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SYNTHESIS AND STRUCTURE OF TRIDENTATE 2,6-BIS(IMINO)PYRIDYL RUTHENIUM(II) COMPLEXES WITH 2,3,6-TRIMETHYL BENZENAMINE LIGAND

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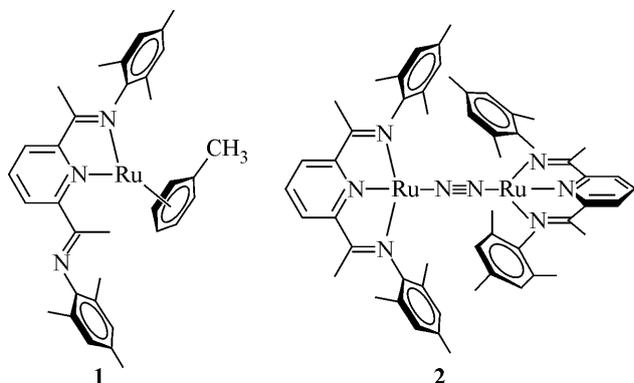
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New tridentate 2,6-bis(imino)pyridyl ruthenium (II) complexes, $[\text{N}_3]\text{Ru}(\text{H})(\text{Cl})(2,4,6\text{-(CH}_3)_3\text{C}_6\text{H}_2\text{NH}_2)$ (**4**) and $[\text{N}_3]\text{Ru}(\text{Cl})_2(2,4,6\text{-(CH}_3)_3\text{C}_6\text{H}_2\text{NH}_2)$ (**5**) ($[\text{N}_3] = 2,6\text{-(2,4,6-(CH}_3)_3\text{C}_6\text{H}_2\text{N=CCH}_3)_2\text{C}_5\text{H}_3\text{N}$) have been synthesized and characterized. The crystal of complex **4**, which has been grown in benzene/cyclohexane by evaporation at room temperature, was studied by X-ray diffraction. Complex **4** crystallizes in the monoclinic space group $C2/c$ with cell parameters of $a = 42.243(4) \text{ \AA}$, $b = 8.1480(5) \text{ \AA}$, $c = 23.662(2) \text{ \AA}$, $\beta = 117.998(1)^\circ$, $V = 7191.1(10) \text{ \AA}^3$, and $Z = 8$. The molecular structure of **4** shows a six coordinate, pseudo-octahedral geometry with a *trans* arrangement of hydrogen and chloride ligands. The mesityl ligands are oriented approximately perpendicular to the 2,6-bis(imino)pyridyl ligand plane, and form a pocket surrounding the 2,4,6-trimethyl benzenamine ligand. The coordinated 2,4,6-trimethyl benzenamine ($2,4,6\text{-(CH}_3)_3\text{C}_6\text{H}_2\text{NH}_2$) is in the *cis* to chloride and hydride, *trans* to pyridine ligand, and lies parallel to one of the mesityl rings.

Keywords: 2,6-bis(imino)pyridyl ligand, ruthenium(II) complex, 2,4,6-trimethyl benzenamine ligand, *trans* arrangement of hydrogen and chloride ligands.

INTRODUCTION

Late transition metal complexes bearing bis(imino)pyridine ligands have drawn much attention recently due to their possible use in catalysis and in other chemical transformations [1–4]. Tridentate bis(imino)pyridine ligands form *mer* geometry, which provides spatial advantages for the interaction of transition metal centers with other ligands. In particular, novel low-valent ruthenium complexes of



the 2,6-bis(imino)pyridine ligands, $[\eta^2\text{-N}_3]\text{Ru}(\eta^6\text{-Ar})$ (**1**) and $\{[\text{N}_3]\text{Ru}\}_2(\mu\text{-N}_2)$ (**2**), where $\text{Ar} = \text{C}_6\text{H}_6$ or $\text{C}_6\text{H}_5(\text{CH}_3)$, and $[\text{N}_3] = 2,6\text{-(2,4,6-(CH}_3)_3\text{C}_6\text{H}_2\text{N=CCH}_3)_2\text{C}_5\text{H}_3\text{N}$, have been synthesized and characterized (Fig. 1) [5, 6]. These low-valent Ru(0) complexes show interesting reactivities toward Si—Cl bond activation as a route to silyl and silylene complexes

Fig. 1. Low-valent ruthenium complexes of the 2,6-bis(imino)pyridine ligands, **1** and **2**

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[6], and the coordination of other donating ligands (e.g. CO and PR₃) to generate a variety of [N₃]Ru(II) families [5].

