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Dimeric Chlorohydrido(tris-triphenylphosphine)ruthenium(II), $\{[(C_6H_5)_3P]_3RuHCl\}_2$, its Preparation, Characterization, and Chemical Reactivity

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*Treatment of dihydridotetrakis(triphenylphosphine)ruthenium(II), I, with 1,4-dichlorooctaphenyltetrasilane, II, in the presence of triethylamine affords dimeric chlorohydrido(*tris*-triphenylphosphine)ruthenium(II), III. The combined analytical and spectroscopic characterization of III are consistent with a bis(μ-chloro) geometry with structurally equivalent PPh_3 moieties on the NMR time scale. III undergoes spontaneous decomposition in benzene and THF with quantitative evolution of dihydrogen and formation of $[(PPh_3)_3RuCl]_x$. The hydrolysis of III is described at both 80 °C and 100 °C with the former temperature affording a stoichiometric quantity of dihydrogen while the latter temperature affords additional dihydrogen.*

Introduction

In an effort to further our understanding of cyclometallo polysilane systems, we have investigated several chemical reactions in an effort to prepare species containing the backbone, $\overline{\mathrm{Ru{-}Si{-}Si{-}Si{-}Si}}$. Our previous investigation in this area have afforded species with the backbones, $\overline{\mathrm{Pt{-}Si{-}Si{-}Si{-}Si}}$ [2] and $\overline{\mathrm{Ti{-}Si{-}Si{-}Si{-}Si}}$ [3]. With regard to analogous cyclopolysilaneruthenium systems, none have been reported to date – including this investigation. However, one of our attempts at synthesis of such a system afforded an extremely interesting new Ru(II) derivative – $\{[(C_6H_5)_3P]_3RuHCl\}_2$. As will be developed in this report, this species is not the well known $[(C_6H_5)_3P]_3RuHCl\cdot$solvate [4].

Result and Discussion

Treatment of dihydridotetrakis(triphenylphosphine)ruthenium(II), $(Ph_3P)_4RuH_2$, (I) with 1,4-dichlorooctaphenyltetrasilane, $Cl_2Si_4Ph_8$, (II) results in the formation of dimeric chlorohydridotris(triphenylphosphine)ruthenium(II), (III) as described by eqn. 1.

$$(Ph_3P)_4RuH_2 + Cl_2Si_4Ph_8 \rightarrow \frac{1}{2}[(Ph_3P)_3RuHCl]_2 + PPh_3 + \frac{1}{2}H_2 + \text{silane product} \qquad (1)$$

Formation of III requires the addition of a benzene solution of II to a benzene suspension of I in the presence of excess NEt_3. Under these reaction conditions an immediate solution color change takes place from yellow to burgandy red accompanied by precipitation of red III and evolution of H_2, eqn. 1.

The silane product (not discussed in this report) and displaced PPh_3, eqn. 1, were removed from III by filtration employing Et_2O (III is insoluble in Et_2O). III was subsequently shown to be analytically pure, dimeric in $CHCl_3$, and diamagnetic.

Spectroscopic Investigation of III, Infrared

The infrared spectrum of III, Fig. 1, contains absorptions attributable to P–Ph vibrations [5] (3045, 1485, 1433, 1092, 748, 696, 678, 522, 512, 499, 490, *etc.*) and Ru–H [6–10] (2030 cm^{-1}), but no absorption unambiguously assignable to Ru–Cl [4]. One possible formulation for III consistent with the analytical data, an *ortho*-metallated species, is not consistent with the infrared data (or with the ^{31}P NMR data). Absorptions in the regions 1500–1600 [11], 1400–1450 [12], 1100 [13], and 730 cm^{-1} [11] have been assigned as arising from the *ortho-*

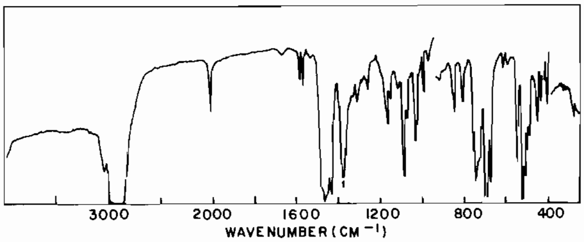

Fig. 1. Infrared spectrum of $[(PPh_3)_3RuHCl]_2$, nujol.

