Figure 3. Electronic and circular dichroism spectra of Cu(hmc-L-hisOCH$_3$)OAc (—), Cu(hmc-D-hisOCH$_3$)OAc (- - -), and Cu-(hmchimNCH$_3$)OAc (⋯) in chloroform solution.

The contribution of the chiral centers of the camphor moiety to the optical activity of the complexes is of only minor entity and can be evaluated by the low-intensity Cotton effects exhibited by the copper(II) complexes of the ligands II, i.e., Cu(hmchim)[14] and Cu(hmchimNCH$_3$)OAc. The CD spectra of these latter complexes display bands of the same sign pattern as Cu(hmc-L-hisOCH$_3$)OAc and Cu(hmc-D-his) (Figure 3). Therefore, the camphor chiral centers induce a chirality of sign δ in the six-membered, N–N bonded, chelate ring of the histamine derivatives. This accounts for the lower amplitude of the CD bands in Cu(hmc-D-hisOCH$_3$)OAc than in Cu-(hmc-L-hisOCH$_3$)OAc. It can be noted that chirality of the same sign (δ) is induced by the camphor chiral centers also in the five-membered, N–O bonded, amino acid chelate ring of the Cu(hmcaa) complexes, as it has been found in the CD spectrum of Cu(hmcgly).[10]

Experimental Section

Elemental analyses were from the microanalytical laboratory of the University of Milano. Electronic and circular dichroism spectra were recorded on Beckman DK-2A and on Jobin-Yvonne Mark III instruments, respectively. Infrared spectra were recorded on a Perkin-Elmer 621 spectrophotometer. EPR spectra were recorded on a Varian E-109 spectrometer operating at X-band frequencies.

Preparation of the Complexes. Cu(hmc-L-his), Cu(hmc-D-his). Equimolar amounts of (+)-(hydroxymethylene)camphor and L- or D-histidine (1 mmol) were refluxed in anhydrous methanol (50 mL) for about 2 h. Cupric acetate (1 mmol) was then added to the hot solution, and green precipitates of the complexes readily formed. The products were collected by filtration, washed with a small amount of methanol–water (1:1), and dried under vacuum.

Cu(hmc-L-his)·H$_2$O. Anal. Calcd for CuC$_{17}$H$_{23}$N$_3$O$_4$: C, 51.43; H, 5.84; N, 10.59. Found: C, 51.53; H, 5.99; N, 10.23.

Cu(hmc-D-his)·CH$_3$OH. Anal. Calcd for CuC$_{18}$H$_{25}$N$_3$O$_4$: C, 52.61; H, 6.13; N, 10.23. Found: C, 53.00; H, 6.05; N, 10.17.

The pyridine adduct of Cu(hmc-D-his) was obtained by slow evaporation of a pyridine solution of the complex at room temperature.

Cu(hmc-D-his)·py. Anal. Calcd for CuC$_{22}$H$_{26}$N$_4$O$_3$: C, 57.70; H, 5.68; N, 12.24. Found: C, 56.94; H, 5.27; N, 11.91.

Cu(hmc-L-hisOCH$_3$)OAc, Cu(hmc-D-hisOCH$_3$)OAc, Cu-(hmchimNCH$_3$)OAc. Equimolar amounts of (+)-(hydroxy-methylene)camphor and free L- or D-histidine methyl ester, or N^r-methylhistamine[15] (1 mmol) (obtained from the corresponding hydrochloride salts and sodium methoxide in absolute ethanol) in methanol (50 mL) were refluxed for about 2 h. Cupric acetate (1 mmol) was then added to the warm solution. The solution was concentrated to a small volume and chromatographed on a Sephadex LH-20 (methanol as eluant). A single main fraction was obtained in each case. This was collected and evaporated to dryness under vacuum.

Cu(hmc-L-hisOCH$_3$)OAc·2H$_2$O. Anal. Calcd for CuC$_{20}$H$_{31}$N$_3$O$_7$: C, 49.13; H, 6.34; N, 7.59. Found: C, 49.83; H, 6.30; N, 7.27.

