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Addition reactions of alkenyl complexes $[Ru(CO)Cl(HC{=}CHR)(PPh_3)_2]$ ($R = n\text{-}C_3H_7$, CMe_3, $SiMe_3$, Ph) with 3,5-dimethylpyrazole. The crystal structure of $[Ru(CO)Cl(HC{=}CHC_3H_7)(PPh_3)_2(Me_2Hpz)]$

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Abstract

The five-coordinated complexes $[Ru(CO)Cl(HC{=}CHR)(PPh_3)_2]$ ($R = n\text{-}C_3H_7$, CMe_3, $SiMe_3$, Ph) react with 3,5-dimethylpyrazole (Me_2Hpz) to give the addition products $[Ru(CO)Cl(HC{=}CHR)(PPh_3)_2(Me_2Hpz)]$. The crystal structure of the complex with $R = n\text{-}C_3H_7$ has been determined by X-ray crystallography, which shows that the two phosphine molecules, the pyrazole and alkenyl ligands, and CO and Cl occupy *trans*-positions in a slightly distorted octahedron. This simple addition reaction always occurs in $CH_2Cl_2/EtOH$, but in $Et_2O/EtOH$ there is a simultaneous transformation of the alkenyl to an alkynyl ligand in the case of the complex with R = Ph.

Introduction

In earlier papers [1,2] we described the five-coordinated complexes $[Ru(CO)Cl(R'C{=}CHR)(PPh_3)_2]$ formed by insertion of alkynes into the Ru–H bond of $[Ru(CO)ClH(PPh_3)_3]$ and elimination of one PPh_3 molecule. This more general reaction takes place in CH_2Cl_2 as solvent, but in EtOH phenylacetylene reacts to give the six-coordinated complex $[Ru(CO)Cl(PhH{=}CH_2)(PPh_3)_3]$ [1]. We present here the results of a study of the ability of the coordinatively unsaturated 5-coordinate complexes to increase their coordination number by addition of 3,5-dimethylpyrazole.

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Results and discussion

Reactions in $CH_2Cl_2/EtOH$

The previously described [1,2] five-coordinated alkenyl complexes [Ru(CO)Cl(HC=CHR)(PPh_3)$_2$] react in CH_2Cl_2/EtOH (1/1) with 3,5-dimethylpyrazole (Me_2Hpz) to give crystalline addition products of general formula [Ru(CO)Cl(HC=CHR)(PPh_3)$_2$(Me_2Hpz)] (R = n-C_3H_7, CMe_3, $SiMe_3$, Ph).

The IR spectra of all these complexes show clearly the characteristic bands of the pyrazole ligand, ν(N–H) (3250–3280 cm^{-1}) and ν(C=N) (ca. 1565 cm^{-1}), together with those of the remaining ligands. The ν(CO) stretching bands are slightly displaced towards the higher frequencies with respect to those of the relevant starting complexes.

The ^1H NMR spectra also show clearly the signals of the CH_3, CH, and NH protons of the pyrazole ligand, together with the signals of the phosphine and alkenyl ligand protons. Relative to the signals of the starting complexes there are increases in the δ and J values for the alkenylic protons of the HC=CHR ligand by 0.05–0.30 ppm and 4–5 Hz, respectively, and shifts towards the higher fields of the signals from the R group. The last effect is especially noticeable for the complexes with R = CMe_3 ($\Delta\delta = -0.973$ ppm) and $SiMe_3$ ($\Delta\delta = -2.18$ ppm), the signal of the methyl groups appearing in the $SiMe_3$ case at δ -0.415. Both effects may be due either to modification in the electronic charge upon introduction of an additional pyrazole ligand, the extent of which must depend on the relative positions of the pyrazole and alkenyl ligands in the adducts, or to a change in shielding effects of the phenyl groups of the PPh_3 ligands. The two PPh_3 molecules are mutually *trans* in the starting five-coordinated complexes [1,2], and their phenyl groups "sandwich" the metal and the remaining ligands in the equatorial plane. The phosphine phenyl signals from the pyrazole adducts are similar in appearance to those from the starting complexes, but are also shifted towards higher field ($\Delta\delta = -0.1$ to $+0.3$ ppm for the complexes with R = n-C_3H_7, CMe_3, and Ph, and $\Delta\delta = -0.5$ ppm for that with R = $SiMe_3$).

