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# Synthesis, characterization and structures of the novel rhenium(IV) complexes trans-[ReCl<sub>4</sub>{PPh<sub>2</sub>(OMe)}<sub>2</sub>], trans-[ReCl<sub>4</sub>{PPh<sub>2</sub>(OEt)}<sub>2</sub>] and trans-[ReCl<sub>4</sub>(OPPh<sub>3</sub>)<sub>2</sub>]

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#### Abstract

*trans*-Tetrachlorobis(methyl-diphenylphosphinite)rhenium(IV) and *trans*-tetrachlorobis(ethyl-diphenylphosphinite)rhenium(IV) were obtained by recrystallization of the corresponding rhenium(III) phosphinite complexes, [ReCl<sub>3</sub>L<sub>3</sub>], in chlorinated solvents. The structures of the compounds consist of discrete molecules with *trans*-octahedral coordination geometry around the rhenium atom, having Re–P distances between 2.534(1) and 2.555(2) Å and Re–Cl distances in the range 2.321(1)–2.336(1) Å. *trans*-[ReCl<sub>4</sub>(OPPh<sub>3</sub>)<sub>2</sub>] is formed during the reaction of triphenyloxonium tetrafluoroborate with [ReNCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>]. P–O bond lengths of 2.033(4) Å have been found, while the Re–Cl bonds are 2.336(2) and 2.344(2) Å, respectively. © 1999 Elsevier Science Ltd. All rights reserved.

## 1. Introduction

The coordination chemistry of rhenium phosphine complexes is well developed. In contrast, reports of the phosphinite and phosphine oxides derivatives are exceedingly rare, despite their uneven reactivities as a consequence of their different electronic and steric properties [1].

[ReX<sub>4</sub>L<sub>2</sub>] derivatives (L=phosphine) have been known for a long time and some of them have been structurally characterized [2–8]. These Re(IV) complexes are usually obtained by reduction of rhenium(V) chloride or oxochloride precursors [5,9–11] or by oxidation of rhenium(III) halides [12]. The latter process may be performed by the solvents when they are chlorinated [13]. Alternative methods are ligand exchange reactions of coordinated solvent molecules vs. phosphine ligands [14] or bond cleavage reactions of binuclear complexes [3].

In this paper, we report the synthesis and structural characterization of the two new rhenium(IV) phosphinite complexes  $[\text{ReCl}_4\{\text{PPh}_2(\text{OMe})\}_2]$  and  $[\text{ReCl}_4\{\text{PPh}_2(\text{OEt})\}_2]$  together with those of the phosphine oxide compound  $[\text{ReCl}_4(\text{OPPh}_3)_2]$ .

#### 2. Experimental

 $[\text{ReCl}_{3}\{\text{PPh}_{2}(\text{OMe})\}_{3}], [\text{ReCl}_{3}\{\text{PPh}_{2}(\text{OMe})\}_{3}]$  and  $[\text{ReNCl}_{2}(\text{PPh}_{3})_{2}]$  have been obtained by reported methods [18,24].  $(\text{Ph}_{3}\text{O})(\text{BF}_{4})$  was supplied commercially (Fluka).

# 2.1. trans-[ $ReCl_4$ { $PPh_2(OMe)$ }]

[ReCl<sub>3</sub>{PPh<sub>2</sub>(OMe}<sub>3</sub>] (500 mg, 0.53 mmol) was dissolved in 10 ml of a CH<sub>2</sub>Cl<sub>2</sub>–CCl<sub>4</sub> mixture (1:4, v/v) and stirred for 24 h. The resulting violet powder was filtered off and recrystallized from CH<sub>2</sub>Cl<sub>2</sub>. Yield, 38%. M.p.: 207–209°C. Found: C, 40.7; H, 3.3%.  $C_{26}H_{26}Cl_4O_2P_2Re$  requires C, 41.1; H, 3.5%. <sup>1</sup>H NMR (CD<sub>2</sub>Cl<sub>2</sub>): 18.4 (m, 8H, C<sub>6</sub>H<sub>5</sub> ortho), 10.2 (m, 4H, C<sub>6</sub>H<sub>5</sub> para), 8.8 (m, 8H, C<sub>6</sub>H<sub>5</sub> meta) and 7.2 (d, 6H, OCH<sub>3</sub>).