Cu(hmc-D-hisOCH$_3$)OAc·2H$_2$O. Anal. Calcd for CuC$_{20}$H$_{31}$N$_3$O$_7$: C, 49.13; H, 6.34; N, 7.59. Found: C, 49.56; H, 6.46; N, 7.31.

Cu(hmchimNCH$_3$)OAc·2H$_2$O. Anal. Calcd for CuC$_{19}$H$_{31}$N$_3$O$_5$: C, 51.30; H, 6.97; N, 9.45. Found: C, 51.96; H, 6.40; N, 9.42.

Cu(hmchim). Equimolar amounts of (+)-(hydroxymethylene)-camphor and free histamine (obtained from the dihydrochloride salt and sodium methoxide in absolute ethanol) (1 mmol) in anhydrous methanol (50 mL) were refluxed for about 2 h. Upon addition of cupric acetate (1 mmol) a brown precipitate immediately formed. This was collected by filtration, washed with methanol, and dried under vacuum. Anal. Calcd for CuC$_{16}$H$_{22}$N$_3$O: C, 57.22; H, 6.56; N, 12.52. Found: C, 57.42; H, 6.30; N, 12.57.

Registry No. Cu(hmc-L-his), 75847-55-1; Cu(hmc-D-his), 75880-25-0; Cu(hmc-D-his)·py, 75847-56-2; Cu(hmc-L-hisOCH$_3$)OAc, 75847-57-3; Cu(hmc-D-hisOCH$_3$)OAc, 75880-26-1; Cu-(hmchimNCH$_3$)OAc, 75847-58-4; Cu(hmchim), 75847-59-5; (+)-(hydroxymethylene)camphor, 14681-31-3; D-histidine, 351-50-8; L-histidine, 71-00-1; L-histidine methyl ester, 1499-46-3; D-histidine methyl ester, 17720-12-6; N-methylhistamine, 501-75-7; histamine, 51-45-6.

(14) The complex containing a deprotonated imidazole, Cu(hmchim), was obtained as the derivative of histamine. This compound is probably polymeric and completely insoluble in chloroform. However its CD spectrum in pyridine is identical with that of Cu(hmchimNCH$_3$)OAc.

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Thionitrosyl-Bridged Rhodium Complexes: Replacement of NO with NS from Transition-Metal Nitrosyl Complexes

K. K. Pandey and U. C. Agarwala*

In recent years there has been considerable speculation about the synthetic and structural studies of metal–thionitrosyl complexes.[1-11] Current interest in the reactions of metal–

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Table I. Analytical Data of Complexes

compd	mp, °C	% C calcd	% C found	% H calcd	% H found	% N calcd	% N found	% S calcd	% S found	% Cl calcd	% Cl found	% P/As calcd	% P/As found	% Rh calcd	% Rh found	yield, %
[Rh(NS)Cl$_2$(PPh$_3$)]$_2$	220	44.7	44.8	3.3	3.1	3.0	2.9	6.8	6.6	14.9	14.7	6.4	6.4	21.1	21.3	25
[Rh(NS)Cl$_2$(AsPh$_3$)]$_2$	220	41.1	41.0	2.9	2.8	2.8	2.6	6.1	6.0	13.7	13.5	14.0	14.2	18.6	19.5	32
Rh(NS)Cl$_2$(PPh$_3$)$_2$	160	58.2	58.0	4.1	4.0	2.1	1.9	4.1	4.3	9.7	9.5	8.2	8.3	13.6	13.8	60
Rh(NS)Cl$_2$(PPh$_3$)·(AsPh$_3$)	165	54.7	54.8	4.0	3.8	1.8	1.8	4.0	4.1	9.2	9.2			13.0	13.1	62
Rh(NS)Cl$_2$(AsPh$_3$)$_2$	169	51.9	51.8	3.6	3.6	1.7	1.7	4.0	3.9	8.7	8.5	17.9	18.0	12.4	12.3	60

