Complexes of WOCl$_4$ and WSCl$_4$ with neutral N- and O-donor ligands: Synthesis, spectroscopy and structures

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**Abstract**

The complexes [WOCl$_4$(L)] and [WSCl$_4$(L)] (L = OPPh$_3$, OMe$_3$, pyridine, 2,2'-bipyridyl), [[WOCl$_4$](µ-L-L)] and [[WSCl$_4$](µ-L-L)] (L = Ph$_3$P(OR)(CH$_2$)$_n$P(OR)Ph$_2$ (n = 1, 2)) have been prepared from WOCl$_4$ or WSCl$_4$ and the ligands in anhydrous CH$_2$Cl$_2$ solution, and characterised by microanalysis, IR and NMR (¹H, ³¹P [¹H]) spectroscopy. X-Ray crystal structures are reported for [WOCl$_4$(OPPh$_3$)], [[WOCl$_4$](µ-Ph$_3$P(OR)(CH$_2$)$_n$P(OR)Ph$_2$)] and [[WSCl$_4$](µ-Ph$_3$P(OR)(CH$_2$)$_n$P(OR)Ph$_2$)]. All, except those of 2,2'-bipyridyl, are six-coordinate with the neutral donor trans to W=O or W=S. Spectroscopic data suggest that the [WOCl$_4$(2,2'-bipy)] and [WSCl$_4$(2,2'-bipy)] are seven-coordinate.

**Keywords:** Tungsten oxide tetrachloride, Tungsten sulfide tetrachloride, Phosphine oxide, 2,2'-Bipyridyl, X-ray structures

1. Introduction

The oxide and sulfide halides of the early d-block metals in their higher oxidation states are Lewis acidic and form complexes with a variety of neutral ligands [1]. In the solid state tungsten oxide, which is bridged to form dimers [Cl$_4$SW(O)-Cl]$_2$WCl$_3$S [3]. A second polymorph of WSCl$_4$ is a tetramer [Cl$_4$SW(O)-Cl]$_2$WCl$_4$-S[µ-Cl]WSCl$_4$ in which the dimer core is linked by single chlorine bridges to two other WSCl$_4$ units [4]. Neutral ligand complexes of WOCl$_4$, were first reported some years ago, obtained by direct reaction conditions, and other products include complexes of fragmented ligands and tungsten(V) complexes containing WCL$_4$ or WCO$_3$ [10,11,15,16]. A few complexes of WOBr$_4$ have been described [1,5,6,7], along with the fluoro complexes, [WOF$_4$(L)] ([L = MeCN, OPPh$_3$, thf, dmso, py, PMe$_3$] and [WOF$_4$(diphosphine)] (diphosphine = o-C$_6$H$_4$(PMe$_2$)$_2$ or Me$_2$PCH$_2$CH$_2$PMe$_2$) that were reported very recently [17,18].

Some corresponding tungsten(V) sulfide tetrachloride complexes, including [WSCl$_4$(L)] (L = thf, py, RCN), [[WSCl$_4$](µ-1,4-dioxane)] and [[WSCl$_4$](µ-Me(S)(CH$_2$)SMe)] have been prepared, but appear to be less stable than those of WOCl$_4$ [1,5,19,20]. Possible applications of complexes of metal sulfide halides include their use as reagents for the chemical vapour deposition of the layered metal dichalcogenide thin film materials [21,22]. We report here a comparison of the complexes of WOCl$_4$ and WSCl$_4$ with a series of neutral N- and O-donor ligands.