Recently, we have also reported the reaction of low-valent bis(imino)pyridyl ruthenium complexes **1** and **2** with amine hydrochlorides to form [N₃]Ru(H)(Cl)(amine) [7]. Commercially available amine hydrochloride salts have been used to generate ruthenium complexes with amine and hydride ligands. In this paper, we report new tridentate 2,6-bis(imino)pyridyl ruthenium (II) complexes, [N₃]Ru(H)(Cl)(2,4,6-(CH₃)₃C₆H₂NH₂) (**4**) and [N₃]Ru(Cl)₂(2,4,6-(CH₃)₃C₆H₂NH₂) (**5**), which were generated on the synthetic way for [η²-N₃]Ru(η⁶-Ar) (**1**) (for **4**) and in the reaction of [η²-N₃]Ru(η⁶-Ar) (**1**) with 2,4,6-trimethylaniline hydrochloride, **6** (for **5**).

EXPERIMENTAL

General Methods. All manipulations were performed in Schlenk-type glassware on a dual-manifold Schlenk line or in a nitrogen-filled Vacuum Atmospheres glove box [13]. All glassware was oven-dried prior to use. ¹H NMR spectra were obtained at 300, 360 and 500 MHz on Bruker DMX-300, AM-360, and AMX-500 FT NMR spectrometers, respectively. ³¹P{¹H} NMR spectra was recorded with broadband ¹H decoupling at 121.5 MHz, on Bruker DMX-300. All NMR spectra were recorded at 303 K unless stated otherwise. Chemical shifts are reported relative to tetramethylsilane for ¹H and external 85 % H₃PO₄ for ³¹P resonances.

Materials. Hydrocarbon solvents were dried over Na/K alloy-benzophenone. Benzene-*d*₆ and toluene-*d*₈ were dried over Na/K alloy. Triethylsilane (Aldrich) was dried over Na prior to use. Ruthenium complexes [η²-N₃]Ru(η⁶-MeC₆H₅) (**1**) [5, 6], {[N₃]Ru}₂(μ-N₂) (**2**) [5, 6], [N₃]Ru(Cl)₂ × (CH₂CH₂) (**3**) [8], P(CH₃)₃ [14] was synthesized according to the literature procedures. 2,4,6-trimethyl aniline (Acros) and HCl (12N, Fisher) were used as received. Abbreviations used: [N₃] = [2,6-(2,4,6-(CH₃)₃C₆H₂N=C(CH₃)₂C₅H₃N)], Mes (mesityl) = 2,4,6-(CH₃)₃C₆H₂—.

[N₃]Ru(H)(Cl)(2,4,6-(CH₃)₃C₆H₂NH₂) (4**).** A toluene solution (15 mL) of [N₃]Ru(Cl)₂(CH₂CH₂) (**3**), (0.41 g, 0.7 mmol) and (CH₃CH₂)₃SiH (1.5 mL, 1.01 g, 9.25 mmol) was stirred under nitrogen for 4 days at room temperature. The reaction mixture was then reduced in volume to approximately 5 mL in vacuo. The product was recrystallized from pentane/toluene at -78 °C using a swivel frit under inert conditions, and then washed with cold pentane and dried in vacuo, yielding a mixture of **4** (about 80 % of Ru complexes) and **1** (about 20 % of Ru complexes). Further recrystallization was done in toluene to give pure **4**. The final total yield of **4** was 38 % (0.18 g). ¹H NMR of **3** (benzene-*d*₆): 7.20 (d, ³J_{HH} = 7.3 Hz, 2H, Pyridyl-*H*(*meta*)), 6.92 (t, ³J_{HH} = 7.4 Hz, 1H, Pyridyl-*H*(*para*)), 6.72 and 6.46 (s, each 2H, Mes-*H*(*meta*)), 6.22 and 6.94 (broad, 2,4,6-trimethyl benzenamine *ring protons*), 2.62, 2.13, 1.90, and 1.71 (s, each 6H, Mes-CH₃(*ortho and para*), N=C—H₃, 2.4—1.8 (broad, 2,4,6-trimethyl benzenamine CH₃ groups), -9.50 (s, 1H, Ru—H). Suitable X-ray quality crystals of **4**, were grown in benzene/cyclohexane by evaporation at room temperature.

