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### The Synthesis, Crystal, Molecular and Electronic Structure of [ReCl<sub>2</sub>(NO)(py)<sub>3</sub>]

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## THE SYNTHESIS, CRYSTAL, MOLECULAR AND ELECTRONIC STRUCTURE OF $[\text{ReCl}_2(\text{NO})(\text{py})_3]$

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(Received 3 September 2002)

In line with our investigations of rhenium nitrosyl complexes, we have studied the reaction of  $[\text{ReCl}_3(\text{NO})(\text{OPPh}_3)(\text{PPh}_3)]$  with pyridine. The  $[\text{ReCl}_2(\text{NO})(\text{py})_3]$  complex obtained in this reaction has been characterised by IR, electronic spectra and magnetochemical measurements; ligand field parameters and the electronic structure have been determined. The crystal and molecular structure of  $[\text{ReCl}_2(\text{NO})(\text{py})_3]$  has been solved by the heavy atom method. Crystals of  $[\text{ReCl}_2(\text{NO})(\text{py})_3]$  contain distorted octahedral molecules with the pyridine ligands in the *mer*-arrangement. The nitrosyl group is coordinated linearly to the rhenium atom as  $\text{NO}^+$ .

**Keywords:** Rhenium complexes; Nitrosyl; X-ray structure; Electronic structure; Nitric oxide

### INTRODUCTION

Due to catalytic activities, capacity for pollution control and structural properties of transition-metal nitrosyl derivatives, interest in these compounds has been growing for many years [1–8]. The recent discoveries of the key role of nitric oxide in many physiological and pathophysiological processes and the development of technetium and rhenium radiopharmaceuticals have fostered interest in the chemistry of nitrosyl complexes for these metals [9–13]. However, only few pyridino nitrosyl complexes of rhenium have been reported so far [14–20].

The goal of this article was to investigate the reaction of  $[\text{ReCl}_3(\text{NO})(\text{OPPh}_3)(\text{PPh}_3)]$  with pyridine. Here, we present the spectroscopic characterisation and the crystal, molecular and electronic structure of  $[\text{ReCl}_2(\text{NO})(\text{py})_3]$ , obtained in the reaction.

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## EXPERIMENTAL

### Materials

Triphenylphosphine and  $\text{NH}_4\text{ReO}_4$  were purchased from Aldrich Chemical Co. and used without further purification.  $[\text{ReCl}_3(\text{NO})(\text{OPPh}_3)(\text{PPh}_3)]$  was synthesised according to a literature method [21]. Solvents were dried and deoxygenated prior to use in the usual way. The reaction, all preparations and the recrystallisation were performed under an argon atmosphere.

### $[\text{ReCl}_2(\text{NO})(\text{py})_3]$ , (**1**)

$[\text{ReCl}_3(\text{NO})(\text{OPPh}_3)(\text{PPh}_3)]$  (0.22 g, 0.24 mmol) was heated in dry pyridine ( $10\text{ cm}^3$ ) at reflux for 4 h. The solution was evaporated to the volume of  $2\text{ cm}^3$  and an excess of ethanol added. The dark red solid which precipitated was recrystallised from  $\text{CH}_2\text{Cl}_2/\text{C}_2\text{H}_5\text{OH}$  to give prismatic crystals (0.1 g, 80%).

### Physical Measurements

Infrared spectra were recorded on a Nicolet Magna 560 spectrophotometer in the range  $2000\text{--}400\text{ cm}^{-1}$  with the samples in the form of potassium bromide pellets. Electronic spectra were measured on a Beckman 5240 spectrophotometer in the range from 800 to 220 nm in deoxygenated dichloromethane solution. Magnetic susceptibilities were measured at 296 K by the Faraday method.

