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LIGATING PROPERTIES OF THIONITROSOAMINES

I. NEUTRAL MONONUCLEAR *N*-THIONITROSODIMETHYLAMINE-PALLADIUM(II) AND -PLATINUM(II) COMPLEXES

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Summary

Me_2NNS (Me = methyl) reacts with $Pd(diene)Cl_2$ (diene = 1,5-cyclooctadiene, norbornadiene, 1,3,5,7-cyclooctatetraene, dicyclopentadiene) to give $Pd(Me_2NNS)_2Cl_2$ and with M(1,5-cyclooctadiene)Cl_2 ($M = Pd^{II}$ or Pt^{II}) in the presence of PR_3 ($PR_3 = PPh_3$, *p*-tolylPPh_2) to give $M(Me_2NNS)(PR_3)Cl_2$. The course of the reactions is, probably, controlled by the chelate effect of the diene. $Pd(Me_2NNS)_2Cl_2$ reacts with L ($L = PPh_3$, *p*-tolylPPh_2, P(*o*-tolyl)$_3$, $AsPh_3$, $SbPh_3$) to form $Pd(Me_2NNS)LCl_2$. All the complexes show *cis*-geometry in the solid state. Me_2NNS brings about bridge-splitting reactions with $[Pd(MeO\text{-}diene)Cl]_2$ (diene = 1,5-cyclooctadiene, 1,3,5,7-cyclooctatetraene, dicyclopentadiene) to form Pd(MeO-diene)(Me_2NNS)Cl. Metathetical reactions with KSCN or KSeCN have been used to prepare $Pd(Me_2NNS)_2(XCN)_2$ (X = S or Se) and $M(Me_2NNS)(PPh_3)(SCN)_2$ ($M = Pd^{II}$ or Pt^{II}). In all the Me_2NNS complexes the unstable *N*-thionitrosodimethylamine is *S*-bonded to the metal and stabilized by this coordination.

Introduction

In recent years there has been considerable interest in the transition metal complexes containing N–S ligands [1–3]. Monodentate N–S ligands are, generally, coordinated to the transition metal via the nitrogen atom [4–6].

Thionitrosoamines are unstable organic species which contain the N=S group [7]. They may, in principle, coordinate to the metal through either the S or N atom or by η^2-N=S coordination. The synthesis of the stable $(Me_2NNS)Cr(CO)_5$ complex (Me = methyl) was recently reported and a crystal structure determination has shown that in this complex the *N*-thionitrosodimethylamine ligand is *S*-bonded to the metal [8].

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In this paper we describe the first stage of a systematic investigation on the influence of the nature of the thionitrosoamine ligands and of the metal in determining the bonding mode of these ligands and the stability of their complexes. One of the main stimulants for such a study was the observation of two resonance forms contribute to the overall electronic structure of the free thionitrosoamine ligands [7].

The study was concerned with the reactions of *N*-thionitrosodimethylamine ligand with the complexes M(diene)Cl_2 (M = Pd^{II} or Pt^{II}; diene = 1,5-cyclooctadiene, norbornadiene, 1,3,5,7-cyclooctatetraene, dicyclopentadiene). These reactions gave novel thionitrosoamine complexes of palladium(II) and platinum(II) in which the ligand is *S*-bonded.

Results and discussion

The complexes Pd(diene)Cl_2 (diene = 1,5-cyclooctadiene, norbornadiene, 1,3,5,7-cyclooctatetraene, dicyclopentadiene) react with *N*-thionitrosodimethylamine to give an orange compound which, on the basis of the analytical data, was formulated as $Pd(Me_2NNS)_2Cl_2$ (I). This compound is stable for some weeks in the solid state decomposes rapidly in dimethylsulfoxide solution. The very low solubility in all the common organic solvents prevented determination of satisfactory ^1H NMR spectra. In the infrared spectrum the bands associated with the *N*-thionitrosodimethylamine ligand are observed at 1492s, 1375s, 1250w, 1132vs, 1028w, 860m, 772vs, 720w, 535m and 415w cm^{-1} and 1132vs is assigned to ν(N–N), 772vs to ν(N–S), suggesting that the coordination to the metal occurs through the sulfur atom. The *S*-coordination of *N*-thionitrosodimethylamine was confirmed by the X-ray structure determination undertaken on the *cis*-$Pd(Me_2NNS)(AsPh_3)Cl_2$ (VII). The results of this X-ray analysis and the synthesis of the other Pd^{II} and Pt^{II} complexes will be described in a future paper.

