Journal of Organometallic Chemistry, 398 (1990) 197–204
Elsevier Sequoia S.A., Lausanne
JOM 21197

^{15}N and ^{13}C NMR studies on cyclopalladated complexes. Synthetic palladium cyanide chemistry leading to organic nitriles

Samuel Affolter and Paul S. Pregosin

Laboratorium für anorganische Chemie ETH-Z, Universitätstrasse 6, CH-8092 Zürich (Switzerland)

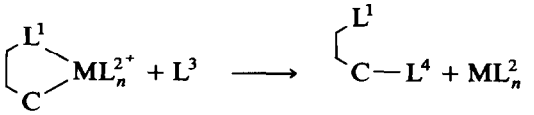
(Received May 14th, 1990)

Abstract

^{15}N NMR spectroscopy has been used to determine whether the ^{15}NO group is coordinated in the cyclopalladated nitrosoamine complex $[(Pd(\mu\text{-}OAc)(O{=}NN\text{-}(CH_3)C_6H_4)]_2$, $PdCl\{O{=}NN(CH_3)C_6H_4\}\{P(OMe)_3\}$ and $K_2[Pd(CN)_3(O{=}NN\text{-}(CH_3)C_6H_4)]$, **11**, as well as in several platinum(II) analogs. ^{13}C NMR studies on **11**, the cyclopalladated *N,N*-dimethylbenzylamine analog $K_2[Pd(CN)_3(Me_2NCH_2C_6\text{-}H_4)]$, **12**, and the dicyano cyclopalladated phosphite anion $[Pd(CN)_2\{(PhO)_2P)\text{-}OC_6H_4)\}]$, **13**, are useful in determining the coordination sphere of these molecules, and afford the novel coupling constants $^2J(^{13}C\text{–}Pd\text{–}^{13}C)$ *cis* and *trans*, between one cyanide and the palladated carbon. The complexes **11–13** are used as models to rationalize the reactions of **11** and **12** with $(Bu^n_4N)CN$ and diphos to form a 2-cyano-*N*-nitrosoaniline and a 2-cyano-*N,N*-dimethylbenzylamine.

Introduction

Reactions of cyclometallated complexes to afford organic products, e.g., reaction 1, represent the last step in the transformation of selected monosubstituted aryl



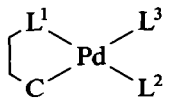
(1)

($L^4 = L^3$ or modified L^3)

compounds into *ortho*-disubstituted derivatives [1–3]. For complexes of palladium a

0022-328X/90/$03.50

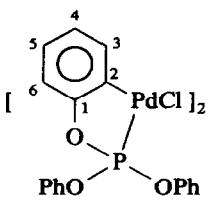
number of intermediates, e.g., **2**, have been postulated [3–7] in which L^3 coordinates to the palladium before subsequent transformation to product.



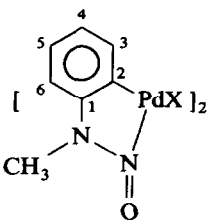
(2)

The nature of **2**, i.e., its coordination number, geometry, charge, etc., is important since the insertion of L^3 into a Pd–C bond and/or a reductive elimination step might be readily achieved if the Pd–L^1 bond were labile.

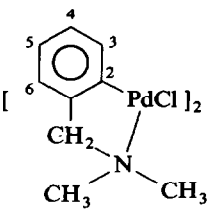
In the course of our synthetic studies on cyclopalladated phosphite [8], nitrosoamine [9], and *N,N*-dimethylbenzylamine [10] ligands, we have found it useful to



(3)



(4a, X = OAc
4b, X = Cl)



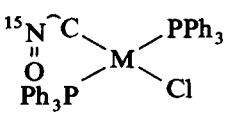
(5)

prepare complexes containing enriched ^{15}N ligands, e.g., **4**, with > 95 atom% $^{15}N{=}O$, and/or with enriched (96.8 atom%) $^{13}CN^-$. These molecules are of interest in that their NMR spectra are rich in structural information and also afford access to relatively rare spin–spin couplings, e.g., $^2J(^{13}C\text{–}M\text{–}^{13}C)$. We report here on the ^{15}N characteristics of several derivatives of **4** and their platinum analogs as well as ^{13}C NMR properties of bis and tris anionic cyano complexes arising from complexes **3–5**.