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disubstituted phenyl ring of an *ortho*-metallated phosphine, and although III does have absorptions in some these regions, the absorptions most characteristic of *ortho*-metallation in similar complexes, *e.g.*, $(PPh_3)_2RuH(o\text{-}C_6H_4PPh_2)Et_2O$, at *ca.* 1550 and *ca.* 1410 cm^{-1} [10], are not present in the infrared spectrum of III. As has been noted recently, absorptions at 1100 and 730 cm^{-1}, often invoked to identify *ortho*-metallated complexes, are complicated by the presence of vibrations from the mono substituted phenyl rings in triphenylphosphine complexes [10].

NMR Spectroscopy

Were one to predict the ^1H NMR spectrum of III, it would contain structural envelopes at 2.5–3.0 τ, due to the phenyl ring hydrogens, and only one other feature – a hydride resonance upfield from TMS. This is indeed what is found for III in C_6D_6, Fig. 2.

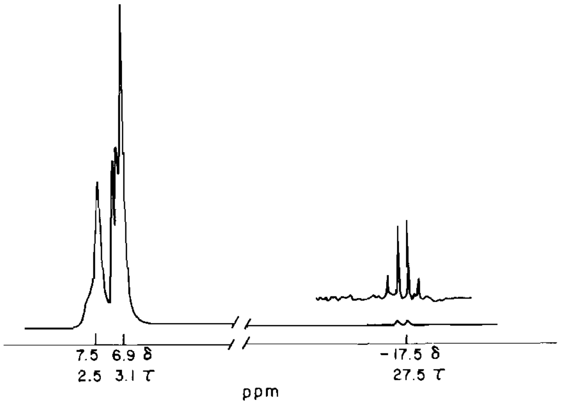

Fig. 2. ^1H NMR spectrum of $[(PPh_3)_2RuHCl]_2$.

Two envelopes of absorptions are centered at 2.5 τ and 3.1 τ respectively and a hydride resonance at 27.5 τ split into a quarter with J_{PH} = 26 Hz. The hydride resonance requires that all three phosphines, associated with III are equivalent on the nmr time scale at room temperature. Unfortunately III decomposes in solution with evolution of H_2 (to be subsequently discussed) and accompaning loss of the hydride resonance with time. Consequently the relative ratio of phenyl hydrogen to hydride hydrogen, obtained by integration, ranges from nearly the expected 45:1 to greater than 1000:1 depending on the amount of time the sample is held at room temperature before the NMR spectrum is obtained. For example, if a saturated solution of III, in C_6D_6, is subjected to nmr data collection immediately upon preparation, integration values of 51:1 are obtained. This procedure requires that the sample remain at room temperature for 45 min. Within a few hours, the relative area under the hydride resonance is decreased noticeably. An additional hindrance to accurate integration data arises from the presence of trance amounts of C_6H_6 in C_6D_6. Separation of the area due to trace C_6H_6 resonance from the area due to PPh_3 resonances is not always unambiguous.

The ^{31}P nmr spectrum of III, in C_6D_6, shows one feature, a single absorption centered at 56.9 ppm downfield from Ext H_3PO_4, in the range expected for PPh_3 on Ru. (The insolubility of III in toluene precluded low temperature studies). The three phosphines are thus equivalent on the nmr time scale at room temperature.