A slow decomposition in solution was observed in $CDCl_3$ by ^1H NMR spectroscopy for the complexes with R = Ph and $SiMe_3$. In the first case new signals from Me_2Hpz (δ(ppm) 1.450, 1.639, Me; 5.140, CH; 11.348, NH) and a hydridic hydrogen signal (δ(ppm) -13.523,t,J(HP) 20 Hz,1H) appear along with those of the original adduct. This may be due to formation of a hydrido pyrazole complex, [Ru(CO)ClH(PPh_3)$_2$(Me_2Hpz)], which may be an isomer of a previously described complex [3]. In the case of the complex with R = $SiMe_3$, after several days the ^1H NMR spectrum in $CDCl_3$ shows the signals of the same hydrido pyrazole complex noted in the other case, but with the signals of the alkenyl ligand HC=CHR with R = $SiMe_3$ replaced by those of an alkenyl pyrazole complex with R = H (δ(ppm): 1.639, 1.821 (s,3H,Me from Me_2Hpz), 4.668 (d,J 20 Hz,1H,HC=), 5.344 (s,1H,CH from Me_2Hpz), 5.430 (d,J 13 Hz,1H,=CH), 8.11 (dd,J 20,13 Hz,1H,=CH), 10.89 (s,1H,NH from Me_2Hpz)). This can be attributed to a slow hydrolytic cleavage of the C–Si bonds by traces of water in the solvent.

Crystal structure of the complex with R = n-C_3H_7

Selected bond lengths and angles are given in Table 1. Figure 1 shows the molecular structure. The crystal consists of individual molecules of [Ru(CO)Cl(HC=CHC_3H_7)(PPh_3)$_2$(Me_2Hpz)] held together by Van der Waals forces. The Ru

Table 1

Important bond lengths (Å) and angles (°) for [Ru(CO)Cl(HC=CHC$_3$H$_7$)(PPh$_3$)$_2$(Me$_2$Hpz)] (e.s.d.'s in parentheses)

Ru–Cl	2.500(3)	N(1)–N(2)	1.35(1)
Ru–P(1)	2.319(3)	N(1)–C(9)	1.35(2)
Ru–P(2)	2.511(3)	N(2)–C(7)	1.34(2)
Ru–N(1)	2.24(1)	O(1)–C(1)	1.18(1)
Ru–C(1)	1.79(1)	C(2)–C(3)	1.32(2)
Ru–C(2)	2.05(1)	C(3)–C(4)	1.52(2)
P(1)–C(101)	1.83(1)	C(4)–C(5)	1.48(3)
P(1)–C(111)	1.84(1)	C(5)–C(6)	1.43(4)
P(1)–C(121)	1.86(1)	C(7)–C(71)	1.51(2)
P(2)–C(201)	1.82(1)	C(7)–C(8)	1.36(2)
P(2)–C(211)	1.84(1)	C(8)–C(9)	1.38(3)
P(2)–C(221)	1.81(1)	C(9)–C(91)	1.49(2)
Mean C–C distances in benzene rings: 1.38 Å			
C(1)–Ru–C(2)	90.0(5)	Ru–N(1)–C(9)	135.7(8)
N(1)–Ru–C(2)	173.8(4)	Ru–N(1)–N(2)	119.6(8)
N(1)–Ru–C(1)	95.2(5)	N(1)–N(2)–C(7)	112(1)
P(2)–Ru–C(2)	85.4(3)	N(2)–N(1)–C(9)	105(1)
P(2)–Ru–C(1)	92.6(4)	Ru–C(1)–O(1)	177(1)
P(2)–Ru–N(1)	90.9(3)	Ru–C(2)–C(3)	134(1)
P(1)–Ru–C(2)	90.0(3)	C(2)–C(3)–C(4)	126(1)
P(1)–Ru–C(1)	88.9(3)	C(3)–C(4)–C(5)	115(1)
P(1)–Ru–N(1)	93.5(3)	C(4)–C(5)–C(6)	119(2)
P(1)–Ru–P(2)	175.2(1)	N(2)–C(7)–C(71)	120(1)
Cl–Ru–C(2)	90.2(3)	N(2)–C(7)–C(8)	106(1)
Cl–Ru–C(1)	178.3(3)	N(1)–C(9)–C(91)	122(1)
Cl–Ru–N(1)	84.5(2)	C(7)–C(8)–C(9)	107(1)
Cl–Ru–P(2)	85.7(1)	C(8)–C(9)–C(91)	128(1)
Cl–Ru–P(1)	92.9(1)	N(1)–C(9)–C(8)	110(1)
Mean C–P–C: 102.19°			
Mean Ru–P–C: 116.04°			
Mean C–C–C in benzene rings: 119.99°			