### Crystal Structure Determination and Refinement

Dark red crystals of **1** suitable for X-ray diffraction were grown by slow evaporation of a dichloromethane/ethanol solution (1 : 1). Three-dimensional X-ray intensity data were collected on a Kuma KM-4 diffractometer, using  $\text{MoK}\alpha$  radiation (graphite monochromator) in the  $\omega\text{--}2\theta$  scan mode, at room temperature. Details concerning crystal data and refinement are given in Table I. Lorentz, polarization and empirical absorption corrections were applied. The structure was solved by means of Patterson and Fourier methods. All non-hydrogen atoms were refined anisotropically using full-matrix, least-squares techniques. The hydrogen atom positions of the phenyl rings were found from subsequent difference Fourier syntheses and were treated as riding on the adjacent carbon atom and refined with individual isotropic temperature factors equal to 1.2 times the values of equivalent temperature factors of the parent carbon atoms with the C–H distance free to refine. SHELXL97 and SHELXTL programs [22,23] were used for all calculations. Atomic scattering factors were those incorporated in the computer programs.

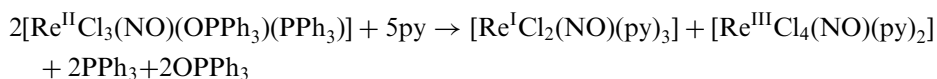
## RESULTS AND DISCUSSION

Refluxing pyridine with  $[\text{ReCl}_3(\text{NO})(\text{OPPh}_3)(\text{PPh}_3)]$  produced Complex **1**. The reaction yield depends on two factors, purity of pyridine (the presence of moisture in the pyridine leads to a drastic fall in yield), and reaction time (the yield of **1** is considerably lower if

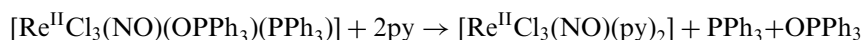
TABLE I Crystal data and structure refinement details for **1**

Empirical formula	C <sub>15</sub> H <sub>15</sub> Cl <sub>2</sub> N <sub>4</sub> ORe
Formula weight	524.41
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	monoclinic, <i>P</i> <sub>2</sub> <sub>1</sub> / <i>n</i>
Unit cell dimensions	<i>a</i> = 9.177(2) <i>b</i> = 12.663(3), <i>c</i> = 15.358(3) Å <i>β</i> = 91.96(3)°
Volume	1783.7(7) Å <sup>3</sup>
<i>z</i> , Calculated density	4, 1.953 Mg/m <sup>3</sup>
Absorption coefficient	7.120 mm <sup>-1</sup>
<i>F</i> (000)	1000
Crystal size	0.45 × 0.24 × 0.11 mm
Theta range for data collection	2.08–25.00°
Index ranges	−10 ≤ <i>h</i> ≤ 10, −1 ≤ <i>k</i> ≤ 15, −1 ≤ <i>l</i> ≤ 18
Reflections collected/unique	3712/3129 [ <i>R</i> (int) = 0.0242]
Completeness to 2θ = 25.00	95.0%
Max. and min. transmission	0.5081 and 0.1419
Refinement method	Full-matrix least-square on <i>F</i> <sup>2</sup>
Data/constraints/parameters	3129/2/252
Goodness-of-fit on <i>F</i> <sup>2</sup>	1.226
Final <i>R</i> indices [ <i>I</i> > 2σ( <i>I</i> )]	<i>R</i> 1 = 0.0262, <i>wR</i> 2 = 0.0609
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0537, <i>wR</i> 2 = 0.0734
Largest diff. peak and hole	1.031 and −0.845 e Å <sup>-3</sup>

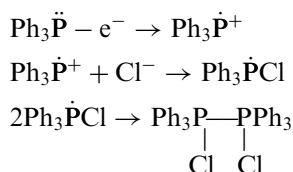
the reaction is carried out for less than four hours). The lack of a second product in the reaction and the high yield of **1** (80%) exclude the disproportionation reaction shown below.



