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Synthesis and X-ray structure of cationic methallyl palladium complexes supported by unsymmetrical β-diimine ligands

Najoua Belhaj Mbarek Elmkacher [a], Taha Guerfel [b], Igor Tkatchenko [c], Faouzi Bouachir [a,*]

[a] Laboratoire de Chimie de Coordination, Département de Chimie, Faculté des Sciences de Monastir, Avenue de l'Environnement, 5019 Monastir, Tunisia
[b] Laboratoire de Chimie du Solide, Département de Chimie, Faculté des Sciences de Monastir, Avenue de l'Environnement, 5019 Monastir, Tunisia
[c] Laboratoire de Synthèse et Electrosynthèse Organométalliques, Faculté des Sciences Mirandes, 9 Allée Alain Savary, BP 47870, 21078 Dijon Cedex, France

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Abstract

Treatment of the unsymmetrical β-iminoamine ligands [PhCN(Ar)CHCNH(Ar)Me] with the zerovalent complex $Pd(dba)_2$ in the presence of the methallyloxyphosphonium salt, gives high yields of the cationic β-diimine complexes $[PhCN(Ar)CH_2CN(Ar)(Me)Pd(\eta^3\text{-}C_4H_7)]^+[PF_6]^-$ (Ar = 2-Me-C_6H_4 (**7**); 2-MeO-C_6H_4 (**8**); 2,6-Me_2-C_6H_3 (**9**); 2,6-iPr_2-C_6H_3 (**10**)). All the new complexes have been characterised by NMR and IR spectroscopy. The structure of the cationic methallyl palladium complex (**10**) has been solved by X-ray crystallography.

Keywords: Palladium; Cationic methallyl complexes; Unsymmetrical β-diimine ligands

1. Introduction

Since the first diketiminate complexes appeared in 1968 [1], there has been sporadic use of this ligand class, otherwise known as diazapentadienyl, vinamidine, β-iminatoaminate *etc*., in many different scenarios [2]. However, the introduction of diaryl diketiminates possessing extreme ortho bulk in 1997 [3], followed rapidly by the first demonstrations of their use as N–N bidentate, monoanionic ligands of remarkable steric control in 1998 [4], prompted a relative explosion of effort in the field.

The study of the anionic ligands β-diketiminate for various main groups or transition metal chemistry has been explored [5–13], however their uses as a neutral donor have been paid less attention [3,14–17].

In polymerization, catalyst design both ligand electronics and sterics play important roles in determining the catalyst activity and polymer molecular weight.

The molecular weights of the polyethylene generated with $(DAD(CH_3,CH_3))NiBr_2$ are higher than those formed using $(DAD(H,H))NiBr_2$. (DAD = 1,4-diazadiene ligands derived from glyoxal (H, H) and diacetyl (CH_3, CH_3)). This may suggest that sterically more hindered nickel center of the catalytic species derived from $(DAD(CH_3,CH_3))NiBr_2$ are longer-lived and may suppress termination by associative olefin exchange of the monomer.

The difference in ligand structure also influences the number of branches N, which represent the number of methyl groups per 1000 carbon atoms. This number is higher for all polymers produced by catalyst $(DAD(CH_3, CH_3))NiBr_2$ compared to the polymers generated with $(DAD(H,H))$-$NiBr_2$ under the same polymerization conditions. Branches are formed when β-hydride elimination of the growing polymer chain, followed by isomerization, occurs.

Isomerization is possible when the polymer chain at the nickel center undergoes β-hydride elimination forming an intermediate olefin hydride complex and the olefin reinserts in a 2,1-manner into the nickel–hydride bond before insertion of the next monomer takes place. More isomerization and branching occur if there is less space at the catalytic

* Corresponding author. Fax: +216 71 704 329.
E-mail address: bouachirf@yahoo.de (F. Bouachir).

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site. Thus with the sterically more hindered site, higher branching frequencies are observed [18].