The far-infrared spectra of I shows two strong bands at 315 and 295 cm^{-1} attributable to terminal ν(Pd–Cl) suggesting a *cis*-configuration [9]. These assignments were confirmed by the IR spectra of the complexes $Pd(Me_2NNS)_2(SCN)_2$ (XII) and $Pd(Me_2NNS)_2(SeCN)_2$(XIII) obtained by metathetical reaction of I with KSCN or KSeCN. The *cis*-configuration of I and of the various $Pd(Me_2NNS)LCl$ (L = PPh_3, *p*-tolylPPh_2, P(*o*-tolyl)$_3$, $AsPh_3$, $SbPh_3$) described is noteworthy, since the *trans*-configuration is usual for PdL_2X_2 complexes in which L is a neutral ligand and X an anionic ligand [10,11], and there are only a few reports of *cis*-PdL_2X_2 complexes [12–16]. It is now accepted that steric requirements are very important in determining the solid state geometry of the complexes PdL_2X_2 and that small ligands are required for the formation of *cis*-complexes [17,18]. The effect of the overcrowding shown by the crystal structure determinations on the *cis*-PdL_2Cl_2 (L = Me_2PPh, P{n-C_3H_7}$_3$) supports these considerations [19,20]. If L is small, electronic factors become important and π-acceptor ligands tend to favour *cis*-geometry in the solid state [18,21]. The formation of the complexes *cis*-$Pd(Me_2NNS)_2Cl_2$ and *cis*-$Pd(Me_2NNS)LCl_2$ can be associated with the sterically undemanding nature ot the *N*-thionitrosodimethylamine ligand and by its electronic structure, which may promote back donation from the metal.

The reactions of M(diene)Cl_2 complexes with the *N*-thionitrosoamine ligand depend on the nature of the diene and of the metal. Thus $Pd(C_{10}H_{12})Cl_2$ reacted with Me_2NNS, molar ratio 1/2, almost immediately at ca. −10°C, whereas the

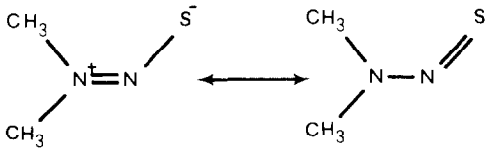
reactions of $Pd(C_7H_8)Cl_2$ or $Pd(1,3,5,7\text{-}C_8H_8)Cl_2$ with Me_2NNS required about 2 h at room temperature, the reaction of $Pd(1,5\text{-}C_8H_{12})Cl_2$ was even slower, only 20% of the diene complex being transformed into I in 2 h. Under these conditions the $Pt(diene)Cl_2$ complexes (diene = 1,5-cyclooctadiene, norbornadiene, 1,3,5,7-cyclooctatetraene, dicyclopentadiene) did not react and, the starting complexes were recovered unchanged after 24 h. (Decomposition of Me_2NNS prevented use of longer times of reaction or more drastic conditions.)

Kinetic studies of displacement of dienes from transition metal complexes have shown that the mechanism involves opening of the chelate ring followed by competition between ring closure and displacement of the diene [22,23]. As recently reported, this mechanism also operates when $Pt(1,5\text{-cyclooctadiene})Cl_2$ reacts with CO and tertiary phosphines (PR_3) to give *cis*-$Pt(PR_3)(CO)Cl_2$ [24]. This process is constrained by the chelate effect, the influence of which depends on the nature of the diene and of the substrate [23–26]. The good agreement between these data and the order of reactivity found in the reactions of $M(diene)Cl_2$ with Me_2NNS ($M = Pd^{II}$ or Pt^{II}), considered along with the observation that catalytic amounts of $AgPF_6$ do not increase the rates of this type of reaction [24], suggests that such a mechanism may operate in the reactions we have studied.