Triphenylphosphine liberated from the coordination sphere in the first step of the reaction



plays the role of reducing agent for the central atom,  $\text{Re}^{\text{II}} \rightarrow \text{Re}^{\text{I}}$ . It has been known for a long time that the cation-radical  $\text{Ph}_3\dot{\text{P}}^+$  [24] is susceptible to nucleophilic attack [25] and this allows us to propose a reduction process occurring with participation of phosphine as follows.



The complex **1** belongs to space group *P*<sub>2</sub><sub>1</sub>/*n* and all atoms occupy general position. Accurate cell parameters, crystal data and details concerning the structure of **1** are given

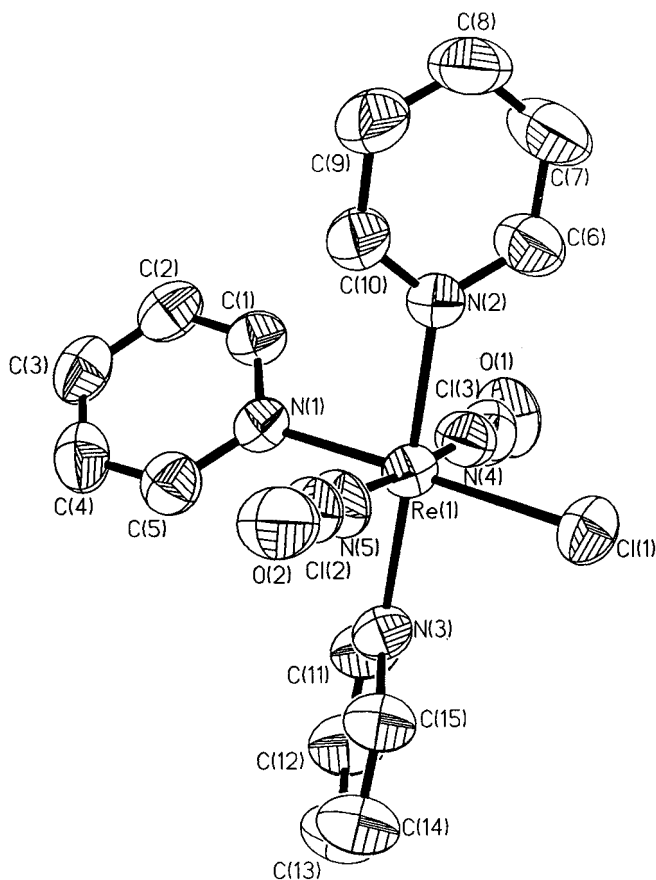


FIGURE 1 Molecular structure of **1** with disordered NO and Cl ligands. Hydrogen atoms are omitted for clarity.

in Table I. NO and Cl ligands coordinated in *trans* positions are disordered in **1** (Fig. 1). Occupancy factors for the atoms Cl(2), Cl(3), N(4), O(1) and N(5), O(2) were refined (see Table II), so the molecular structure of **1** may be presented in a simplified way as shown in Fig. 2. Site occupancy factors were 0.57(2) and 0.43(2) for Cl(2) and Cl(3), respectively, 0.56(2) for the nitrosyl group N(4)=O(1) and 0.43(2) for the nitrosyl group N(5)=O(2), respectively. The coordination around rhenium is distorted octahedral and pyridine ligands are arranged in a *meridional* fashion. The angles between *cis*-ligands vary from 82.6(8) to 92.4(11)°, while the *trans*-angles are between 171.3(8) and 179.4(2)°, so that the departure from the ideal octahedron ascribed to mutual repulsion of the ligands is very limited.

