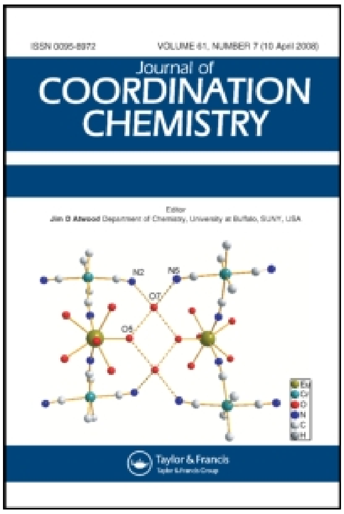

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Different coordination modes of tetradentate Schiff bases in monomeric and dimeric oxorhenium(V) complexes

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Different coordination modes of tetradentate Schiff bases in monomeric and dimeric oxorhenium(V) complexes

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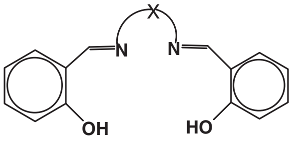
The reactions of the tetradentate amine-phenol type Schiff bases H_2sal_2en (1,2-ethylenebis(salicylideneimine) and H_2sal_2mp (1,2-benzylenebis(salicylideneimine)) with *trans*-$[ReOCl_3(PPh_3)_2]$ or (n-Bu_4N)$[ReOCl_4]$ in air gave the products (μ-O)$[ReO(sal_2en)]_2$ (**1**) and $[ReOCl(sal_2mp)]$ (**2**), respectively. X-ray and spectroscopic studies have shown that **1** contains the linear O=Re–O–Re=O grouping, with the four donor atoms of sal_2en^{2-} coordinating in the square plane *cis* to the oxo ions. In **2**, a *cis* oxo-chloro arrangement is observed with a phenolic oxygen being coordinated *trans* to the oxo group. The terminal Re=O bond lengths in **1** and **2** are 1.709(4) and 1.683(3) Å, respectively.

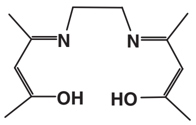
Keywords: Oxorhenium(V); Tetradentate N_2O_2 Schiff bases; Crystal structure

1. Introduction

Rhenium(V) and technetium(V) complexes of classical tetradentate amine-phenol and amine-acetylacetone Schiff bases have been well studied during the last 25 years, and the realization that this particular field has now been fully explored is accepted [1–5].



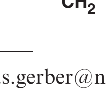
H_2sal_2nxn



H_2acac_2en

X=CH_2CH_2 H_2sal_2en

=



H_2sal_2mp

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It was shown that complexes of the general formula [MOCl(sal$_2$nxn)] and [MOCl(acac$_2$nxn)] (M = Re, Tc) are readily obtained by reaction of *trans*-[ReOCl$_3$(PPh$_3$)$_2$] or (*n*-Bu$_4$N)[ReOCl$_4$] with H$_2$sal$_2$nxn and H$_2$acac$_2$en, respectively, if air and moisture are excluded from the reaction mixtures [2, 4]. It was shown by X-ray and spectroscopic studies [2–4] that these complexes exhibit distorted octahedral coordination geometry, with a *trans* oxo-chlorometallate(V) core. Another study, however, has shown that complexes of the [ReOCl(sal$_2$nxn)] type may also have the *cis* oxo-chlororhenate arrangement if the bridging unit X contains three carbon atoms [5]. However, if reactions are carried out in air and wet solvents—so that some water is always present—complexes of the type [Re$_2$O$_3$L$_2$] (L = sal$_2$nxn^{2-}, acac$_2$en^{2-}) are formed, with a linear O=Re–O–Re=O grouping.

We have studied the reactions of the tetradentate ligands H$_2$sal$_2$en and H$_2$sal$_2$mp with *trans*-[ReOCl$_3$(PPh$_3$)$_2$] and (*n*-Bu$_4$N)[ReOCl$_4$] in air. With the former ligand, which contains two carbons bridging the imino groups, the μ-oxo dimer [Re$_2$O$_3$(sal$_2$en)$_2$] (**1**) was isolated. With the latter, the monomeric complex [ReOCl(sal$_2$mp)] (**2**) was obtained. An oxo-bridged dimer of formula [Re$_2$O$_3$(sal$_2$mp)$_2$] could not be isolated. Complex **1** was reported earlier [1], but, to our knowledge, its molecular structure has not been published.