Comparison of III with $(PPh_3)_3RuHCl$, IV

Although the suggested formulation for $\{(PPh_3)_3\text{-}RuHCl\}_2$, III, is consistent with the analytical and spectroscopic data the most likely alternative formulation is, $(PPh_3)_3RuHCl\cdot$solvate, IV. This latter material does indeed have three equivalent phosphines, one Cl and one H *per* Ru. The nmr spectra obtained for III is very similar to that absorbed for IV, however, III is not IV as a thorough comparison of their physical, analytical, and spectral properties indicate. III is a red solid, mp 168 °C, while IV is a violet-black solid, mp 218–220 °C [4]. From an analytical standpoint, III and IV differ only by the added solvent molecule benzene. This difference, nevertheless, has a significant effect on the analytical results, Table I. Furthermore, hydrolysis of III results in the formation of one mol H_2 per 9.3 × 10^2 g III (923.97 g/mol monomer III) rather than one mol H_2 per 1002.0 g, the theoretical molecule weight of IV, *i.e.*, $(PPh_3)_3RuHCl\cdot C_6H_6$.

TABLE I. Comparison of Analytical Data for $[(Ph_3P)_3\text{-}RuHCl]_2$, III, and $[Ph_3P)_3RuHCl\cdot C_6H_6$, IV.

	wt.%		
	C	H	Cl
Calcd., III	70.19	4.98	3.84
Found, III	70.16	5.02	3.84
Calcd., IV	71.91	5.19	3.54

Spectroscopic differences between III and IV also exist. Although the infrared spectra of both compounds are dominated by PPh_3 absorptions, the modes due to Ru–H are slightly different, 2035 cm^{-1} for III and 2020 cm^{-1} for IV [4]. In addition ν Ru–Cl at 282 cm^{-1} is assigned in IV [4] and while an absorption appears near this frequency for III, 284 cm^{-1}, it cannot unambiguously be assigned ν Ru–Cl because we have observed an absorption at this frequence in infrared spectrum of the products of reactions between $(PPh_3)_3RuH_2$ with both Si_4Ph_8 and $H_2Si_4PH_8$; obviously these latter reaction products cannot contain ν Ru–Cl.

The ^1H nmr spectrum of III *in C_6D_6* has been described and is different from that reported for IV.

For example, III shows a split pattern of resonances in the phenyl region in contrast to the one broad absorption for IV. Also, IV does not afford an upfield resonance, *in* C_6D_6, even for supersaturated solution, whereas III exhibits a quartet at 27.4 τ, $J_{PH} = 26$ Hz (IV does exhibit a quartet centered at 28.08 τ in CH_2Cl_2) [4]. A single broad ^{31}P nmr absorption appears at 56.9 ppm for III and at 59.0 ppm for IV [14]. These small but discernible differences in the nmr spectra substantiate the differences between III and IV.

An important chemical difference between III and IV is the solution decomposition of III to quantitatively afford H_2; this chemical property has not been reported for IV.

Solution Decomposition of $[(Ph_3P)_3RuHCl]_2$, *III, Formation of* $[Ph_3P)_3RuCl]_x$, *V*

III results from the reaction of I with II, eqn. 1, employing benzene as the solvent, in the presence of excess NEt_3. Under these reaction conditions, III is only slightly soluble in benzene and precipitates as a red crystalline material and maybe isolated after washing with toluene to remove non-volatile reaction by-products. With regard to the reaction described by eqn. 1, the presence of NEt_3 is necessary for the indicated chemical transformation, however, NEt_3 may be subsequently quantitatively isolated. No $NHEt_3^+Cl^-$ is formed, eqn. 1, when one employs high purity NEt_3. When solutions of III are prepared in benzene H_2 evolution immediately commences, however, the presence of NEt_3 greatly retards this reaction. The solubility of III is much greater in THF than benzene, and solutions of III in the former are rapidly transformed at 25 °C according to eqn. 2.

