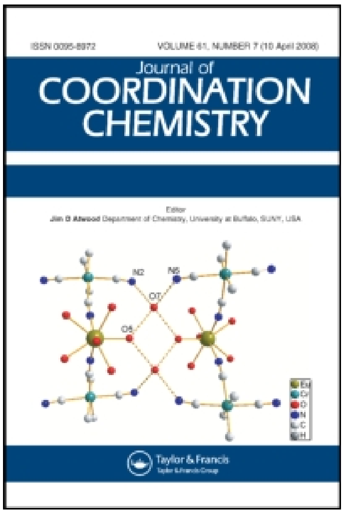

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Synthesis, spectroscopic investigation, crystal and molecular structure of $[ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2]$

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Synthesis, spectroscopic investigation, crystal and molecular structure of [$ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2$]

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[$ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2$] has been obtained in the reaction of benzoylhydrazido-Re(V) with an excess of nicotine. The [$ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2$] complex crystallizes in the monoclinic space group $P2_1/n$. The complex was characterized by infrared, Ultaviolet-visible, ^{1}H NMR and magnetic measurements.

Keywords: Rhenium; Organodiazenido; X-ray structure; Electronic structure

1. Introduction

Benzoylhydrazido-Re(V) [$ReCl_2(\eta^2\text{-}N_2COPh\text{-}N',O)(PPh_3)_2$] is a well-known starting material for the syntheses of a variety of Re complexes with significance in the fields of nitrogen fixation and for preparing nitrogen-containing organic compounds [1]. Organodiazenido ligands, as well as others which contain metal-nitrogen multiple bonds, have been of interest because of their amphoteric nature and close relationship to dinitrogen and nitrosyl ligands. Similar to nitrosyl, organodiazenido (–NNR) displays a variety of geometries: singly bent, doubly bent and bridging. Structural and synthetic studies of organodiazenido transition metal complexes have shown that the chemistry of these compounds is varied and interesting [2–4]. Diazenido Re complexes bearing *N,O*-ligands can catalyze peroxidate oxidation of cycloalkanes which occurs via intermediate peroxo and/or Re species [5, 6].

Widespread interest in the development of rhenium radiopharmaceuticals is a result of the ideal nuclear properties of $^{186/188}$Re isotopes (strong β-emission energies of 1070 keV and 2120 keV and long half-lives of 90 h and 17 h, respectively) which are

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suitable for target radiotherapy of cancers [7–10]. Here we present the synthesis, spectroscopic, and structural characterization of [$ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2$].

2. Experimental

2.1. *General procedure*

Ammonium perrhenate and triphenylphosphine were purchased from Aldrich Chemical and nicotine purchased from Fluka AG, and used without further purification. The [$ReCl_2(\eta^2$-N_2COPh-N',O)($PPh_3)_2$] complex was prepared according to the literature method [11]. The solvent used was argon saturated.

2.2. *Synthesis of [$ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2$]*

[$ReCl_2(\eta^2$-N_2COPh-N',O)($PPh_3)_2$] (1 g, 1.1 mmol) was added to nicotine (0.5 ml, 1.2 mmol) in acetone (50 ml) and the reaction mixture was refluxed for 12 h. The brown crystalline precipitate was collected by filtration and crystals suitable for X-ray structure determination were obtained by recrystallization from mixture of chloroform and methanol and the yield was 75%.

Infrared (IR) (KBr, cm^{-1}): 3053(m), 1643(s), 1517(vs), 1483(vs), 1434(vs), 1309(m), 1234(vs), 1170(s), 1090(m), 14044(s), 1020(s), 746(m), 733(m), 695(vs), 638(m), 516(vs), 495(m). ^1H NMR (400 MHz) 2.19 (3H, s, CH_3), 1.8–2.15 (7H, m, $3 \times CH_2 + CH$), 7.1–7.8 (39H, m, Ar–H). Calcd for $C_{53}H_{49}Cl_2N_4OP_2Re$: C, 59.1%; H, 4.59%; N, 5.2%. Found: C, 59%; H, 4.6%; N, 5.3%.

