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# The synthesis, spectroscopic investigation, crystal and molecular structure of the [ReBrCl<sub>2</sub>(SbPh<sub>3</sub>)<sub>3</sub>] complex

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### Abstract

The  $[\text{ReBrCl}_2(\text{SbPh}_3)_3]$  complex has been obtained in the reaction of  $[\text{Bu}_4^t\text{N}]_2[\text{ReBr}_3\text{Cl}_2(\text{NO})]$  with an excess of triphenylstibine. The halide ions and stibine molecules of  $[\text{ReBrCl}_2(\text{SbPh}_3)_3]$  are arranged in a *meridional* fashion. The substitution reactions of the nitrosyl ligand are very rare so the formation of the  $[\text{ReBrCl}_2(\text{SbPh}_3)_3]$  compound in this reaction is particularly interesting. © 2003 Elsevier Science B.V. All rights reserved.

Keywords: Rhenium(III); Stibine; Nitrosyl complexes; X-ray structure

# 1. Introduction

The widespread contemporary interest in the coordination chemistry of rhenium is motivated by the perspective of replacing technetium-99m with rhenium-186/188 for use as a diagnostic or radiotherapeutic agent [1–5]. Due to key role of nitric oxide in human cardiovascular and nervous systems and in immune response to pathogen invasion [6–9] the rhenium nitrosyl complexes are of special importance.

Previously, we investigated the reactivity of  $[ReOX_3L_2]$ compounds (X = Cl and Br, L = PPh<sub>3</sub> and AsPh<sub>3</sub>) towards gaseous nitric oxide and the reactions of  $[Bu_4^tN]_2[ReX_5$ (NO)] with an excess of triphenylphosphine and triphenylarsine [10–18]. The  $[ReOX_3L_2]$  complexes react with NO to give various products, depending on the reaction conditions:  $[ReX_3(NO)(OPPh_3)_2]$ ,  $[ReX_3(NO)(PPh_3)_2]$ ,  $[ReX_2(NO)_2(PPh_3)_2]$ ,  $[ReBr_3(NO)(MeCN)(PPh_3)]$ , [Re- $Cl_3(NO)(PPh_3)(OPPh_3)]$ ,  $[ReCl_4(PPh_3)_2]$ ,  $[ReBr_3(NO)(dppe)]$  $[ReBr_4(dppe)]$ ,  $[ReX_3(NO)(OAsPh_3)_2]$ ,  $[ReCl_4(OAsPh_3)_2]$ and  $[ReCl_3(NO)(AsPh_3)_2][ReCl_4(AsPh_3)_2]$ . The reactions of  $[Bu_4^tN]_2[ReX_5(NO)]$  with PPh<sub>3</sub> and AsPh<sub>3</sub> lead to  $[ReBr_3(NO)(PPh_3)_2]$ ,  $[ReCl_3(NO)(PPh_3)_2]$ ,  $[ReBr_3(NO)$  $(AsPh_3)_2]$  and  $[ReCl_3(NO)(AsPh_3)_2][ReCl_4(AsPh_3)_2]$ , respectively. Now we are extending our studies to the examination of the reaction of  $[Bu_4^tN]_2[ReBr_3Cl_2(NO)]$  with triphenylstibine. The formation of the  $[ReBrCl_2(SbPh_3)_3]$ compound in this reaction is particularly interesting since substitution reactions of the nitrosyl ligand are very rare observed [19–21]. Here, we present the synthesis, spectroscopic and structural characterisation of the  $[ReBrCl_2(SbPh_3)_3]$  complex.

## 2. Experimental

Triphenylstibine, triphenylphosphine and NH<sub>4</sub>ReO<sub>4</sub> were purchased from Aldrich Chemical Co. and used without further purification. The [ReO(OEt)Cl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>] complex was synthesised according to the literature method [22]. Gaseous NO, obtained in the reaction: 2NaNO<sub>2</sub>+3H<sub>2</sub>SO<sub>4</sub>+FeSO<sub>4</sub>  $\rightarrow$  2NO+2NaHSO<sub>4</sub>+Fe<sub>2</sub> (SO<sub>4</sub>)<sub>3</sub> + 2H<sub>2</sub>O, was purified by passing through the washers with concentrated KOH solution and over solid NaOH. The reaction was performed under argon atmosphere.