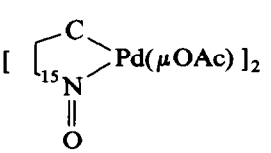
Results and discussion

It is useful to abbreviate the cyclometallated ligands and henceforth the cyclometallated species from **3–5** will be written as O=N^C, N^C and P^C, respectively, e.g., **4a** = $[Pd(\mu\text{-}OAc)(O{=}N\text{^}C)]_2$.

Complex **4a** can be readily prepared in ca. 90% yield [9] from the enriched ligand and $Pd(OAc)_2$. Indeed, there are a number of stable cyclopalladated nitrosoamines, and two derivatives of **4b**, PdCl(O=N^C))(PPh_3), **6**, and *trans*-PdCl(O=N^C)$(PPh_3)_2$, **7**, have been crystallized by Constable et al. [11] and their structures determined.



(**7**, M = Pd, $\delta^{15}N = 538.8$
8, M = Pt, $\delta^{15}N = 546.2$)



(**4a**, $\delta^{15}N = 416.6$)

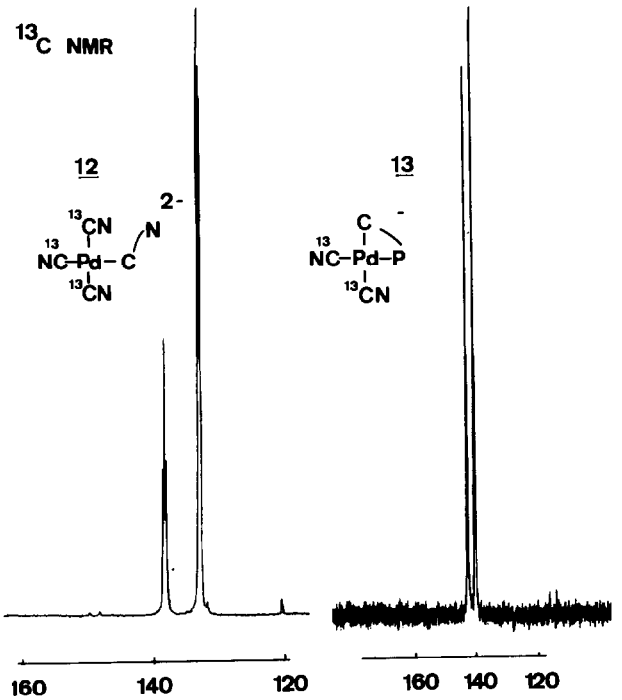
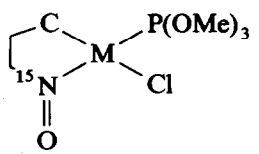


Fig. 1. ^{13}C spectrum of left: complex **12**, enriched in $^{13}CN^-$ and right: ^{31}P NMR spectrum of complex **13**, enriched in $^{13}CN^-$.

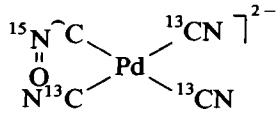
The usefulness of the nitrogen-15 chemical shift, $\delta^{15}N$, in differentiating between coordinated and uncoordinated sp^2 nitrogen atoms has been recognized in the coordination chemistry of azo [12,13], Schiffs' base [14], thiocyanate [15], nitrosyl [16–18], diazenido [19] and other ligands [20], and is nicely illustrated by compari-



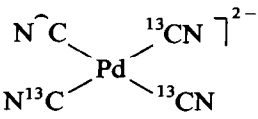
(**9**, M = Pd, $\delta^{15}N = 440.1$
10, M = Pt, $\delta^{15}N = 422.8$)

son of $\delta^{15}N$ for **4a** with $\delta^{15}N$ for **7**, which is known [11] to be the four-coordinate *trans* isomer shown. Complex **7** has an ^{15}N shift of 538.8, close to that of the free ligand, 548.2, whereas complex **4a** with bound nitrogen experiences the expected [12–20] large high field (low frequency) shift due to involvement of the nitrogen lone-pair in the palladium nitrogen bond. As we have shown on several occasions [14,21], the magnitude of this high field shift depends on the ligand *trans* to the coordinated nitrogen such that, for sp^2 nitrogen, there is a low field shift with increasing *trans* influence. This effect is illustrated by **9** in which $P(OMe)_3$ is coordinated *trans* to ^{15}N, relative to **4a** with μ-OAc *trans* to ^{15}N. Complexes **8** and **10** are platinum analogs of **7** and **9**, respectively. The *trans* orientation of the ^{15}N and ^{31}P spins in **9** and **10** is supported by the $^2J(P,N)$ values of 83.5 and 95.2 Hz, respectively, in keeping with earlier reports on this subject [14,15,21–23].