2.3. *Physical measurements*

Infrared spectra were recorded on a Nicolet Magna 560 spectrophotometer in the spectral range of 4000–400 cm^{-1} with samples in the form of potassium bromide pellets. Electronic spectra were measured on a Lab Alliance ultraviolet-visible 8500 spectrophotometer in the range of 800–220 nm in deoxygenated dichloromethane. The magnetic susceptibility was determined with a superconducting quantum interference device (SQUID, Quantum Design) magnetometer. The NMR spectra were recorded with a Bruker AM400 spectrometer at 25°C using 10% solutions in $CDCl_3$. Elemental analyses (C, H, N) were performed on a Perkin–Elmer CHN-2400 analyzer.

2.4. *Crystal structure determination and refinement*

The crystals of [$ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2$] suitable for X-ray structure determination were obtained by recrystallization from mixture of chloroform and methanol. A red-brown crystal was mounted on a KM-4-CCD automatic diffractometer equipped with CCD detector and used for data collection. X-ray intensity data were collected with graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) at room temperature with ω scan mode. A 25 s exposure time was used and all Ewald sphere was collected up to $2\theta = 50.26°$. The unit cell parameters were determined from least-squares refinement of the setting angles of 13,477 strongest reflections. Details concerning crystal data and refinement for $[ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2]$ are given in table 1. The structure was solved by Patterson method and subsequently completed by difference Fourier recycling. All non-hydrogen atoms were refined anisotropically using full-matrix, least-squares. All hydrogens were found on different Fourier synthesis and refined as "riding" on their parent carbons with geometry idealization after each cycle and assigned isotropic temperature factors of 1.2 (non-methyl H atoms) or 1.5 (methyl H atoms) times the value of the parent carbon atom temperature factor. The methyl group was allowed to rotate about its local three-fold axis. SHELXS-97 [12], SHELXL-97 [13], and SHELXTL [14] programs were used for all the calculations.

3. Results and discussion

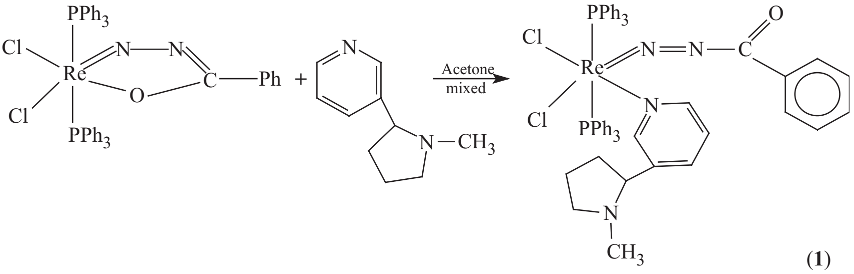
The reaction of $[ReCl_2(\eta^2\text{-}N_2COPh\text{-}N',O)(PPh_3)_2]$ with nicotine results in opening of the chelate ring through displacement of coordinated carbonyl by nicotine [equation (1)] and lead to $[ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2]$.

The complex crystallizes in the monoclinic space group $P2_1/n$ and its crystal structure is composed of discrete monomers with all atoms located in general positions

Table 1. Crystal data and structure refinement for $[ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2]$.

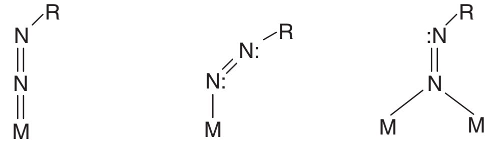
Empirical formula	$C_{53}H_{49}Cl_2N_4OP_2Re$
Formula weight	1077.00
Temperature (K)	291.0(2)
Wavelength (λ)	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions (Å, °)	
a	15.1460(11)
b	16.8038(6)
c	20.2027(13)
α	90
β	111.857(7)
γ	90
Volume (Å^3)	4772.2(5)
Z	4
Density (calculated) (Mg m^{-3})	1.499
Absorption coefficient (mm^{-1})	2.768
$F(000)$	2168
Crystal size (mm^3)	0.593 × 0.317 × 0.164
θ range for data collection (°)	3.14–25.13
Index ranges	$-18 \le h \le 18$ $-20 \le k \le 0$ $-24 \le l \le 24$
Reflections collected	46,884
Independent reflections	8487 ($R_{int} = 0.0726$)
Max. and min. transmission	0.787 and 0.414
Completeness to θ (%)	96.0
Data/restraints/parameters	8487/0/607
Goodness-of-fit on F^2	1.113
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0438$ $wR_2 = 0.0883$
R indices (all data)	$R_1 = 0.0485$ $wR_2 = 0.0937$
Largest diff. peak and hole (e Å^{-3})	1.488 and −1.228

(shown in figure 1). The 1-methyl-2-pyrrolidine of the nicotine substituent is disordered over two positions with 0.5104 : 0.4896 participation.