It is also known that when two different nucleophiles are present, the displacement of the diene involves, an initial opening of the chelate ring by the more powerful nucleophile; after the chelate effect has been removed, the displacement by a poor nucleophile can be a very rapid proccess [24]. This result led us to attempt the syntheses of the mixed ligand complexes $M(Me_2NNS)LCl_2$ where $M = Pd^{II}$ or Pt^{II} and L is a Group VB ligand. The reactions of $M(1,5\text{-}C_8H_{12})Cl_2$, at room temperature with equimolar amounts of tertiary phosphines in the presence of an excess of Me_2NNS led rapidly and in high yields to the $M(Me_2NNS)(PR_3)Cl_2$ ($M = Pd^{II}$; $PR_3 = PPh_3$ (II), *p*-tolylPPh$_2$ (III); $M = Pt^{II}$; $PR_3 = PPh_3$ (IV), *p*-tolylPPh$_2$(V)). Attempts to prepare the $M(Me_2NNS)(ER_3)Cl_2$ complexes where M is Pd^{II} or Pt^{II} and ER_3 is a bulky ligand as PCy_3 (Cy = cyclohexyl) or P(*o*-tolyl)$_3$ or $AsPh_3$ by this route failed, a mixture of products being obtained. From the reactions between $Pd(1,5\text{-}C_8H_{12})Cl_2$ and ER_3, in the presence of an excess of Me_2NNS we obtained I, *trans*-$Pd(ER_3)Cl_2$ and unidentified products while $Pt(1,5\text{-}C_8H_{12})Cl_2$ gave a mixture of unidentified Pt-phosphine complexes. The palladium complexes were also obtained when an acetone suspension of *cis*-$Pd(Me_2NNS)_2Cl_2$ along with an equimolar amount of the appropriate ligand was kept at room temperature overnight this method also gave the complexes $Pd(Me_2NNS)(ER_3)Cl_2$ (ER_3 = P(*o*-tolyl)$_3$ (VI), $AsPh_3$ (VII), $SbPh_3$ (VIII)).

All the mixed-ligand complexes prepared are yellow or orange solids, stable for some weeks in the solid state and almost insoluble in common organic solvents, although the complexes $M(Me_2NNS)$(*p*-tolylPPh$_2$)Cl_2 ($M = Pd^{II}$ (III); $M = Pt^{II}$ (V)) are slightly soluble in chlorinated solvents. All the complexes show the same pattern of bands as I for the Me_2NNS moiety. The IR region between 270–325 cm^{-1} shows two strong bands assigned to the stretching M–Cl, suggesting *cis*-configurations for all the complexes, as confirmed by the X-ray structure analysis on complex VII. The ^1H NMR spectra of the complexes III and V show, in addition to the resonance of the phenyl groups, a singlet in the range 7.5–8τ and two singlets in the range 6.5–7τ (see Table 1). The first singlet is assigned to the methyl of the tolyl group and the other two to the methyl group of the Me_2NNS entity. The ^1H NMR

spectrum of free *N*-thionitrosodimethylamine shows that two resonance forms, the dipolar (1) and the covalent (2), contribute to the overall electronic structure [7]. The presence of two peaks for the methyl of *N*-thionitrosodimethylamine *S*-bonded to the metal indicates that when the ligand is *S*-coordinated there is still restriction of rotation about the N–N bond.



(1) (2)

We also examined the reactions of $[Pd(MeOdiene)Cl]_2$ with Me_2NNS. Bridge-splitting reactions occurred readily at room temperature in acetone to give the complexes $Pd(MeOdiene)(Me_2NNS)Cl$ (diene = 1,5-cyclooctadiene (IX), 1,3,5,7-cyclooctatetraene (X), dicyclopentadiene (XI)). These are orange solids stable for some days at room temperature and for some weeks at ca. −15°C. They are very soluble in acetone or chlorinated solvents, insoluble in diethyl ether or saturated hydrocarbons, and non-conducting in acetone. The low value of the ν(Pd–Cl) frequency (255–270 cm^{-1}) suggests a configuration in which the chlorine atom is *trans* to the Pd–C σ-bond [27]. The IR spectra also display a strong band at 1095 cm^{-1} due to ν(C–O) [28,29]. In the methoxycyclooctadiene derivative this band is split, to give two bands at 1062 and 1082 cm^{-1}. Since the ^1H NMR spectrum of this complex does not show the presence of isomers, this splitting may arise from a packing effect. The ^1H NMR spectra of complexes IX, X and XI show, the CH_3O resonance in the range 6.6–6.9τ and the CH_3 *N*-bonded, as two singlets, in the range 5.6–6.3τ.

Metathetical reactions with KSCN or KSeCN gave the complexes XII, XIII, XIV and XV. They are orange solids, stable for some weeks, and slightly soluble in acetone or chlorinated solvents. They show the ν(C–N) band at about 2100 cm^{-1}. The thiocyanate complexes show the δ(SCN) band at 430 cm^{-1}, and the selenocyanate complex shows the δ(SeCN) band at 375 cm^{-1} and the ν(C–Se) band at 510 cm^{-1}. On the basis of literature data [18,30,31], and bearing in mind that sterically small ligands favour *cis*-geometry and the presence of the Pd–SCN bond [18] we formulated these complexes as *cis*-$Pd(Me_2NNS)_2(SCN)_2$, *cis*-$Pd(Me_2NNS)_2(SeCN)_2$, *cis*-$Pd(Me_2NNS)(PPh_3)(SCN)_2$ and *cis*-$Pt(Me_2NNS)(PPh_3)(SCN)_2$.