#### 2.1. Synthesis of $[NBu_4]_2[ReBr_3Cl_2(NO)]$

The  $[Bu_4^tN]_2[ReBr_3Cl_2(NO)]$  complex was obtained according to the modified synthesis of Giusto and coworkers [23].  $[ReO(OEt)Cl_2(PPh_3)_2]$  (1.5 g, 1.8 mmol)

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was added to a solution of  $[Bu_4^tN]Br$  (3.30 g, 10 mmol) in ethanol (60 cm<sup>3</sup>). Nitrogen oxide was bubbled under reflux for 3 h. The reaction solution was evaporated to the volume of 10 cm<sup>3</sup> and green precipitate of  $[Bu_4^tN]_2[ReBr_3Cl_2(NO)]$  was formed by an addition of 60 cm<sup>3</sup> of diethylether and after overnight it was filtered off. The product was washed with cold ether and dried in vacuo. The yield 25%.

IR (KBr, cm<sup>-1</sup>) 3049 (m), 2999(w) 1736 (s), 1481 (br, m), 1438 (sh), 1381 (m), 1261 (w), 1155 (m), 1119 (w), 1093 (w), 1029 (w), 882 (m) 803 (m), 748 (w), 725 (m), 695 (m), 545 (m), 534 (m).

Anal. Found: C, 14.48; H, 2.77; N, 6.35. Calc. for C<sub>8</sub>H<sub>18</sub>N<sub>3</sub>OBr<sub>3</sub>Cl<sub>2</sub>Re: C, 14.36; H, 2.71; N, 6.28.

#### 2.2. Synthesis of $[ReBrCl_2(SbPh_3)_3]$

A mixture of  $[Bu_4^tN]_2[ReBr_3Cl_2(NO)]$  (1 g, 1.5 mmol) and SbPh<sub>3</sub> (5.3 g, 15 mmol) in ethanol (50 cm<sup>3</sup>) was refluxed for 12 h. The formed yellow-green precipitate was filtered and washed with ether. X-ray quality crystals were obtained by recrystallization from a mixture of chloroform and ethanol. Yield 65%.

IR (KBr, cm<sup>-1</sup>) 3052 (m), 2898 (w), 1480 (s), 1431 (s), 1332 (w), 1303 (w), 1264 (w), 1186 (w), 1158 (w), 1067 (s), 1020 (m) 998 (w), 728 (s), 692 (s), 658 (w), 451 (s).

Found: C, 46.32; H, 3.23. Calc. for C<sub>54</sub>H<sub>45</sub>Sb<sub>3</sub>BrCl<sub>2</sub> Re: C, 46.45; H, 3.25.

#### 2.3. Physical measurements

UV–VIS spectra were recorded on a spectrophotometer Lab Alliance UV–VIS 8500 in the range 800– 220 nm in deoxygenated dichloromethane solution. IR spectra were recorded as KBr pellets with a Nicolet Magna 560 spectrophotometer in the spectral range 4000–400 cm<sup>-1</sup>. Elemental analyses (C H N) were performed on a Perkin–Elmer CHN-2400 analyzer. Magnetic susceptibilities were measured at 296 K by the Faraday method.