# 2.2. trans-[ $ReCl_4$ { $PPh_2(OEt)$ }]

This compound was prepared as outlined above for the methyl derivative starting from  $[\text{ReCl}_3\{\text{PPh}_2(\text{OMe})\}_3]$ . Yield, 47%. M.p.: 202–208°C. Found: C, 41.8; H, 3.9%. C<sub>28</sub>H<sub>30</sub>Cl<sub>4</sub>O<sub>2</sub>P<sub>2</sub>Re requires C, 42.7; H, 3.8%. <sup>1</sup>H NMR (CD<sub>2</sub>Cl<sub>2</sub>): 18.4 (m, 8H, C<sub>6</sub>H<sub>5</sub> ortho), 10.1 (m, 4H, C<sub>6</sub>H<sub>5</sub> para), 8.8 (m, 8H, C<sub>6</sub>H<sub>5</sub> meta), 7.4 (d, 4H, OCH<sub>2</sub>) and 3.8 (m, 6H, CH<sub>3</sub>).

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# 2.3. trans- $[ReCl_4(OPPh_3)_2]$

[ReNCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>] (80 mg, 0.1 mmol) was suspended in 200 ml of CH<sub>2</sub>Cl<sub>2</sub> and (Ph<sub>3</sub>O)(BF<sub>4</sub>) (330 mg, 1 mmol) was added. The mixture was refluxed for 24 h. During this time, the adduct was only partially dissolved to give a red-brown solution. The remaining solid (essentially [ReNCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>] with a few percent of [ReOCl<sub>3</sub>(PPh<sub>3</sub>)<sub>2</sub>]) was filtered off and the filtrate was reduced in volume to about 50 ml and stored overnight in a refrigerator, which resulted in the deposition of red plates at the glass walls. More product could be obtained by further evaporation of the solution. Yield, 18 mg (20%). Found: C, 48.1; H, 3.6%. C<sub>36</sub>H<sub>30</sub>Cl<sub>4</sub>O<sub>2</sub>P<sub>2</sub>Re requires C, 48.9; H, 3.4%. IR:  $\nu$ (P=O) 1106 cm<sup>-1</sup>. Magnetic moment (SQUID): 4.21 B.M.

## 2.4. X-ray data collection, structure and refinement

Crystallographic measurements were performed on a CAD4 Enraf-Nonius diffractometer with molybdenum K $\alpha$  radiation. Crystal data and experimental conditions are listed in Table 3. Data were corrected for polarization and Lorentz effects.  $\Psi$ -scan absorption corrections were also applied [25].

Structure analyses were carried out by the heavy atom method [26] followed by difference Fourier techniques until all non-hydrogen atoms were located. All non-H atoms have been anisotropically refined. The positions of H-atoms have been calculated geometrically and included in structure factor calculations. Scattering factors and anomalous dispersion terms have been taken from reference [27]. Most calculations were performed with the programs SHELXS97 [18] and SHELXL97 [28].

## 3. Results and discussion

derivatives  $(L=PPh_2{OMe})$ The  $[\text{ReCl}_4\text{L}_2]$ and  $PPh_{2}{OEt}$  are obtained in yields >30% by recrystallization of the corresponding  $[ReCl_3L_3]$  compounds in chlorinated solvents. The oxidation of the rhenium(III) compounds follows a reaction pattern that has previously been found for phosphine complexes of rhenium [12] and technetium [15]. The mechanism of this reaction has not been elucidated. Oxygen may be ruled out as an oxidant in the case of our reactions. No evidence has been found for oxidation products of the readily oxidisable phosphinite ligands that have been potentially released during the reaction. The activation of halogenated solvents by rhenium or molybdenum complexes, however, and their participation in the formation of the products has also been reported for other reactions [16,17] and suggests a remarkable catalytic activity of the transition metal complexes applied.