The cationic methallyl complexes with symmetrical β-diimine ($R_1 = R_2 = CH_3$) are excellent catalysts for the oligomerization of the styrene, they yield 1,3-diphenylbut-1-ene with selectivity ⩾98%. However cationic methallyl complexes with α-diimine ligand produce polymer. This reflects the effect of change of rigidity of the ligand toward the catalytic activity of the complex [19]. Different chemistry can be carried out with subtle changes of steric demand in the R_1 and R_2 positions.

The present work is a continuation of our study in β-diimine allylic complexes. In this paper we describe a one-pot synthesis of new η^3-allyl cationic complexes of palladium (II) supported by new ligands, the unsymmetrical β-diimine [20].

2. Results and discussion

These compounds $[(PhCNAr)CH_2(CH_3CNAr)Pd(allyl)]PF_6$ which are isolated as stable salts, can be easily obtained in high yields by an oxidative addition of methallyloxyphosphonium salt **6** to the zerovalent compound $Pd(dba)_2$ **5** in methylene chloride in the presence of a β-iminoamine ligand (Scheme 1).

The new complexes **7**–**10** which are soluble in methylene chloride and sparingly soluble in diethyl ether gave satisfactory analysis and were characterized by ^1H, ^{13}C $\{^1H\}$ NMR and IR spectroscopy.

The NMR data of ligands **1**–**4** are consistent with the hydrogen-bridged β-iminoamine structure [20], although complexes **7**–**10** incorporating these ligands as a β-diimine tautomer.

The ^1H NMR spectra of these compounds show two resonance signals for the allyl protons (H_{syn} and H_{anti}) which appear as a multiplet in contrast with the complexes of the symmetrical β-diimine ($R_1 = R_2 = CH_3$) which appear as a singlet [16]. The multiplicity of these signals is explained by the unsymmetrical diimine backbone and the *syn*/*syn* and *anti*/*anti* interconversion due to the dynamic behaviour of the allyl protons [21].

Thus the two resonances are located, respectively, at 2.19, 2.09, 2.58, 2.54 ppm for H_{anti} and at 2.59, 2.39, 2.88, 2.63 ppm for H_{syn} for complexes **7**–**10**.

In ^{13}C NMR spectra, the allylic carbon C(11) and C(13) appears at (62.51 and 64.54); (63.57 and 64.26); (64.41 and 64.94) and (62.33 and 65.72 ppm), respectively, for complexes **7**–**10**.

The imine carbons show two signals between 172 and 177 ppm.

The IR spectrum of compounds **7**–**10** indicated a shift in the C=N stretching frequency (1645; 1603, 1641; 1623, 1660 and 1616, 1647 cm^{-1}) relative to the respectively corresponding stretching frequency in the free ligands **1**–**4** (1622; 1622; 1610, 1649 and 1615 cm^{-1}). Moreover, the C=N stretching frequency appears at low frequency compared with their value for the compounds with the symmetrical β-diimine.

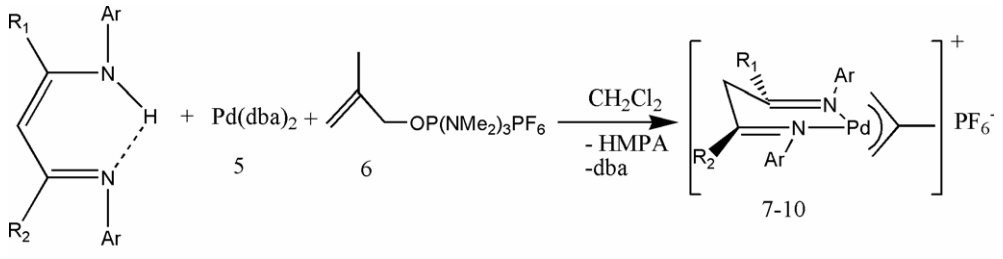
Complexes **7**–**10** exhibit the frequencies of the counter-ion PF_6^- at 838, 846, 832 and 837 cm^{-1}.