2. Results and discussion

2.1. Phosphine oxide complexes.

The WOCl$_4$ and WSCl$_4$ precursors were made by minor modifications of the literature route from WCl$_6$ and O(SiMe$_3$)$_2$ or S(SiMe$_3$)$_2$, respectively, in CH$_2$Cl$_2$ [23]. The complexes were subse-
quently synthesised by reaction of the appropriate ligand with the WECl₄ (E = O or S) in anhydrous CH₂Cl₂. Attempts to prepare the complexes by “one pot” reactions, i.e. by adding the O(SiMe₃)₂ or S(SiMe₃)₂ to WCl₆ in CH₂Cl₂, followed by addition of the ligand, were less successful and often gave impure products or mixtures. The complexes are very readily hydrolysed with formation of the robust [WO₂Cl₂(L)ₙ] (and other products) and use of anhydrous ligands and solvents with Schlenk and glove box techniques is essential to obtain pure complexes. In several cases, adventitious hydrolysis led to the isolation of [WO₂Cl₂(L)ₙ] complexes, as seen in other WOX₄/WO₂X₂ systems [1,17]. The reaction of WOCl₄ with one molar equivalent of OPPh₃ or OPMe₃ or 0.5 molar equivalents of Ph₂P(O)(CH₂)ₙP(O)Ph₂ (n = 1, dppmO₂; n = 2, dppeO₂) in anhydrous CH₂Cl₂ solution gave orange or yellow complexes [WOCl₄(-OPR₃)] (R = Me, Ph) or [(WOCl₄)₂(L-L)] (L-L = dppmO₂, dppeO₂).

Scheme 1. Synthesis of the complexes of WECl₄.
ligands. The IR spectrum of [WOCl₄(OPPh₃)] has been reported twice, but with poor agreement [13, 24]; our data are in agreement with the report of Behzadi et al. [24]. The ³¹P{¹H} NMR spectra show substantial high frequency shifts for the phosphoryl group upon coordination and for the complexes of [WOCl₄(OPR₃)] are very similar to those observed for the corresponding [WOCl₄(OPR₃)] [17]; (the [WOCl₄₂(diphosphine dioxide)] complexes have not been reported). The report [13] that [WOCl₄(OPPh₃)] exhibits a low frequency coordination shift of -4 ppm is clearly in error.

The reaction of WSCl₄ with OPPh₃, OPMe₃, dpmpO₂ and dppeO₂ in anhydrous CH₂Cl₂ solution, produced brown powders, [WSCl₄(OPR₃)] (R = Me, Ph) and [WSCl₄₂(L-L)] (L-L = dpmpO₂, dppeO₂). The X-ray crystal structure of [WSCl₄₂(dppeO₂)] (Fig. 3) shows a centrosymmetric dimer with the tungsten lying out of the WCl₄ plane towards the sulfide group. The W–O₁ = 2.159(2) Å, is similar to the average of the corresponding links in [WOCl₄₂(dppeO₂)], (2.155 (6) Å).

2.2. Complexes of N-heterocycles

To compare the complexes of the O-donor phosphine oxides with those of N-donor ligands, the complexes with pyridine and 2,2'-bipyridyl were examined. The yellow [WOCl₄(py)] and brown [WSCl₄(py)] complexes [7, 19] were made by reacting the constituents in a 1:1 molar ratio in dry CH₂Cl₂ solution. The products are diamagnetic and the spectroscopic properties are unexceptional. Under reflux or with longer reaction times, reduction to W(V) species occurs [19]. The reaction of WOCl₄₂ with 2,2'-bipyridyl has been reported before [7, 25], the green solid being variously formulated as [WOCl₄₂(2,2'-bipy)] or the paramagnetic W(V) complex, [WOCl₄₂(2,2'-bipy)]. By conducting the reaction in CH₂Cl₂ solution at room temperature and with a relatively short reaction time, we obtained a green complex [WOCl₄(2,2'-bipy)], which was diamagnetic and with the ¹H NMR spectrum showing equivalent pyridyl rings. Repeated attempts to obtain crystals for an X-ray study have been unsuccessful, but it is highly likely that the complex [WOCl₄(2,2'-bipy)], contains seven-coordinate tungsten, probably pentagonal bipyramidal with axial O/Cl. Similar seven-coordination is established in [WOCl₄₀-C₆H₄(AsMe₂)] [12] and in several complexes of WOF₄ [17, 18, 26]. The ν(W=O) of 970 cm⁻¹ is lower than the values observed in the six-coordinate complexes, but similar to those in [WOCl₄₀-C₆H₄(AsMe₂)] (964 cm⁻¹) [12], [WOCl₄₂(2,2'-bipy)] (968 cm⁻¹) or [WOCl₄(py)₂] (973 cm⁻¹) [26], which supports the assignment of the higher coordination number, and the proposed geometry in which the oxido-group can occupy an axial position. The new complex [WSCl₄₂(2,2'-bipy)] was obtained as a red solid, and is spectroscopically very similar. Attempts to grow crystals of the seven-coordinate W(VI) complex from CH₂Cl₂ solution over several days resulted in crystals being obtained of the previously reported six-coordinate [WSCl₄₂(2,2'-bipy)] [19] (Fig. 4).