nitrosyl complexes has prompted us to undertake the reaction of platinum metal–nitrosyl complexes with trithiazyl trichloride which exists as a solvated monomer NSCl(solvent)$_x$ in THF or CCl$_4$–CHCl$_3$ (1:1) and has been used as a thionitrosylating agent.[1-4] Herein we wish to report a series of environmentally important reactions which suggest that in the complexes with low ν_{NO} (1500–1650 cm^{-1}). NO group is replaced by NS group and convert NO and NS into N$_2$ while no reaction is observed in the complexes with higher ν_{NO} (1650–1875 cm^{-1}).

Experimental Section

All the reagents used were AnalaR or of chemically pure grade. The solvents were dried and freshly distilled before use. Every reaction was carried out under pure, dry argon.

Nitrosyltris(triphenylphosphine)rhodium(I), dichloronitrosylbis-(triphenylarsine)rhodium(III), and trithiazyl trichloride were prepared by the literature methods.[12-14]

(a) Reaction of (NSCl)$_3$ with Rh(NO)(PPh$_3$)$_3$. A yellow solution of (NSCl)$_3$ in CCl$_4$ (15 mL) was added dropwise to a stirred solution of Rh(NO)(PPh$_3$)$_3$ (0.2 g) in CHCl$_3$ (20 mL) at room temperature. After the addition of (NSCl)$_3$ solution was complete, the mixture was stirred for 1 h. Solvent was evaporated under reduced pressure, and the residue was extracted with benzene. On adding petroleum ether to the extract, a brown compound was precipitated out which was centrifuged, washed with petroleum ether and water, and dried in air. It was recrystallized as brown crystals [Rh(NS)Cl$_2$(PPh$_3$)]$_2$ from toluene–dichloromethane (1:1). SPPh$_3$ and OPPh$_3$ were recovered from the washings and analyzed. Evolution of N$_2$ gas is observed by gas chromatography. Similar result was observed by using tetrahydrofuran in place of CCl$_4$–CHCl$_3$.

(b) Reaction of (NSCl)$_3$ with Rh(NO)Cl$_2$(AsPh$_3$)$_2$. A yellow solution of (NSCl)$_3$ in CCl$_4$ (15 mL) was added dropwise to a stirred solution of Rh(NO)Cl$_2$(AsPh$_3$)$_2$ (0.2 g) in CHCl$_3$ (20 mL) at room temperature. Red-brown crystals of [Rh(NS)Cl$_2$(AsPh$_3$)]$_2$ were isolated by a procedure similar to that given in part a.

(c) Reaction of [Rh(NS)Cl$_2$(PPh$_3$)]$_2$ with Triphenylphosphine. Triphenylphosphine (0.4 g) was added to a solution of [Rh(NS)-Cl$_2$(PPh$_3$)]$_2$ (0.2 g) in dichloromethane (20 mL). The resulting solution was refluxed for 2 h. On addition of diethyl ether to it, a red-brown complex Rh(NS)Cl$_2$(PPh$_3$)$_2$ was precipitated out which was centrifuged, washed with ether, and dried in vacuum. It was recrystallized from benzene–ether.

(d) Reaction of [Rh(NS)Cl$_2$(PPh$_3$)]$_2$ with Triphenylarsine. Triphenylarsine (0.5 g) was added to a solution of [Rh(NS)Cl$_2$(PPh$_3$)]$_2$ (0.2 g) in dichloromethane (20 mL). The resulting solution was refluxed for 4 h. Red-brown complex Rh(NS)Cl$_2$(PPh$_3$)(AsPh$_3$) was isolated by a procedure similar to that given in part c.