Reaction of **4 with P(CH₃)₃.** An NMR tube was loaded with a benzene-*d*₆ solution (0.3 mL) of **4** (6 mg, 0.009 mmol), and the solution was degassed in vacuo. At -196 °C, P(CH₃)₃ (0.11 mmol) was added, and the NMR tube was sealed. The reaction mixture was then warmed to room temperature and the reaction was monitored via ¹H NMR spectroscopy. After 5 min, all **4** was consumed, and changed to [N₃]Ru(H)(Cl)(P(CH₃)₃) and 2,4,6-trimethylaniline, **7**. ¹H NMR resonances were compared with those of [N₃^{xy}]Ru complexes isolated [5, 6].

Reaction of [η²-N₃]Ru(η⁶-Ar) (1**) with 2,4,6-trimethylaniline hydrochloride, **6**.** The 2,4,6-trimethylaniline hydrochloride, **6**, was prepared from the reaction of 2,4,6-trimethylaniline and excess HCl (12N) in pentane. The yellow solid of **6** was filtered and washed with pentane and dried overnight in vacuo. An NMR tube was loaded by benzene-*d*₆ solution (0.3 mL) of **1** (6 mg, 0.010 mmol) and excess **6** solids, and the NMR tube was sealed. The reaction was monitored via ¹H NMR spectroscopy. After 4 hours, All **1** was consumed, and converted to [N₃]Ru(Cl)₂(2,4,6-(CH₃)₃C₆H₂NH₂) (**5**). ¹H NMR of **5** (benzene-*d*₆): 7.53 (d, ³J_{HH} = 7.8 Hz, 2H, Pyridyl-*H*(*meta*)), 7.27 (t, ³J_{HH} = 7.8 Hz, 1H, Pyridyl-*H*(*para*)), 7.11 (s, 4H, Mes-*H*(*meta*)), 6.97 and 6.83 (s, each 1H, 2,4,6-trimethyl benzenamine *ring*

Table 1

Crystallographic Data for $[\text{N}_3]\text{Ru}(\text{H})(\text{Cl})(2,4,6\text{-}(\text{CH}_3)_3\text{C}_6\text{H}_2\text{NH}_2)$, **4**

CCDC Deposition Number	CCDC 794056
Formula	$\text{C}_{39}\text{H}_{51}\text{N}_4\text{ClRu}$
Formula weight	712.36
Crystal class	Monoclinic
Space group	$C2/c$ (#15)
Z	8
Cell constants $a, b, c, \text{\AA}$; β , deg.	42.243(4), 8.1480(5), 23.662(2); 117.998(1)
$V, \text{\AA}^3$	7191.1(10)
μ, cm^{-1}	5.43
crystal size, mm	0.42×0.10×0.01
$d_{\text{calc}}, \text{g/cm}^3$	1.316
$F(000)$	2992
Radiation	$\text{MoK}\alpha$ ($\lambda = 0.71069 \text{\AA}$)
Diffractometer	Rigaku Mercury CCD
Scan type	ϕ and ω rotations
Rotation width	0.5°
Exposure, s	30
Total number of images	534
2θ range, deg.	5.12—54.96
hkl collected	$-53 \leq h \leq 51, -8 \leq k \leq 10, -30 \leq l \leq 30$
No. reflections measured	26242
No. unique reflections	8158 ($R_{\text{int}} = 0.0344$)
No. observed reflections	6632 ($F > 4\sigma$)
No. reflections used in refinement	8158
No. parameters	402
Weighting scheme	$w = 1/[\sigma^2(F_0^2) + 0.0416P^2 + 18.3222P]$, where $P = (F_0^2 + 2F_c^2)/3$
R indices ($F > 4\sigma$)	$R_1 = 0.0480, wR_2 = 0.1023$
R indices (all data)	$R_1 = 0.0647, wR_2 = 0.1126$
GOOF	1.109
Final Difference Peaks, $e/\text{\AA}^3$	+1.249, -0.669