2. Experimental

2.1. Reagents *and instrumentation*

Trans-[ReOCl$_3$(PPh$_3$)$_2$] and (*n*-Bu$_4$N)[ReOCl$_4$] were prepared by literature procedures [6, 7]. Salicylaldehyde, 1,2-diaminoethane and 2-aminobenzylamine were obtained commercially (Aldrich). The ligands *N*,*N*′-ethylenebis(salicylideneimine) (H$_2$sal$_2$en) and 1,2-benzylenebis(salicylideneimine) (H$_2$sal$_2$mp) were prepared as previously reported, by condensation of two equivalents of salicylaldehyde with the respective diamine in absolute ethanol [8]. They were recrystallized from ethanol. All other chemicals and solvents were of reagent grade and used as received from commercial sources. All reactions and manipulations were carried out in air. The scientific instrumentation used is the same as reported earlier [9]. IR spectra were obtained using KBr discs and ^{1}H NMR spectra were run in *d*$_6$-DMSO.

2.2. Synthesis *of (μ-O)[ReO(sal$_2$en)]$_2$ (1)*

To a suspension of 0.100 g (120 μmol) of *trans*-[ReOCl$_3$(PPh$_3$)$_2$] in 20 cm^3 of methanol was added 0.064 g (240 μmol) of H$_2$sal$_2$en. The mixture was heated under reflux for approximately 2 h, then cooled to room temperature, to give a green solution. Slow evaporation of the mother liquor over a period of 2 days at room temperature gave dark green crystals, which were suitable for X-ray diffraction studies. The crystals were removed by filtration, washed with toluene (3 × 3 cm^3) and diethylether (3 × 3 cm^3), and dried under vacuum. Yield = 0.038 g (66%, based on Re), m.p. = 307°C. Anal. Calcd (%): C, 40.33; H, 2.96; N, 5.88. Found: C, 40.27; H, 2.76; N, 5.74. IR (cm^{-1}): ν(Re=O) 968; ν(C=N) 1643, 1601; ν(C–O) 1281; ν_{as}(Re–O–Re) 702; ν(Re–N) 475; ν(Re–O) 393. ^{1}H NMR (ppm): 7.96 (s, 2H, *H*(7)*H*(10)), 7.38 (t, 2H, *H*(3)*H*(14)), 6.88

(d, 2H, *H*(2)*H*(15)), 6.78 (d, 2H, *H*(5)*H*(12)), 6.54 (t, 2H, *H*(4)*H*(13)), 2.52 (m, 4H, C(8)H_2, C(9)H_2).

2.3. Synthesis *of [ReOCl(sal$_2$mp)] (2)*

H_2sal$_2$mp (0.112 g, 340 µmol) was dissolved in 10 cm^3 of ethanol and added to 0.100 g (170 µmol) of (*n*-Bu_4N)[$ReOCl_4$] in 10 cm^3 of ethanol. The resulting solution was heated under reflux for 90 min and then cooled to room temperature. A greed solid was collected by filtration, washed with ethanol (3 × 5 cm^3), followed by diethylether (3 × 5 cm^3), and dried under vacuum. X-ray quality crystals were obtained by recrystallization of **2** from acetonitrile/ethanol (2 : 1). Yield = 0.068 g (71%), m.p. = 155°C. Anal. Calcd (%): C, 44.56; H, 2.85; N, 4.95. Found: C, 44.71; H, 3.02; N, 4.86. IR (cm^{-1}): ν(Re=O) 964; ν(C=N) 1614, 1602; ν(C–O) 1301, 1285; ν(Re–N) 486; ν(Re–O) 390(br). ^{1}H NMR (ppm): 10.26 (s, 1H, *H*(15)), 9.05 (s, 1H, *H*(7)), 7.70 (d, 1H, *H*(20)), 7.63–7.68 (m, 4H, *H*(2)*H*(4)*H*(11)*H*(13)), 7.51 (d, 1H, *H*(17)), 7.29 (t, 1H, *H*(18)), 7.25 (d, 1H, *H*(5)), 7.11 (t, 1H, *H*(19)), 6.99 (t, 1H, *H*(3)), 6.95 (t, 1H, *H*(12)), 6.85 (d, 1H, *H*(10)), 2.09 (s, 2H, C(8)H_2).