[ReCl<sub>4</sub>{PPh<sub>2</sub>(OMe)}<sub>2</sub>] and [ReCl<sub>4</sub>{PPh<sub>2</sub>(OEt)}<sub>2</sub>] are crystalline violet products. They are air-stable and soluble in common organic solvents such as chloroform, dichloromethane, alcohols, dimethylsulfoxide (DMSO), etc. The <sup>1</sup>H NMR spectra of the paramagnetic complexes show typical paramagnetic shifts of the signal positions with

Table 1				
Crystal	data	and	structure	refinement

	$[\text{ReCl}_{4}\{\text{PPh}_{2}(\text{OMe})\}_{2}]$	$[\text{ReCl}_4\{\text{PPh}_2(\text{OEt})\}_2]$	$[\operatorname{ReCl}_4(\operatorname{OPPh}_3)_2]$
Empirical formula	$C_{26}H_{26}Cl_4O_2P_2Re$	$C_{28}H_{30}Cl_4O_2P_2Re$	$C_{36}H_{30}Cl_4O_2P_2Re$
Temperature (K)	293(2)	293(2)	223(2)
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system/space group	Monoclinic/C2/c	Monoclinic/P2 $_1/n$	Triclinic/P1
Unit cell dimensions	a=15.998(3) Å	a=9.621(1) Å	a=9.372(4) Å
	b=9.122(1) Å	b=15.756(2) Å	b=9.682(4)Å
	c=19.655(3) Å	c=10.239(2) Å	c = 10.422(5)  Å $\alpha = 102.26(2)$
	$\beta = 101.54(1)^{\circ}$	$\beta = 104.29(1)^{\circ}$	$\beta = 108.75(2)^{\circ}$ $\gamma = 93.47(2)^{\circ}$
Volume $(Å^3)$	2810.3(7)	1504.1(4)	866.6(6)
Ζ	4	2	1
Crystal size (mm <sup>3</sup> )	0.25×0.25×0.15	0.20×0.20×0.15	0.15×0.10×0.05
$\theta$ range for data collection (°)	3–27	3–27	3–25
<i>h</i> , <i>k</i> , <i>l</i> index range	0,21; 0,12; -25,25	0,11; -19,0; -12,12	-1,11; -11,11; -12,12
Reflections collected	3491	3215	3893
Independent reflections $(R_{int})$	3376 (0.0302)	3034 (0.0371)	3057 (0.0247)
Reflections observed, criterion	2397, $I > 2\sigma(I)$	1949, $I > 2\sigma(I)$	2906, I>2σ (I)
Refinement method		Full-matrix least-squares on $F^2$	
Data/restraints/parameters	3376/0/160	3034/0/209	3057/0/205
Goodness-of-fit on $F^2$	1.023	1.022	1.097
Final <i>R</i> indices $R_1$ , $wR_2$ [ $I \ge 2\sigma(I)$ ] $wR_2$ [ $I \ge 2\sigma(I)$ ]	0.0308, 0.0728	0.0307, 0.0542	0.037, 0.0754

respect to those expected for diamagnetic compounds [18]. The paramagnetism of reactives and products prevented us from monitoring the reaction course by NMR spectroscopy.

 $[\text{ReCl}_4(\text{OPPh}_3)_2]$  is formed from the reaction of  $[\text{ReNCl}_2(\text{PPh}_3)_2]$  with  $(\text{Ph}_3\text{O})(\text{BF}_4)$  in low yields. The attempted attack of the triphenyloxonium cation at the nucleophilic nitrido ligand of the rhenium(V) complex could not be observed. During a refluxing period of 24 h in CH<sub>2</sub>Cl<sub>2</sub>, however, a considerable amount of the sparingly soluble  $[\text{ReNCl}_2(\text{PPh}_3)_2]$  dissolved to give a red–brown solution, from which red crystals of  $[\text{ReCl}_4(\text{OPPh}_3)_2]$  have been isolated. The remaining, insoluble solid contained, besides the adduct, small amounts (approximately 5%) of  $[\text{ReOCl}_3(\text{PPh}_3)_2]$ , which has been detected by IR spectroscopy.

[ReCl<sub>4</sub>(OPPh<sub>3</sub>)<sub>2</sub>] is soluble in hot CH<sub>2</sub>Cl<sub>2</sub> and CHCl<sub>3</sub> and can be recrystallized from those solvents to give red plates. The compound is paramagnetic, with an effective magnetic moment of  $\mu_{eff}$ =4.21 B.M. The infrared spectrum shows the  $\nu$ (P=O) frequency at 1106 cm<sup>-1</sup>. This value is comparable with those that have been found for [Re(NCS)<sub>4</sub>(PPh<sub>3</sub>)(OPPh<sub>3</sub>)] (1116 cm<sup>-1</sup>) [19] and a number of Re<sup>11</sup> nitrosyl complexes containing triphenylphosphine oxide ligands with  $\nu$ (P=O) bands between 1105 and 1220 cm<sup>-1</sup> [20].