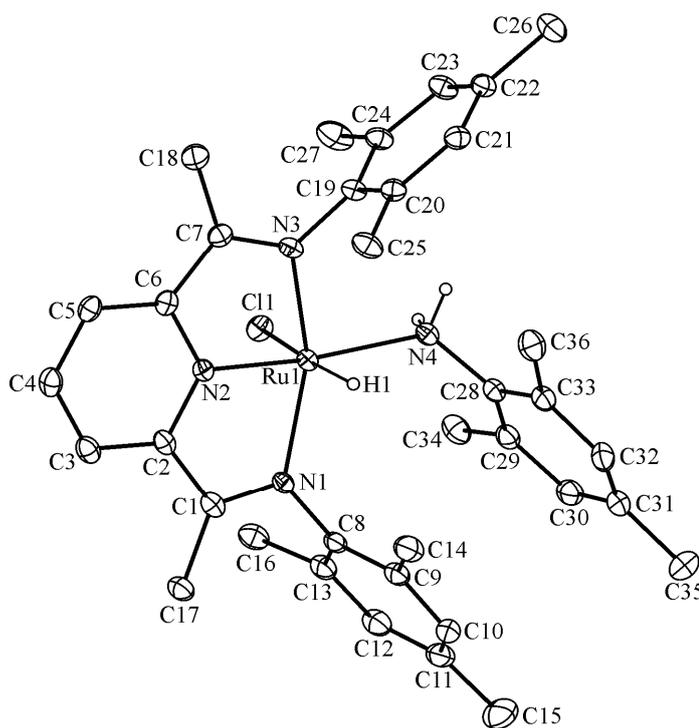
protons), 3.51 (broad s, 2H, NH_2), 2.70 (s, 12H, Mes- CH_3 (*ortho*)), 2.57, 2.56, and 2.47 (s, each 6H, Mes- CH_3 (*para*), $\text{N}=\text{C}-\text{CH}_3$, and aniline- CH_3 (*ortho*)), 2.34 (s, 3H, 2,4,6-trimethyl benzenamine- CH_3 (*para*)). The NMR solution was filtered under N_2 and the filtrate was collected to another NMR tube, and degassed in vacuo. At -196°C , $\text{P}(\text{CH}_3)_3$ (0.08 mmol) was added, and the NMR tube was sealed. The reaction mixture was then warmed to room temperature and the reaction was monitored via ^1H NMR spectroscopy. After 5min, all **5** was consumed, and changed to $[\text{N}_3]\text{Ru}(\text{Cl})_2(\text{P}(\text{CH}_3)_3)$ with 2,4,6-trimethylaniline, **7**.

Single Crystal X-ray Diffraction Analysis of $[\text{N}_3]\text{Ru}(\text{H})(\text{Cl})(2,4,6\text{-}(\text{CH}_3)_3\text{C}_6\text{H}_2\text{NH}_2)$ (4**).** CIF file containing complete information on the studied structure was deposited with CCDC, deposition number 794056, and is freely available upon request from the following web site: www.ccdc.cam.ac.uk/data_request/cif.