2.4. X-ray *structure*

Intensity data collection was performed at 200 K on a Nonius Kappa CCD detector system using the ω scan technique with Mo Kα radiation. Intensities were corrected for Lorentz and polarization effects. The structures were solved by direct methods and were refined by full-matrix least-squares procedures using the SHELXL-97 package [10]. Crystal and structure refinement data for **1** and **2** are given in table 1. Non-hydrogen atoms were refined with anisotropic displacement parameters; hydrogen atoms were idealized and fixed. Selected bond lengths and angles are given in tables 2 (for **1**) and 3 (for **2**).

3. Results and discussion

3.1. Synthesis

The complex (µ-O)[ReO(sal$_2$en)]$_2$ (**1**) was synthesized by reaction of *trans*-[$ReOCl_3(PPh_3)_2$] with two molecular equivalents of H_2sal$_2$en in refluxing methanol, in air with trace amounts of water present. Under the same reaction conditions, but with (*n*-Bu_4N) [$ReOCl_4$] and H_2sal$_2$mp as reactants in ethanol, the monomeric complex [ReOCl(sal$_2$mp)] (**2**) was obtained as the sole product. This intimates that the four donor atoms of sal$_2$en^{2-} in **1** coordinate in the square plane *cis* to the oxo group, with a labile chloride in the *trans* site, which is then substituted by a water molecule, the double deprotonation of which leads to complex **1**. In **2**, the *trans* position to the oxo group is occupied by a deprotonated phenolic oxygen, preventing formation of the linear O=Re–O–Re=O moiety. In fact, complexes **1** and **2** were obtained regardless of starting complexes [$ReOCl_3(PPh_3)_2$] or [$ReOCl_4$]$^-$. With equimolar quantities of reactants, [$ReO(OR)Cl_2(PPh_3)_2$] (R = Me, Et) was obtained as the sole product, suggesting that the second equivalent of H_2sal$_2$nxn acts as a base to deprotonate the phenolic oxygen atom. Both complexes are diamagnetic and non-electrolytes in DMF, and are only soluble in the polar solvents acetonitrile, DMSO and DMF.

Table 1. Crystal data and structure refinement details for **1** and **2**.

	1	**2**
Formula	$C_{32}H_{28}N_4O_7Re_2$	$C_{12}H_{16}ClN_2O_3Re$
Formula weight	953	566.02
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	$P\bar{1}$
Unit cell dimensions (Å,°)		
a	10.287(1)	9.7610(2)
b	11.710(1)	10.1150(2)
c	12.747(1)	10.1584(4)
α		82.297(1)
β	105.92(1)	87.063(1)
γ		67.410(2)
Volume (Å^3)	1476.6(3)	917.66(5)
Z	2	2
Density (calc.; Mg m^{-3})	2.144	2.049
Crystal size (mm)	0.09 × 0.16 × 0.30	0.0 × 0.08 × 0.16
Absorption coefficient (mm^{-1})	8.247	6.793
$F(000)$	908	544
Theta range	2.1–28.1	3.4–26.0
Index ranges	$-13 \le h \le 13$	$-11 \le h \le 12$
	$-15 \le k \le 14$	$-11 \le k \le 12$
	$-16 \le \ell \le 16$	$-12 \le \ell \le 12$
Reflections collected/unique/R(int)	12434/3531/0.072	10845/3562/0.049
Observed data [$I > 2.0\sigma(I)$]	2470	3046
Data/parameters	3531/205	3562/253
Goodness-of-fit on F^2	0.86	0.98
R, wR_2	0.0311, 0.0611	0.0290, 0.0485
Largest diff peak/hole (e Å^{-3})	0.64/−1.66	1.24/−0.82

Table 2. Selected bond lengths (Å) and angles (°) for **1**.

