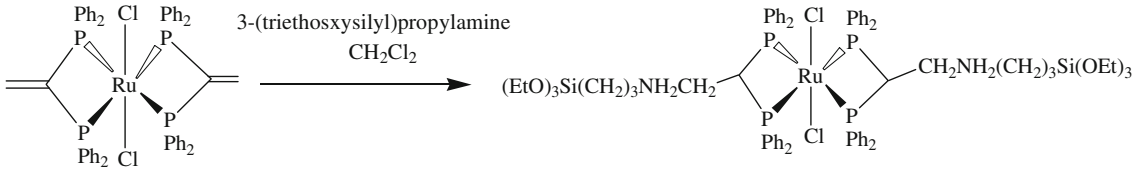
Fig. 1 $^{31}P\{^1H\}$ NMR spectroscopy of the reaction between complex **2** and excess two equimolar of 3-(trimethoxysilyl)propylamine ligand in $CDCl_3$ to produce complex **5**

co-ligands in complexes **3–5**; the chlorides must be mutually *trans* in all of these complexes.

The $^{13}C\{^1H\}$ NMR spectra also corroborate the structures given in Scheme 1. Resonances between 40–120 ppm are attributed to the aliphatic part of the phosphine ligand in complex **2.** Additional ^{13}C peaks between 5–60 ppm were observed for the amine ligands in the complexes **3–5.** AX2 splitting patterns were observed for the aliphatic and aromatic carbon atoms directly attached to phosphorus. The 135 DEPT $^{13}C\{^1H\}$ NMR spectra of complex **5** represented in Fig. 2 is a typical example to differentiate the C, CH, CH_2, and CH_3 carbon types. The chemical shifts of the corresponding fragments are in agreement with those of the free ligands.

In the IR spectrum of complex **2**, characteristic absorptions in the ranges 3272–3205 cm^1 can be assigned to C–H stretching vibrations of C=CH_2, CH_2-P and Ph, additional peaks detected at 3392–3350 cm^{-1} for complexes **3–5** are assigned to the stretching vibrations of NH_2. All other characteristic bands due to the other functional groups are also present in the expected regions.

Complex **2** has also been characterized by X-ray crystallography. The molecular structure of $[RuCl_2(dppme)_2]$ is shown in Fig. 3. Selected bond distances and bond angles are listed in Table 1.



Scheme 3 Nucleophilic addition reaction at $RuCl_2(dppen)_2$ complex

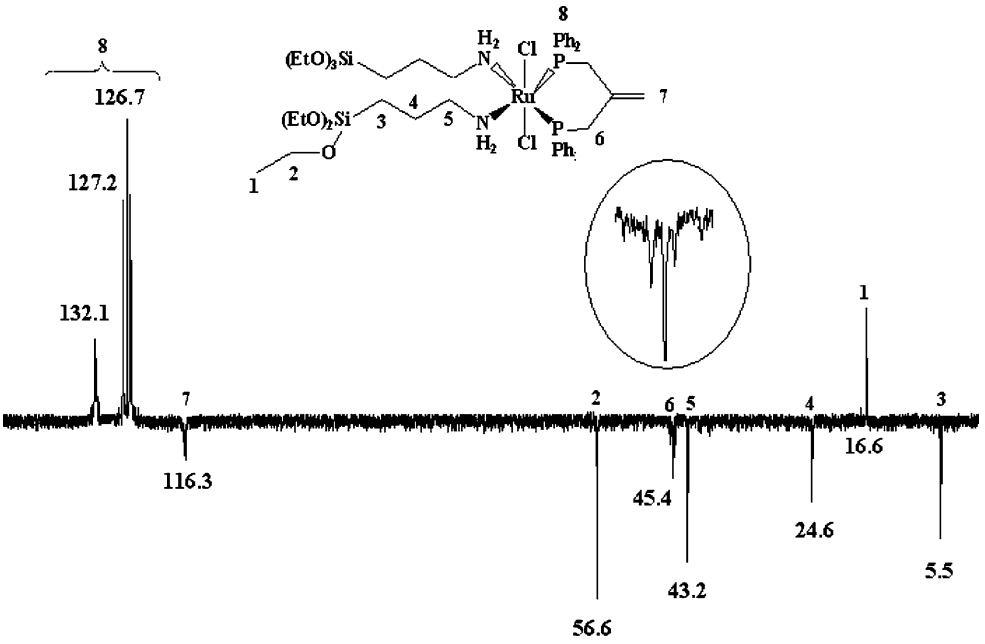


Fig. 2 The 135 DEPT $^{13}C\{^1H\}$ NMR spectrum (ppm) corroborates the structure of **5**

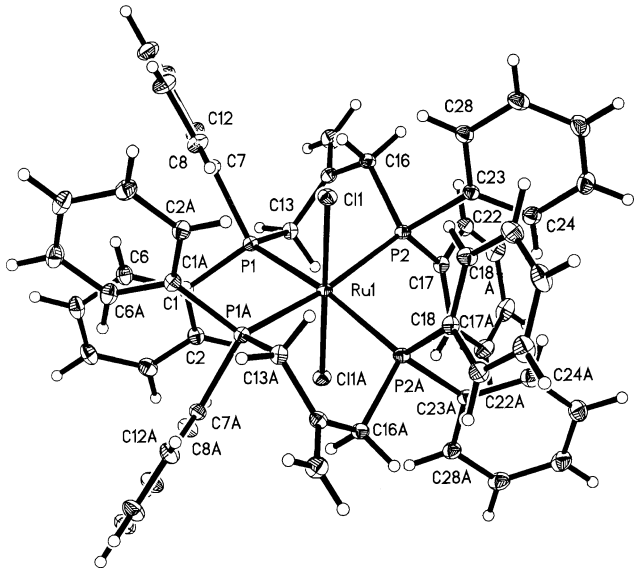


Fig. 3 Molecular structure of *trans*-$Cl_2Ru(dppme)_2$. Diethyl ether and $CHCl_3$ solvent molecules are omitted for clarity. Empirical formula $C_{60}H_{56}Cl_{14}P_4Ru$, Formula weight 1498.30, α (°) 90, β (°) 111.833(14), γ (°) 90, μ (mm^{-1}) 0.949, $F(000)$ 3032, θ range (°) 2.10 to 27.51, Reflections collected/unique 29226/7508 [$R_{int} = 0.0351$], Absorption correction empirical, Final R indices [$I > 2\sigma(I)$], R_1/wR_2 0.0633/0.1646, R indices (all data), R_1/wR_2 0.0660/0.1663, Extinction coefficient 0.00139(13)

The overall geometry around the ruthenium is best described as distorted octahedral. The two six-membered rings formed by the four PPh_2 units of the dppme ligands place to the ruthenium center in the equatorial plane. The four PPh_2 units are displaced alternately above and below the equatorial plane. The *trans* P(2)–Ru(1)–P(1A) angle is 169.81° concave toward Cl(1) and 190.19° convex toward Cl(1A), while the *trans* P(1)–Ru(1)–P(2A) angle is 169.81° concave toward Cl(1A) and 190.19° convex toward Cl(1); this distortion appears to relieve steric congestion. The Ru–Cl, Ru–P(1) and Ru–P(2) bond lengths average 2.414, 2.419, and 2.412Å, respectively. Coordination of diphosphine and two amine ligands to the $RuCl_2$ unit can lead to *cis* and *trans*-$RuCl_2$ geometry [11–14]. Although X-ray quality crystals of complexes **3–5** could not be obtained, NMR studies involving comparisons to related systems confirm that these complexes are exclusively formed as their *trans*-$RuCl_2$ isomers.

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