$$\frac{1}{2}[(Ph_3P)_3RuHCl]_2 \xrightarrow[\text{or THF}]{\text{benzene}} \frac{1}{2}H_2 + [(Ph_3P)_3RuCl]_x \qquad (2)$$

Characterization of $[(Ph_3P)_3RuCl]_x$, *V*

The ^{31}P NMR spectrum of the solution resulting in the transformation of III to V, eqn. 2, contains only a single broad absorption at 57 ppm from external H_3PO_4; no free PPh_3 is present and there is no spectropic indication that *o*-metallation has taken place. The latter type species would be expected to exhibit multiple ^{31}P absorptions, *e.g.* $(C_6H_4PPh_2)(PPh_3)_2Ru(H)(Et_2O)$ exhibits ^{31}P absorptions at 54.4, 20.0 and 26.1 ppm, $J_{PP} = 23.7$, 17.7 and 17.1 Hz [10].

The ^1H NMR spectrum of V shows only features common to PPh_3 metal complexes-absorptions centered at 2.3 and 2.7 τ; no hydride resonance is present. Analysis of the integration data indicates that if a hydride resonance was present it would be clearly visible.

The infrared spectrum of V is also consistent with the absence of an RuH derivative; there are no absorptions in the range 1600–2100 cm^{-1} [6–9], νRuH, or at *ca.* 1550 cm^{-1} and 1410 cm^{-1} indicative of an *ortho*-metallated species [10].

Possible Geometries for III and V

The combined analytical and molecular weight data obtained for III are consistent with a dimeric formulation and the ^{31}P NMR data indicate equivalent PPh_3 ligands. Furthermore, the high field ^1H NMR quartet (1:3:3:1) is indicative of three equivalent PPh_3 ligands, while the observe νRuH frequency is in the range for terminal rather than bridging hydride.

The most likely geometry for III, in solution at 25 °C, consists of a bis-μ-chloro bridged species with Ru in a pseudo octahedral environment and with PPh_3 exchange taking place.

It is interesting to note the recent preparation of bis(μ-chloro)bis-triphenylphosphene-ruthenium(II), $[(PPh_3)_2RuCl_2]_2$, as a result of the presence of pyridine derivatives interacting with $(PPh_3)_3RuCl_2$ [15]. Recall the synthesis of III takes place in the presence of NEt_3, *i.e.* under reaction conditions similar to those related to formation of dimeric $[(PPh_3)_2RuCl_2]_2$. Dimerization is not limited to those species which would be four coordinate if monomeric, *e.g.*, $(MeCN)(PPh_3)_2RuCl_2$ exist as a dimer [16].

With regard to V, this species contains three equivalent PPh_3 ligands in solution, based on ^{31}P NMR data, and the geometry most likely also involves chloro bridges. This latter possibility is solely based on the tendency for chlororuthenium species to associate *via* μ-Cl bonding. Future investigations of V will include a molecular weight determination as well as magnetic measurements.

Hydrolysis of III

Treatment of III with D_2O, in the presence of piperidine, affords only trace amounts of noncondensable gas at 25 °C, however, reaction at 80 °C for 3 hours affords one mol of the mixture, $H_2/HD/D_2$ (in the mol ratios 8.5:16:4.5), per mol Ru. Based on the gaseous product mol ratios, H–D exchange has taken place. No further dihydrogen evolution is observed during a 24 hr time period. When the temperature of a hydrolysis mixture, employing H_2O, is raised to 110 °C additional dihydrogen is evolved. Repeated heating to 100 °C and cooling to 25 °C with removal of H_2, through 8 cycles affords 4.6 mol H_2 per mol of Ru initially present as III. After 8 thermal cycles, there was no evidence to suggest termination of H_2 evolution.

