

Bis(pentafluorophenyl)nickel(II) complexes with α -diimines and iminophosphines

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Received 17 February 1998; accepted 6 May 1998

Abstract

New bis(pentafluorophenyl) derivatives of nickel(II) have been prepared by reaction between cis -[Ni(C₆F₅)₂(PhCN)₂] and bidentate chelate ligands L₂. The new complexes of general formula [Ni(C₆F₅)₂L₂] containing mixed-donor bidentate ligands (L₂ = iminophosphine: P(*o*-C₆H₄CH=N-R); R = Me, Et, ⁿPr, ⁱPr, ^tBu, Ph, NH-Me) or symmetrical ligands (L₂ = α -diimine: R₁N=CR-CR=NR₁; R = H, R₁ = Ph, C₆H₄Me-*o* or C₆H₄Me-*p*; R = Me, R₁ = Ph, C₆H₄Me-*o* or C₆H₄Me-*p*) have been characterized by partial elemental analyses and spectroscopic methods (IR, and ¹H, ³¹P and ¹⁹F NMR). © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Chelate complexes; Pentafluorophenylnickel complexes; Iminophosphines; α -Diimines

1. Introduction

The labile complexes cis -[M(C₆F₅)₂(PhCN)₂] (M = Ni or Pd) have shown to be convenient precursors for the preparation of other pentafluorophenyl compounds [1–4]. This paper is concerned with the preparation of complexes [Ni(C₆F₅)₂L₂], where L₂ is one of the following ligands: L₂ = iminophosphine: P(*o*-C₆H₄CH=N-R); R = Me, Et, ⁿPr, ⁱPr, ^tBu, Ph, NH-Me); L₂ = α -diimine: R₁N=CR-CR=NR₁; R = H, R₁ = Ph, C₆H₄Me-*o* or C₆H₄Me-*p*; R = Me, R₁ = Ph, C₆H₄Me-*o* or C₆H₄Me-*p*. The new complexes have been prepared from cis -[Ni(C₆F₅)₂(PhCN)₂] via the corresponding substitution reactions in dichloromethane or acetone as solvents.

The chemistry of transition metal complexes of α -diimines is dominated by the versatile coordination behaviour of this type of organic ligands and the interesting properties of their metal complexes [5–8], thus, some α -diimine complexes of nickel(II) [9] and palladium(II) [10,11] have been used as catalysts for oligomerization and hydrogenation processes.

On the other hand, there has recently been considerable interest in the chemistry of polydentate ligands with both hard and soft donor atoms. In this sense, the metal complexes with N and P donor atoms display a variety of coordination well beyond those of P–P or N–N ligands [12–14]. Here we report the preparation of some pentafluorophenyl nickel(II)

derivatives with bidentate ligands, α -diimines (N,N-donors) and iminophosphines (N,P-donors). The iminophosphine ligands were prepared via Schiff-base reaction between *o*-(diphenylphosphino)benzaldehyde and the corresponding amine [15]. The new complexes have been characterized by partial elemental analyses, and spectroscopic methods (IR, and ¹H, ³¹P and ¹⁹F NMR).

2. Experimental

2.1. Physical measurements and materials

C, H and N analyses were carried out with a Perkin-Elmer 240C microanalyser. IR spectra were recorded on a Perkin-Elmer 1430 spectrophotometer using Nujol mulls between polyethylene sheets. NMR data were recorded on a Bruker AC 200E (¹H) or a Varian Unity 300 (¹⁹F, ³¹P) spectrometer.

The precursor cis -[Ni(C₆F₅)₂(PhCN)₂] was prepared as described in the literature [16]. The α -diimine [17–19] and iminophosphine [15] ligands were prepared according to reported procedures and all the solvents were dried by standard methods before use.

2.2. Preparation of the iminophosphine complexes

The complexes were obtained by treating [Ni(C₆F₅)₂(PhCN)₂] with the corresponding iminophosphine (molar

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ratio 1:1) in acetone according to the following general method. To an acetone (5 ml) solution of the $[\text{Ni}(\text{C}_6\text{F}_5)_2(\text{PhCN})_2]$ (0.15 g, 0.25 mmol) was added the calculated amount of an iminophosphine acetone solution previously prepared. The reaction was stirred at room temperature for 30 min. Upon complete solvent evaporation, the crude product was collected with hexane/*i*-PrOH and then filtered off, washed with hexane and air-dried. Yellow crystals of the compounds (1–7) were obtained in high yield by recrystallization from dichloromethane-hexane.