atom displays a distorted octahedral coordination, with Ru, Cl, C(1), C(2) and N(1) atoms in the equatorial plane (largest deviation from mean plane −0.0421(5) Å in Ru atom) and two P atoms of PPh$_3$ ligands occupy the axial positions (P(1)–Ru–P(2) angle is 175.2(1)°). The Ru–C(1) (1.79(1) Å and Ru–C(2) (2.05(1) Å) distances are similar to those found in [Ru(CO)Cl(PhC=CHPh)(PPh$_3$)$_3$] [1] (1.79(1) and 2.03(1) Å). The C92)–C(3) bond length of 1.32(1) Å is indicative of a double bond and similar to that found in the complex just mentioned, 1.37(2) Å. The bond lengths Ru–Cl, 2.499(2) Å, and Ru–N(1), 2.24(1) Å, are as expected [1,2]. The C(2)–C(3)–C(4)–C(5) and C(3)–C(4)–C(5)–C(6) torsion angles, −119(2) and 167(2)°, correspond to the zig-zag geometry of the pentenyl ligand. The Ru–P(1) distance, 2.319(3) Å, falls in the usual range [1–3], but the Ru–P(2) distance, 2.511(3) Å, is longer. Similar differences have been found previously [4,5]. The pyrazole ring is planar (largest deviation from mean plane −0.04(2) Å in C(71) atom) and has the usual features [3].

Reactions in Et$_2$O / EtOH

Reaction of [Ru(CO)Cl(HC=CHPh)(PPh$_3$)$_2$] with Me$_2$Hpz in Et$_2$O/EtOH leads