Re–O(1)	2.033(4)	Re–O(2)	2.035(4)
Re–O(3)	1.709(4)	Re–O(4)	1.9176(3)
Re–N(1)	2.056(4)	Re–N(2)	2.057(5)
N(1)–C(7)	1.278(8)	N(2)–C(10)	1.278(9)
C(1)–O(1)	1.327(7)	C(16)–O(2)	1.327(8)
N(1)–C(8)	1.466(7)	N(2)–C(9)	1.489(7)
C(8)–C(9)	1.518(9)	C(6)–C(7)	1.446(8)
O(3)–Re–O(4)	171.3(1)	Re–O(4)–Re	180.00
O(3)–Re–O(1)	97.6(2)	O(3)–Re–O(2)	96.3(2)
O(3)–Re–N(1)	94.3(2)	O(3)–Re–N(2)	89.3(2)
N(1)–Re–N(2)	81.9(2)	O(1)–Re–O(2)	90.2(2)
N(1)–Re–O(1)	92.9(2)	N(2)–Re–O(2)	93.8(2)
O(1)–Re–N(2)	171.6(2)	N(1)–Re–O(2)	168.5(2)
C(7)–N(1)–C(8)	122.8(4)	C(9)–N(2)–C(10)	123.8(5)
N(1)–C(7)–C(6)	124.7(5)	N(2)–C(10)–C(11)	125.7(6)
Re–O(1)–C(1)	121.0(3)	Re–O(2)–C(16)	122.8(4)

3.2. Spectroscopy

The IR spectrum of **1** displays the Re=O stretch as a weak band at 968 cm^{-1}. Weak or medium intensity absorptions corresponding to ν(Re=O) have been observed previously for dimeric Re(V) species [4, 11]. However, the dominant feature of the spectrum is the presence of a strong and intense absorption at 702 cm^{-1}, ascribed to the asymmetric Re–O–Re stretch [4, 11]. The tetradentate coordination of sal$_2$en^{2-} in **1**

Table 3. Selected bond lengths (Å) and angles (°) for **2**.

Re–O(3)	1.683(3)	Re–O(2)	1.997(3)
Re–O(1)	2.003(3)	Re–N(1)	2.080(3)
Re0–Cl	2.420(1)	Re–N(2)	2.105(4)
N(1)–C(7)	1.295(6)	N(2)–C(15)	1.292(6)
N(1)–C(8)	1.487(6)	C(15)–C(16)	1.445(6)
O(1)–C(1)	1.320(6)	O(2)–C(21)	1.334(6)
C(8)–C(9)	1.518(6)	N(2)–C(14)	1.451(5)
O(2)–Re–O(3)	170.5(1)	O(3)–Re–O(1)	101.1(1)
O(3)–Re–N(1)	94.5(1)	O(3)–Re–N(2)	91.0(1)
O(3)–Re–Cl	92.0(1)	Re–O(2)–C(2)	131.2(3)
O(1)–Re–N(2)	167.8(1)	N(1)–Re–Cl	173.5(1)
N(1)–Re–N(2)	86.0(1)	N(1)–Re–O(1)	91.8(1)
O(1)–Re–Cl	86.1(1)	C(7)–N(1)–C(8)	117.3(3)
C(6)–C(7)–N(1)	128.0(4)	N(1)–C(8)–C(9)	113.7(3)
C(14)–N(2)–C(15)	116.3(4)	N(2)–C(15)–C(16)	124.6(4)

is manifested by ν(Re–N) at 475 cm^{-1} and ν(Re–O) at 393 cm^{-1}. The IR spectrum of **2** shows ν(Re=O) at 964 cm^{-1} as a strong peak, with ν(Re–N) and ν(Re–O) appearing as medium intensity bands at 486 and 390 cm^{-1}, respectively. Complexes **1** and **2** show sharp, well-resolved peaks in their ^{1}H NMR spectra. There are no detectable paramagnetic shifts or line broadening of signals. The appearance of a single set of resonances (triplet–doublet–doublet–triplet) for all four *N*,*O*-fractions of the two sal$_2$en^{2-} ligands in **1** proves the magnetic equivalence of the protons of the four phenyl rings. The inequivalence of the three phenyl rings in **2** leads to a complex spectrum, in which the resonances of twelve separate resonances for the twelve aromatic protons could be detected, showing the asymmetric arrangement of the sal$_2$np fragments.