X-ray single-crystal analysis reveals that compound **10** exhibits some interesting features compared with the symmetrical β-diimine complexes. Suitable prismatic and colourless single crystals of **10** were obtained by crystallization from CH_2Cl_2/*n*-hexane. Complex **10** crystallizes in the monoclinic unit cell $P2_1/n$ group (Table 1).

An ORTEP-plot shown in Fig. 1 confirms the identity of complex **10**.

The $[(\eta^3\text{-}C_4H_7)Pd(NN)]^+$ cation of **10** adopts the usual slightly distorted square planar arrangement and this distortion is illustrated by the N(1)–Pd–N(2) bond angle equal to 90.9° (Fig. 1b). We note that this distortion is more attenuated than in the complex with the symmetrical β-diimine (N(1)–Pd–N(2) = 90.6°).

Selected bond lengths and angles are listed in Tables 2 and 3.



$R_1 = CH_3$, R_2 = Ph
(7) : Ar = 2-Me-C_6H_4
(8) : Ar = 2-MeO-C_6H_4
(9): Ar = 2,6-Me_2-C_6H_3
(10) : Ar = 2,6-iPr_2-C_6H_3
dba = dibenzylideneacetone
HMPA = Hexamethylphosphotriamide

Scheme 1. Synthesis of $[(\eta^3\text{-}C_4H_7)Pd((PhCNAr)CH_2(MeCNAr))]^+[PF_6]^-$ complexes.

Table 1
Crystallographic data and structure refinement for **10**

Empirical formula	$C_{38}H_{51}F_6N_2PPd$
Formula weight	787.18
Crystal system	monoclinic
Space group	$P2_1/n$
Z	4
a (Å)	12.495(5)
b (Å)	13.697(5)
c (Å)	22.244(5)
α(°)	90.000(5)
β (°)	92.230(5)
γ (°)	90.000(5)
V (Å^3)	3804(2)
D_{calc} (g cm^{-3})	1.374
Crystal size (mm^3)	0.11 × 0.18 × 0.25
Temperature (K)	293(2)
Wavelength (Å)	0.71069
F(000)	1632
Absorption coefficient (mm^{-1})	0.587
θ Range for data collection (°)	1.75–27.03
Limiting indices	$-15 \leqslant h \leqslant 15$, $0 \leqslant k \leqslant 17$, $-2 \leqslant l \leqslant 28$
Reflections collected/unique [R(int)]	9354/ 8271 [0.1933]
Completeness to $\theta = 27.03$	99.2%
Absorption correction	empirical (DIFABS)
Maximum and minimum transmission	0.5729 and 0.1077
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	8271/0/433
Goodness-of-fit on F^2	0.941
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0907$, $wR_2 = 0.2328$

$R_1 = \sum(||F_o| - |F_c||)/\sum|F_o|$.
$wR_2 = [\sum w(F_o^2 - F_c^2)^2/\sum(F_o^2)^2]^{1/2}$, where $w = 1/[\sigma^2(F_o^2) + (0.17P)^2]$, where $P = (F_o^2 + 2F_c^2)/3$.

Table 2
Selected interatomic distances

	Bond distances (Å) for **10**	Bond distances (Å) for the symmetric complex ($R_1 = R_2 = CH_3$)
Pd–N(1)	2.103(1)	2.105(3)
Pd–N(2)	2.130(1)	2.106(3)
Pd–C(11)	2.091(1)	2.129(4)
Pd–C(13)	2.117(1)	2.115(4)
Pd–C(12)	2.144(1)	2.153(3)
N(2)–C(3)	1.263(1)	1.284(4)
C(1)–N(1)	1.296(1)	1.284(4)
N(2)–C(15)	1.410(1)	1.462(4)
N(1)–C(27)	1.478(1)	1.459(4)
C(1)–C(2)	1.473(1)	1.509(4)
C(3)–C(2)	1.543(1)	1.504(4)