(e) Reaction of [Rh(NS)Cl$_2$(AsPh$_3$)]$_2$ with L (L = PPh$_3$, AsPh$_3$). L (PPh$_3$, AsPh$_3$) (0.5 g) was added to a solution of Rh(NS)Cl$_2$-(AsPh$_3$)$_2$ (0.2 g) in dichloromethane (20 mL). The resulting solution

was refluxed for 3 h. Red-brown complex [Rh(NS)Cl$_2$(AsPh$_3$)L] (L = PPh$_3$ or AsPh$_3$) was isolated by a procedure similar to that given in part c.
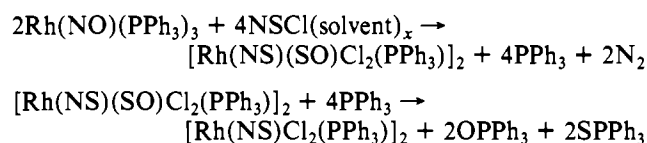
C, H, and N analyses were performed by the Microanalytical Section of the Indian Institute of Technology, Kanpur, India. Chloride, sulfur, phosphorus, and metal analyses were done by the standard methods.[15,16] The IR spectra of the samples were recorded with the Perkin-Elmer Model 580 spectrophotometer in the range 4000–4250 cm^{-1}. Samples were prepared as KBr pellets and CsI pellets. The magnetic measurements were made on a Gouy balance. Molecular weights were determined cryoscopically. Conductivity were measured by the Toshniwal conductometer. Analytical data of the complexes are given in Table I.

Results and Discussion

Reaction of (NSCl)$_3$ in THF or CCl$_4$–CHCl$_3$ with Rh-(NO)(PPh$_3$)$_3$ gave a novel complex [Rh(NS)Cl$_2$(PPh$_3$)]$_2$ in the form of brown shining crystals. Total product analysis revealed that in addition to complex [Rh(NS)Cl$_2$(PPh$_3$)]$_2$, the only other products of the reaction are triphenylphosphine oxide, triphenylphosphine sulfide and nitrogen gas. The reaction may therefore be represented as

$$2Rh(NO)(PPh_3)_3 + 4NSCl(solvent)_x \rightarrow$$
$$\underset{I}{[Rh(NS)Cl_2(PPh_3)]_2} + 2OPPh_3 + 2SPPh_3 + 2N_2$$
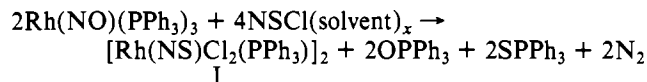
A plausible mechanism involves the prior formation of the six-coordinate intermediate [Rh(NS)(SO)Cl$_2$(PPh$_3$)]$_2$ (ν_{SO}, 1035 cm^{-1}) which reacts readily with triphenylphosphine to give complex I, SPPh$_3$, and OPPh$_3$.

$$2Rh(NO)(PPh_3)_3 + 4NSCl(solvent)_x \rightarrow$$
$$[Rh(NS)(SO)Cl_2(PPh_3)]_2 + 4PPh_3 + 2N_2$$

$$[Rh(NS)(SO)Cl_2(PPh_3)]_2 + 4PPh_3 \rightarrow$$
$$[Rh(NS)Cl_2(PPh_3)]_2 + 2OPPh_3 + 2SPPh_3$$

The formation of OPPh$_3$ and SPPh$_3$ in the reaction of sulfur monoxide complex [Ir(SO)$_2$(dppe)$_2$]Cl with triphenylphosphine have been reported by Schmid and Ritter.[17,18]