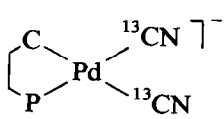
Reaction of either **4a** or **4b** with three equivalents of enriched $^{13}CN^-$ per palladium affords complex **11**. The ^{15}N resonance, $\delta = 529.5$, is again consistent



(**11**, $\delta^{15}N = 529.5$)



(**12**)



(**13**)

with an uncoordinated nitroso function, and in support of this we observe two ^{13}C resonances, a triplet a $\delta = 146.6$ and a doublet at $\delta = 144.6$ in the ratio of 1 : 2, with a $^2J(^{13}C,^{13}C)$ value of 7.3 Hz.

The cyclopalladated carbon, C-2, $\delta = 149.2$, is split by the *trans* $^{13}CN^-$ into a doublet: $^2J(^{13}C,^{13}C)_{trans} = 42.0$ Hz. Although we do not observe a corresponding *cis* coupling, these data seem to support the generally observed [24] trend: $^2J(X,Y)_{trans} > {}^2J(X,Y)_{cis}$.

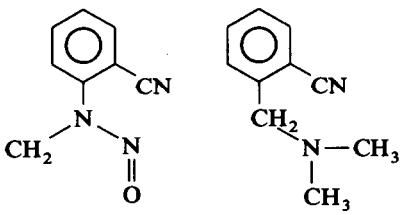
The analogous enriched cyclopalladated *N,N'*-dimethylbenzylamine dianion, $Pd(^{13}CN)_3(C\widehat{\ }N)^{2-}$, **12**, also revealed two $^{13}CN^-$ signals in a 1 : 2 ratio at δ 149.4 and 146.7 with $^2J(^{13}C,^{13}C)_{cis} = 7.0$ Hz, see Fig. 1. The palladated carbon, C-2, $\delta = 154.8$, couples to both $^{13}CN^-$ ligands, $^2J(^{13}C,^{13}C)_{trans} = 37.6$ Hz and $^2J(^{13}C,^{13}C)_{cis} = 0.5$ Hz, so that in this case the expectation of $^2J_{trans} > {}^2J_{cis}$ is confirmed. Consequently, we assume that the amine nitrogen is not complexed. For both **11** and and **12** the aqueous solutions used for the measurements showed no signs of decomposition over several hours suggesting only a relatively slow reductive elimination possibility. We note that Nast et al. [25] have prepared $(PPh_4)_2[Pd(CN)_3Ph]$.

The cyclopalladated phosphite, **3**, reacts with two equivalents of $^{13}CN^-$ to afford **13**, in which the phosphite ligand remains bound. In **13** there are two $^{13}CN^-$ resonances in a 1 : 1 ratio at $\delta = 141.8$ and 135.7 with $^2J(^{31}P,^{13}C)$ values of 199 and 21 Hz, respectively, see Fig. 1, thereby allowing the low field signal to be assigned as $^{13}CN^-$ *trans* to phosphite. The $^2J(^{13}C,^{13}C)_{cis}$ value for the two CN^- ligands is 8.6 Hz. The cyclometallated carbon, C-2, is found at $\delta = 139.6$ with a $^2J(^{31}P,^{13}C)$ value of 4.6 Hz. Finally we note that the $^2J(^{13}C,^{13}C)_{cis}$ values in the $Fe(CN)_5(NO)^{2-}$ dianion and in the $Fe(CN)_5(NO_2)^{4-}$ tetraanion are 17.6 and 9 Hz, respectively [26].

A number of attempts to coordinate an additional CN^- to **13** were essentially fruitless (see Experimental section); however, in one experiment, involving 1 equivalent of **13** and 1 equivalent $K^{13}CN$ in $CDCl_3/CD_3OD$, 1 : 1, the ^{13}C resonance for the cyanide *cis* to phosphite remains visible, but with loss of ^{13}CN *cis*-coupling, whereas the ^{13}C resonance for cyanide *trans* to phosphite disappears—as does the ^{13}C signal for the uncoordinated $^{13}CN^-$ (ca. 164 ppm). It seems that the phosphite ligand labilizes the *trans* position sufficiently to induce a relatively selective CN^- exchange reaction. In the table and Experimental section we give addition supporting data for **7–10** and **11–13**.