Table 3
Selected angles (°) for **10**

N (1)–Pd–N(2)	90.9(4)	C(11)–Pd–N (1)	101.3(6)
N (1)–Pd–C(13)	166.3(6)	C(11)–Pd–N (2)	166.3(6)
C (13)–Pd–N(2)	101.2(6)	N (1)–Pd–C(12)	133.1(6)
N (2)–Pd–C(12)	133.3(6)	C(11)–Pd–C(13)	66.0(7)
C(11)–Pd–C(12)	37.7(6)	C(11)–Pd–C(12)	37.7(6)
C(13)–Pd–C(12)	38.2(6)	C(3)–N(2)–C(15)	123.3(1)
N(1)–C(1)–C(4)	124.5(1)	N(1)–C(1)–C(2)	123.0(1)
N(2)–C(3)–C(5)	124.1(1)	N(2)–C(3)–C(2)	122.0(1)
C(1)–N(1)–C(27)	120.7(1)	C(3)–N(2)–Pd	123.8(1)
C(15)–N(2)–Pd	112.8(8)	C(1)–N(1)–Pd	123.1(1)
C(27)–N(1)–Pd	116.1(8)	C(16)–C(15)–N	119.3(1)
C(20)–C(15)–N(2)	119.7(1)	C(32)–C(27)–N(1)	120.4(1)
C(28)–C(27)–N(1)	117.4(1)	C(12)–C(11)–Pd	73.2(9)
C(12)–C(13)–Pd	71.9(9)		

If we define a plane containing the Pd and the two nitrogen atoms, then the terminal carbons are 0.24 (C11) and 0.25 Å (C13) below the plane, whereas the central allyl carbon is 0.44 Å above.

The allyl ligand is bonded almost symmetrically to the palladium centre Pd–C(11) = 2.09 Å and Pd–C(13) = 2.11 Å.

The N1–Pd–N2 plane is tilted by 63.74° with regard to the C11–C12–C13 plane. The methyl on the allyl group is slightly tilted out of the allyl plane by about 9.1° as indicated by the torsion angle of 170.1° of C(11)–C(12)–C(13)–C(14). The allyl plane (C(11), C(12), C(13)) makes an angle of 67.65° with the palladium coordinative least-square plane (N(1), N(2), C(11), C(13))

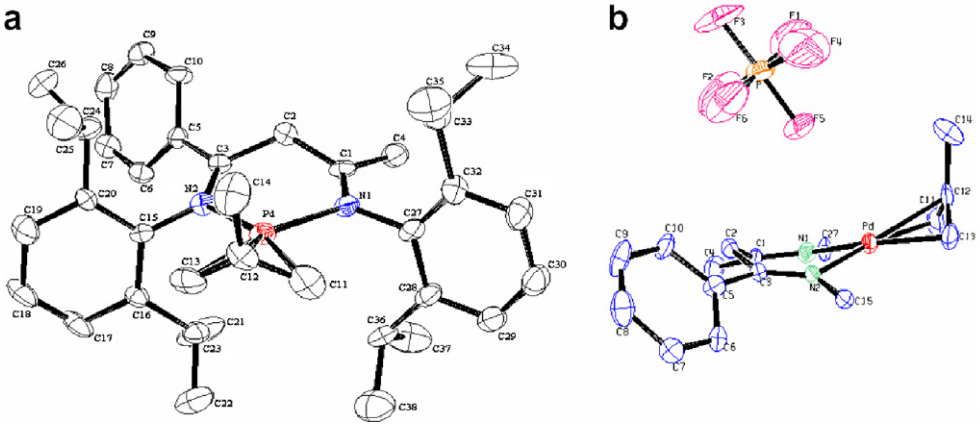


Fig. 1. (a) Perspective ORTEP diagram of the cation of **10**. Thermal ellipsoids are at 30% probability. Hydrogen atoms and anion are omitted for clarity. (b) View of the chelate ring in **10**. Hydrogen atoms and aryl ring are omitted for clarity.