The most important bond lengths and angles for **1** are reported in Table III. A large difference in Re–NO bond lengths and Re–N–O angles of two nitrosyl groups with nearly half occupancy factor is caused by the disordered structure [Re(1)–N(4) = 1.92(2), Re(1)–N(5) = 1.84(2) Å, Re(1)–N(4)–O(1) = 160(3) and Re(1)–N(5)–O(2) = 174(9)°]. The mean Re–NO and N–O bond distances of **1** [Re–N = 1.88(2) and N–O = 1.11(1) Å] are in good agreement with values previously found by

TABLE II Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for **1**.  $U_{\text{eq}}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor. S.o.f is given if different from 1

	$x/a$	$y/b$	$z/c$	s.o.f.	$U_{\text{eq}}$
Re(1)	137(1)	944(1)	2566(1)		47(1)
Cl(1)	-1206(2)	-74(1)	1466(1)		60(1)
Cl(2)	761(11)	-598(7)	3332(7)	0.571	64(2)
Cl(3)	-400(14)	2470(7)	1824(6)	0.429	67(3)
N(4)	-250(4)	2320(2)	2090(13)	0.565	75(8)
O(1)	-480(2)	2953(14)	1607(11)	0.565	92(5)
N(5)	580(6)	-280(3)	3170(3)	0.435	82(11)
O(2)	880(3)	-955(19)	3593(18)	0.435	95(8)
N(1)	1277(5)	1830(4)	3545(3)		49(1)
C(1)	2188(6)	2615(5)	3338(4)		54(2)
C(2)	2892(7)	3238(5)	3952(5)		67(2)
C(3)	2704(8)	3046(6)	4809(5)		69(2)
C(4)	1783(8)	2242(6)	5032(5)		71(2)
C(5)	1110(7)	1655(6)	4397(4)		61(2)
N(2)	2118(5)	796(4)	1877(3)		53(1)
C(6)	2177(8)	1084(6)	1026(5)		71(2)
C(7)	3428(10)	1022(7)	572(6)		94(3)
C(8)	4697(9)	686(7)	988(6)		83(2)
C(9)	4646(8)	375(7)	1830(6)		79(2)
C(10)	3364(7)	446(6)	2259(5)		65(2)
N(3)	-1813(5)	1076(4)	3267(3)		50(1)
C(11)	-2370(7)	2026(5)	3494(5)		62(2)
C(12)	-3591(8)	2138(6)	3953(5)		74(2)
C(13)	-4308(9)	1249(7)	4240(6)		81(2)
C(14)	-3776(9)	286(6)	4013(5)		78(2)
C(15)	-2552(7)	223(6)	3533(5)		64(2)

others, for example in  $[\text{ReBr}_4(\text{NO})(\text{EtOH})]^-$  [1.723(15), 1.19(2) Å] [26] and in *mer*- $[\text{ReCl}_2(\text{NO})(\text{PMePh}_2)_3]$  [1.775(10), 1.182(14) Å] [14]. The N–O bond lengths, 1.10 to 1.11 (1) Å are as expected for  $\text{NO}^+$ . The Re–Cl (in the range 2.289(9) to 2.428(2) Å, mean 2.335 Å) and Re–N<sub>py</sub> (in the range 2.123(5) to 2.144(5) Å, mean 2.131 Å) bond lengths are comparable to those found for similar complexes (*trans*- $[\text{ReCl}_4(\text{NO})(\text{py})]^-$  2.35 to 2.38 Å for Re–Cl and 2.22 Å for Re–N [27]; *mer*- $[\text{ReCl}_3(3,5\text{-lut})_3]$  2.35 to 2.40 Å for Re–Cl and 2.12 to 2.13 Å for Re–N [28]; *trans*- $[\text{ReO}_2(\text{py})_4]^+$  2.14 to 2.17 Å for Re–N [30];  $[\text{ReOCl}_2(\text{OEt})(\text{py})_2]$  2.37, 2.44 Å for Re–Cl and 2.13, 2.14 Å for Re–N [31];  $[\text{ReOCl}_2(\text{OSiMe}_3)(\text{py})_2]$  2.39, 2.41 Å for Re–Cl and 2.13 Å for Re–N [32]; *trans*- $[\text{ReO}_2(4\text{-picoline})_4]^+$  2.11 to 2.15 Å for Re–N [33]).

