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Note

Synthesis and cyclization reactions of platinum(II)-coordinated arsonium-substituted phenyl isocyanides, *o*-($I^-R_3As^+$–CH_2)$C_6H_4N{\equiv}C$

Giacomo Facchin [a], Rino A. Michelin [b,*], Mirto Mozzon [b], Paolo Sgarbossa [b], Augusto Tassan [b]

[a] *Istituto di Scienze e Tecnologie Molecolari, ISTM-C.N.R., c/o Dipartimento di Processi Chimici dell'Ingegneria, Università di Padova, Via F. Marzolo 9, Padua 35131, Italy*
[b] *Dipartimento di Processi Chimici dell'Ingegneria, Università di Padova, Via F. Marzolo 9, Padua 35131, Italy*

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Abstract

The arsonium-substituted isocyanides, *o*-(I^- $^+R_3AsCH_2$)$C_6H_4N{\equiv}C$ ($AsR_3 = AsPh_3$, $\mathbf{L_1}$; $AsMePh_2$, $\mathbf{L_2}$; $AsMe_2Ph$, $\mathbf{L_3}$), were prepared by reaction of *o*-(chloromethyl)phenyl isocyanide, *o*-(CH_2Cl)$C_6H_4N{\equiv}C$, with a slight molar stoichiometric amount of the arsine in the presence of a 3-fold excess of NaI in acetone at room temperature. The isocyanides $\mathbf{L_1}$–$\mathbf{L_3}$ coordinate to some Pt(II) complexes such as *trans*-[PtX{*o*-(I^- $^+R_3AsCH_2$)C_6H_4NC}$(PPh_3)_2$] [BF_4] ($AsR_3 = AsPh_3$, **1**; $AsMePh_2$, **2**; $AsMe_2Ph$, **3**; X = Cl, I) and [PtX{*o*-(I^- $^+R_3AsCH_2$)C_6H_4NC}($Ph_2PCH{=}CHPPh_2$)] [BF_4] ($AsR_3 = AsMePh_2$, **4**; X = Cl, I). Complexes **2**–**4** are converted in CH_2Cl_2 at room temperature in the presence of NEt_3 to the corresponding indolidin-2-ylidene derivatives *trans*-[PtX{$\overline{CN(H)–o–C_6H_4C}$($AsR_3$)}$(PPh_3)_2$]$BF_4$] ($AsR_3 = AsPh_3$, **5**; $AsMePh_2$, **6**; $AsMe_2Ph$, **7**) and [PtX{$\overline{CN(H)–o–C_6H_4C}$($AsMePh_2$)}($Ph_2PCH{=}CHPPh_2$)][$BF_4$] (**8**).

Keywords: Functional isocyanides; Arsonium-substituted isocyanides; N-heterocyclic carbene complexes; Platinum(II) complexes

1. Introduction

The organometallic chemistry of several functionalized isocyanides has been often addressed [1,2] to the formation of N-heterocyclic carbenes (NHC), which have been found to be promising alternative ligands to the commonly used phosphines and phosphites and also for their favorable application in homogeneous catalysis [3]. The NHC ligands are obtained through an intramolecular 1,2-addition of the functional moiety, usually a protic nucleophile such as an hydroxy, an amino or an ylide group, across the C≡N triple bond. Typical representative examples of functional isocyanides leading to NHC ligands upon metal coordination are illustrated in Chart A. They include the hydroxyalkyl isocyanides **I**, reported by Fehlhammer et al. [4], the β-functional phenyl isocyanides **II**, investigated by Hahn and his group [2] and the γ-functional phenyl isocyanides **III**, synthesized by some of us [1,5–12].

As an extension to these latter studies, we wish to report herein the synthesis and the platinum(II)-promoted cyclization reactions of some arsonium-substituted phenyl isocyanides shown in Chart B.

2. Experimental

All reactions were carried out under dinitrogen using standard Schlenk techniques. All solvents were dried by conventional methods and distilled prior to use. Infrared spectra in CH_2Cl_2 solution or nujol mulls were recorded

* Corresponding author. Tel.: +39-49-827-5522; fax: +39-49-827-5525.
E-mail address: rino.michelin@unipd.it (R.A. Michelin).