We consider the difference in chemistry for **13** relative to **11** and **12** to be of interest in light of the following cyanation reactions:

(i) Both, **4a** and **5a** react with $(Bu^n_4N)CN$ and diphos, $Ph_2PCH_2CH_2PPh_2$, to afford reasonable yields of the *ortho* cyano compounds **14** and **15**, respectively.



(14) (15)

with the chemistry as shown:

Complex **4a** (or **5**) + 2 diphos + $(Bu^n_4N)CN \xrightarrow[CH_2Cl_2]{}$

+ **14** (or **15**) $Pd(diphos)_2 + Bu^n_4N(OAc)$ (or Bu^n_4NCl)

The presence of the Pd(O) complex $Pd(diphos)_2$ is confirmed by its ^{31}P resonance at $\delta = 32.6$. This type of reaction has been reported previously for azobenzene compounds [27].

(ii) The comparable phosphite chemistry starting from **3** gave no readily detectable nitrile compound (see Experimental section for further comment).

It is possible that the failure to isolate a phosphite analog of **14** or **15** is related to the ease with which CN^- coordinates and/or the rate of the presumed subsequent reductive elimination. The coordination is, as we have shown, not so facile for derivatives of **3** as for complexes of **4** and **5**. Moreover, since theoretical [28] and experimental work [29] have shown that the reductive elimination from square planar Pd^{II} is facilitated by ligand dissociation, the tendency for the phosphite to remain coordinated could well increase the activation energy for this process as well. In any case, the use of various NMR probes to help in the elucidation of the palladium coordination sphere should prove increasingly valuable for the understanding of these and subsequent transformations involving such metallocycles.

Experimental

NMR spectra were recorded an Bruker WM-250 and AM-200 spectrometers. Details of the individual measurements are given in Table 1. IR spectra were recorded on a Perkin Elmer 883 spectrophotometer. Mass spectra and elemental analyses were carried out by the analytical laboratory of the ETH Zürich.

$Na^{15}NO_2$ and $K^{13}CN$ were purchased from Stohler Isotope Chemicals. Complexes **3**–**5** were prepared by published methods [8–10], as were **7** [7] and **9** and **10** [9]. Complexes **9** and **10** were prepared in 95 and 90% yields, respectively, from their bridging chlorides (see Table 1 for analytical data).

Preparation of 11. The cyclopalladated nitrosoamine **4a** (30.6 mg, 0.05 mmol) and $K^{13}CN$ (198.8 mg, 0.30 mmol—19.5 mg for the normal KCN reaction) were stirred in 0.5 ml of D_2O. After 30 min the clear solution formed was used for the NMR studies. The IR data were obtained by removal of the D_2O and examination of the resulting solid as a KBr pellet.

Preparation of 12. The cyclopalladated benzylamine **5** (27.6 mg, 0.05 mmol) and $K^{13}CN$ (19.8 mg, 0.30 mmol—for the normal KCN reaction 19.5 mg) were stirred in 0.5 ml D_2O. After 30 min the clear solution which resulted was used for the NMR

Table 1. Selected spectroscopic data [a] for **9–15**

	9 [b]		**10** [c]
IR, Pd-Cl	247, 291		
^{31}P	117.2		84.3, $^{1}J(Pt,P) = 6815$
	^{1}H	^{13}C	^{1}H
N–CH_3	3.60	30.7	3.58 [2.5]
1		114.2 (3.2)	
2		140.3 (1)	
3	7.67 (6.6)	137.8 (9.6)	7.76 (1.8) [63.8]
4	7.07	126.6 (6.1)	7.09
5	7.26	126.3	7.26
6	7.02	114.0	7.00
$P(OMe)_3$	3.80	53.7 (1.7)	3.86 (12.8) [3.0]