$[\text{Ni}(\text{C}_6\text{F}_5)_2\{\text{P}(o\text{-C}_6\text{H}_4\text{-CH=NR})\text{Ph}_2\}]$ (R = Me) (1) was obtained in 88% yield. Anal. Calcd. for $\text{C}_{32}\text{F}_{10}\text{H}_{18}\text{NPNi}$: C, 55.2; H, 2.6; N, 2.0. Found: C, 55.1; H, 2.5; N, 2.0%. IR (cm^{-1}): 1630 (C=N str).

$[\text{Ni}(\text{C}_6\text{F}_5)_2\{\text{P}(o\text{-C}_6\text{H}_4\text{-CH=NR})\text{Ph}_2\}]$ (R = Et) (2) was obtained in 95% yield. Anal. Calcd. for $\text{C}_{33}\text{F}_{10}\text{H}_{20}\text{NPNi}$: C, 55.8; H, 2.8; N, 2.0. Found: C, 55.9; H, 2.9; N, 2.1%. IR (cm^{-1}): 1626 (C=N str).

$[\text{Ni}(\text{C}_6\text{F}_5)_2\{\text{P}(o\text{-C}_6\text{H}_4\text{-CH=NR})\text{Ph}_2\}]$ (R = *n*Pr) (3) was obtained in 75% yield. Anal. Calcd. for $\text{C}_{34}\text{F}_{10}\text{H}_{22}\text{NPNi}$: C, 56.4; H, 3.1; N, 1.9. Found: C, 56.6; H, 3.0; N, 2.0%. IR (cm^{-1}): 1632 (C=N str).

$[\text{Ni}(\text{C}_6\text{F}_5)_2\{\text{P}(o\text{-C}_6\text{H}_4\text{-CH=NR})\text{Ph}_2\}]$ (R = *i*Pr) (4) was obtained in 82% yield. Anal. Calcd. for $\text{C}_{34}\text{F}_{10}\text{H}_{22}\text{NPNi}$: C, 56.4; H, 3.1; N, 1.9. Found: C, 56.3; H, 2.9; N, 1.9%. IR (cm^{-1}): 1630 (C=N str).

$[\text{Ni}(\text{C}_6\text{F}_5)_2\{\text{P}(o\text{-C}_6\text{H}_4\text{-CH=NR})\text{Ph}_2\}]$ (R = *t*Bu) (5) was obtained in 88% yield. Anal. Calcd. for $\text{C}_{35}\text{F}_{10}\text{H}_{24}\text{NPNi}$: C, 56.9; H, 3.3; N, 1.9. Found: C, 56.8; H, 3.4; N, 2.1%. IR (cm^{-1}): 1620 (C=N str).

$[\text{Ni}(\text{C}_6\text{F}_5)_2\{\text{P}(o\text{-C}_6\text{H}_4\text{-CH=NR})\text{Ph}_2\}]$ (R = Ph) (6) was obtained in 87% yield. Anal. Calcd. for $\text{C}_{37}\text{F}_{10}\text{H}_{20}\text{NPNi}$: C, 58.6; H, 2.7; N, 1.9. Found: C, 58.6; H, 2.9; N, 2.0%. IR (cm^{-1}): 1624 (C=N str).

$[\text{Ni}(\text{C}_6\text{F}_5)_2\{\text{P}(o\text{-C}_6\text{H}_4\text{-CH=NR})\text{Ph}_2\}]$ (R = NHMe) (7) was obtained in 92% yield. Anal. Calcd. for $\text{C}_{32}\text{F}_{10}\text{H}_{19}\text{N}_2\text{PNi}$: C, 54.0; H, 2.7; N, 3.9. Found: C, 53.9; H, 2.9; N, 4.0%. IR (cm^{-1}): 1632 (C=N str), 3418 (N–H str).

2.3. Preparation of the α -diimine complexes

To a solution of complex $[\text{Ni}(\text{C}_6\text{F}_5)_2(\text{PhCN})_2]$ (0.15 g, 0.25 mmol) in dichloromethane was added the corresponding α -diimine (0.25 mmol) previously prepared as described elsewhere [17–19]. After stirring for 30 min at room temperature, the solution was concentrated under reduced pressure. Addition of hexane caused precipitation of the complexes (8–13). These were filtered off, air dried and recrystallized from dichloromethane-hexane.