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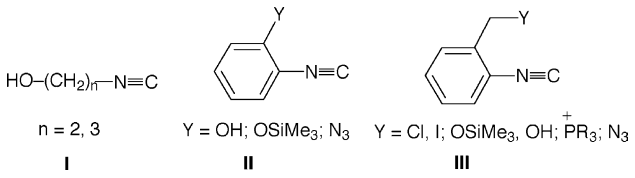
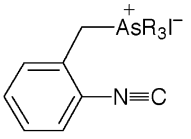


Chart A.



AsR_3 = $AsPh_3$ (**L_1**), $AsPh_2Me$ (**L_2**), $AsPhMe_2$ (**L_3**)

Chart B.

on a Perkin–Elmer 983 or Nicolet FT-IR Avatar spectrometers. NMR spectra were run on a Bruker AC 200 (^{1}H at 200.13 MHz, ^{31}P at 81.01 MHz and ^{13}C at 50.32 MHz), the chemical shifts (δ in ppm) are referred to Me_4Si (^{1}H and ^{13}C) and external 85% H_3PO_4 (^{31}P). The spectra of all nuclei (except ^{1}H) were ^{1}H decoupled.

2.1. Starting materials

The arsines were Stream Chemicals products and used as purchased. The compounds *cis*-[$PtCl_2(PPh_3)_2$] [13], [$PtCl_2(Ph_2PCH{=}CHPPh_2)$] [14] and *o*-(choloromethyl)phenyl isocyanide [8a] were prepared according to literature methods.

2.2. Synthesis of arsonium-substituted phenyl isocyanides *o*-($I^-R_3As^+$–CH_2)$C_6H_4N{\equiv}C$ ($AsR_3 = AsPh_3$ (**L_1**), $AsMePh_2$ (**L_2**), $AsMe_2Ph$(**L_3**))

A typical procedure for the preparation of ligands **L_1**–**L_3** is reported herein for **L_1**. Triphenylarsine (0.73 g, 2.4 mmol) and an excess of NaI (0.89 g, 6.0 mmol) were added to an acetone solution of *o*-($ClCH_2$)C_6H_4NC (0.3 g, 2.0 mmol). The reaction mixture was stirred at room temperature for 6 h and then taken to dryness. The residue was dissolved in dichloromethane (40 ml) and the resulting solution filtered off and concentrated to a small volume (10 ml). Addition of Et_2O (50 ml) gave a white product, which was filtered off and recrystallized from CH_2Cl_2/Et_2O. **L_1**. Yield: 38%. IR: (CH_2Cl_2), ν(N≡C) 2120 cm^{-1}; (nujol) 2118 cm^{-1}. ^{1}H NMR (δ, $CDCl_3$): 5.56 (s, CH_2). **L_2**. Yield: 89%. IR: (CH_2Cl_2), ν(N≡C) 2120 cm^{-1}; (nujol) 2121 cm^{-1}. ^{1}H NMR (δ, $CDCl_3$): 5.14 (s, CH_2), 2.85 (s, CH_3). **L_3**. Yield: 86%. IR: (CH_2Cl_2), ν(N≡C) 2119 cm^{-1}; (nujol) 2119 cm^{-1}. ^{1}H NMR (δ, $CDCl_3$): 4.76 (s, CH_2), 2.64 (s, CH_3). ^{13}C{^{1}H} NMR (δ, DMSO-d_6): 6.70 (s, *C*H_3), 28.5 (s, *C*H_2), 168.2 (s, N≡C).