Suitable X-ray quality crystals of **4**, were grown in benzene/cyclohexane by evaporation at room temperature. Compound **4** crystallizes in the monoclinic space group $C2/c$ (systematic absences hkl : $h + k = \text{odd}$ and $h0l$: $l = \text{odd}$) with $a = 42.243(4) \text{\AA}$, $b = 8.1480(5) \text{\AA}$, $c = 23.662(2) \text{\AA}$, $\beta = 117.998(1)^\circ$,

Fig. 2. ORTEP drawing of complex **4** with 30 % probability thermal ellipsoids

$V = 7191.1(10) \text{ \AA}^3$, $Z = 8$ and $d_{\text{calc}} = 1.316 \text{ g/cm}^3$. X-ray intensity data were collected on a Rigaku Mercury CCD area detector employing graphite-monochromated MoK radiation ($\lambda = 0.71069 \text{ \AA}$) at a temperature of 143°K. Preliminary indexing was performed from a series of twelve 0.5° rotation images with exposures of 30 seconds. A total of 534 rotation images were collected with a crystal to detector distance of 36 mm, a 2θ swing angle of -12° , rotation widths of 0.5° and exposures of 75 seconds: scan number 1 was a ϕ -scan from 210° to 420° at $\omega = 10^\circ$ and $\chi = 20^\circ$; scan number 2 was a ω -scan from -20° to 5° at $\phi = 45^\circ$ and $\chi = -90^\circ$; and scan number 3 was an ω -scan from -20° to 12° at $\chi = -90^\circ$ and $\phi = 135^\circ$. Ro-



tation images were processed using CrystalClear [15], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the CrystalStructure [16] program package for further processing and structure solution on a Dell Pentium III computer. A total of 26242 reflections were measured over the ranges $5.12 \leq 2\theta \leq 54.96^\circ$, $-53 \leq h \leq 51$, $-8 \leq k \leq 10$, $-30 \leq l \leq 30$ yielding 8158 unique reflections ($R_{\text{int}} = 0.0344$). The intensity data were corrected for Lorentz and polarization effects and for absorption using REQAB (minimum and maximum transmission: 0.810 and 1.000).

The structure was solved by direct methods (SIR97) [17]. The asymmetric unit includes 1/2 of a molecule of cyclohexane solvent (the cyclohexane molecule lies on a crystallographic 2-fold axis). Refinement was done by full-matrix least squares based on F^2 using SHELXL-97 [18]. All reflections were used during refinement (F^2 's that were experimentally negative were replaced by $F^2 = 0$). The weighting scheme used was $w = 1/[\sigma^2(F_0^2) + 0.0416P^2 + 18.3222P]$, where $P = (F_0^2 + 2F_c^2)/3$.

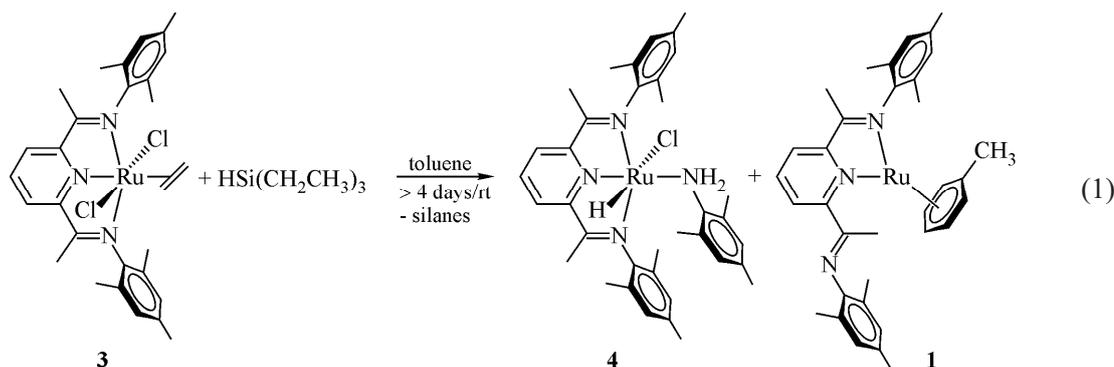
Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a "riding" model, except hydride hydrogen atoms which were refined isotropically. Refinement converged to $R_1 = 0.0480$ and $wR_2 = 0.1023$ for 6632 reflections for which $F > 4\sigma(F)$ and $R_1 = 0.0647$, $wR_2 = 0.1126$ and GOF = 1.109 for all 8158 unique, non-zero reflections and 421 variables. The maximum Δ/σ in the final cycle of least squares was 0.003 and the two most prominent peaks in the final difference Fourier were $+1.249$ and -0.669 e/\AA^3 .