(1)

The coordination geometry about the rhenium center exhibits *trans* triphenylphosphine molecules minimizing steric congestion and chloride *trans* to the monodentate organodiazenido ligand and to nicotine molecule which displaced the carbonyl oxygen of the diazenido substituent. Selected bond lengths and angles are summarized in table 2. The angular distortions from an ideal octahedron are caused by the presence of multiply bonding ligand (–NNR) in *cis* position to nicotine. Structural studies have confirmed that the organodiazenido ligand may display a variety of geometries: "singly bent," "doubly bent," and "bridging."



Singly bent Doubly bent Bridging

The structural types of organodiazenido ligands.

Complexes containing "singly bent" ligand typically have metal–nitrogen bond lengths of 1.7–1.8 Å, indicative of multiple bonding, and nitrogen–nitrogen multiple bonds shown by bond lengths of 1.2–1.3 Å. The metal–nitrogen–nitrogen bond angle in this structural type is relatively linear [15].

An example of complex containing "double bent" ligand is $[RhCl(N_2C_6H_5)(C_6H_5P((CH_2)_3P(C_6H_5)_2)_2)][PF_6] \cdot CH_2Cl_2$. The Re–N bond length is 1.961(7) Å and is longer than complexes containing "single bent" type at about 0.16 Å. The N–N bond length is 1.172(9) Å, intermediate between triple and double bond. The rhenium–nitrogen–nitrogen bond angle is 125° [16]. An example of "bridging" complex is $[PhN{=}NMn(CO)_4]_2$. The Mn–N bond lengths are 2.031(2) and 2.021(2) Å, the N–N distance is 1.234(3) Å for a double bond [17].

The Re(1)–N(1)–N(2) angles are essentially linear and the N(1)–N(2)–C(37) angles are near 120°, indicating "singly bent" benzoyldiazenido complexes. The short Re–N(1) and N(1)–N(2) distances suggest extensive delocalization and multiple bonding throughout the N_2COPh unit, confirmed by the virtual planarity of the entire Re–N–gtNCOPh and the linearity of the Re–N–N unit. The Re–N(1), N(1)–N(2) and N(2)–C bond lengths and N(1)–N(2)–C(37), Re–N(1)–N(2) angles are in good agreement with values found by other "singly bent" organodiazenido complexes [4, 18, 19]. The significant lengthening of the Re–Cl bond results from the *trans* location of chloride to the strongly π-interacting benzoyldiazenido and nicotine. The Re–Cl(2)

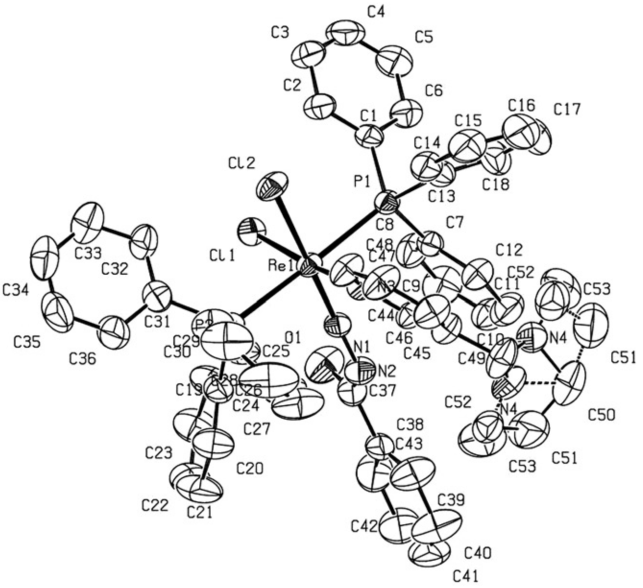


Figure 1. The molecular structure of [$ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2$]. Displacement ellipsoids are drawn at 50% probability level. Hydrogen atoms are omitted for clarity. Disordered part is indicated by dashed lines.

Table 2. Selected bond lengths (Å) and angles (°) for [$ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2$].