2.3. Synthesis of the complexes trans-[$Pt(L)X(PPh_3)_2$][BF_4] ($L = L_1$ (**1**); L_2 (**2**), L_3 (**3**); X = Cl, I)

Complexes **1**–**3** were prepared by a similar procedure which is described herein for **1**. *cis*-[$PtCl_2(PPh_3)_2$] (0.54 g, 0.69 mmol) and $NaBF_4$ (0.30 g, 2.76 mmol) were dissolved in acetone (50 ml). The solution was then treated dropwise with an acetone solution (30 ml) of **L_1** (0.38 g, 0.69 mmol) over a period of 45 min and stirred overnight. The solvent was then removed under vacuum and the residue treated with CH_2Cl_2. After filtration, the solution was concentrated under reduced pressure to a small volume (10 ml). Addition of Et_2O gave a white-cream precipitate, which was filtered off and then recrystallized from CH_2Cl_2/Et_2O. **1**. Yield: 73%. IR (nujol): ν(N≡C) 2179 cm^{-1}. ^{1}H NMR (δ, $CDCl_3$): 3.80 (s, CH_2). ^{31}P{^{1}H} NMR (δ, $CDCl_3$): 11.0 (s, PPh_3, $^1J_{P-Pt}$ 2148 Hz), 19.3 (s, PPh_3, $^1J_{P-Pt}$ 2165 Hz). **2**. Yield: 70%. IR (nujol): ν(N≡C) 2181 cm^{-1}. ^{1}H NMR (δ, DMSO-d_6): 3.50 (s, CH_2), 2.10 (s, CH_3). ^{31}P{^{1}H} NMR (δ, DMSO-d_6): 19.1 (s, PPh_3, $^1J_{P-Pt}$ 2210 Hz). ^{13}C{^{1}H} NMR (δ, DMSO-d_6): 5.20 (s, *C*H_3), 27.9 (s, *C*H_2), 153.6 (s, N≡C). **3**. Yield: 88%. IR (nujol): ν(N≡C) 2181 cm^{-1}, (CH_2Cl_2) 2188 cm^{-1}. ^{1}H NMR (δ, DMSO-d_6): 3.50 (s, CH_2), 2.10 (s, CH_3). ^{31}P{^{1}H} NMR (δ, DMSO-d_6): 19.1 (s, PPh_3, $^1J_{P-Pt}$ 2207 Hz), 11.1 (s, PPh_3, $^1J_{P-Pt}$ 2174 Hz. ^{13}C{^{1}H} NMR (δ, DMSO-d_6): 6.62 (s, *C*H_3), 28.0 (s, *C*H_2).

2.4. Synthesis of [$PtCl(L_2)(Ph_2$ $PCH{=}CHPPh_2)$] (**4**)

This compound was prepared using a similar procedure reported above for complex **1**. [$PtCl(L_2)$ ($Ph_2PCH{=}CHPPh_2$)] (0.37 g, 0.55 mmol), $AgBF_4$ (0.24 g, 2.2 mmol) and **L_2** (0.27 g, 0.55 mmol). Yield: 81%. IR (nujol): ν(N≡C) 2197 cm^{-1}. ^{1}H NMR (δ, $CDCl_3$): 4.88 (s, CH_2), 2.60 (s, CH_3). ^{31}P{^{1}H} NMR (δ, $CDCl_3$): 50.1 (d, P *trans* to halide, $^1J_{P-Pt}$ 3586 Hz, $^2J_{P-P}$ 10.6 Hz), 53.2 (d, P *trans* to isocyanide, $^1J_{P-Pt}$ 3448 Hz, $^2J_{P-P}$ 10.6 Hz).

2.5. Cyclization reactions of the Pt(II)-coordinated isocyanides. Synthesis of trans-[$PtX\{\overline{CN(H)\text{–}o\text{–}C_6H_4C}(AsR_3)\}(PPh_3)_2$][$BF_4$] ($AsR_3 = AsPh_3$ (**5**), $AsMePh_2$ (**6**), $AsMe_2Ph$ (**7**)) and trans[$PtX\{\overline{CN(H)\text{–}o\text{–}C_6H_4C}(AsMePh_2)\}(Ph_2PCH{=}CHPPh_2)$][$BF_4$] (**8**), ($X$ = Cl, I)

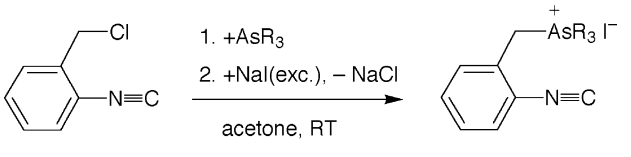
These compounds were prepared by a standard procedure, which is described herein for complex **5**. A solution of **1** (0.67 g, 0.46 mmol) in acetone (20 ml) was reacted with NEt_3 (0.65 ml, 4.6 mmol) and stirred at room temperature. The progress of the reaction was monitored by following the decrease of the C≡N absorption at ca. 2200 cm^{-1}. When the band was completely disappeared, the solution was taken to dryness and the residue washed with several 10 ml portions of H_2O to remove the inorganic salts. The remaining solid