#### 3.1. Crystal structures

The molecular structure of the complexes *trans*-[Re- $Cl_4{PPh_2(OMe)}_2$ ] and *trans*-[ReCl\_4{PPh\_2(OEt)}\_2] are shown in Figs. 1 and 2 and important bond distances and angles are listed in the accompanying Table 1. A ZORTEP representation [21] of *trans*-[ReCl\_4(OPPh\_3)\_2] is given in Fig. 3, and the corresponding bond lengths and angles are summarized in Table 2.

In all three structures, the rhenium atoms are situated on symmetry centers imposing the essentially  $D_{4h}$  symmetry in the complexes. Therefore, all *trans* angles are equal to 180° and the *cis* angles are all close to 90°.

The Re–Cl and Re–P distances within the ReCl<sub>4</sub>P<sub>2</sub> octahedra of the phosphinite complexes are similar to those found in the structurally characterized tetrachlorobis(phosphine)rhenium(IV) derivatives *trans*-[ReCl<sub>4</sub>(PMe<sub>2</sub>Ph)<sub>2</sub>] [6], *trans*-[ReCl<sub>4</sub>(PPh<sub>3</sub>)<sub>2</sub>] [4,5], *trans*-[ReCl<sub>4</sub>(PEt<sub>3</sub>)<sub>2</sub>] [3] and *trans*-[ReCl<sub>4</sub>{P(m-tol)<sub>3</sub>}<sub>2</sub>] [8] and also agree with those in the only *cis*-diphosphine derivative [ReCl<sub>4</sub>(dppe)] [2]. All of these complexes, including those of the present work, show Re–Cl distances ranging between 2.32 and 2.40 Å and Re–P distances ranging from 2.51 to 2.55 Å. The latter, as has been reported by Engelhardt et al. ([6], see also ref. [22]), suggests that effects other than electronic effects should dominate the chlorine attachment to rhenium.

Although the rhenium kernel is very similar in the two



Fig. 1. ZORTEP [21] representation of the molecular structure of  $[ReCl_{4}{PPh_{2}(OMe)}_{2}]$ . Thermal ellipsoids represent 50% probability.

phosphinite derivatives, the ligands are oriented differently with respect to the  $\text{ReCl}_4$  plane. The vector P(1)-O(1) lies almost on the vector  $\text{Re}-\text{Cl}(2)^i$  as can be seen in the value



Fig. 2. ZORTEP [21] representation of the molecular structure of  $[ReCl_{4}{PPh_{2}(OEt)}_{2}]$ . Thermal ellipsoids represent 50% probability.

Table 2 Selected bond lengths (Å) and angles (°) for  $[ReCl_4{PPh_2(OMe)}_2]$  and  $[ReCl_4{PPh_2(OEt)}_2]$ 

	$[\operatorname{ReCl}_{4}\{\operatorname{PPh}_{2}(\operatorname{OMe})\}_{2}]^{a}$	$[\operatorname{ReCl}_{4}\{\operatorname{PPh}_{2}(\operatorname{OEt})\}_{2}]^{b}$
Re–Cl(1)	2.3209(13)	2.3273(14)
Re-Cl(2)	2.3364(12)	2.325(2)
Re-P(1)	2.5342(13)	2.555(2)
P(1) - O(1)	1.598(3)	1.600(4)
P(1)-C(21)	1.829(5)	1.802(6)
P(1) - C(11)	1.808(5)	1.821(6)
O(1)–C(1)	1.460(6)	1.441(8)
C(1)–C(2)		1.427(10)
$Cl(2)-Re-Cl(1)^{i}$	89.40(5)	90.11(6)
Cl(2)-Re-Cl(1)	90.60(5)	89.89(6)
Cl(2)-Re-P(1)	87.66(4)	90.63(6)
Cl(1)-Re-P(1)	90.10(5)	89.77(5)
$Cl(2)-Re-P(1)^{i}$	92.34(4)	89.37(6)
$Cl(1)-Re-P(1)^{i}$	89.90(5)	90.23(5)
O(1) - P(1) - C(21)	105.4(2)	100.6(3)
O(1) - P(1) - C(11)	100.3(2)	105.9(3)
C(21)-P(1)-C(11)	103.0(2)	104.8(3)
O(1)–P(1)–Re	112.7(2)	113.9(2)
C(21)-P(1)-Re	120.0(2)	112.8(2)
C(11)-P(1)-Re	113.1(2)	117.1(2)
C(1)-O(1)-P(1)	119.4(3)	123.9(4)
C(2)-C(1)-O(1)		111.6(7)