#### 2.4. Crystal structures determination and refinement

The X-ray intensity data were collected with graphite monochromated Mo-K<sub> $\alpha$ </sub> radiation ( $\lambda = 0.71073$  Å) at room temperature on a Kuma Diffraction KM4 diffractometer and processed with Omnibus-BLP data processing program [24]. Details concerning crystal data and refinement are given in Table 1. Lorentz polarization, empirical absorption corrections were applied. The structures were solved by means of the Patterson and Fourier methods. All the non-hydrogen atoms were refined anisotropically using full-matrix, least-squares technique. The hydrogen atoms of the phenyl rings were treated as "riding" on their parent carbon atoms [d(C-H) = 0.96 Å] and assigned isotropic temperature factors

Γal	ble	1	

Crystal data and structure refinement for [ReBrCl<sub>2</sub>(SbPh<sub>3</sub>)<sub>3</sub>]

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Empirical formula	C54H45Sb3BrCl2Re	
Formula weight	1396.16	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	$a = 13.410(3) \text{ Å}; \alpha = 90^{\circ}$	
	$b = 16.215(3) \text{ Å}; \beta = 90^{\circ}$	
	$c = 22.683(5) \text{ Å}; \gamma = 90^{\circ}$	
Volume	4932.3(18) Å <sup>3</sup>	
Ζ	4	
Density (calculated)	1.880 M g/m <sup>3</sup>	
Absorption coefficient	$5.025 \text{ mm}^{-1}$	
F(000)	2664	
Crystal size	0.32×0.26×0.21 mm	
$\theta$ range for data collection	1.54–25.06°	
Index ranges	$-2 \leq h \leq 15$	
	$0 \leq k \leq 19$	
	$-2 \leq l \leq 27$	
Reflections collected	6210	
Independent reflections	5912 ( $R_{\rm int} = 0.0331$ )	
Max. and min. transmission	0.4184 and 0.2962	
Completeness to $2\theta$	99.9%	
Data/restraints/parameters	5912/0/551	
Goodness-of-fit on $F^2$	1.131	
Final <i>R</i> indices $[I > 2\sigma(I)]$	R1 = 0.0504	
	wR2 = 0.1013	
R indices (all data)	R1 = 0.0760	
	wR2 = 0.1134	
Absolute structure parameter	-0.024(13)	
Largest diff. peak and hole	1.435 and $-0.877 \text{ e} ^{-3}$	

equal 1.2 times the value of equivalent temperature factor of the parent carbon atom. SHELXL97 [25] and SHEL-XTL [26] programs were used for all the calculations.

#### 3. Results and discussion

The reactions of  $[Bu_4^t N]_2 [ReX_5(NO)]$  (X = Cl or Br) with an excess of PPh<sub>3</sub> and AsPh<sub>3</sub> in ethanol produced  $[ReCl_3(NO)(PPh_3)_2], [ReBr_3(NO)(PPh_3)_2], [ReBr_3(NO)$  $(AsPh_3)_2$  and  $[ReCl_3(NO)(AsPh_3)_2][ReCl_4(AsPh_3)_2]$ complexes, respectively [15,17,18]. The [ReX<sub>3</sub>(NO)  $(EPh_3)_2$  complexes (E = P or As) have pseudooctahedral geometry with trans-phosphine or arsine ligands, meridional halide ligands and a linear nitrosyl group. The nitrosyl stretching frequencies in [ReX<sub>3</sub>(NO)(EPh<sub>3</sub>)<sub>2</sub>] compounds are nearly independent of E, it depends only on X ligands: 1776 cm<sup>-1</sup> for [ReBr<sub>3</sub>(NO)(PPh<sub>3</sub>)<sub>2</sub>], 1773  $cm^{-1}$  for [ReBr<sub>3</sub>(NO)(AsPh<sub>3</sub>)<sub>2</sub>], 1734 cm<sup>-1</sup> for [Re- $Cl_3(NO)(PPh_3)_2$  and 1738 cm<sup>-1</sup> for [ReCl<sub>3</sub>(NO)  $(PPh_3)_2$ ][ReCl<sub>4</sub>(AsPh<sub>3</sub>)<sub>2</sub>]. The lower values of  $v_{NO}$  for chloro-compounds than for the corresponding bromocomplexes is in accordance with the greater  $\pi$ -acceptor character of bromo ion than chloro one.