Thoughts on the Mechanism of Formation and Decomposition of III

Previous investigations of the lability of I with respect to PPh_3 dissociation have resulted in the conclusions [17]: 1. dissociation of I is negligible in non-polar solvents, 2. I dissociated extensively in polar solvents with the solvent occupying the single vacant coordination site (in picoline, pyridine, dimethylamine, ammonia or DMF), 3. the dissociation of PPh_3 is reversed with solvent removed, and 4. the *cis*-dihydrido configuration for $(Ph_3P)_4{}_3RuH_2$ is maintained in polar and non-polar solvents. Therefore, with respect to the reaction represented by eqn. 1, the initial step most likely involves a weak NEt_3–I interaction facilitating H–Cl exchange (between I and $Cl_2Si_4Ph_8$) and subsequent dimer formation, *i.e.* $[(Ph_3P)_3RuHCl]_2$. As previously indicated, the Ru reaction product, eqn. 1, is stable with respect to H_2 evolution in the presence of excess NEt_3. When the reaction mixture, eqn. 1, is subjected to high vacuum, benzene and NEt_3 are completely removed to afford $[(Ph_3P)_3RuHCl]_2$, III.

With regard to the stability of III towards H_2 evolution, III is quite stable in contact with large excesses of both toluene and diethylether (III is insoluble in both of these reagents), but evolves H_2 in benzene and more rapidly in THF. These relative rates of decomposition parallels the relative solubilities in the indicated solvent systems. The presence of NEt_3 during the initial synthesis would appear to decrease the solubility of (III) in benzene thereby facilitation isolation as a stable precipitate.

The presently favored geometry for III involves a bis-μ-chloro system with 3 equivalent PPh_3 ligands on the NMR time scale. A possible decomposition intermediate, eqn. 2, is a triple bridged dimer with bis-μ-chloro-mono-μ-hydrido bridge bonding. This species would possess both six and seven coordinate ruthenium with the latter ruthenium associated with a terminal hydrido moiety. Stable seven coordinate ruthenium complexes have recently been established, $(Ph_3P)_3RuH_3SiR_3$ [18–20]. With regard to the suggested intermediate dimer, reductive elimination of H_2 would be expected to be accompanied by formation of material of composition, $[(Ph_3P)_3RuCl]_x$, *i.e.* that observed for V.

We are currently investigating the chemical reactivity of III as related to hydrogenation catalysis and will report these results in the future.

Experimental

Equipment

Materials were handled employing standard vacuum line and dry box techniques. The latter utilized Burdett Prepurified, 99.98%, dinitrogen dried and deoxygenated by Linde 13x molecular sieves and Fisher Scientific Ridox respectively. Filtrations were carried out, *in vacuo*, employing a medium porosity glass frit while all detachable glass to glass seals were *via* Fusion-Urry o-ring joints utilizing Viton A o-rings. Molecular weights were determined with a Model 301 A Vapor Pressure Osmometer manufactured by Mechrolab, Incorporated. Infrared spectra were recorded on a Perkin-Elmer Model 457 grating spectrophotometer accurate to ±2 cm^{-1} (calibrated against polystyrene). Solids were examined as Nujol or Fluorolube mulls between either potassium bromide or cesium iodide plates. The ^1H nmr spectra were recorded with Varian Associates A-60 or EM-360 spectrometer operating at 60 MHz. Fourier transformer ^{31}P, ^1H nmr spectra were recorded on a Bruker HX-90 spectrometer. All ^{31}P nmr spectra were obtained at 36.4 MHz, all protons decoupled, referenced to external 85% H_3PO_4 while ^1H nmr spectra were run at 90 MHz and referenced either to tetramethyl silane or the solvent. Reactive samples were prepared by condensation of the solvent onto samples, *in vacuo*, and maintaining the sample at −78 °C prior to spectroscopic investigations.

Analyses

Elemental analyses were performed by Galbraith Laboratories, Inc., Knoxville, Tennessee, Alfred Bernhardt Mikroanalytisches Laboratorium, Elbach, West Germany, and Schwarzkopf Microanalytical Laboratory, Woodside, New York. Evolved dihydrogen was measured in a calibrated Toepler pump assembly and characterized by oxidation over CuO at 300 °C. H_2, HD, D_2 mixtures were separated and characterized by low temperature chromatography employing a $FeCl_3$–alumina solid phase [21–23].