$$[Ir(SO)_2(dppe)_2]Cl + 4PPh_3 \rightarrow$$
$$[Ir(dppe)_2]Cl + 2OPPh_3 + 2SPPh_3$$
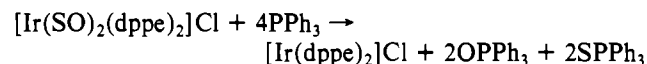
Similar reactions of (NSCl)(solvent)$_x$ with Rh(NO)(AsPh$_3$)$_3$, Rh(NO)Cl$_2$(PPh$_3$)$_2$, and Rh(NO)Cl$_2$(AsPh$_3$)$_2$ afforded thionitrosyl-bridged complexes [Rh(NS)Cl$_2$(MPh$_3$)]$_2$ (I, M = P; II, M = As).
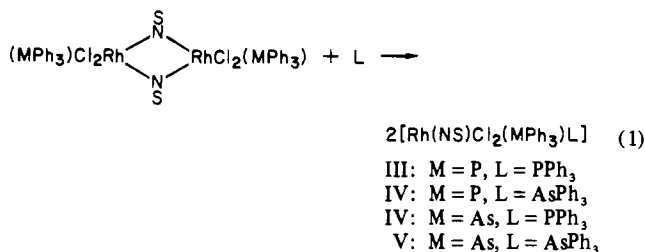
Both complexes I and II are air stable, nonelectrolytic in nitrobenzene, soluble in THF, benzene dichloromethane, and ethanol, and insoluble in hexane and ether are diamagnetic solids. The molecular weights for I and II were found to be 948 and 1040, respectively. The IR spectra of I and II showed a slightly broad band at 840 cm^{-1}. We attribute the band at 840 cm^{-1} to the bridging thionitrosyl groups. The absence of

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a band in the region 1610–1630 cm^{-1} due to ν_{NO} and the appearance of a new band at 840 cm^{-1} indicate the replacement of the nitrosyl group by NS. It may be noted that the difference between the 1620 and 840 cm^{-1} is of the same order as that between nonbridging and bridging NO groups. For the two bridging groups the NS frequencies will be very close together and may be superimposed.

In order to prove further the bridging nature of the NS group we have carried out reactions of I and II with PPh$_3$ and AsPh$_3$ which resulted in the bridge cleavage (eq 1). The IR

$$(MPh_3)Cl_2Rh \underset{\underset{S}{\overset{\|}{N}}}{\overset{\overset{S}{\overset{\|}{N}}}{\diamondsuit}} RhCl_2(MPh_3) \; + \; L \; \longrightarrow$$

$$2[Rh(NS)Cl_2(MPh_3)L] \quad (1)$$

III: M = P, L = PPh$_3$
IV: M = P, L = AsPh$_3$
IV: M = As, L = PPh$_3$
V: M = As, L = AsPh$_3$

spectra of these complexes show absorption band in the region 1116–1120 cm^{-1} which are in close agreement with the values reported previously for similar complexes. This supports that the band at 840 cm^{-1} in complexes I and II, which disappeared in the spectra of complexes III–V, is a characteristic band of bridging thionitrosyl groups. Similar results are observed in iridium nitrosyl complexes,[19] but in contrast to Rh and IR complexes, [M(NO)X$_3$(PPh$_3$)$_2$] (M = Ru, Os; X = Cl, Br) show no reaction with NSCl(solvent)$_x$. The reactions of NSCl(THF)$_x$ with the neutral complexes [(η^5-C$_5$H$_5$)M-(CO)$_2$(NO)] (M = Cr or Mo) in THF or CH$_2$Cl$_2$ afforded (η^5-C$_5$H$_5$)Cr(NO)$_2$Cl and [(η^5-C$_5$H$_5$)Mo(NO)Cl]$_2$.[8]

On the basis of these reactions, we conclude that the NO group is replaced by the NS group in metal–nitrosyl complexes having low frequencies (ν_{NO} = 1500–1650 cm^{-1}).

Registry No. I, 76136-88-4; II, 76136-89-5; III, 76136-90-8; IV, 76136-91-9; V, 76136-92-0; (NSCl)$_3$, 5964-00-1; Rh(NO)(PPh$_3$)$_3$, 21558-94-1; Rh(NO)Cl$_2$(AsPh$_3$)$_2$, 27411-16-1.