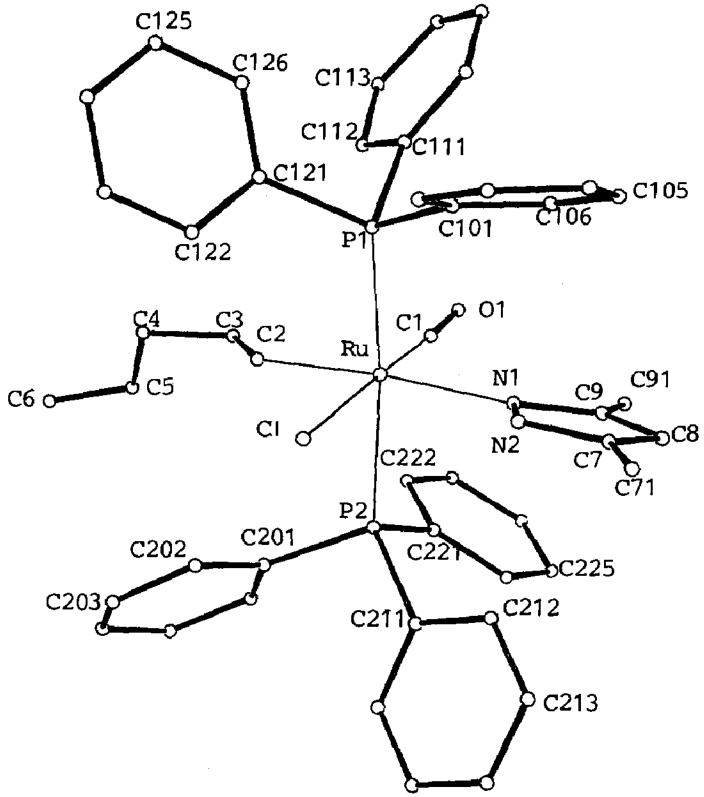

Fig. 1. ORTEP drawing of the molecular structure of [Ru(CO)Cl(HC=CHC$_3$H$_7$)(PPh$_3$)$_2$(MeHpz)]. The atom numbering is the same as that in Table 3.

to formation of an alkynyl complex of composition [Ru(CO)Cl(C≡CPh)(PPh$_3$)$_2$-(Me$_2$Hpz)]. The dehydrogenation of the alkenyl ligand may occur via an intermediate η-phenylacetylene hydride complex. The presence of this intermediate has been postulated previously for the interconversion of [Ru(CO)Cl(PhC=CH$_2$)-(PPh$_3$)$_3$] and [Ru(CO)Cl(HC=CHPh)(PPh$_3$)$_2$] in MeOH/CH$_2$Cl$_2$ [1].

This behaviour was not observed in the reactions of the other five-coordinated alkenyl complexes here studied, which give the same adducts in Et$_2$O/EtOH as in CH$_2$Cl$_2$/EtOH.

From our results we conclude that all the five-coordinate complexes [Ru(CO)Cl(HC=CHR)(PPh$_3$)$_2$] here studied react in CH$_2$Cl$_2$/EtOH with 3,5-dimethylpyrazole to give six-coordinated species [Ru(CO)Cl(HC=CHR)(PPh$_3$)$_2$(Me$_2$-Hpz)] in which the two phosphine molecules, the Me$_2$Hpz and alkenyl ligands, and CO and Cl, are mutually *trans*.

The presence of [Ru(CO)ClH(Me$_2$Hpz)(PPh$_3$)$_2$] signals in the ^{1}H NMR spectrum of [Ru(CO)Cl(HC=CHPh)(PPh$_3$)$_2$(Me$_2$Hpz)] in CDCl$_3$ after several days and the formation of an alkynyl complex in the reaction of [Ru(CO)Cl(HC=CHPh)-(PPh$_3$)$_2$] with Me$_2$Hpz in Et$_2$O/EtOH can be related to the existence of an equilibrium between several species in solution (eq. 1):

$$[\mathrm{Ru(CO)Cl(HC{=}CHPh)(PPh_3)_2(Me_2Hpz)}] \rightleftarrows (\mathrm{Ru(CO)ClH(PPh_3)_2(Me_2Hpz)}) + \mathrm{PhC{\equiv}CH} \rightarrow (\mathrm{Ru(CO)Cl(C{\equiv}CPh)(PPh_3)_2(Me_2Hpz)}) + \mathrm{H_2} \quad (1)$$

Experimental

General comments

The ^1H NMR spectra were recorded on a Bruker WM 360 spectrometer at 360 MHz; shifts are relative to TMS. IR spectra (KBr discs) were recorded in a Pye-Unicam SP3-300S instrument. The $[Ru(CO)Cl(HC{=}CHR)(PPh_3)_2]$ complexes were prepared as previously described ($R = C_3H_7$, Ph [1]; $R = CMe_3$, $SiMe_3$ [2]).