The nitrosyl [ReBrCl<sub>2</sub>(NO)(SbPh<sub>3</sub>)<sub>2</sub>] complex has not been isolated in the reaction of *mer*,*trans*-[Bu<sup>t</sup><sub>4</sub>N]<sub>2</sub>

[ReBr<sub>3</sub>Cl<sub>2</sub>(NO)] with triphenylstibine in ethanol but the formation of the nitrosyl has been confirmed by monitoring of the reaction via IR spectroscopy. However, the presence of  $v_{\rm NO}$  at much lower frequencies (1680 cm<sup>-1</sup>) suggests the different ligands' arrangement around the rhenium center in the stibine nitrosyl in comparison with phosphine and arsine [ReX<sub>3</sub>(NO)(EPh<sub>3</sub>)<sub>2</sub>] complexes. It is most likely that the stibine ligands are in cis-geometry and the nitosyl group occupies the trans-position to SbPh<sub>3</sub> molecule. The *cis*-arrangement of SbPh<sub>3</sub> molecules in [ReCl(CO)<sub>3</sub>(SbPh<sub>3</sub>)<sub>2</sub>] was confirmed by a crystal structure determination [27]. The PPh<sub>3</sub> molecules of the analogous [ReCl(CO)<sub>3</sub>(PPh<sub>3</sub>)<sub>2</sub>] carbonyl are in transgeometry [28]. It is known that the increase of M-E and E-C bond lengths in the order P>As>Sb leads to the decrease of the EPh<sub>3</sub> cone angles [29]. The smaller size of SbPh<sub>3</sub> in comparison with PPh<sub>3</sub> and AsPh<sub>3</sub> can be used to explain the *cis*-arrangement of SbPh<sub>3</sub> molecules in  $[ReCl(CO)_3(SbPh_3)_2]$  and the formation of  $[ReBrCl_2]$  $(SbPh_3)_3$  in the reaction of  $[Bu_4^t N]_2$  [ReBr<sub>3</sub>Cl<sub>2</sub>(NO)] with SbPh<sub>3</sub>. The Re-E and E-C bond lengths and C-E-C angles (E = P or As) for selected rhenium complexes are summarised in Table 2.

The [ReBrCl<sub>2</sub>(SbPh<sub>3</sub>)<sub>3</sub>] complex crystallises in the orthorhombic space group  $P2_12_12_1$  and its structure consists of discrete and well-separated monomers. Fig. 1 shows the molecular structure of this compound and the selected bond lengths and angles for [ReBrCl<sub>2</sub>(SbPh<sub>3</sub>)<sub>3</sub>] are summarized in Table 3. The two groups of ligands of [ReBrCl<sub>2</sub>(SbPh<sub>3</sub>)<sub>3</sub>] (halide ions and stibine molecules) are arranged in a meridional fashion. Due to steric repulsion of three bulky SbPh<sub>3</sub> molecules the octahedral Re geometry shows the expected angular distortions. The angles between *cis*-ligands vary between 85.12(6)° and 96.00(10)°, while the *trans*-angles are between  $170.00(4)^{\circ}$  and  $178.13(16)^{\circ}$ . The conformations of [Re-BrCl<sub>2</sub>(SbPh<sub>3</sub>)<sub>3</sub>] molecules are stabilised by two C- $H \cdots Cl$  weak intramolecular hydrogen bonds [31]: C(14)–H(14A)···Cl(2) with D···A distance equal 3.56(2)A and D-H···A angle –  $153.3^\circ$ ; C(8)-H(8A)···Cl(1) with D···A distance equal 3.52 (2) Å and D-H···A angle - 152.9°.