A complex [WOCl₄₁.10-phen] has been claimed [25], but with no reported data. Our attempts using similar reaction conditions to those used for [WOCl₄₂(2,2'-bipy)] produced a mixture of products, one of which was identified by an X-ray structure determination as [WOCl₄₁.10-phen] CH₂Cl₂ by comparison of the unit cell parameters with the literature [27]. It is possible that the more rigid 1,10-phenanthroline is poorly matched to the seven-coordinate tungsten centre. It is notable that attempts to isolate [WOCl₄₁.10-phen] from reaction of [WOCl₄ (MeCN)] with 1,10-phen failed, with [WOCl₄₁.10-phen] being identified as one major product [17].

As indicated above, decomposition (probably hydrolysis) of some of the WCl₄ complexes to WOCl₄ species was noted, whereas under more forcing condition (higher temperatures, excess ligand or extended reaction time) reduction to tungsten (V) species sometimes occurs [1, 7, 19, 28]. During attempts to grow crystals from the [WOCl₄₂(OPR₃)] by slow evaporation from CH₂Cl₂ solutions. A few white crystals were isolated, which proved to be cyclic [W₄d₁uO₈] species. Several batches of white crystals were obtained, most with the crystal quality too poor to merit report, but a few good crystals were obtained on one occasion from the OPM₃ system and the structure is shown in Fig. 5.

The crystals contain W(VI) with a distorted octahedral coordination environment composed of WOCl₄(OPMe₃) units linked...
Fig. 5. The structure of \([\text{WCl}_4\text{O}_3\text{OPMe}_3]_2\) showing the atom numbering scheme. The Me groups on O3 show disorder over two sites, only one is shown. H atoms and the lattice CH2Cl2 molecules are omitted for clarity. Selected bond lengths (Å) and angles (°) are: W1–Cl2 = 2.3491(8), W1–Cl1 = 2.3397(8), W1–O9 = 2.0817(18), W1–O1 = 2.0817(18), W1–O3 = 1.7651(18), W1–O2 = 1.710(2), W2–Cl4 = 2.3528(7), W2–Cl3 = 2.3522(8), W2–O4 = 2.0715(18), W2–O6 = 1.7669(19), W2–O3 = 2.2301(18), W2–O5 = 1.7167(19), W3–Cl5 = 2.3522(8), W3–Cl6 = 2.3549(8), W3–O9 = 1.7539(18), W3–O6 = 2.2233(18), W3–O7 = 2.0681(19), W3–O8 = 1.715(2), W–O6–W = 152.97(11), 154.90(11), O6–W–O9 = 86.07(8), 85.01(8), 86.07(8).

4. Experimental

Syntheses were performed by using standard Schlenk and glove-box techniques under a dry N2 atmosphere. WCl6 (Acros organics), O(SiMe3)2 and S(SiMe3)2 (Sigma–Aldrich) were used as received. Solvents were dried by distillation from CaH2 (CH2Cl2, MeCN) or Na/benzophenone ketyl (toluene, n-hexane). Ligands (2,2’-bipy, 1,10-phen, OPPh3) were obtained from Sigma–Aldrich and dried by heating in vacuo before use. Pyridine was dried over sodium and freshly distilled. The diphosphine dioxides, dppeO2, were made by dry air oxidation of the corresponding diphosphines in CH2Cl2 solution, catalysed by SnI4 [29] and were checked for purity by 31P{1H} NMR spectroscopy. They were also dried by heating in vacuo.