3.3. Structures

3.3.1. (μ-O)[ReO(sal$_2$en)]$_2$ (1). The molecular structure of **1** is illustrated in figure 1. The axis of the molecule is formed by the O=Re–O–Re=O moiety, with the bridging O(4) atom lying on a crystallographic inversion centre. Therefore, the Re–O–Re angle is exactly 180°, and the two O=Re–O angles are equal (171.3(1)°), showing significant deviation from linearity. Each rhenium atom is centred in an octahedron with the equatorial plane formed by a N_2O_2 donor set. The octahedron is severely distorted, with three of the equatorial donor atoms [O(1), O(2) and N(1)] being displaced away from the Re=O bond. The result of these distortions is an O(3)–Re–O(4) angle of 171.3(1)°, with the Re atom displaced from the mean equatorial plane by 0.16 Å towards the O(3) oxo atom. Axial Re=O and Re–O distances of 1.709(4) and 1.9176(3) Å, respectively, are typical of this backbone. Re–N(1) and Re–N(2) bond distances are identical, as are Re–O(1) and Re–O(2) bond lengths. These compare well with those found earlier in similar complexes (Re–N average 2.07 Å; Re–O average 2.03 Å) [3–5, 12].

The molecule adopts a staggered conformation in which the ReN_2O_2 planes are mutually rotated by 180° about the O=Re–O–Re=O axis. The two phenyl rings of each NO-half of sal$_2$en are nearly coplanar (dihedral angle = 9.83°) and the dihedral angle between the phenyl rings of the two sal$_2$en chelates is exactly 0°. Intraligand bond

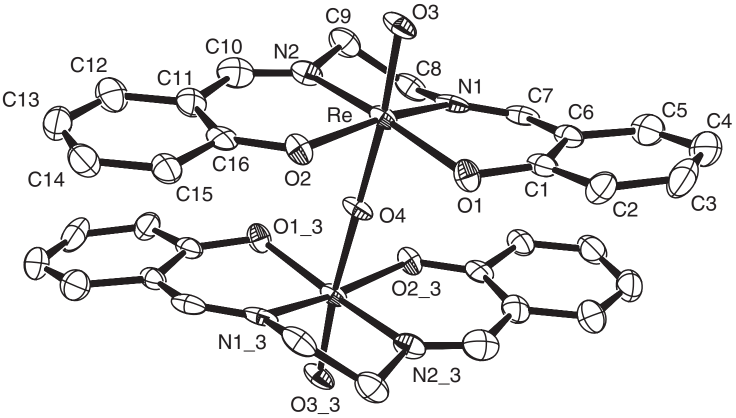

Figure 1. The molecular structure of $[Re_2O_3(sal_2en)_2]$ (**1**), showing the atom numbering scheme with thermal ellipsoids drawn at the 40% probability level.