$[\text{Ni}(\text{C}_6\text{F}_5)_2(\text{R}_1\text{N=CRCR=NR}_1)]$ (R = H, R_1 = Ph) (8) was obtained in 58% yield. Anal. Calcd. for $\text{C}_{26}\text{F}_{10}\text{H}_{12}\text{N}_2\text{Ni}$: C, 51.9; H, 4.7; N, 2.0. Found: C, 51.8; H, 4.7; N, 2.0%. IR (cm^{-1}): 1594 (C=N str).

$[\text{Ni}(\text{C}_6\text{F}_5)_2(\text{R}_1\text{N=CRCR=NR}_1)]$ (R = H, R_1 = *o*-MeC₆H₄) (9) was obtained in 60% yield. Anal. Calcd. for

$\text{C}_{28}\text{F}_{10}\text{H}_{16}\text{N}_2\text{Ni}$: C, 53.4; H, 4.4; N, 2.6. Found: C, 53.4; H, 4.4; N, 2.4%. IR (cm^{-1}): 1538 (C=N str).

$[\text{Ni}(\text{C}_6\text{F}_5)_2(\text{R}_1\text{N=CRCR=NR}_1)]$ (R = H, R_1 = *p*-MeC₆H₄) (10) was obtained in 65% yield. Anal. Calcd. for $\text{C}_{28}\text{F}_{10}\text{H}_{16}\text{N}_2\text{Ni}$: C, 53.4; H, 4.4; N, 2.6. Found: C, 53.3; H, 4.4; N, 2.5%. IR (cm^{-1}): 1606 (C=N str).

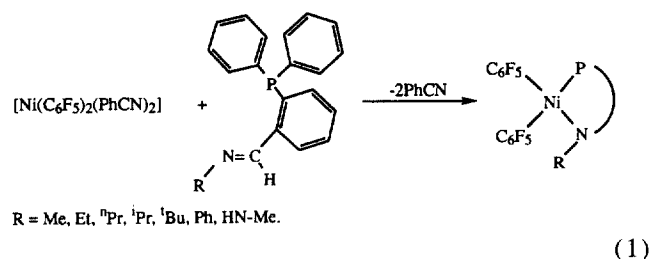
$[\text{Ni}(\text{C}_6\text{F}_5)_2(\text{R}_1\text{N=CRCR=NR}_1)]$ (R = Me, R_1 = Ph) (11) was obtained in 87% yield. Anal. Calcd. for $\text{C}_{28}\text{F}_{10}\text{H}_{16}\text{N}_2\text{Ni}$: C, 53.4; H, 4.5; N, 2.6. Found: C, 53.4; H, 4.5; N, 2.7%. IR (cm^{-1}): 1592 (C=N str).

$[\text{Ni}(\text{C}_6\text{F}_5)_2(\text{R}_1\text{N=CRCR=NR}_1)]$ (R = Me, R_1 = *o*-MeC₆H₄) (12) was obtained in 62% yield. Anal. Calcd. for $\text{C}_{30}\text{F}_{10}\text{H}_{20}\text{N}_2\text{Ni}$: C, 54.8; H, 4.3; N, 3.0. Found: C, 54.7; H, 4.2; N, 3.1%. IR (cm^{-1}): 1602 (C=N str).

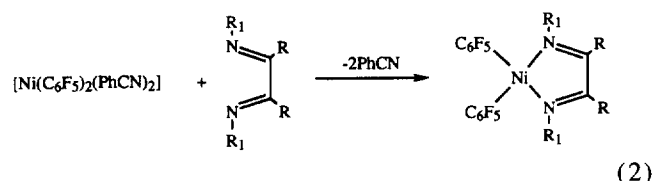
$[\text{Ni}(\text{C}_6\text{F}_5)_2(\text{R}_1\text{N=CRCR=NR}_1)]$ (R = Me, R_1 = *p*-MeC₆H₄) (13) was obtained in 82% yield. Anal. Calcd. for $\text{C}_{30}\text{F}_{10}\text{H}_{20}\text{N}_2\text{Ni}$: C, 54.8; H, 4.3; N, 3.0. Found: C, 54.7; H, 4.1; N, 3.0%. IR (cm^{-1}): 1614 (C=N str).