was dissolved in CH_2Cl_2 (50 ml) and dried over anhydrous Na_2SO_4. The mixture was then filtered off, concentrated to a small volume and treated with Et_2O (50 ml) to give a white precipitate. **5**. Yield: 50%. IR (nujol): ν(NH) 3356 cm^{-1}. ^1H NMR (δ, DMSO-d_6): 2.50 (s, CH_3), 11.4 (s, NH). $^{31}P\{^1H\}$ NMR (δ, DMSO-d_6): 11.6 (s, PPh_3, $^1J_{P-Pt}$ 2757 Hz), 16.3 (s, PPh_3, $^1J_{P-Pt}$ 2815 Hz). **6**. Yield: 50%. IR (nujol): ν(NH) 3355 cm^{-1}. ^1H NMR (δ, DMSO-d_6): 2.50 (s, CH_3), 11.2 (s, NH). $^{31}P\{^1H\}$ NMR (δ, DMSO-d_6): 14.7 (s, PPh_3, $^1J_{P-Pt}$ 2706 Hz), 18.8 (s, PPh_3, $^1J_{P-Pt}$ 2769 Hz). **7**. Yield: 63%. IR (nujol): ν(NH) 3345 cm^{-1}. ^1H NMR (δ, $CDCl_3$): 2.10 (s, CH_3), 11.2 (s, NH). $^{31}P\{^1H\}$ NMR (δ, $CDCl_3$): 16.4 (s, PPh_3, $^1J_{P-Pt}$ 3011 Hz), 20.6 (s, PPh_3, $^1J_{P-Pt}$ 3068 Hz). $^{13}C\{^1H\}$ NMR (δ, $CDCl_3$): 7.8 (s, *C*H_3). **8**. Yield: 93%. IR (nujol): ν(NH) 3355 cm^{-1}. ^1H NMR (δ, DMSO-d_6): 2.10 (s, CH_3), 11.7 (s, NH). $^{31}P\{^1H\}$ NMR (δ, DMSO-d_6): 49.0 (d, P *trans* to halide, $^1J_{P-Pt}$ 3632 Hz, $^2J_{PP}$ 8.8 Hz), 57.0 (d, P *trans* to carbene, $^1J_{P-Pt}$ 1955 Hz, $^2J_{PP}$ 8.8 Hz). $^{13}C\{^1H\}$ NMR (δ, DMSO-d_6): 8.7 (s, *C*H_3), 163.8 (dd, $C_{carbene}$, $^2J_{C-Pcis}$ 7.6 Hz, $^2J_{C-Ptrans}$ 129.2 Hz).

3. Results and discussion

3.1. Synthesis of the ligands

Eq. (1) reports the synthesis of the arsonium-substituted isocyanide ligands $\mathbf{L_1}$–$\mathbf{L_3}$. They are obtained in moderate to high yield (ca. 38–89%) by reaction of the known *o*-(chloromethyl)phenyl isocyanide [8a], *o*-$(CH_2Cl)C_6H_4N{\equiv}C$, with a ca. 20% molar amount of the arsine AsR_3 ($AsR_3 = AsPh_3$, $AsMePh_2$, $AsMe_2Ph$) in the presence of a 3-fold excess of NaI in acetone at room temperature for 6 h (Eq. (1)). The use of sodium iodide is necessary since the arsines are not nucleophilic enough to react directly with *o*-$(CH_2Cl)C_6H_4N{\equiv}C$ to give the corresponding arsonium chloride salts. As previously reported [8a], NaI initially reacts with the chloromethylisocyanide to afford the more reactive iodomethyl analog, *o*-$(CH_2I)C_6H_4N{\equiv}C$, which then produces the observed products.



$\mathbf{L_1}$ - $\mathbf{L_3}$ (1)

Ligands $\mathbf{L_1}$–$\mathbf{L_3}$ are crystalline, pale cream, odorless solids, which are stable in the solid state and in solution. No decomposition is observed upon exposure to light over a period of months. They are soluble in DMSO, slightly soluble in chlorinated solvents and insoluble in diethyl ether and other common organic solvents. Diagnostic spectral features (see Section 2) are the $\tilde{\nu}$(N≡C) wavenumber, which is observed around 2120 cm^{-1} (CH_2Cl_2 solution or nujol mull), as also found for the analogous phosphonium-substituted ligands, *o*-$(BF_4^-R_3P^+–CH_2)C_6H_4N{\equiv}C$ [8a]. The methylene resonance of the $R_3As^+–CH_2–$ moiety shows up in the ^1H NMR spectra as a singlet in the range δ 5.56–4.76, with the highest and lowest downfield shifts for the $AsPh_3$ and $AsMe_2Ph$ derivatives, respectively. The isocyanide carbon resonance in the $^{13}C\{^1H\}$ NMR spectra was detected only for $\mathbf{L_3}$, which was found in DMSO-d_6 as a broad singlet at 168.2 ppm, a value that matches those observed for other isocyanide ligands such as *o*-$(Me_3SiOCH_2)C_6H_4N{\equiv}C$ (δ(N≡*C*) 167.5, $CDCl_3$ solution) [9], *o*-$(HOCH_2)C_6H_4N{\equiv}C$ (δ(N≡*C*) 166.8, $CDCl_3$ solution) [9], *o*-$(N_3CH_2)C_6H_4N{\equiv}C$ (δ(N≡*C*) 168.8, $CDCl_3$ solution) [10].