The Re(1)–Cl(1) bond length *trans* to the pyridine ligand [2.428(2) Å] is longer than Re(1)–Cl(2) and Re(1)–Cl(3) distances [2.341(6) and 2.289(9) Å, respectively]. A similar trend is observed in the *mer*- $[\text{ReCl}_3(3,5\text{-lut})_3]$  complex (where 3,5-lut = 3,5-dimethylpyridine); the chloride ligand *trans* to 3,5-dimethylpyridine is lengthened by 0.04 Å compared to the mutually *trans*-ones. Variation of Re–N<sub>lut</sub> distances in *mer*- $[\text{ReCl}_3(3,5\text{-lut})_3]$ , similar to the variation of Re–N<sub>py</sub> in **1**, is within experimental error [28].

The IR spectrum of **1** shows  $\nu(\text{NO})$  at  $1655\text{ cm}^{-1}$ . This is consistent with the linear NO range of Haymore and Ibers [29] [ $\nu(\text{NO})$  above  $1620\text{--}1610\text{ cm}^{-1}$  assigned to linear M–N–O systems;  $\nu(\text{NO})$  below  $1610\text{ cm}^{-1}$  assigned to bent M–N–O systems]. Most nitrosyl rhenium (I) complexes of the type  $[\text{ReCl}_2(\text{NO})\text{L}_3]$ , where L is a nitrogen or phosphorus donor ligand, show  $\nu(\text{NO})$  very close to  $1650\text{ cm}^{-1}$  (for example

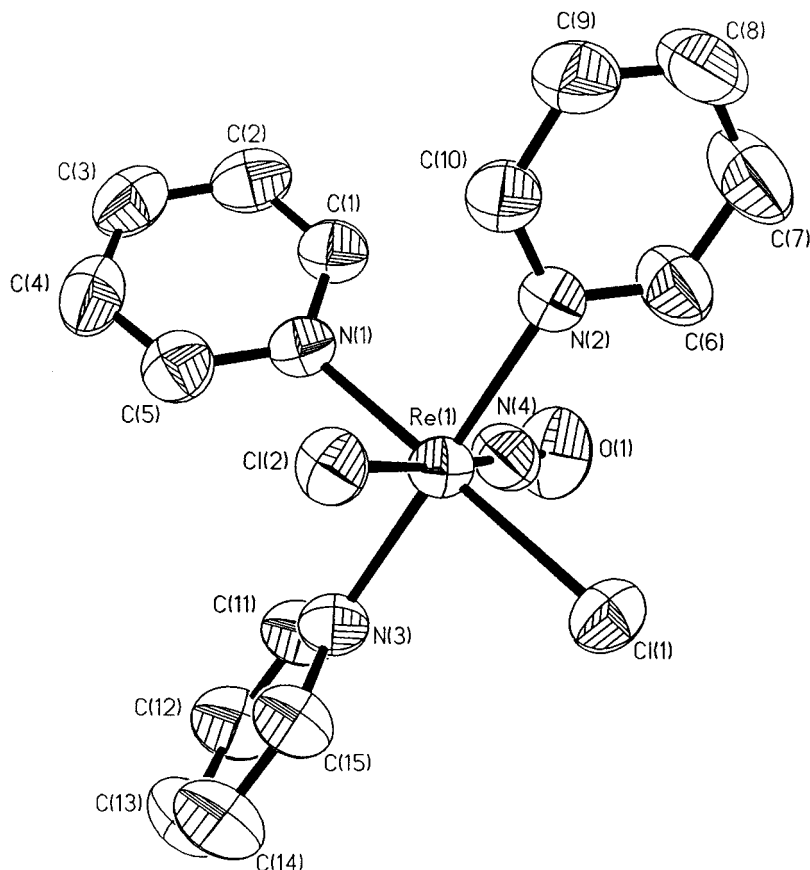


FIGURE 2 Simplified molecular structure of **1** (without disordered NO and Cl ligands). Hydrogen atoms are omitted for clarity.