The β-iminoamine ligand is bounded in a unsymmetrical fashion as the β-diimine tautomer, the C(1)–N(1) and C(3)–N(2) distances are 1.29 Å and 1.263 Å, respectively.

This ligand forms a six-membered ring with the palladium atom with Pd–N (sp^2) bond length of 2.10 Å and 2.13 Å in agreement with known values in the literature for related complexes [15,22–24]. Bond distances within the six-member chelate ring are consistent with the localized β-diimine structure drawn in Fig. 1, while the ring itself adopts a boat conformation as indicated in Fig. 1b. The aryl rings C(15–20) and C(27–32) are tilted out of the (N(1), C(1), C(3), N(2)) least-square plane by 70° and 69.49°, respectively.

3. Conclusion

New cationic methallyl palladium complexes supported by unsymmetrical β-diimine have been described. The application of these compounds in catalytic reactions such as dimerization and polymerization of alkenes and functional alkenes is in current study in order to compare them with the symmetric ones.

4. Experimental

4.1. General

All manipulations were carried out under an atmosphere of dry argon using standard Schlenk techniques.

Diethyl ether was distilled from sodium benzophenone; methylene chloride and hexane were distilled over P_2O_5. 2-Methylaniline, 2,6-dimethylaniline, 2-methoxyaniline and 2,6-diisopropylaniline were distilled from potassium hydroxide prior to use. $Pd(dba)_2$ [25,26] methallyloxyphosphonium salt [27] and β-iminoamine ligands [20] were prepared according to literature methods. All other reagents were obtained from standard commercial vendors and used as received. NMR spectra were recorded on a Bruker AC-300 spectrometer. H and C chemical shifts are given in ppm and referenced to the residual solvent resonance relative to TMS. Infrared spectra were recorded on a Bruker Victor 22 (Golden Gate Technique).

4.2. General procedure for the preparation of [(β-diimine)Pd(η^3-C_4H_7)]$^+PF_6^-$ complexes

In an inert atmosphere, $Pd(dba)_2$ complex (1 equiv.) was dissolved in 20 cm^3 anhydrous CH_2Cl_2. Methallyloxyphosphonium salt $C_4H_7OP^+(NMe_2)_3PF_6^-$ and β-iminoamine ligands were added (1 equiv.). The black-red solution was stirred at room temperature for 24 h. The supernatant was separated by filtration through a celite filter, and the solvent was removed under vacuum to afford the oil compound. This was washed with diethyl ether (3×15 cm^3) and dried in vacuum. The solid was crystallized from methylene chloride/*n*-hexane solution. Yields and spectral data of compounds **7**–**10** are reported below.

4.2.1. [(2-CH_3-C_6H_4NC(Ph)CH_2C(Me)NC_6H_4-2-CH_3)Pd(η^3-C_4H_7)]$^+PF_6^-$ (7)

Following the general procedure, from [2-CH_3-C_6H_4-NC(Ph)CHC(Me)NHC_6H_4-2-CH_3] (0.059 g, 0.173 mmol), $Pd(dba)_2$ (0.1 g, 0.173 mmol) and 2-methylallyloxyphosphonium (0.173 mmol, 0.065 g) was obtained 0.097 g of **7** as a pale yellow solid after crystallization from a mixture (CH_2Cl_2/*n*-hexane: 1/1).