	11 [e]		**12** [f]		**13** [g]
IR [d]	2135, 2116		2110, 2130		2128
	^{1}H	^{13}C	^{1}H	^{13}C	^{13}C
N–CH_3	3.74	38.2	2.34	47.3	
CH_2			3.87	70.7	
1		151.1		146.2	159.0 (2.7)
2		149.2		154.8	139.6 (4.5)
3	7.66	142.6	7.42	140.5	142.8 (9)
4		130.5		131.5	122.8
5		127.5		128.7	126.9
6		126.4		126.3	110.8 (20)
*C*N		146.6, 144.6		149.4 t, 146.7, d	141.8 $J(P,C) = 199$ 135.7 $J(P,C) = 21$

	14 [h]		**15**	
IR: ν(C≡N)	2226		2228	
ν(N=O)			1460	
ν(N–N)	1081			
	^{1}H	^{13}C	^{1}H	^{13}C
1		144.5		143.2
2		107.4		112.8
3	[i]	134.0 [j]	7.63	132.8 [j]
4		128.6	7.32	127.6 [j]
5		134.4 [j]	7.55	133.0 [j]
6		124.4	7.55	130.3 [j]
CH_2			3.62	62.0
N–CH_3	353 [d]	34.4	2.28	45.6
C≡N		116.4		117.8
MS: M^+	161. (5.7) [k]		160 (10.5) [k]	

[a] δ values in ppm, J values in Hz. Values in parenthesis are couplings to ^{31}P, in square brackets, $J(Pt,H)$, $CDCl_3$. [b] $C_{10}H_{16}ClN_2O_4Pd$ Calcd.: C, 30.13; H, 4.11; N, 6.88; Cl, 8.69. Found: C, 29.93; H, 3.99; N, 6.98; Cl, 8.84. There are ^{15}N couplings to: NCH_3, 1.75; NCH_3, 1.0; C-1, 6.1; C-2, 0.5; C-5, 0.6; C-6, 2.8. [c] $C_{10}H_{16}ClN_2O_4Pt$ Calcd: C, 24.26; H, 3.09; N, 5.60; Cl, 7.40. Found: C, 24.51; H, 3.26; N, 5.72; Cl, 7.24. $^{3}J(^{15}N,CH_3) = 1.6$. [d] See ref. 25 for IR of $[Pd(Cn)_3(Ph)]^-$. Numbers in parentheses are couplings to ^{31}P. [e] D_2O solution, $^{2}J(^{13}C,^{13}C)$ for cyanides = 7.3 Hz, $^{2}J(^{13}C,^{13}C)_{trans} = 42$ Hz. H-4–H-6 not resolved. [f] D_2O solution, ^{1}H resonances of H-4 through H-6 not resolved. $^{2}J(^{13}C,^{13}C)$ *trans* and *cis* = 37.6 and 0.5 Hz, respectively. [g] $CDCl_3$ solution, $\delta^{31}P = 142.4$ (CD_3OD), 141.1 ($CDcl_3$) as crown-ether salt. 13 ^{1}H resonances between 6.55 and 7.40, δ H-3 = 8.08, see text for $^{2}J(^{13}C,^{13}C)$ values. [h] There are two isomers of **14** in the ratio 97/3 with NO *cis* to CH_3 as the major component. ^{1}H–NCH_3 for minor isomer = 4.21 ppm. [i] 7.56, m, 2H; 7.80, m, 2H. [j] Tentative assignment. [k] Values in parentheses are relative percentages.

studies. The IR data were obtained by removal of the D_2O and examination of the resulting solid as a KBr pellet.

Preparation of 13. (a) The cyclopalladated phosphite **3** (36 mg, 0.04 mmol), KCN (10.4 mg, 0.16 mmol) and 42.6 mg 18-crown-6 (to solubilize the KCN) were stirred in 2 ml CD_2Cl_2 for 1 h and the resulting clear solution used for the NMR studies. The conversion into **13** is essentially quantitative. (b) Complex **3** (36 mg, 0.04 mmol) and KCN (10.4 mg, 0.16 mmol) were dissolved in 1 ml CD_2Cl_2/CD_3OD, 1 : 1, and the resulting solution used for NMR studies. The conversion was again quantitative. Removal of the solvent gave a solid from which the IR data were obtained.