$[Ru(CO)Cl(HC{=}CHR)(PPh_3)_2(Me_2Hpz)]$ complexes

An excess of Me_2Hpz was added to the red-orange solution of $\{Ru(CO)Cl(HC{=}CHR)(PPh_3)_2\}$ (0.3 g) in CH_2Cl_2/EtOH (1/1) (10 ml) until the solution became light yellow. After 10 min stirring the solution was concentrated. Light green or yellow crystalline products were obtained by slow evaporation from an EtOH solution in air. All these products are stable to air (yield 80–90%). The crystals of the compound with $R = C_3H_7$ were suitable for X-ray structure determination.

Complex with $R = C_3H_7$ (yellow). (Found: C, 66.6; H, 5.4; N, 3.3.

Table 2

Crystal data, data collection and structure refinement

Crystal data	
Formula	$C_{47}H_{47}N_2OP_2Ru$
Crystal habit	Transparent yellow parallelepiped
Crystal size (mm)	$0.20 \times 0.16 \times 0.16$
Symmetry	Monoclinic, $\mid P2_1 \mid a$
Unit cell dimensions	18.530(2), 12.623(2), 19.379(2) Å 90.0, 110.328(8), 90.0°
Packing: V (Å^3), Z	4252.5(7), 4
D_c (g cm^{-3}), M, $F(000)$	1.335, 854.4, 1768
μ (cm^{-1})(Mo-K_α)	5.34
Experimental data	
Technique	Four circle diffractometer: Enraf–Nonius CAD-4 Bisecting geometry Graphite-oriented monochromator: Mo-K_α
Scanning range for θ	$2 < \theta < 26$
Number of reflexions:	
Measured	8700
Observed	4555 ($2\sigma(I)$ criterion)
Absorption	Empirical absortion correction [6]
Solution and refinement	
Solution	Patterson and Fourier synthesis
Refinement	Anisotropic for all atoms
H atoms	Geometrical calculation
w-scheme	Empirical as to give no trends in $\langle w\Delta^2F\rangle$ vs. $\langle \mid F_o \mid \rangle$ and $\langle \sin\theta/\lambda \rangle$ [8]
Final R and Rw	8.2, 7.9
Computer and programs	Vax 11/750, XRAY76 [7], ORTEP [9].
Scattering factors	Int. Tables for X-Ray Crystallography [10]
Anomalous dispersion	Applied for Ru, Cl, P and C. Int. Tables X-Ray Crystallography [10]