Re(1)–N(1)	1.758(4)	N(1)–Re(1)–N(3)	89.87(18)
Re(1)–N(3)	2.210(4)	N(1)–Re(1)–Cl(2)	175.33(15)
Re(1)–Cl(2)	2.4397(12)	N(3)–Re(1)–Cl(2)	85.87(11)
Re(1)–Cl(1)	2.4405(13)	N(1)–Re(1)–Cl(1)	95.59(15)
Re(1)–P(2)	2.4614(14)	N(3)–Re(1)–Cl(1)	174.26(11)
Re(1)–P(1)	2.4899(14)	Cl(2)–Re(1)–Cl(1)	88.73(5)
N(1)–N(2)	1.261(6)	N(1)–Re(1)–P(2)	89.11(14)
N(2)–C(37)	1.395(7)	N(3)–Re(1)–P(2)	90.95(12)
C(37)–O(1)	1.233(8)	Cl(2)–Re(1)–P(2)	89.03(5)
N(3)–C(44)	1.342(7)	Cl(1)–Re(1)–P(2)	90.91(5)
N(3)–C(48)	1.354(6)	N(1)–Re(1)–P(1)	91.89(14)
C(49)–N(4)	1.316(12)	N(3)–Re(1)–P(1)	90.56(11)
C(49)–N(4)	1.404(12)	Cl(2)–Re(1)–P(1)	90.08(5)
C(50)–N(4)	1.477(14)	Cl(1)–Re(1)–P(1)	87.49(4)
N(4)–C(53)	1.47(5)	P(2)–Re(1)–P(1)	178.18(5)
N(4)–C(53)	1.38(3)	C(7)–P(1)–Re(1)	108.01(16)
		C(1)–P(1)–Re(1)	119.82(18)
		C(13)–P(1)–Re(1)	119.6(2)
		C(25)–P(2)–Re(1)	112.8(2)
		C(31)–P(2)–Re(1)	120.1(2)
		C(19)–P(2)–Re(1)	113.48(18)
		N(2)–N(1)–Re(1)	169.3(4)
		N(1)–N(2)–C(37)	120.6(5)
		O(1)–C(37)–N(2)	124.3(6)
		O(1)–C(37)–C(38)	123.5(6)
		N(2)–C(37)–C(38)	112.1(6)
		C(44)–N(3)–Re(1)	118.4(3)
		C(48)–N(3)–Re(1)	123.5(4)
		N(3)–C(44)–C(45)	122.7(5)
		C(44)–N(3)–C(48)	118.0(5)

Table 3. Hydrogen-bond geometry for $[ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2]$ (Å, °).

D–H···A	H···A	D···A	<(DHA)
C2–H2···Cl2	2.69	3.530(6)	150.7
C24–H24···Cl1	2.79	3.641(7)	153.2
C32–H32···Cl1	2.77	3.442(6)	129.6
C32–H32···Cl2	2.72	3.405(7)	131.1
C39–H39···N2	2.44	2.776(9)	101.3
C44–H44···N1	2.50	2.983(7)	112.7
C48–H48···Cl2	2.57	3.141(6)	120.3

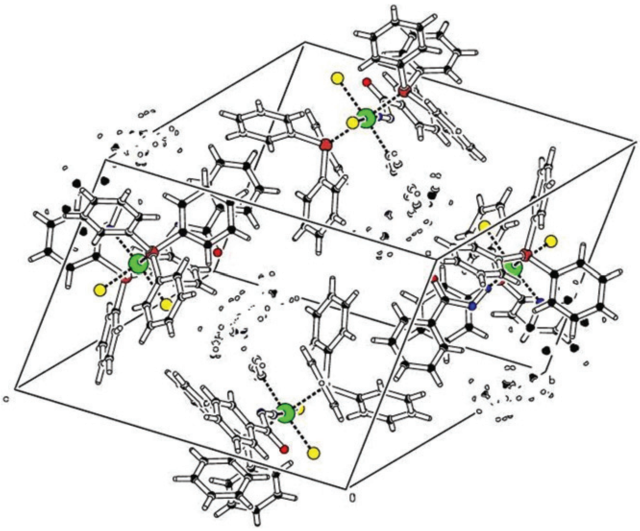


Figure 2. The packing diagram of $[ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2]$.