There are very few Re–Sb distances reported in the Cambridge structural database with [ReCl<sub>2</sub>(NO)<sub>2</sub>





The Re–E and E–C bond lengths and C–E–C angles (E=P, As or Sb) for selected rhenium complexes

Complex	Re–E bond length (average, [Å])	E–C bond length (average, [Å])	C-E-C angle (average, [°])	Reference
[Re(NO)Cl <sub>3</sub> (PPh <sub>3</sub> ) <sub>2</sub> ]	2.530	1.822	104.3	[15]
$[Re(NO)Br_3(PPh_3)_2]$	2.540	1.822	104.3	[17]
[Re(NO)Cl <sub>3</sub> (AsPh <sub>3</sub> ) <sub>2</sub> ]	2.612	1.942	103.4	[18]
[Re(NO)Br <sub>3</sub> (AsPh <sub>3</sub> ) <sub>2</sub> ]	2.595	1.935	103.7	[18]
[ReCl(CO) <sub>3</sub> (SbPh <sub>3</sub> )]	2.728	2.137	100.1	[27]
$[\text{ReCl}_2(\text{NO})_2(\text{SbPh}_3)_2]$	2.711	2.114	102.5	[30]



Table 3 Selected bond lengths  $[\mathring{A}]$  and angles  $[^\circ]$  for  $[ReBrCl_2(SbPh_3)_3]$ 

Bond lengths		Angles	
Re(1)-Br(1)	2.5962(19)	Cl(2)-Re(1)-Cl(1)	178.13(16)
Re(1)-Cl(1)	2.466(5)	Cl(2)-Re(1)-Br(1)	87.27(14)
Re(1)-Cl(2)	2.407(7)	Cl(1)-Re(1)-Br(1)	91.06(11)
Re(1)-Sb(1)	2.6694(13)	Cl(2)-Re(1)-Sb(1)	85.66(13)
Re(1)-Sb(2)	2.6830(13)	Cl(1)-Re(1)-Sb(1)	96.00(10)
Re(1)-Sb(3)	2.6942(14)	Br(1)-Re(1)-Sb(1)	172.91(7)
Sb(1)-C(1)	2.121(17)	Cl(2)-Re(1)-Sb(2)	90.60(14)
Sb(1)–C(7)	2.152(17)	Cl(1)-Re(1)-Sb(2)	88.44(10)
Sb(1)–C(13)	2.180(18)	Br(1)-Re(1)-Sb(2)	85.83(6)
Sb(2)–C(19)	2.119(16)	Sb(1)-Re(1)-Sb(2)	93.66(4)
Sb(2)-C(25)	2.130(18)	Cl(2)-Re(1)-Sb(3)	93.20(14)
Sb(2)–C(31)	2.132(14)	Cl(1)-Re(1)-Sb(3)	87.50(10)
Sb(3)-C(37)	2.132(16)	Br(1)-Re(1)-Sb(3)	85.12(6)
Sb(3)–C(43)	2.134(18)	Sb(1)-Re(1)-Sb(3)	95.85(4)
Sb(3)-C(49)	2.104(14)	Sb(2)-Re(1)-Sb(3)	170.00(4)
		C(1)-Sb(1)-C(7)	100.9(7)
		C(1)-Sb(1)-C(13)	98.4(7)
		C(7)–Sb(1)–C(13)	99.3(7)
		C(1)-Sb(1)-Re(1)	115.8(4)
		C(7)-Sb(1)-Re(1)	123.4(5)
		C(13)-Sb(1)-Re(1)	115.0(5)
		C(19)-Sb(2)-C(25)	103.1(7)
		C(19)-Sb(2)-C(31)	94.1(7)
		C(25)–Sb(2)–C(31)	99.6(7)
		C(19)-Sb(2)-Re(1)	119.4(4)
		C(25)-Sb(2)-Re(1)	117.2(5)
		C(31)-Sb(2)-Re(1)	119.4(4)
		C(43)-Sb(3)-C(49)	101.3(7)
		C(43)-Sb(3)-C(37)	99.2(6)
		C(49)-Sb(3)-C(37)	95.7(7)
		C(43)-Sb(3)-Re(1)	117.2(4)
		C(49)-Sb(3)-Re(1)	116.5(4)
		C(37)-Sb(3)-Re(1)	122.8(5)

observed in *mer*-[OsBr<sub>3</sub>(SbPh<sub>3</sub>)<sub>3</sub>] [33], in which the Os–Br *trans* to SbPh<sub>3</sub> [2.522(2) Å] is considerable longer than the others 2.507(2) and 2.508(2) Å.