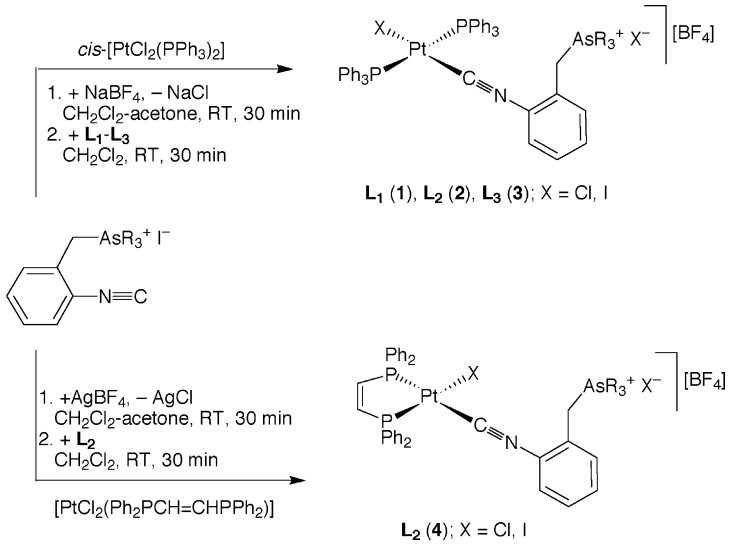
3.2. Platinum(II) complexes of $\boldsymbol{L_1}$–$\boldsymbol{L_3}$

The coordinating ability of the isocyanide ligand $\mathbf{L_1}$–$\mathbf{L_3}$ has been tested in a series of reactions with some Pt(II) metal complexes as illustrated in Scheme 1.

Complexes **1**–**3** are obtained in good yield (70–80%) from the dichloro-platinum derivative *cis*-$[PtCl_2(PPh_3)_2]$ by initial treatment with 1 equiv. of $AgBF_4$ in CH_2Cl_2-acetone and then, after filtration of AgCl, with 1 equiv. of the required $\mathbf{L_1}$–$\mathbf{L_3}$ ligand. Compound **4** was obtained in ca. 80% yield by a similar procedure starting from $[PtCl_2(Ph_2PCH{=}CHPPh_2)]$ and the isocyanide $\mathbf{L_2}$. They are white, air-stable solids, which have been characterized by IR, ^1H and $^{31}P\{^1H\}$ NMR (Section 2). The IR spectra (nujol mull) show a strong absorption around 2180 cm^{-1} corresponding to $\tilde{\nu}$(N≡C), which is shifted to lower wavenumbers of ca. 10 cm^{-1} with respect to the parent phosphonium-substituted isocyanide complexes *trans*-$[PtCl\{o\text{-}BF_4^-R_3P^+–CH_2\}C_6H_4N{\equiv}C)(PPh_3)_2][BF_4]$ ($PR_3 = PPh_3$, $PMePh_2$, PMe_2Ph, PMe_3) and of ca. 20 cm^{-1} compared to the corresponding *o*-(chloromethyl)phenyl isocyanide derivatives *trans*-[PtCl(*o*-$ClCH_2C_6H_4N{\equiv}C)(PPh_3)_2][BF_4]$ [8b]. The observed decrease in the wavenumber of the N≡C moiety was explained [8b] with the presence of the bulkier *o*-$R_3E^+–CH_2$ (E = P, As) group, which somewhat sterically hinders the coordination of the isocyanide. Release of some steric strain as in compound **4**, where two *trans*-PPh_3 ligands are replaced by a less encumbering diphosphine, shifts the $\tilde{\nu}$ (N≡C) to higher wavenumbers (2197 cm^{-1}, nujol mull).