distance *trans* to NNR ligand is shorter than the Re–Cl(1) *trans* to nicotine, indicating the stronger *trans* influence of heterocycle molecule in comparison with benzoyldiazenido. The Re–N(3) bond lengths (2.210(4) Å) agree well with values found previously in similar rhenium compounds containing monodentate *N*-heterocyclic ligands [20, 21]. The bond valences were computed as $\nu_{ij} = \exp[(R_{ij} - d_{ij})/b]$ [22–24], where R_{ij} is the bond-valence parameter (in the formal sense the R_{ij} parameter value can be considered as the idealized single-bond length between i and j atoms). The $R_{\text{Re–N}}$, $R_{\text{Re–P}}$, and $R_{\text{Re–Cl}}$ were taken as 2.06, 2.46, and 2.23 [25], respectively, and b was taken as 0.37 [22]. The computed bond valences of the rhenium are $\nu_{\text{Re–N(nicotine)}} = 0.67$, $\nu_{\text{Re–N(N2COPh)}} = 2.26$, $\nu_{\text{Re–P}} = 0.92$ and 1.00; $\nu_{\text{Re–Cl}} = 0.57$ and 0.57 v.u. (valence units) which means that $\text{Re–N}_{(\text{N2COPh})}$ bond is three to four times stronger than other bonds and can be formally considered a double bond, the Re–Cl bonds are the weakest, and the strength of $\text{Re–N}_{(\text{nicotine})}$ bond is between Re–Cl and Re–P bond strength.

The structure is stabilized by multiple weak intramolecular hydrogen bonds [26, 27] (table 3). The packing diagram of $[ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2]$ is depicted in figure 2. The possibility of stacking interactions between complex

Table 4. Band positions, molar absorption coefficients and assignments for $[ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2]$.

Band position [cm^{-1}]	ε	Assignment
18,954	430	$^1A_1 \rightarrow {}^3T_1$, 5d $\rightarrow \pi^*_{\text{nicotine}}$
26,137	11,593	$^1A_1 \rightarrow {}^3T_1$, 5d $\rightarrow \pi^*_{N_2COPh}$
34,843	26,504	$^1A_1 \rightarrow {}^1T_2, \pi \rightarrow \pi^*_{C_6H_5}$
43,592	66,225	$\pi^b_{C_6H_5} \rightarrow 3d(P)$, $\pi \rightarrow \pi^*_{N_2COPh}$, $\pi \rightarrow \pi^*_{\text{nicotine}}$

Note: ε = molar absorption coefficient [$dm^3 mol^{-1} cm^{-1}$].

molecules rejected on the basis of shortest distance between parallel aromatic rings is 5.829 Å.

In contrast to the starting $[ReCl_2(\eta^2\text{-}N_2COPh\text{-}N',O)(PPh_3)_2]$, the IR spectrum shows several bands in the 1550–1650 cm^{-1} range assigned to ν(N=N), ν(C–N), and ν(C=O), confirming the opening of the chelate ligand and the presence of nicotine [4]. The complex shows the characteristic pair of bands at approximately 1430 and 1480 cm^{-1} for triphenylphosphine. Table 4 presents the positions, molar absorption coefficients of electronic bands, and the electronic transitions assigned to the bands for $[ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2]$. Diamagnetism of the complex and linear Re=N=N– configuration indicate that N_2COPh is a four-electron donor, and Re(I) has a low-spin d^6 configuration. Taking into account the possible d–d, charge transfer and intra-ligand transitions, the absorption bands were ascribed as shown in table 4. Charge transfer transitions between Re(I) and the ligands are only of MLCT character, and N_2COPh is a stronger acceptor than nicotine, proved by higher intensity of the band at 26,137 cm^{-1} in comparison with the band at 18,954 cm^{-1}. This conclusion is confirmed by the bond length of Re–N(1) being shorter than Re–N(3), and the bond length of Re–Cl(1) longer than Re–Cl(2). More 5d electron delocalization on N_2COPh than on nicotine is also proved by the longer N(2)–C(37) bond length than N(3)–C(44). The data of table 4 indicate a pseudo-octahedral electron configuration of Re(I) with similar covalency degree as in the analog with pyridine [28], $10D_q = 28{,}313\ cm^{-1}$, $B = 544\ cm^{-1}$ and $C = 2{,}176\ cm^{-1}$.

Supplementary data

Supplementary data of $[ReCl_2(N_2COPh)(C_{10}H_{14}N_2)(PPh_3)_2]$ are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, on request quoting deposition number CCDC 679697.

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