[ReCl<sub>2</sub>(NO)(PMe<sub>2</sub>Ph)<sub>3</sub>] 1665 cm<sup>-1</sup>, [ReCl<sub>2</sub>(NO)(PEt<sub>2</sub>Ph)<sub>3</sub>] 1660 cm<sup>-1</sup>, [ReCl<sub>2</sub>(NO)(py)(PPh<sub>3</sub>)<sub>2</sub>] [14] 1670 cm<sup>-1</sup>, [ReBr<sub>2</sub>(NO)(4-picoline)<sub>3</sub>] [20] 1660 cm<sup>-1</sup>). This makes it exceedingly likely that all these complexes have essentially linear Re–N–O systems. The band at 1605 cm<sup>-1</sup> and the series of weak bands centred at approximately 1460 cm<sup>-1</sup> and 730 cm<sup>-1</sup> are due to the pyridine ligand. Magnetochemical measurements confirm the lack of unpaired electrons in Complex **1**.

The positions [cm<sup>-1</sup>] and molar absorption coefficients [dm<sup>3</sup>/mol<sup>-1</sup>/cm<sup>-1</sup>] of electronic bands for **1** are as follows: 15450 (120), 18700 (450), 26900 (3000) and 30600 (2700). The absorption spectrum of **1** is interpreted by comparison with the other Re(I) complexes with ligands structurally related to those in Compound **1** [34–36], considering splitting of *d* orbitals in the ligand field of C<sub>s</sub> symmetry. First, two absorption bands in the electronic spectrum of **1** correspond to typical low-intensity electron transitions between *d* orbitals. They correspond to the *d*<sub>xz</sub> → *d*<sub>x<sup>2</sup>-y<sup>2</sup></sub> and *d*<sub>yz</sub> → *d*<sub>x<sup>2</sup>-y<sup>2</sup></sub> transitions, respectively. The band at 30600 cm<sup>-1</sup> is also a *d*–*d* transition, a result of the *d*<sub>xy</sub> → *d*<sub>x<sup>2</sup>-y<sup>2</sup></sub> transition. However, its intensity is much higher than those for the bands at 15450 cm<sup>-1</sup> and 18700 cm<sup>-1</sup>, caused by effects of *d*<sub>xy</sub> → π\*<sub>NO</sub> and *d*<sub>xy</sub> → *d*<sub>x<sup>2</sup>-y<sup>2</sup></sub>

TABLE III Selected bondlengths [Å] and angles [°] for **1**

<i>Bonds lengths</i>	
Re(1)–N(5)	1.843(19)
Re(1)–N(4)	1.92(2)
Re(1)–N(1)	2.123(5)
Re(1)–N(3)	2.125(5)
Re(1)–N(2)	2.144(5)
Re(1)–Cl(3)	2.289(9)
Re(1)–Cl(2)	2.341(6)
Re(1)–Cl(1)	2.4276(16)
N(4)–O(1)	1.107(10)
N(5)–O(2)	1.104(10)
N(1)–C(1)	1.345(7)
N(1)–C(5)	1.341(8)
N(2)–C(6)	1.359(9)
N(2)–C(10)	1.343(8)
N(3)–C(15)	1.346(8)
N(3)–C(11)	1.358(8)
<i>Bond angles</i>	
N(5)–Re(1)–N(4)	171.7(19)
N(5)–Re(1)–N(1)	89.4(18)
N(4)–Re(1)–N(1)	82.6(8)
N(5)–Re(1)–N(3)	89.2(17)
N(4)–Re(1)–N(3)	88.4(11)
N(1)–Re(1)–N(3)	90.17(19)
N(5)–Re(1)–N(2)	89.9(17)
N(4)–Re(1)–N(2)	92.4(11)
N(1)–Re(1)–N(2)	89.44(18)
N(3)–Re(1)–N(2)	179.08(18)
N(5)–Re(1)–Cl(3)	179.4(19)
N(1)–Re(1)–Cl(3)	89.9(3)
N(3)–Re(1)–Cl(3)	90.9(3)
N(2)–Re(1)–Cl(3)	90.0(4)
N(4)–Re(1)–Cl(2)	171.3(8)
N(1)–Re(1)–Cl(2)	88.8(3)
N(3)–Re(1)–Cl(2)	90.4(3)
N(2)–Re(1)–Cl(2)	88.8(3)
Cl(3)–Re(1)–Cl(2)	178.2(4)
N(5)–Re(1)–Cl(1)	90.3(18)
N(4)–Re(1)–Cl(1)	97.6(8)
N(1)–Re(1)–Cl(1)	178.88(13)
N(3)–Re(1)–Cl(1)	88.74(14)
N(2)–Re(1)–Cl(1)	91.65(14)
Cl(3)–Re(1)–Cl(1)	90.4(2)
Cl(2)–Re(1)–Cl(1)	90.9(3)
O(1)–N(4)–Re(1)	160(3)
Cl(2)–N(5)–Re(1)	174(9)