Experimental

The following compounds were prepared by published methods: $Pd(1,5\text{-}C_8H_{12})Cl_2$ [32], $Pd(C_7H_8)Cl_2$ [33], $Pd(1,3,5,7\text{-}C_8H_8)Cl_2$ [34], $Pd(C_{10}H_{12})Cl_2$ [33], $[Pd(MeOC_8H_{12})Cl]_2$ [35], $[Pd(MeOC_8H_8)Cl]_2$ [28], $[Pd(MeOC_{10}H_{12})Cl]_2$ [29], $Pt(C_8H_{12})Cl_2$ [32], $Pt(C_7H_8)Cl_2$ [32], $Pt(1,3,5,7\text{-}C_8H_8)Cl_2$ [32], $Pt(C_{10}H_{12})Cl_2$ [32], $Pd(C_6H_5CN)_2Cl_2$ [36] and Me_2NNS [7]. Other reagents were commercial products, and used without purification.

Elemental analyses were carried out by the Microanalytical Laboratory of the Organic Chemistry Institute of Milan and by Malissa & Reuter Analytische Labora-

TABLE 1. ANALYTICAL AND CHARACTERISTIC IR AND ^{1}H NMR DATA

Complex		Found (calcd.) (%)				IR data (cm^{-1}) [a]		^{1}H NMR data(τ,ppm) [b]	
		C	H	N	S	ν(M–Cl)	ν(C–O)	δ(N–CH_3)	Others
(I)	*cis*-$Pd(Me_2NNS)_2Cl_2$	13.50	3.42	15.69	18.00	315s			
		(13.43)	(3.41)	(15.67)	(17.93)	295s			
(II)	*cis*-$Pd(Me_2NNS)(PPh_3)Cl_2$	45.35	3.96	5.25		325s			
		(45.34)	(3.99)	(5.29)		280s			
(III)	*cis*-$Pd(Me_2NNS)(p\text{-tolyl}PPh_2)Cl_2$	46.35	4.29	5.11		318s		6.72	7.65 ($C_6H_4CH_3$)
		(46.38)	(4.26)	(5.15)		282s		6.60	
(IV)	*cis*-$Pt(Me_2NNS)(PPh_3)Cl_2$	38.80	3.45	4.50		325s			
		(38.84)	(3.42)	(4.53)		285s			
(V)	*cis*-$Pt(Me_2NNS)(p\text{-tolyl}PPh_2)Cl_2$	39.90	3.70	4.50		322s		6.90	7.77 ($C_6H_4CH_3$)
		(39.88)	(3.66)	(4.42)		285s		6.75	
(VI)	*cis*-$Pd(Me_2NNS)(P(o\text{-tolyl})_3)Cl_2$	48.30	4.78	4.95		318s			
		(48.31)	(4.76)	(4.90)		285s			
(VII)	*cis*-$Pd(Me_2NNS)(AsPh_3)Cl_2$	41.90	3.75	4.85		323s			
		(41.87)	(3.69)	(4.88)		282s			
(VIII)	*cis*-$Pd(Me_2NNS)(SbPh_3)Cl_2$	38.60	3.41	4.50		318s			
		(38.71)	(3.41)	(4.51)		270s			
(IX)	$Pd(MeOC_8H_{12})(Me_2NNS)Cl$	35.60	5.75	7.58		270m	1082s	6.23	6.78 (O-CH_3)
		(35.59)	(5.70)	(7.54)			1062s	5.98	
(X)	$Pd(MeOC_8H_8)(Me_2NNS)Cl$	35.90	4.70	7.65		270m	1095s	6.29	6.64 (O-CH_3)
		(35.98)	(4.66)	(7.62)				5.98	
(XI)	$Pd(MeOC_{10}H_{12})(Me_2NNS)Cl$	39.55	5.30	7.00		255m	1095s	6.27	6.86 (O-CH_3)
		(39.50)	(5.35)	(7.08)				5.99	
(XII)	*cis*-$Pd(Me_2NNS)_2(SCN)_2$	17.90	3.05	20.85	31.80	2100s [c]	430m [d]	6.13 [e]	
		(17.88)	(3.00)	(20.86)	(31.83)	2090s [c]		5.88 [e]	
(XIII)	*cis*-$Pd(Me_2NNS)_2(SeCN)_2$	14.50	2.45	16.90		2095s [c]	375m [f]		
		(14.51)	(2.43)	(16.92)			510w [g]		
(XIV)	*cis*-$Pd(Me_2NNS)_2(PPh_3)(SCN)_2$	45.95	3.70	9.70		2105s [c]	430m [d]	6.25	
		(45.95)	(3.68)	(9.74)				6.00	
(XV)	*cis*-$Pt(Me_2NNS)_2(PPh_3)(SCN)_2$	39.80	3.20	8.40		2110s [c]	430m [d]		
		(39.81)	(3.19)	(8.44)		2100s [c]			