3. Results and discussion

In acetone, *cis*- $[\text{Ni}(\text{C}_6\text{F}_5)_2(\text{PhCN})_2]$ reacts with iminophosphines (molar ratio 1:1) under mild conditions to give the corresponding square-planar neutral complexes $[\text{Ni}(\text{C}_6\text{F}_5)_2\text{L}_2]$, according to Eq. (1).



The addition of the corresponding α -diimine to *cis*- $[\text{Ni}(\text{C}_6\text{F}_5)_2(\text{PhCN})_2]$ in dichloromethane leads to the formation of the expected organo-complexes (Eq. (2)).



The new pentafluorophenyl-nickel derivatives with iminophosphines are air-stable yellow solids, while the α -diimine complexes present a deep purple colour. Infrared spectra of all compounds show the characteristic absorptions of the C₆F₅ group [20] at ca. 1630 m, 1490 vs, 1050 s and 950 vs cm^{-1} . The presence of two bands in the 800–780 cm^{-1} for the so-called 'X-sensitive' mode of C₆F₅ is characteristic of

Table 1
 ^1H , ^{19}F and ^{31}P NMR data for the nickel iminophosphine complexes ^{a,b}

Compound	R	^1H δ [ppm (SiMe ₄)]	^{19}F δ [ppm (CFCl ₃)]	^{31}P δ [ppm (H ₃ PO ₄)]
(1)	Me	3.3 (s, 3H, Me) 7.4 (m, 11H) 7.7 (m, 1H) 7.8 (m, 2H) 8.2 (s, 1H, CH=N)	-116.3 (d, 2F _o , J=30.5) -118.0 (m, 2F _o) -163.4 (t, 1F _p , J=20.0) -165.2 (m, 1F _p + 2F _m) -166.3 (m, 2F _m) -168.4 (m, 2F _m)	31.7 (s, 1P)
(2)	Et	1.4 (t, 3H, CH ₃ , J=7.2) 3.3 (q, 2H, CH ₂ , J=7.2) 7.5 (m, 11H) 7.7 (m, 1H) 7.9 (m, 2H) 8.3 (d, 1H, CH=N, J=3.4)	-116.4 (d, 2F _o , J=29.1) -117.7 (m, 2F _o) -163.4 (t, 1F _p , J=19.8) -165.2 (m, 1F _p + 2F _m) -166.3 (m, 2F _m)	28.9 (s, 1P)
(3)	ⁿ Pr	0.6 (t, 3H, CH ₃ , J=7.3) 1.7 (m, 2H, CH ₂) 3.2 (t, 2H, CH ₂ N, J=8.2) 7.4 (m, 11H) 7.7 (m, 1H) 7.9 (m, 2H) 8.3 (d, 1H, CH=N, J=3.5)	-116.2 (d, 2F _o , J=29.1) -118.0 (m, 2F _o) -163.3 (t, 1F _p , J=19.8) -165.1 (m, 1F _p + 2F _m) -166.3 (m, 2F _m)	28.6 (s, 1P)
(4)	ⁱ Pr	1.0 (d, 6H, CH ₃ , J=6.6) 3.9 (sep, 1H, CH, J=6.6) 7.5 (m, 11H) 7.7 (m, 1H) 7.9 (m, 2H) 8.3 (d, 1H, CH=N, J=4.1)	-116.3 (d, 2F _o , J=27.4) -117.9 (d, 2F _o , J=29.1) -163.4 (t, 1F _p , J=19.8) -165.0 (t, 1F _p , J=19.8) -165.3 (m, 2F _m) -166.3 (m, 2F _m)	30.1 (s, 1P)
(5)	^t Bu	1.2 (s, 9H, CH ₃) 6.9 (m, 2H) 7.3 (m, 11H) 7.7 (m, 1H) 7.9 (m, 2H) 8.3 (d, 1H, CH=N, J=5.5)	-114.6 (br, 1F _o) -116.3 (br, 1F _o) -118.5 (br, 2F _o) -163.5 (t, 1F _p , J=19.8) -164.9 (m, 1F _p + 1F _m) -166.7 (br, 3F _m)	30.1 (s, 1P)
(6)	Ph	7.2 (m, 4H) 7.5 (m, 12H) 7.8 (t, 1H, J=7.6) 7.9 (t, 1H, J=7.6) 8.1 (m, 1H, CH=N)	-115.9 (d, 2F _o , J=24.3) -118.4 (m, 2F _o) -164.2 (t, 1F _p , J=19.8) -164.9 (t, 1F _p , J=19.8) -165.9 (m, 2F _m) -166.2 (m, 2F _m)	28.5 (s, 1P)
(7)	NH-Me	2.7 (d, 3H, Me, J=4.5) 6.6 (q, 1H, NH, J=4.5) 7.5 (m, 12H) 7.7 (m, 2H) 8.5 (d, 1H, CH=N, J=3.4)	-115.9 (d, 2F _o , J=24.3) -118.4 (m, 2F _o) -164.2 (t, 1F _p , J=19.8) -164.9 (t, 1F _p , J=19.8) -165.9 (m, 2F _m) -166.2 (m, 2F _m)	31.3 (s, 1P)