transitions. Considering the fact that  $d_{xz}$  and  $d_{yz}$  orbitals have different symmetries than the  $\pi^*_{\text{NO}}$  orbital (positioned in  $xy$  plane), this effect does not occur for the  $d_{xz} \rightarrow d_{x^2-y^2}$  and  $d_{yz} \rightarrow d_{x^2-y^2}$  transitions.

The band at  $26900 \text{ cm}^{-1}$  is a charge transfer transition corresponding to promotion of  $d_{xy}$  electrons to the  $\pi^*_{\text{NO}}$  orbital. A simplified MO diagram for **1** is presented in Fig. 3. and values of ligand field parameters for Complex **1** are  $Dq = 3058$ ,  $D_s = -4447$  and  $Dt = 378 \text{ cm}^{-1}$ . In the nitrosyl Complex **1**, the Re ion is present in the +1 oxidation state, and its electronic configuration, considering splitting of  $d$  orbitals in  $C_s$  symmetry is  $(d_{xy})^2(d_{yz})^2(d_{xz})^2$ .



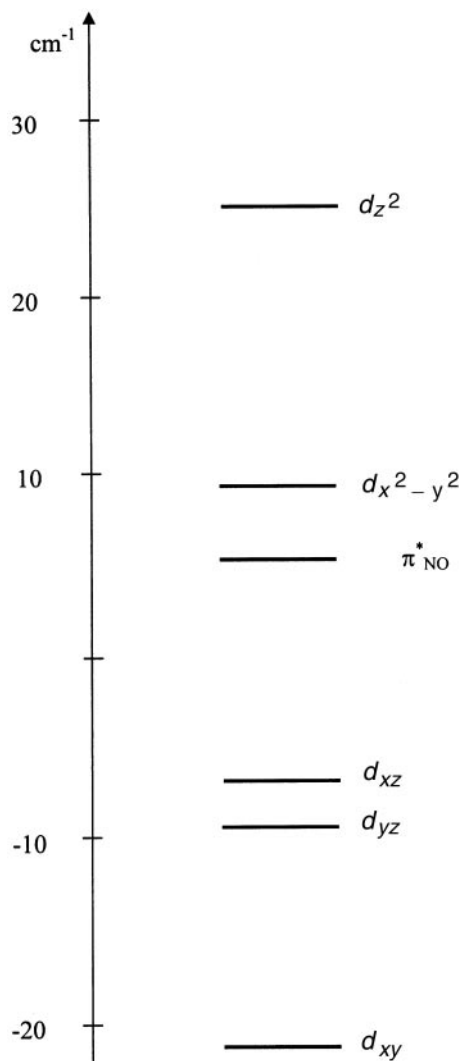


FIGURE 3 Molecular orbital diagram for 1.

### *Acknowledgments*

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### *Supplementary data*

Full list of bond lengths and angles with lists of atomic coordinates, anisotropic thermal parameters, H-atom parameters and structure factors for  $[\text{ReCl}_2(\text{NO})(\text{py})_3]$  have been deposited and are available on request from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK, quoting number CCDC160779.

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