Reagents

Benzene, Anal. Reagent, Mallinckrodt Chemical Works, was dried over lithium aluminum hydride and distilled from the hydride just prior to use. *Benzene-d_6*, 99.5%, ICN Corp., was stored over Linde 4x molecular sieves. *Chloroform*, Certified A.C.S., Fisher Scientific Co., was dried over Lithium Aluminum hydride. *Chloroform-d_1*, 99.8%, Diaprep, Inc., was used as received. *Deuterium oxide*, D_2O, Isotopes, Inc., was used as received. *Dichloromethane-d_2* 99.5%, Merck and Co., was used as received. *Dichlorodiphenylsilane*, PCR, Inc., was used as received. *Ethanol*, Absolute IMC Chemical Group, Inc., was used as received. *Heptane*, Fisher Scientific Co., was dried over lithium aluminum hydride and distilled from it just prior to use. *Lithium*, Lump, Research Organic/Inorganic Chemical Corp., was used as received. *Lithium aluminum hydride*, $LiAlH_4$, Alfa Inorganics, Inc., was used without further purification. *Piperidine*, 99.2%, Fisher Scientific Co., was dried over Linde 4x Molecular Sieves, and distilled under vacuum just prior to use. *Ruthenium tri-*

chloride hydrate, Ventron, Inc., was used as received. *Tetrahydrofuran,* Certified, Fisher Scientific Co., was dried over lithium aluminum hydride. *Tetrahydrofuran-d_8*, 99%, Merck and Co., was used as received. *Tetramethylsilane,* PCR, Inc., was used as received. *Toluene,* Certified A.C., Fisher Scientific Co., was dried over lithium aluminum hydride and distilled just prior to use. *Toluene-d_8*, 99%, Merck and Co., Inc., was dried over NaK and distilled from it prior to use. *Triethyl aluminum,* Texas Alkyls, was used as received. *Triethylamine,* Matheson, Coleman and Bell, was stored over lithium aluminum hydride and distilled from it just before use. *Triphenylphosphine,* Chemical Samples Co., was used as received. *1,4-Dichlorooctaphenyltetrasilane* was prepared by the literature method [24, 25], m.p. 182 °C, lit. 182–183 °C [24]. *Dihydrididotetrakis(triphenylphosphine)-ruthenium(II)* was prepared by the literature method [6].

Treatment of Dichlorotetrakis(triphenylphosphine)-ruthenium(II), $(Ph_3P)_4RuCl_2$, with 1,4-Dihydride-octaphenyltetrasilane, $H_2Si_4Ph_8$

A 50 ml flask was charged with 0.200 g of $(Ph_3P)_4RuCl_2$ (0.164 mmol) and 0.168 g of $H_2Si_4Ph_8$ (0.230 mmol). The flask was evacuated and approximately 25 mL of benzene was distilled into it and the solution refluxed overnight at approximately 50 °C. No change in the brown solution color of the starting complex was apparent. The solvent was removed, *in vacuo,* and as the last traces of solvent were removed at 70 °C, the brown material turned red; no HCl or H_2 was evolved.