Various attempts to coordinate an additional CN^- ligand using **13** and CN^- in a 1 : 1 ratio gave the following results:

	CN^- source	Solvent	NMR probe	Result
(i)	$(Bu^n_4N)CN$	$CDCl_3$	^{31}P	only **13** ($\delta = 142.4$) observed
(ii)	K^+CN^- (as 18-crown ether salt)	$CDCl_3$	^{31}P	only **13** ($\delta = 141.1$) observed
(iii)	K^+CN^- (as 18-crown ether salt)	$CDCl_3$	^{13}C	uncoordinated ^{13}CN ($\delta =$ 164.1) observed
(iv)	KCN	$CDCl_3/CD_3OD$	^{31}P	only **13** initially observed, slow (hours) alcoholysis
(v)	$K^{13}CN$	$CDCl_3/CD_3OD$	^{13}C	cyanide exchange, see text.

2-(N-methyl-N-nitrosoamino)benzonitrile, 14. A solution of complex **4a** (120 mg, 0.20 mmol) in ca. 5 ml of CH_2Cl_2 was treated with solid diphos (320 mg, 0.80 mmol). Stirring for 1 h was followed by addition of solid $(Bu^n_4N)CN$ (108 mg, 0.40 mmol). The mixture was stirred for 3 days at room temperature, the solvent then removed, and the residue repeatedly extracted with 10 ml portions of hexane. The hexane extracts were combined and concentrated under vacuum to give 35 mg (54%) of crude product. An identical procedure starting from 0.20 mmol of **5** afforded 46 mg (72%) of product **15**.

An analogous sequence starting from 180 mg of **3** gave no significant amount of hexane-soluble material. The ^{31}P NMR spectrum of the non-hexane soluble fraction showed at least six different components. On the assumption that the phosphite might be coordinated to Pd^0, basic hydrolysis was attempted, but work-up of the organic products gave no evidence for 2-hydroxybenzonitrile.

N-methyl-N(15N-nitroso)aniline. To a solution of *N*-methylaniline (4.3 g, 40 mmol) in 6 ml of concentrated HCl with 16 g of ice at 0 °C was slowly added a solution of $Na^{15}NO_2$ (2.9 g, 40 mmol) in 10 ml of H_2O. Stirring for one hour was followed by extraction of the oily layer with 2 × 50 ml of ether. The ethereal extract was dried (Na_2SO_4) and the solvent removed under vacuum. Fractional distillation of the residue gave 5.2 g (95%) of product.

Acknowledgements

We thank the Swiss National Science Foundation and the ETH, Zürich, for support and the Johnson–Matthey Research Centre, England, for the loan of platinum metals.