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Syntheses of Carbonyl Halides of Late Transition Elements in Thionyl Chloride as Solvent. Carbonyl Complexes of Palladium(II)

Fausto Calderazzo* and Daniela Belli Dell'Amico

Palladium has been extensively used as a catalyst of several reactions involving CO such as the snythesis of dialkyl oxalates,[1] alkyl isocyanates from nitro aromatics,[2] and esters from olefins and alcohols.[3] In spite of this, the chemistry of halo–carbonyl complexes of palladium is still far from a complete understanding. An earlier report by Manchot and König[4] described a compound of analytical composition PdCl$_2$(CO) obtained by carbonylation of PdCl$_2$ in methanol; this formulation was later questioned[5] and the product was

Table I. IR Carbonyl Stretching Vibrations of Carbonyl Complexes of Palladium(II) and Platinum(II)

| compd | $\tilde{\nu}_{CO}$, cm^{-1} | | | | |
	cyclo-hexane	C$_6$H$_6$	CH$_2$Cl$_2$	Nujol	ref
Pd$_2$Cl$_4$(CO)$_2$	2159	2162	2166	2167	this work
Pt$_2$Cl$_4$(CO)$_2$	2128	2129	2139	2146	this work
				2146	6
[PdCl$_3$(CO)]$^-$			2146	2141	this work
			2132		20
[PtCl$_3$(CO)]$^-$		2105	2109		this work
		(toluene)		2106	21

suggested to be [PdCl(CO)]$_n$ contaminated with PdCl$_2$. A carbonyl absorption at 1976 cm^{-1} was measured[6] for the product reported by Manchot and König, and a strong absorption at 1947 cm^{-1} was recently reported[7] for PdCl$_2$-(CO)(PhCN). However, these values are unusually low for terminal carbonyl groups of late transition elements and they are not in agreement with the values reported[6] for Pt$_2$Cl$_4$(CO)$_2$, for *cis*-PtCl$_2$(CO)$_2$ (2177 and 2136 cm^{-1} in thionyl chloride as solvent), and for some cationic carbonyl complexes of palladium(II), which were found[9] to absorb around 2140 cm^{-1}. On the basis of the surprisingly low values of ν_{CO} reported[6] by Irving and Magnusson, a stronger π back-donation was suggested for palladium(II) in comparison with platinum(II).[10] A proposal was also offered[11] that the palladium compound might be dimeric as its platinum analogue was suggested[6] to be, but with carbonyl bridges rather than chloride bridges, an unprecedented feature for unsubstituted halometal carbonyls.[12]

We have recently discovered a new method for preparing halo carbonyls of gold(I) and platinum(II).[8,13] We want now to report that application of this method to PdCl$_2$ did in fact lead to genuine palladium(II)–halo–carbonyl derivatives.

Experimental Section

PdCl$_2$ of various origins gave similar results.

Pd$_2$Cl$_4$(CO)$_2$ was prepared by treating PdCl$_2$ with CO at super atmospheric pressure in thionyl chloride. In a typical experiment, PdCl$_2$ (3.1 g, 17.48 mmol) was introduced in a Hastelloy C autoclave together with SOCl$_2$ (20 mL) and stirred at about 120 °C for 12 h with CO under pressure (initial P_{CO} = 50 atm). The yellow-orange solution was filtered under a CO atmosphere, and the filtrate was added to heptane pretreated with some drops of thionyl chloride. The yellow-orange microcrystalline precipitate was briefly dried in a stream of CO and kept in vials sealed under CO (2.26 g, 63% yield). The compound is soluble in dichloromethane, thionyl chloride, and benzene (about 1%) and slightly soluble in saturated hydrocarbons. Under strictly anhydrous conditions it is reasonably stable either in the solid state or in solution at room temperature: decomposition occurred immediately upon contact with trace amounts of water, finally yielding a black precipitate, presumably palladium metal, through the intermediacy of unidentified orange solids. Anal. Calcd for Pd$_2$Cl$_4$(CO)$_2$

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