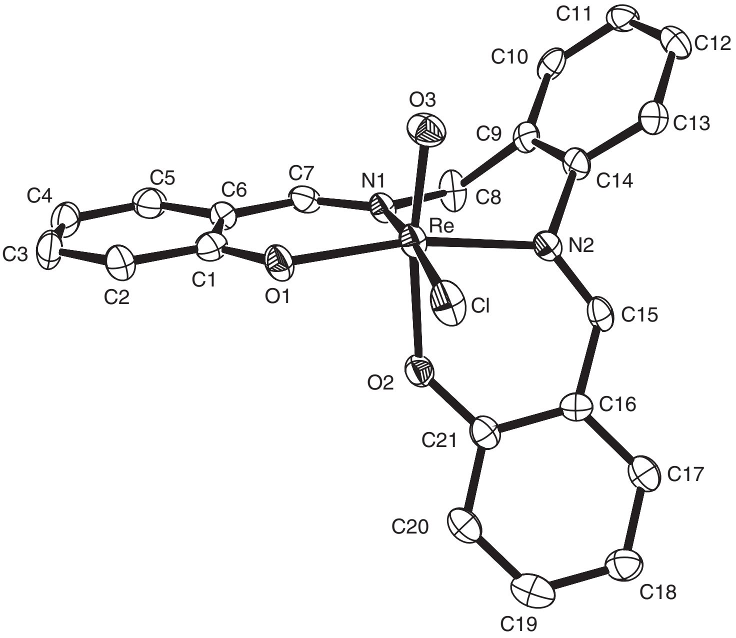

Figure 2. An ORTEP view (at 40% probability) of $[ReOCl(sal_2mp)]$ (**2**).

distances illustrate π-delocalization in the phenyl rings (average C–C distance = 1.396 Å). The C(7)–N(1) and C(10)–N(2) bond lengths are identical at 1.278(9) Å, illustrating their double bond nature. Angles around N(1) and N(2) show the sp^2 hybridization of the imine nitrogens.

3.3.2. $[ReOCl(sal_2mp)]$ (2). An ORTEP view of the asymmetric unit of **2**, along with the atom numbering scheme, is given in figure 2. The rhenium atom lies at the centre of a distorted octahedron. The basal plane is defined by O(1), the imine nitrogens N(1) and N(2), and the chloride. The deprotonated phenolic oxygen O(2) and oxo group O(3) lie in *trans* axial positions. The rhenium(V) ion deviates from the mean equatorial plane by 0.17 Å towards O(3), and the N_2OCl donor atoms deviate by some 0.05 Å from this plane as well. Deviation of rhenium results in O(3)–Re–O(1), O(3)–Re–N(1), O(3)–Re–N(2) and O(3)–Re–Cl angles being larger than 90° (101.1(1), 94.5(1), 91.0(1) and 92.0(1)°, respectively). The O(3)–Re–O(2) angle deviates considerably from linearity at 170.5(1)°. Interestingly, the equatorial N(1)–Re–O(1) 'bite' angle of 91.8(1)° is significantly larger than the axial N(2)–Re–O(2) angle of 80.7(1)°. In the N_2O_3Cl polyhedron, the rhenium ion lies 0.97 Å from the O(3)N(1)O(1) plane, and 1.32 Å from O(2)N(2)Cl plane, the dihedral angle being 3.20°.

The Re=O(3) distance of 1.683(3) Å implies some triple bond character and is typical of complexes of this kind [5, 11, 13]. The difference between the bond distances Re–O(1) (2.003(3) Å) and Re–O(2) (1.997(3) Å), *trans* to the oxo group, is insignificant, and these lengths fall in the observed range 1.98(1)–2.09(1) Å [12, 13]. The two Re–N distances are unequal (Re–N(1) = 2.080(3), Re–N(2) = 2.105(4) Å) and typical of Re–N (imine) bonds, which vary in the range 2.005(6)–2.131(5) Å [2–5]. All other bond distances and angles are normal. Both C(1)C(6) and C(16)C(21) phenyl rings are planar with a dihedral angle of 63.3°. The C(9)C(14) phenyl ring makes a dihedral angle of 47.9° with the mean equatorial plane.

Previous studies of H_2sal_2en and other potentially tetradentate dianionic Schiff base ligands H_2sal_2pp (*N*,*N*′-*o*-phenylenebis(salicylideneimine)) and H_2acac_2en reported the Schiff base-bridged species $[ReOX_2(PPh_3)]_2(sal_2en)$ (X = Cl, Br), $[ReOCl_3(PPh_3)]_2(H_2acac_2en)$, $\{[ReOCl_3]_2(sal_2en)\}^{2-}$ and $\{[ReOCl_4]_2(H_2sal_2en)\}^{2-}$ as well as the mononuclear complexes [ReOCl(L)] (L = sal_2en, sal_2pp, sal_2en) [14–16].

Supplementary material

Files CCDC-265234 (for **1**) and CCDC-265235 (for **2**) contain the crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Rd, Cambridge CB2 1EZ, UK; Fax: +44 (0) 1223 336033; Email: deposit@ccdc.cam.ac.uk.

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