^a In CDCl₃.

^b J in Hz.

the *cis*-Ni(C₆F₅)₂ fragment, [21] which obviously appears in compounds containing bidentate chelate ligands.

The most relevant IR bands assigned to the ligands in both the α -diimine and iminophosphine complexes appear in the 1640–1500 cm⁻¹ region. Thus the IR spectra of the complexes show a single strong band in this region attributed to the C=N stretching vibrations, shifted to lower frequencies than in the free ligands.

The ^1H , ^{19}F and ^{31}P -NMR data of iminophosphine (1–7) and α -diimine (8–13) complexes are collected in Tables 1

and 2, respectively. The ^{31}P -NMR of iminophosphine compounds consist of singlets with chemical shifts in the typical range of Ni(II) complexes, while the appreciable coupling (3–6 Hz) to the phosphorus atom exhibited by the CH=N protons is the only remarkable aspect of their ^1H -NMR spectra.

The most outstanding feature in the ^1H -NMR spectra of α -diimine complexes is found in compounds 9 and 12 (R₁N=CRCR=NR₁; R=H, R₁=*o*-MeC₆H₄ 9, R=Me, R₁=*o*-MeC₆H₄ 12), that exhibit two resonance signals for

Table 2
 ^1H and ^{19}F NMR data for the nickel complexes with α -diimines ^{a,b}

Compound	R	R ₁	^1H δ [ppm (SiMe ₄)]	^{19}F δ [ppm (CFCl ₃)] ^c
(8)	H	Ph	7.1 (m, 6H, Ph) 7.3 (m, 4H, Ph) 8.6 (s, 2H, CH=N)	-117.6 (d, 4F _o , J=26.0) -163.3 (t, 2F _p , J=18.3) -166.1 (m, 4F _m)
(9)	H	<i>o</i> -MeC ₆ H ₄	2.5 (s, 6H, Me) 7.1 (m, 8H) 8.6 (s, 1H, CH=N)	-115.7 (br, 1F _o , J=29.1) -116.3 (br, 1F _o , J=28.9) -117.4 (br, 1F _o , J=23.1) -118.2 (br, 1F _o , J=27.4) -163.4 (t, 2F _p , J=18.3) -166.2 (br, 4F _m)
(10)	H	<i>p</i> -MeC ₆ H ₄	2.2 (s, 6H, Me) 7.0 (d, 4H, C ₆ H ₄ , J=8.2) 7.1 (d, 4H, C ₆ H ₄ , J=8.2) 8.5 (s, 2H, CH=N)	-117.3 (d, 4F _o , J=22.0) -163.0 (t, 2F _p , J=19.2) -165.7 (m, 4F _m)
(11)	Me	Ph	2.2 (s, 6H, Me) 6.7 (m, 4H, Ph) 7.1 (m, 6H, Ph)	-117.2 (d, 4F _o , J=23.1) -164.3 (t, 2F _p , J=18.3) -166.6 (m, 4F _m)
(12)	Me	<i>o</i> -MeC ₆ H ₄	2.1 (s, 3H, Me) 2.2 (s, 3H, Me) 2.4 (s, 3H, <i>o</i> -MeC ₆ H ₄) 2.5 (s, 3H, <i>o</i> -MeC ₆ H ₄) 7.0 (m, 2H, C ₆ H ₄) 7.2 (m, 6H, C ₆ H ₄)	-115.2 (br, 1F _o) -116.1 (br, 1F _o) -116.7 (br, 1F _o) -117.9 (br, 1F _o) -164.3 (t, 2F _p , J=18.1) -166.5 (br, 4F _m)
(13)	Me	<i>p</i> -MeC ₆ H ₄	2.2 (m, 12H, Me) 6.7 (d, 4H, C ₆ H ₄ , J=8.0) 7.1 (d, 4H, C ₆ H ₄ , J=8.0)	-117.3 (d, 4F _o , J=22.0) -163.0 (t, 2F _p , J=19.2) -165.7 (m, 4F _m)

^a In CDCl₃.