Table 1 lists cell information, data collection parameters, and refinement data. Fig. 2 is an ORTEP [19] representation of the molecule with 30 % probability thermal ellipsoids displayed.

RESULTS AND DISCUSSION

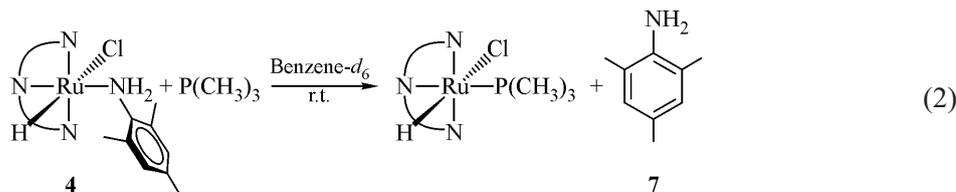
The synthetic methods of **1** have been well established [5]. In general, treatment of $[\text{N}_3]\text{Ru(II)}$ ethylene adduct, $[\text{N}_3]\text{Ru}(\text{Cl})_2(\text{CH}_2\text{CH}_2)$ (**3**) [8] with excess $(\text{CH}_3\text{CH}_2)_3\text{SiH}$ in an arene solvent (e.g., toluene or benzene) for 1 day at room temperature leads to formation of **1**, with the concurrent formation of $(\text{CH}_3\text{CH}_2)_3\text{SiCl}$, $(\text{CH}_3\text{CH}_2)_4\text{Si}$, and ethane [5]. However, when this reaction was allowed to proceed for longer than 3 days, a variety of side products were formed, and this is mainly due to the reactions of complex **1** to other silane compounds. For example, reactions of complex **1** with various alkylsilanes [9] or alkylchlorosilanes [10] have been reported.

2,6-Bis(imino)pyridyl ruthenium (II) complex with 2,4,6-trimethyl benzenamine ligand, $[\text{N}_3]\text{Ru}(\text{H})(\text{Cl})(2,4,6\text{-(CH}_3)_3\text{C}_6\text{H}_2\text{NH}_2)$ (**4**) was obtained in a synthetic experiment intended to produce $[\eta^2\text{-N}_3]\text{Ru}(\eta^6\text{-Ar})$ (**1**). The reaction of **3** with excess $(\text{CH}_3\text{CH}_2)_3\text{SiH}$ (13 equiv.) in toluene was conducted under nitrogen for 4 days at room temperature, and then the product was recrystallized from pentane/toluene at -78°C . The initially recrystallized compounds were the mixture of an aniline coordinated complex, $[\text{N}_3]\text{Ru}(\text{H})(\text{Cl})(2,4,6\text{-(CH}_3)_3\text{C}_6\text{H}_2\text{NH}_2)$ (**4**) (about 80 % of Ru complexes) and **1** (about 20 % of Ru complexes) (Eq. 1). Complex **4** was less soluble in toluene than **1**, therefore repeated recrystallizations from toluene solution lead to the isolation of pure complex **4** (38 % yield).



The solid-state structure of **4** was determined by a single-crystal X-ray diffraction study (Fig. 2 and Table 1), and exhibits a six coordinate, pseudo-octahedral geometry with a *trans* arrangement of hydride and chloride ligands ($\text{H1-Ru1-Cl1} = 167.5(12)^\circ$). Tridentate bis(imino)pyridyl ligands enforce the *mer* geometry. The *trans* N1-Ru-N3 bond angle is $155.99(10)^\circ$, which reflects a typical bis(imino)pyridyl complex and shows the constraints of the chelating ligand [5, 11, 12]. The mesityl ligands are oriented approximately perpendicular to the $[\text{N}_3]$ ligand plane, and form a pocket surrounding the trimethylaniline ligand. The coordinated 2,4,6-trimethylaniline ligand ($2,4,6\text{-(CH}_3)_3\text{C}_6\text{H}_2\text{NH}_2$) is *cis* to chloride and hydride ($\text{H1-Ru1-N4} = 86.3(12)^\circ$, $\text{Cl1-Ru1-N4} = 82.33(7)^\circ$) and *trans* to the pyridine ($\text{N2-Ru1-N4} = 173.78(10)^\circ$). The coordinated 2,4,6-trimethylaniline ligand lies approximately parallel to one of the mesityl rings.