[a] Nujol mulls. [b] CD_3Cl solutions. [c] ν(C–N). [d] δ(SCN). [e] Acetone-d_6 solutions. [f] δ(SeCN). [g] ν(C–Se).

torien, Elbach, Germany. Conductivity measuraments were carried out with a WTW LBR conductivity meter. Infrared spectra were recorded on a Perkin–Elmer 577 spectrophotometer using CsI plates. Proton NMR spectra were recorded on a Perkin–Elmer R 24B spectrometer with tetramethylsilane as internal standard.

Analytical and characteristic IR and ^1H NMR data are reported in Table 1.

Preparation of cis-Pd(Me_2NNS)$_2Cl_2$ (I)

N-Thionitrosodimethylamine (225 mg, 2.5 mmol) was added to a solution of $Pd(C_{10}H_{12})Cl_2$ (309.5 mg, 1 mmol) in acetone or dichloromethane (ca. 50 cm^3). An orange precipitate was formed immediately, and after 5 min was filtered off, washed with acetone and diethyl ether, and dried (Yield 80%). The reactions of Me_2NNS with $Pd(C_7H_8)Cl_2$, $Pd(1,3,5,7\text{-}C_8H_8)Cl_2$, or $Pd(1,5\text{-}C_8H_{12})Cl_2$ proceeded similarly, but longer reaction times were required (see Discussion).

Preparation of the complexes M(Me_2NNS)(PR_3)Cl_2 (II–V)

To a stirred solution of $M(1,5\text{-}C_8H_{12})Cl_2$ (M = Pd or Pt) (1 mmol) in acetone or dichloromethane (50 cm^3) 270 mg; (3 mmol) of Me_2NNS in rapid succession, and 1 mmol of PR_3 (PR_3 = PPh_3; *p*-tolylPPh_2) were added. The yellow precipitate was filtered off, washed with acetone and diethyl ether and dried (Yield ca. 95%).

Preparation of the complexes Pd(Me_2NNS)LCl_2 (II, III, VI–VIII)

To a suspension of *cis*-Pd$(Me_2NNS)_2Cl_2$ (357 mg, 1 mmol) in acetone (80 cm^3) was added 1 mmol of L (L = PPh_3, *p*-tolylPPh_2, P(*o*-tolyl)$_3$, $AsPh_3$, $SbPh_3$). The mixture was stirred overnight then the yellow (II, III, VI) or orange (VII, VIII) product was filtered off, washed several times with acetone and diethyl ether, and dried (Yield ca. 70%).

Preparation of the complexes Pd(MeOdiene)(Me_2NNS)Cl (IX–XI)

To a filtered solution of $[Pd(MeOdiene)Cl]_2$ (0.5 mmol) (diene = 1,5-cyclooctadiene,1,3,5,7-cyclooctatetraene, dicyclopentadiene) in dichloromethane (ca. 50 cm^3) was added Me_2NNS (100 mg, 1.1 mmol). After a few minutes stirring, the solution was filtered and concentrated to ca. 10 cm^3. Addition of n-hexane gave a yellow-orange precipitate, which was filtered off, washed with diethyl ether, and dried (Yield ca. 80%).

Preparation of the complexes Pd(Me_2NNS)$_2$(XCN)$_2$ (XII, XIII)

The KXCN (X = S or Se) (2.1 mmol) was added to a suspension of *cis*-Pd$(Me_2NNS)_2Cl_2$ (357 mg, 1 mmol) in acetone (100 cm^3) and the mixture was stirred for 6 h. The orange solution was filtered and concentrated (10 cm^3) under reduced pressure. Addition of diethyl ether gave an orange precipitate, which was filtered off and washed with diethyl ether. The product was recrystallized from acetone/diethyl ether (Yield ca. 70%).

Preparation of the complexes M(Me_2NNS)(PPh_3)Cl_2 (XIV, XV)

The KSCN (102 mg, 1.05 mmol) was added to a suspension of *cis*-M$(Me_2NNS)(PPh_3)$ Cl_2 (M = Pd or Pt) (0.5 mmol) in acetone (100 cm^3) and the mixture was stirred for 6 h. The orange solution was filtered and evaporated to 20 cm^3 under reduced pressure. Addition of diethyl ether gave an orange precipitate. The product was recrystallized from chloroform/diethyl ether (Yield ca. 70%).

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