^b J in Hz.

^c At 20°C.

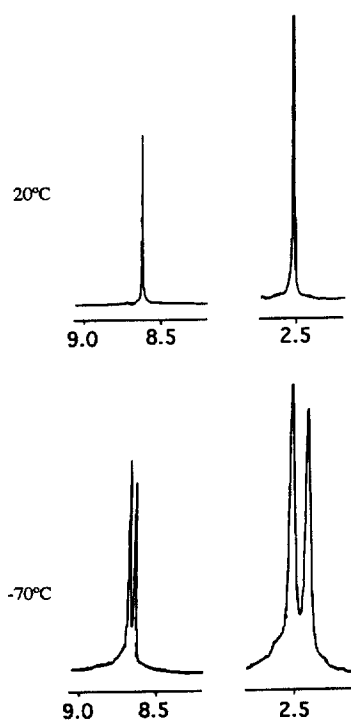


Fig. 1. ^1H NMR spectra of complex **9** in the aliphatic and CH=N regions at 20 and -70°C .

both the R substituents and the *o*-methyl groups. This behaviour is detected at room temperature in the spectrum of **12**, while the splitting of peaks in **9** is observed at -70°C . (Fig. 1) and suggests frozen rotation of the *o*-MeC₆H₄ substituents around the C–N bond.

The ^{19}F spectra show the expected patterns of three resonance signals for the α -diimine complexes, corresponding to two equivalent fluorophenyl groups rotating freely around the N–C bond (intensity ratio 2:1:2), and duplicated resonances for the iminophosphine complexes, consistent with the presence of two nonequivalent C₆F₅ groups, one *trans* to N and one *trans* to P. Thus, there are two different *o*-fluorine resonances: the lower field doublet attributed to the C₆F₅ ring *trans* to N, and a multiplet signal caused by additional coupling with the *trans* P-atom.

The ^{19}F -NMR spectra of complexes **5**, **9** and **12** at room temperature show broad resonances in the *o*-fluorine region, suggesting that the bulky *o*-tolyl and *t*-butyl groups slow down the 'free' rotation of the C₆F₅ rings. Fig. 2 shows the spectra of complex **9** as a function of temperature. At -20°C , the rotation is sufficiently sluggish to make the *o*-fluorine atoms distinguishable by their different chemical shifts, and four 1:1:1:1 doublet resonances of equal intensity (caused by coupling to the *m*-fluorine atom) are observed. As the tem-

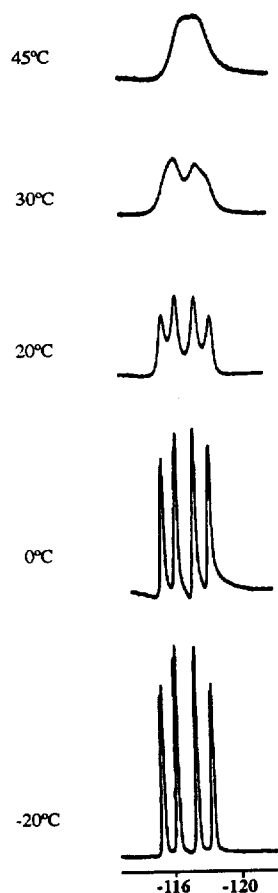


Fig. 2. Variable-temperature ^{19}F NMR spectra of complex **9** in the *o*-fluorine region.

perature is raised, the signals broaden and coalesce into two groups of resonances is observed at 30°C, while a single broad signal is obtained at 45°C. This condition of fast exchange was attained at 40°C in the previously reported nickel and palladium salicylaldiminate pentafluorophenyl derivatives [22,23].

Acknowledgements

We thank the Consejería de Cultura y Educación, project

PCOM-17/96 EXP (Comunidad Autónoma de Murcia, Spain) for financial support. J.L. Serrano thanks the Obra Social y Cultural de Cajamurcia for a research grant.

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