Synthesis of $\{(PPh_3)_3RuHCl\}_2$, III

A typical reaction involved a solution of 0.48 g of $Cl_2Si_4Ph_8$ (0.60 mmol) in 20 mL of benzene added to a suspension of 0.70 g $(Ph_3P)_4RuH_2$ (0.61 mmol) in 25 mL of benzene and 1 ml (7.2 mmol) of Et_3N. Gas evolution and an immediate color change of the solution to red accompanies the addition. After completion of the addition of reagents, the volatile materials were removed and 0.29 mmol of H_2 isolated. Twenty mL of benzene was distilled onto the reaction mixture and the solution *immediately* filtered (III is only moderately soluble in benzene, while PPh_3 and the by-product silane are very soluble in benzene). The red precipitate was washed with two portions of absolute EtOH and two portions of dry Et_2O. 0.49 g of the red solid, $\{(PPh_3)_3RuHCl\}_2$, III, were isolated (88% yield). *Anal.* Calcd. for $\{(PPh_3)_3RuHCl\}$ C, 70.19; H, 4.98; Cl, 3.84. Found C, 70.16; H, 5.02; Cl, 3.84.

III melts with decomp. at 168 °C and exhibits infrared absorptions, in nujol, at 3045 (m), 2030 (m), 1588 (w), 1573 (w), 1482 (vs sh), 1437 (vs), 1433 (vs sh), 1400, 1313 (w), 1262 (w), 1182 (w sh), 1172 (m), 1158 (w), 1118 (w), 1092 (s), 1088 (s sh), 1072 (m sh), 1037 (s), 998 (m), 972 (vw), 852 (m), 809 (m), 757 (s sh), 748 (s), 696 (vs), 678 (vs sh), 618 (w), 551 (s), 526 (vs sh), 522 (vs), 512 (s sh), 499 (s sh), 490 (s sh), 458 (m), 438 (m), 422 (w), 408 (m), and 284 (m) cm^{-1}.

The mass spectrum of III exhibits no parent mass, and no significant masses above 262 (PPh_3). An osmometric molecular weight measurement in $CHCl_3$ gives a value of 1.6×10^3 g/mol (1.848×10^3 theor. for a dimer). Molecular weight measurements in benzene were not reproducible due to incomplete solubility, while III rapidly decomposed in THF. The initial molecular weight determination in $CHCl_3$ was most likely accompanied by a small amount of decomposition as the molecular weight decreased with time. However, initial measurements were reproducible within the indicated accuracy.

Solution Decomposition of III. Synthesis of $[(PPh_3)_3RuCl]_x$, V

A typical reaction involved a solution of 0.125 g III (0.135 mmol monomer) in benzene of THF with rapid (1–3 hr) H_2 evolution, 0.135 mmol. The resulting solution was filtered (benzene), *in vacuo,* and freeze dried to afford red V which exhibited infrared absorptions, in nujol, at 3050 (m), 2494 (w), 1582 (w), 1568 (m), 1485 (s sh), 1436 (vs), 1307 (vw), 1270 (vw), 1185 (w), 1152 (w), 1115 (vw sh), 1088 (s), 1027 (w), 998 (w), 851 (vw), 805 (w), 742 (vs), 720 (m), 697 (vs), 617 (w), 560 (m sh), 545 (m sh), 533 (vs), 465 (w), and 430 (w br) cm^{-1}. V exhibits single ^{31}P NMR absorption at 57 ppm from 85%, H_3PO_4, while the ^1H NMR spectrum consisted only of complex phenyl absorption centered at 2.3 and 2.7 τ.

Hydrolysis of III

A 0.0914 g sample of III (0.0989 mmol monomer) was dissolved in 10 mL of benzene and a solution of 1 mL D_2O and 10 mL piperidine added. Only trace amounts of H_2 were produced by stirring at 25 °C. Heating of the mixture to 80 °C for 3 hr produced 0.098 mmol of H_2. Continued heating of the reaction mixture at 80 °C for 24 hr produced no additional H_2. When the temperature of the reaction mixture was raised to 100 °C, additonal H_2 was evolved. When the sample was alternately heated to 100 °C for 2 hr and cooled to −196 °C for H_2 removal, 8 heat cycles afforded 0.92 mmol H_2 (the isotopic content was not determined) or 9.3 hydrogen per Ru. Residue anal.: Calcd. for $(PPh_3)_3RuCl$ 3.84% Cl.

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