Table 3

Atomic coordinates for $C_{47}H_{47}N_2OP_2Ru$

Atom	x	y	z
Ru	0.1401(1)	0.2097(0)	−0.2402(0)
Cl	0.2564(2)	0.2474(2)	−0.1298(1)
P(1)	0.1575(2)	0.0279(2)	−0.2273(2)
P(2)	0.1308(2)	0.4070(2)	−0.2577(2)
N(1)	0.0767(5)	0.2186(8)	−0.1606(5)
N(2)	0.1163(6)	0.2117(9)	−0.0875(5)
O(1)	0.0028(5)	0.1699(8)	−0.3733(5)
C(1)	0.0561(6)	0.1869(8)	−0.3194(7)
C(2)	0.2062(6)	0.2135(10)	−0.3060(5)
C(3)	0.1911(7)	0.2060(11)	−0.3777(7)
C(4)	0.2488(8)	0.2151(15)	−0.4173(7)
C(5)	0.2337(12)	0.3031(20)	−0.4712(12)
C(6)	0.2919(14)	0.3333(20)	−0.5002(14)
C(7)	0.0701(9)	0.2220(12)	−0.0476(8)
C(71)	0.1036(11)	0.2206(16)	0.0352(9)
C(8)	−0.0023(9)	0.2351(11)	−0.0964(10)
C(9)	0.0025(7)	0.2315(9)	−0.1659(8)
C(91)	−0.0611(7)	0.2441(12)	−0.2381(9)
C(101)	0.1422(6)	−0.0194(8)	−0.1444(6)
C(102)	0.2034(7)	−0.0407(10)	−0.0794(6)
C(103)	0.1902(8)	−0.0660(11)	−0.0145(7)
C(104)	0.1167(10)	−0.0694(12)	−0.0134(8)
C(105)	0.0557(8)	−0.0481(12)	−0.0761(8)
C(106)	0.0680(7)	−0.0218(9)	−0.1416(7)
C(111)	0.0959(7)	−0.0590(9)	−0.3000(7)
C(112)	0.0921(9)	−0.0400(11)	−0.3710(7)
C(113)	0.0489(11)	−0.1040(13)	−0.4297(8)
C(114)	0.0092(11)	−0.1878(14)	−0.4144(10)
C(115)	0.0108(9)	−0.2093(14)	−0.3463(11)
C(116)	0.0549(9)	−0.1451(11)	−0.2871(8)
C(121)	0.2526(7)	−0.0323(9)	−0.2164(6)
C(122)	0.3188(7)	0.0245(10)	−0.1842(8)
C(123)	0.3905(8)	−0.0235(12)	−0.1711(9)
C(124)	0.3963(9)	−0.1251(14)	−0.1919(9)
C(125)	0.3306(10)	−0.1807(11)	−0.2242(9)
C(126)	0.2580(9)	−0.1364(11)	−0.2369(8)
C(201)	0.2047(7)	0.4666(9)	−0.2868(7)
C(202)	0.2806(9)	0.4485(12)	−0.2464(12)
C(203)	0.3375(11)	0.5009(15)	−0.2656(19)
C(204)	0.3202(17)	0.5627(20)	−0.3238(18)
C(205)	0.2461(17)	0.5844(20)	−0.3638(12)
C(206)	0.1870(9)	0.5351(15)	−0.3451(7)
C(211)	0.1354(6)	0.4946(9)	−0.1803(6)
C(212)	0.1060(7)	0.4651(10)	−0.1267(7)
C(213)	0.1074(8)	0.5347(12)	−0.0700(7)
C(214)	0.1379(9)	0.6327(12)	−0.0666(8)
C(215)	0.1653(12)	0.6644(12)	−0.1206(10)
C(216)	0.1653(11)	0.5963(11)	−0.1771(8)
C(221)	0.0405(6)	0.4418(8)	−0.3282(5)
C(222)	0.0257(7)	0.4029(9)	−0.3991(6)
C(223)	−0.0446(8)	0.4215(12)	−0.4533(7)
C(224)	−0.1006(8)	0.4747(11)	−0.4378(7)
C(225)	−0.0880(7)	0.5147(11)	−0.3686(8)
C(226)	−0.0157(7)	0.4975(10)	−0.3126(7)

$C_{47}H_{47}ClN_2OP_2Ru$ calc: C, 66.1; H, 5.6; N, 3.3%). IR ν(CO) 1928vs, ν(C=C) 1650vw; ν(C=N) 1570m, ν(NH) 3250m cm^{-1}. ^1H NMR (ppm): δ 0.524(t, J 7.8 Hz,3H,Me), 0.890(q, J 7.8 Hz,2H,CH_2), 1.665(m,5H,Me from $Me_2Hpz + CH_2$), 1.803(s,3H,Me from Me_2Hpz), 4.650(dt, J 16,7.8 Hz,1H,=CH), 5.338(s,1H,CH from Me_2Hpz), 7.119(d, J 16 Hz,1H,HC=), 7.12–7.25(m,20H,Ph), 7.25–7.40(m,10H,Ph), 10.939(s,1H,NH from Me_2Hpz).