The positive values of $\Delta\tilde{\nu} = \tilde{\nu}(N{\equiv}C)_{coord} - \tilde{\nu}(N{\equiv}C)_{free}$ of ca. 60–80 cm^{-1} observed for **1**–**4** reflect the electrophilicity of the isocyanide carbon, which is therefore a potentially reactive center toward nucleophilic attack [15].

The ^1H NMR spectra of **1**–**3** show the $–CH_2–$ resonance at ca. 3.50 ppm as a singlet, which is shifted to



Scheme 1.

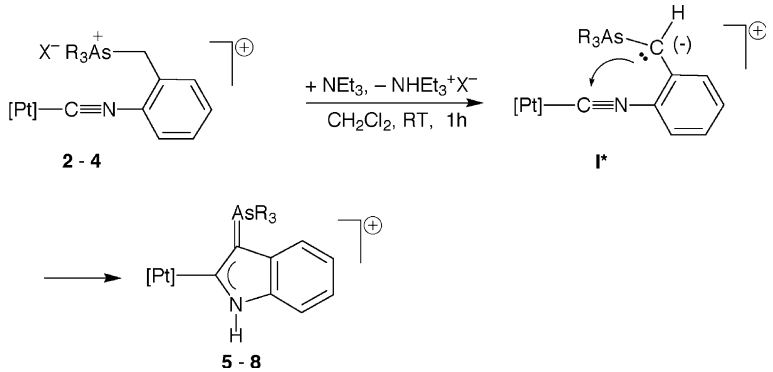
higher fields with respect to the free ligands $\mathbf{L_1}$–$\mathbf{L_3}$. Such a shielding effect likely arises from the presence of the aryl substituents in the metal-coordinated PPh_3 ligands, which are mutually *trans* and *cis* to the isocyanide ligands. Such effect appears to be less pronounced in compound **4** ($\delta(CH_2)$ 4.88 ppm).

The $^{31}P\{^1H\}$ NMR spectra of **1** and **3** show two singlets, flanked by ^{195}Pt satellites, for the PPh_3 ligands likely due to the presence of a mixture of two complexes arising from chloride-iodide exchange occurring in their preparation (Scheme 1). The presence of a singlet resonance for the PPh_3 ligands in the ^{31}P NMR spectra supports the *trans* geometry of the complexes.

3.3. Cyclization reactions of platinum(II)-coordinated L_1–L_3

Complexes **2**–**4** are found to react with a 10-fold excess of NEt_3 in CH_2Cl_2 at room temperature to afford in good yield the C-2 metal bonded indole derivatives **5**–**8** (Scheme 2). As previously reported for the corresponding phosphonium-substituted complexes [8b], the proposed mechanism for their formation entails, in the first step, NEt_3 attacks to the activated methylene group of the $-CH_2AsR_3^+$ arsonium moiety to produce the highly reactive ylide–isocyanide–metal intermediate **I***, which shows the ylidic function in the 1,2-zwitterionic structure. Subsequently, the ylidic carbanion of **I*** intramolecularly adds to the adjacent coordinated isocyanide giving the final 3-(arsonio)indolin-2-ylidene derivatives **5**–**8**. The intermediate **I*** could not be isolated or even detected by spectroscopic techniques, probably owing to its enhanced reactivity toward the highly reactive metal-coordinated isocyanide.

The "ylide-carbene" compounds **5**–**8** are stable in the solid state and in solution. They are soluble in DMSO, slightly soluble in chlorinated solvents and insoluble in other common organic solvents. They have been characterized by elemental analysis, IR, ^1H and $^{31}P\{^1H\}$ NMR (Section 2). The IR spectra (nujol mull) show $\tilde{\nu}$ (NH) around 3350 cm^{-1}, while in the ^1H NMR spectra the N–H resonance falls in the range 8–11 ppm. It is



Scheme 2.

interesting to note that the $^{31}C\{^{1}H\}$ NMR spectrum of **8** shows the carbene carbon resonance at 163.8 ppm ($^{2}J_{CPcis}$ 7.6, $^{2}J_{CPtrans}$ 129.2 Hz), a value that is in agreement with those reported for other platinum(II) carbene systems [16].

Acknowledgements

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