 $a^{i}i=-x+1/2, -y+1/2, -z. b^{i}i=-x, -y, -z.$ 

of the torsion angle O(1)–P(1)–Re–Cl(2)'  $[1.6(2)^{\circ}]$  in *trans*-[ReCl<sub>4</sub>{PPh<sub>2</sub>(OMe)}<sub>2</sub>] (**I**), while the same angle is wider by 24.8(2)° in compound *trans*-[Re-Cl<sub>4</sub>{PPh<sub>2</sub>(OEt)}<sub>2</sub>] (**II**).

The bonding situation in *trans*- $[\text{ReCl}_4(\text{OPPh}_3)_2]$  is

Table 3 Selected bond lengths and angles for  $[\text{ReCl}_4(\text{OPPh}_3)_2]^a$ 

Re–O(1)	2.034(4)	P(1)-C(10)	1.777(6)
$\operatorname{Re}(1) - \operatorname{Cl}(1)$	2.3438(17)	P(1)-C(20)	1.781(7)
Re(1)-Cl(2)	2.3362(16)	P(1)-C(30)	1.787(7)
P(1) - O(1)	1.444(5)		
$O(1)^{i} - Re(1) - O(1)$	180.0	P(1) - O(1) - Re(1)	156.2(3)
$O(1) - Re(1) - Cl(2)^{i}$	89.14(14)	O(1) - P(1) - C(10)	110.8(3)
O(1) - Re(1) - Cl(2)	90.86(14)	O(1) - P(1) - C(20)	106.8(3)
$O(1) - Re(1) - Cl(1)^{i}$	89.32(14)	C(10)-P(1)-C(20)	109.5(3)
$Cl(2)^{i}-Re(1)-Cl(1)^{i}$	90.33(6)	O(1) - P(1) - C(30)	112.1(3)
$Cl(2)-Re(1)-Cl(1)^{i}$	89.67(6)	C(10)-P(1)-C(30)	107.9(3)
$Cl(1)^{i}-Re(1)-Cl(1)$	180.0	C(20)-P(1)-C(30)	109.7(3)

i = -x, -y, -z.

comparable with that of the phosphinite complexes. The Re–O bond length of 2.033(4) Å is slightly longer than that in cis-[Re(NCS)<sub>4</sub>(PPh<sub>3</sub>)(OPPh<sub>3</sub>)], where a length of 2.018 Å has been found [19] but it is similar to that in the mixed halide  $[\text{ReBr}_{2.5}\text{Cl}_{1.5}(\text{OPPh}_3)_2]$  [2.030(7) Å], which was reported recently [23]. This is surprising, since the Re-N bond distances in the rhenium(IV) isothiocyanato complex suggest an increase of the structural trans influence in the order  $OPPh_3 \le NCS^- \le PPh_3$ . Re-O distances between 2.020 and 2.075 Å have been found for a number of rhenium(II) nitrosyl compounds with triphenylphosphine oxide ligands [20]. For those complexes, shorter bonds are also evident when PPh<sub>3</sub> or OPPh<sub>3</sub> are situated in a cis-arrangement to the triphenylphosphine oxide ligand regarded. The longest Re-OPPh3 bond has been found in a *trans* position to NO<sup>+</sup>.



Fig. 3. ZORTEP [21] representation of the molecular structure of [ReCl<sub>4</sub>(OPPh<sub>3</sub>)<sub>2</sub>]. Thermal ellipsoids represent 50% probability.

#### Supporting information available:

More details of the crystal structure determinations have been deposited with the Cambridge Crystallographic Data Center with the deposition numbers 104994 ([ReCl<sub>4</sub>{PPh<sub>2</sub>(OMe)}<sub>2</sub>]), 104995([ReCl<sub>4</sub>{PPh<sub>2</sub>(OEt)}<sub>2</sub>]) and 104996 ([ReCl<sub>4</sub>(OPPh<sub>3</sub>)<sub>2</sub>]).

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