Complex with R = CMe_3 (yellow). (Found: C, 66.2; H, 5.9; N, 3.2. $C_{48}H_{49}ClN_2OP_2Ru$ calc: C, 66.4; H, 5.7; N, 3.2%). IR ν(CO) 1926vs, ν(C=N) 1567m, ν(NH) 3280m cm^{-1}. ^1H NMR (ppm): δ 0.492(s,9H,3Me), 1.667(s,3H,Me from Me_2Hpz), 1.854(s,3H,Me from Me_2Hpz), 4.77(d, J 17.6 Hz,1H,=CH), 5.40(s,1H,CH from Me_2Hpz), 7.08–7.28(m,21H,Ph + HC=), 7.28–7.45(m,10H,Ph), 11.028(s,1H,NH from Me_2Hpz).

Complex with R = $SiMe_3$ (yellow). (Found: C, 63.5; H, 5.7; N, 3.2. $C_{47}H_{49}ClN_2O$ P_2RuSi calc: C, 63.8; H, 5.6; N, 3.2%). IR ν(CO) 1930vs, ν(C=C) 1505m, ν(C=N) 1565m, ν(NH) 3295m cm^{-1}. ^1H NMR (ppm): δ −0.415(s,9H,$SiMe_3$), 1.686, 1.812(s,3H,Me from Me_2Hpz), 5.385(s,1H,CH from Me_2Hpz), 5.481(d, J 19 Hz,1H,=CH), 7.00–7.19(m,20H,Ph), 7.20–7.35(m,10H,Ph), 8.749(d, J 19 Hz,1H,HC=), 10.999(s,1H,NH from Me_2Hpz).

Complex with R = Ph (green). (Found: C, 67.4; H, 5.2; N, 3.1. $C_{50}H_{45}ClN_2OP_2Ru$ calc: C, 67.6; H, 5.1; N, 3.2%). IR ν(CO) 1928vs, ν(C=C) 1550m, ν(C=N) 1575m, ν(NH) 3210 cm^{-1}. ^1H NMR (ppm): δ 1.688, 1.849(s,3H,Me from Me_2Hpz), 5.373(s,1H,CH from Me_2Hpz), 5.639(d, J 17 Hz,1H,=CH), 6.683(d, J 6 Hz,2H,Ph), 6.824(t, J 6 Hz,1H,Ph), 7.002(t, J 6 Hz,2H,Ph), 7.05–7.15(m,12H,Ph), 7.15–7.25-(m,6H,Ph), 7.25–7.35(m,12H,Ph), 8.667(d, J 17 Hz,1H,HC=), 11.016(s,1H,NH from Me_2Hpz).

[Ru(CO)Cl(C≡CPh)(PPh_3)$_2$(Me_2Hpz)] (reaction in EtOH / Et_2O)

When the reaction of the alkenyl compound with R = Ph was performed under the same conditions but in Et_2O/EtOH, yellow crystals were obtained which correspond to an alkynyl complex also containing the pyrazole ligand (yield 85%). (Found: C, 67.6; H, 5.0; N, 3.1. $C_{50}H_{43}ClN_2OP_2Ru$ calc: C, 67.8; H, 4.9; N, 3.2%). IR ν(CO) 1945vs, 1925sh; ν(C≡C) 2070s, ν(C=N) 1548m, ν(NH) 3180m cm^{-1}. ^1H NMR (ppm): δ 1.525, 1.694 (s,3H,Me from Me_2Hpz), 5.192(s,1H,CH from Me_2Hpz), 6.815(d, J 8 Hz,2H,Ph), 6.918(t, J 8 Hz,1H,Ph), 7.003(t, J 8 Hz, 2H,Ph), 7.10–7.40(m,20H,Ph), 7.40–7.80(m,10H,Ph), 10.93(s,1H,NH from Me_2Hpz).

Structure determination

X-ray diffraction data and experimental details on the structure solution and refinement are given in Table 2. The final atomic coordinates are given in Table 3. Lists of structure factors and thermal parameters are available from the authors.

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