Yield 87%. IR (Golden Gate): $\nu_{C=N} = 1645$ cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$, 25 °C, δ [ppm]): $\delta = 1.62$ (s, 3H, Me (allyl)); 1.79 (s, 3H Me on ligand); 1.98 (s, 3H, Me–C_6H_4); 2.11 (s, 3H, Me–C_6H_4); 2.19 (m, 2H, H_{11anti} and H_{13anti}); 2.59 (m, 2H, H_{11syn} and H_{13syn}); 3.83 (m, 2H, ligand–CH_2); 6.97–7.42 (m, 13H, ArH). ^{13}C NMR (75.5 MHz, $CDCl_3$, 25 °C, δ [ppm]): $\delta = 14.25$ (Me (allyl));18.85 (Me on C_6H_4); 19.48 (Me on C_6H_4); 22.31 (Me on ligand); 49.71 (CH_2 on ligand); 62.51 and 64.52 (C(11) and C(13)); 121.37–133.24 (C(ar) and C(12)); 138.13 (C–C on C_6H_5); 148.13 and 150.61 (C–N on C_6H_4); 172.45 and 176.25 (C=N on ligand).

4.2.2. [(2-CH_3O-C_6H_4NC(Ph)CH_2C(Me)NC_6H_4-2-CH_3O)-Pd(η^3-C_4H_7)]$^+PF_6^-$ (8)

Following the general procedure, from [2-CH_3O-C_6H_4NC(Ph)CHC(Me)NHC_6H_4-2-CH_3O] (0.064 g, 0.173 mmol), $Pd(dba)_2$ (0.1 g, 0.173 mmol) and 2-methylallyloxyphosphonium (0.173 mmol, 0.065 g) was obtained 0.104 g of **8** as a pale yellow solid after crystallization from a mixture (CH_2Cl_2/*n*-hexane: 1/1).

Yield = 89%. IR (Golden Gate): $\nu_{C=N} = 1603$ and 1641 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$, 25 °C, δ [ppm]): $\delta = 1.81$ (s, 3H, Me (allyl)); 2.00 (s, 3H, Me on ligand); 2.09 (m, 2H, H_{11anti} and H_{13anti}); 2.39 (m, 2H, H_{11syn} and H_{13syn}); 3.65–3.80 (m, 2H, ligand–CH_2); 3.85–3.91(m, 6H, H_3CO); 6.77–7.60 (m, 13H, ArH). ^{13}C NMR (75.5 MHz, $CDCl_3$, 25 °C, δ [ppm]): $\delta = 14.18$ (Me (allyl)); 22.75 (Me on ligand); 55.88 (CH_3O); 49.95 (CH_2 on ligand); 62.33 and 65.72 (C(11) and C(13)); 111.52–130.64 (C(ar) and C(12)-allyl); 134.44 (C–C on C_6H_5) 139.58 and 144.42 (C–N on C_6H_4); 177.07 and 177.21 (C=N on ligand).

4.2.3. [2,6-$(CH_3)_2$-C_6H_3NC(Ph)CH_2C(Me)NC_6H_3-2,6-$(CH_3)_2$Pd(η^3-C_4H_7)]$^+PF_6^-$ (9)

Following the general procedure, from [2,6-$(CH_3)_2$-C_6H_3NC(Ph)CHC(Me)NHC_6H_3-2,6-$(CH_3)_2$] (0.063 g, 0.173 mmol), $Pd(dba)_2$ (0.1 g, 0.173 mmol) and 2-methylallyloxyphosphonium (0.173 mmol, 0.065 g) was obtained 0.107 g of **9** as a pale yellow solid after crystallization from a mixture (CH_2Cl_2/*n*-hexane: 1/1).