The complex [ReBrCl<sub>2</sub>(SbPh<sub>3</sub>)<sub>3</sub>] shows room temperature magnetic moment of 1.5 BM. This value is characteristic of mononuclear complexes with low-spin rhenium(III) (d<sup>4</sup>) ions in O<sub>h</sub> field [34], and arises because of the large spin–orbit coupling [35]. Magnetic moment corresponding with two unpaired electrons may be achieved only at very low temperature, because it is a condition favoring the triplet state.

The positions and molar absorption coefficients of electronic bands for 1 and the electronic transitions assigned to the bands are shown in Table 4.

[ReBrCl<sub>2</sub>(SbPh<sub>3</sub>)<sub>3</sub>] complex, in spite of its low symmetry (C<sub>2</sub>), behaves as an octahedron, because of a strong spin–orbital coupling ( $\lambda = 4592 \text{ cm}^{-1}$ ). That is why the absorption bands together with their attributions indicate a pseudo-octahedral environment of the central atom. Comparatively intense bands, connected with intercombination electron transitions, are caused by the spin–orbital coupling. As a result of the coupling, term <sup>3</sup>T<sub>1</sub> splits into singlet components, and mixing of

Table 4

Band positions, molar absorption coefficients and assignments for [ReBrCl<sub>2</sub>(SbPh<sub>3</sub>)<sub>3</sub>]

Band position [cm <sup>-1</sup> ]	3	Assignment
23,450	790	${}^{3}T_{1} \rightarrow {}^{1}T_{2}$
26,300	680	$^{3}T_{1}\rightarrow ^{1}A_{1}$
34,980	14,000	${}^{3}T_{1} \rightarrow {}^{3}E; \pi \rightarrow \pi^{*}_{Re-Sb}$
44,030	20,625	${}^{3}T_{1} \rightarrow {}^{3}T_{2}; \pi^{b}_{C_{6}H_{5}} \rightarrow 5d_{Sb}$
45,500	17,425	$\pi \to \pi^*_{\mathrm{C_6H_5}}$

 $\varepsilon = \text{molar absorption coefficient } [\text{dm}^3 \,\text{mol}^{-1} \,\text{cm}^{-1}].$ 

terms  ${}^{1}T_{2}$  and  ${}^{1}A_{1}$  with  ${}^{3}T_{1}$  causes, in some degree, singlet-singlet character of the intercombination transitions. The central ion of Re(III) has the following electron configuration  $(d_{xy})^2(d_{xz})^1(d_{yz})^1$ . Re–Sb bond in trans-position towards Re-Br is ca. 0.023 Å shorter in comparison with the other Re-Sb bonds. This fact confirms formation of  $\pi$ (Re–Sb) bond with participation of donor electrons from  $d_{xz}$  and  $d_{yz}$  orbitals of the rhenium atom. Obviously, the electrons occupying  $d_{xy}$  orbital of the central atom also participate in Re-Sb bonds formation, but the number of donor electrons per one Sb atom, lower by half, results in longer Re–Sb bonds in trans-position. High degree of covalence of the complex is confirmed by values of  $D_q = 3498 \text{ cm}^{-1}$ ,  $B = 440 \text{ cm}^{-1}$ and  $C = 1758 \text{ cm}^{-1}$ . Significantly lower molar extinction coefficient of the band connected with  $\pi^b_{C_6H_5} \rightarrow 5d_{Sb}$ transition, compared with an analogous electron transfer towards  $3d_P$ , also confirms the participation of  $5d_{Sb}$ acceptor orbitals in the formation of  $\pi$ (Re–Sb) bond. It lowers the probability of  $\pi^{b}_{C_{6}H_{5}} \rightarrow 5d_{Sb}$  transition.

#### Supplementary data

Supplementary data of [ReBrCl<sub>2</sub>(SbPh<sub>3</sub>)<sub>3</sub>] complex are available from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK on request, quoting the deposition number 205379.

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