The ^1H NMR spectrum of **4** in solution at room temperature is consistent with the solid state structure, and exhibits four resonances determined as the imine methyls, two pairs of *o*-mesityl methyls, and *p*-mesityl methyls, which is consistent with mirror symmetry in the plane bisecting pyridine from the solid state structure. A single ^1H NMR resonance is observed for the ruthenium hydride ($\delta -19.50$, singlet), and this upfield shift is consistent with other $[\text{N}_3]\text{Ru}$ complexes with a *trans* hydride-chloride arrangement [5, 6]. Reaction of **4** with excess $\text{P}(\text{CH}_3)_3$ (12 equiv.) in benzene- d_6 at room temperature leads to rapid displacement of $2,4,6\text{-(CH}_3)_3\text{C}_6\text{H}_2\text{NH}_2$ (**7**) and formation of $[\text{N}_3]\text{Ru}(\text{H})(\text{Cl})(\text{P}(\text{CH}_3)_3)$ (Eq. 1) [5, 6]. Further reaction with additional $\text{P}(\text{CH}_3)_3$ to form probable Ru(0) complex such as the bis(phosphine) complex $[\text{N}_3]\text{Ru}(\text{P}(\text{CH}_3)_3)_2$ and the 2,4,6-trimethylaniline hydrochloride, **6**, is not observed at room temperature.²¹ This is consistent with the previous results [7].



We could not find alternative synthetic methods for the isolation of **4**. In other efforts, we have synthesized $[\text{N}_3]\text{Ru}(\text{H})(\text{Cl})(\text{amine})$ complexes from the reaction of **1** with various amine hydrochlorides [7]. According to the synthetic scheme for obtaining general $[\text{N}_3]\text{Ru}(\text{H})(\text{Cl})(\text{amine})$ complexes,

we also performed the reaction of **1** with **6** in benzene-*d*₆. In this attempt, formation of a new complex, which is assigned as [N₃]Ru(Cl)₂(2,4,6-(CH₃)₃C₆H₂NH₂) (**5**) by the ¹H NMR spectrum, was observed. The ¹H NMR spectrum of **4** at room temperature exhibits three resonances determined as the *o*-mesityl methyls (12H), imine methyls (6H), and *p*-mesityl methyls (6H), consistent with mirror symmetry in the planes of the [N₃] ligand and the plane bisecting the pyridine. Complex **5** reacts with P(CH₃)₃ to produce [N₃]Ru(Cl)₂(P(CH₃)₃), and 2,4,6-(CH₃)₃C₆H₂NH₂ (**7**).

CONCLUSIONS

In conclusion, new tridentate 2,6-bis(imino)pyridyl ruthenium (II) complexes, [N₃]Ru(H)(Cl)(2,4,6-(CH₃)₃C₆H₂NH₂) (**4**) and [N₃]Ru(Cl)₂(2,4,6-(CH₃)₃C₆H₂NH₂) (**5**) have been synthesized and characterized. The reaction of low-valent ruthenium complex **1** with 2,4,6-trimethylaniline hydrochloride, **6** generates complex **5**, which is a six-coordinate dichloro ruthenium (II) complexes with amine ligands. Complex **4** has been formed in the reaction of [N₃]Ru(Cl)₂(CH₂CH₂) (**3**) with excess (CH₃CH₂)₃SiH for 4 days at room temperature. Complex **4** and **5** show reactivity toward P(CH₃)₃, and the displacement of 2,4,6-trimethylaniline by P(CH₃)₃ occurs in the reaction.

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