Yield 92%. IR (Golden Gate): $\nu_{C=N} = 1623$ and 1660 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$, 25 °C, δ [ppm]): $\delta = 0.87$ (s, 3H, Me (allyl)); 1.56 (s, 3H, Me on ligand); 1.95 (s, 3H, Me on C_6H_3); 2.02 (s, 3H, Me on C_6H_3); 2.19 (s, 3H, Me on C_6H_3); 2.25 (s, 3H, Me on C_6H_3); 2.58 (m, 2H, H_{11anti} and H_{13anti}); 2.88 (m, 2H, H_{11syn} and H_{13syn}); 4.72 (m, 2H, ligand CH_2); 6.92–7.26 (m, 11H, Har). ^{13}C NMR (75.5 MHz, $CDCl_3$, 25 °C, δ [ppm]):

$\delta = 14.20$ (Me (allyl)); 22.72 (Me on ligand); 18.16, 18.37, 18.82 and 19.19(Me on C_6H_3); 49.46 (CH_2 on ligand); 63.57 and 64.26 (C(11) and C(13)); 126.24–135.68 (C(ar) and C(12)); 137.21 (C–C on C_6H_5); 149.84 and 150.51(C–N on C_6H_3); 173.73 and 176.54 (C=N on ligand).

4.2.4. [2,6-(iPr)$_2$-C$_6$H$_3$NC(Ph)CH$_2$C(Me)NC$_6$H$_3$-2,6-(iPr)$_2$Pd(η^3-C$_4$H$_7$)]$^+$PF$_6^-$ (**10**)

Following the general procedure, from [2,6-(iPr)$_2$-$C_6H_3NC(Ph)CHC(Me)NHC_6H_3$-2,6-(iPr)$_2$] (0.083 g, 0.173 mmol), Pd(dba)$_2$ (0.1 g, 0.173 mmol) and 2-methylallyloxyphosphonium (0.173 mmol, 0.065 g) was obtained 0.129 g of **10** as a pale yellow solid after crystallization from a mixture (CH_2Cl_2/*n*-hexane: 1/1).

Yield = 95%. IR (Golden Gate): $\nu_{C=N} = 1616$ and 1647 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C, δ [ppm]): $\delta = 0.88$–1.40 (m, 21 H, Me-iPr and Me on allyl); 1.99 (s, 3H, Me on ligand); 2.17 (d, 6H, $J = 16.8$ Hz, Me-iPr); 2.54 (m, 2H, H_{11anti} and H_{13anti}); 2.63 (m, 2H, H_{11syn} and H_{13syn}); 3.02 (sept, 2H, $J = 7.5$ Hz, H-C-iPr); 3.39 (sept, 1H, $J = 6.9$ Hz, H-C-iPr); 3.76 (sept, 1H, $J = 7.2$ Hz, H-C-iPr); 4.66–5.06 (m, 2H, CH_2 on ligand); 7.02–7.26 (m, 11H, H_{ar}); ^{13}C NMR (75.5 MHz, $CDCl_3$, 25 °C, δ [ppm]): $\delta = 15.87$ (Me-allyl); 22.84 (Me on ligand); 23.84, 23.88, 24.00, 24.21, 24.34, 24.38, 24.71 and 25.66 (Me on iPr group); 28.68, 28.76, 29.20 and 29.43 (CH on iPr group); 49.91 (CH_2 on ligand); 64.41 and 64.9 (C(11) and C(13)); 124.25–138.08 (C(ar) and C(12)); 138.23 (C–C on C_6H_5); 147.26 and 148.14 (C–N on C_6H_3); 172.77 and 177.45 (C=N on ligand).

4.3. X-ray crystallographic study

The X-ray crystallographic study of complex **10** was carried out on a CAD4 Enraf-Nonius diffractometer (Mo Kα). Data were collected at 283 K in the range 1–27° and this gave a total of 9354 reflections, yielding 8271 independent values ($R_{int} = 0.1933$). The structure was solved by direct method and difference Fourier techniques and were refined by full-matrix least-squares analysis. Refinements were based on F^2 and were carried out using all the data (SHELXL-97). All of the non-hydrogen atoms were re-fined anisotropically. The hydrogen atoms were fixed geometrically in their idealized positions. The set of physical and crystallographic characteristics as well as the experimental conditions are listed in Table 1.

Appendix A. Supplementary material

CCDC 272962 contains the supplementary crystallographic data for **10**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033, or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.poly.2007.11.026.

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