References

1 J. Dehand and M. Pfeffer, Coord. Chem. Rev., 18 (1976) 327; G.R. Newcome, W.E. Puckett, V.K. Guptas and G.E. Kiefer, Chem. Rev., 86 (1986) 451.
2 R.F. Heck and J.M. Thompson, J. Organomet. Chem., 40 (1975) 2667; H. Horino and N. Inoue, Tetrahedron Lett., 26 (1979) 2403; idem, J. Organomet. Chem., 46 (1981) 4416; S. Murahashi, Y. Tomba, M. Yamamura and N. Yoshimura, ibid., 43 (1978) 4099; D.R. Fahey, ibid., 27 (1971) 283.
3 (a) P.S. Pregosin and R. Rüedi, J. Organomet. Chem., 273 (1984) 401, (b) A. Albinati, P.S. Pregosin and R. Rüedi, Helv. Chim. Acta, 68 (1985) 2046.
4 K. Hiraki, Y. Fuchita and K. Takechi, Inorg. Chem., 20 (1981) 4316; R.M. Ceder and J. Sales, J. Organomet. Chem., 294 (1985) 389, J. Granell, J. Sales and J. Vilarrasa, Transition Met. Chem., 9 (1984) 203; Y. Yamamoto and H. Yamazaki, Inorg. Chim. Acta, 41 (1980) 229; H. Alper, J. Organomet. Chem., 61 (1973) C62.
5 J. Dupont, M. Pfeffer, M.A. Rotteveel, A. DeClan and J. Fischer, Organometallics, 8 (1989) 116; J. Dupont, M. Pfeffer, J.C. Daran and J. Gouteron, J. Chem. Soc., Dalton Trans., (1988) 2421; F. Maasarani, M. Pfeffer and G. de Borgne, Organometallics, 6 (1987) 2029, 2043, and references therein.
6 S. Trofimenko, Inorg. Chem., 12 (1973) 1215.
7 B.N. Cockburn, D.V. Howe, T. Keating, B.F.G. Johnson and J. Lewois, J. Chem. Soc., Dalton Trans., (1973) 404.
8 A. Albinati, S. Affolter and P.S. Pregosin, Organometallics, 9 (1990) 379.
9 A. Albinati, S. Affolter and P.S. Pregosin, J. Organomet. Chem., 395 (1990) 231.
10 For reactions of this ligand see refs. 2, 4 and A. Bahsoun, J. Dehand, M. Pfeffer, M. Zinsius, S.E. Bouaoud and G. Le Borgne, J. Chem. Soc., Dalton Trans., (1979) 547; M. Pfeffer, J. Fischer and A. Mitschler, Organometallics, 3 (1984) 1531; A. Ryabov, Inorg. Chem., 26 (1987) 1252; A. Ryabov and A.K. Yatsimirsky, Inorg. Chem., 23 (1984) 789; A.D. Ryabov, I.K. Sakodinskaya and A.K. Yatsimirsky, J. Chem. Soc., Perkin Trans., II, (1983) 1511.
11 A.G. Constable, W.S. McDonald and B.L. Shaw, J. Chem. Soc., Dalton Trans., (1980) 2282.
12 P.S. Pregosin and E. Steiner, Helv. Chim. Acta, 59 (1976) 376.
13 K. Gehrig, M. Hugentobler, A.J. Klaus and P. Rys, Inorg. Chem., 21 (1982) 2493.
14 P.S. Pregosin, R. Rüedi and C. Anklin, Magn. Reson. Chem., 24 (1986) 255; H. Motschi and P.S. Pregosin, Inorg. Chim. Acta, 40 (1980) 141; H. van der Poel, G. van Koten, D.M. Grove, P.S. Pregosin and K.H.A. Ostoja and Starzewski, Helv. Chim. Acta, 64 (1981) 1174.
15 P.S. Pregosin, H. Streit and L.M. Venanzi, Inorg. Chim. Acta, 40 (1980) 141.
16 R.E. Bott, B.W.S. Kolthammer, P. Legzdins and J.D. Roberts, Inorg. Chem., 18 (1979) 2049.
17 R.E. Stevens and W.L. Gladfelter, Inorg. Chem., 22 (1983) 2034.
18 J. Bultitute, L.F. Larkworthy, J. Mason, D.C. Povey and B. Sandell, Inorg. Chem., 23 (1984) 3629; P.A. Duffin, L.F. Larkworthy, J. Mason, A.N. Stephens and R.M. Thompson, ibid., 26 (1987) 2034; L.K. Bell, J. Mason, D.M.P. Mingos and D.G. Tew, Inorg. Chem., 22 (1983) 3497.
19 B.L. Haymore, M. Hughes, J. Mason and R.L. Richards, J. Chem. Soc., Dalton Trans., (1988) 2935.
20 J. Mason, Chem. Br., (1983) 654.
21 H. Motschi, P.S. Pregosin and L.M. Venanzi, Helv. Chim. Acta, 62 (1973) 667.
22 A.J. Corty and S.E. Jacobson, J. Chem. Soc., Chem. Commun., (1975) 175.
23 P.F. Kelly and J.D. Woollins, J. Chem. Soc., Dalton Trans., (1988) 1053.
24 P.S. Pregosin and R.W. Kunz, NMR Basic Principles and Progress, Springer–Verlag, Berlin 1989, Vol. 16, p. 28.
25 R. Nast, J. Bülck and R. Kramolowsky, Chem. Ber., 108 (1975) 3461.
26 A.R. Butler, C. Glidewell, A.R. Hyde and J. McGinnis, Inorg. Chem., 24 (1985) 2931.
27 K. Gehrig, A.J. Klaus and P. Rys, Helv. Chim. Acta, 66 (1983) 2603.
28 S. Koniya, T.A. Albright, R. Hoffmann and J.K. Kochi, J. Am. Chem. Soc., 98 (1978) 7255; K. Tatsumi, R. Hoffmann, A. Yamamoto, J.K. Stille, Bull. Chem. Soc. Jpn., 54 (1981) 1857.
29 A. Yamamoto, T. Yamamoto, S. Koniya and F. Ozawa, Pure Appl. Chem., 56 (1984) 1621.