Infrared spectra were recorded on a Perkin–Elmer Spectrum 100 spectrometer in the range 4000–200 cm\(^{-1}\), with samples prepared as Nujol mulls between CsI plates. 1H NMR spectra were recorded using a Bruker AV 400 spectrometer and referenced to the residual protio-resonance of the solvent. 31P{1H} NMR spectra were obtained from CD2Cl2 solutions using a Bruker AV 400 spectrometer and referenced external 85% H3PO4. Microanalyses on new compounds were undertaken by London Metropolitan University or Medac.

4.1. \([\text{WOCl}_4]\)

Following the literature method [23], a solution of [Me2Si]2O (0.82 g, 5.04 mmol) in dichloromethane (5 mL) was added to a stirred solution of WCl6 (2.0 g, 5.04 mmol) in dichloromethane (5 mL). The orange solution was stirred for 1 h and the orange precipitate was filtered off and washed with cold hexane (2 × 1 mL), and dried in vacuo. IR spectrum (Nujol, cm\(^{-1}\)) : 889sh W=O, 875s W=O, 381s, br W–Cl, 326s W–Cl.

4.2. \([\text{WSCl}_4]\)

WCl6 (2.5 g, 6.3 mmol) in toluene (5 mL) was chilled to 0 °C and hexamethyldisilathane (1.35 g, 6.3 mmol) in toluene (5 mL) was added dropwise over 15 min. The red/brown solution formed was stirred for 30 min. and taken to dryness in vacuo, resulting in a brown powder. The brown powder was sublimed in vacuo yielding a red powder on the cold finger. Yield: 0.950 g, 42%. IR spectrum (Nujol, cm\(^{-1}\)): 556 m W=O, 981 s W–Cl. 1H NMR (CDCl3): δ = 7.88 (m, [6H]), 7.70 (m, [3H]), 7.58 (m, [6H]).

4.3. \([\text{WOCl}_4\text{OPPh}_3]\)

A solution of OPPh3 (0.105 g, 0.44 mmol) in dichloromethane (5 mL) was added slowly to a suspension of WOCl4 (0.150 g, 0.44 mmol) in dichloromethane (5 mL). The clear orange solution was then stirred for 1 h, concentrated to ~5 mL in vacuo, filtered, and the resulting orange solid washed with hexane (2 × 1 mL), and dried in vacuo. Yield: 0.130 g, 48%. Required for C18H15Cl4O2PW (433.7): C, 8.31%;
H, 2.09. Found: C, 8.55; H, 2.02%. IR spectrum (Nujol, cm⁻¹): 1089s, br P=O, 957s W=O, 337s W–Cl. 1H NMR (CDCl₃): δ = 1.88 (d, 2⁻, JHH = 13 Hz). 3¹P{¹H} NMR (CDCl₃): δ = +64.2 (s).

4.5. [(WOCl₄)₂(dpdmO₂)]

Powdered dpdmO₂ (0.120 g, 0.29 mmol) in dichloromethane (5 mL) was added slowly to a red suspension of WOCl₄ (0.150 g, 0.28 mmol) in dichloromethane (5 mL). The red/orange solution formed was stirred for 1 h, then concentrated to ~5 mL, filtered, and the orange powder washed with hexane (2 × 1 mL) and dried in vacuo. Yield: 0.264 g, 83%. Required for C₂₅H₂₂Cl₈O₄P₂W₂ (1099.7): C, 27.30; H, 2.02. Found: C, 27.42; H, 2.17. IR spectrum (Nujol, cm⁻¹): 1076m, br P=O, 981s W=O, 338s, br W–Cl. 1H NMR (CDCl₃): δ = 7.81 (m, [8H]), 7.61 (m, [4H]), 7.43 (m, [8H]). 4.62 (t, [2H], JHP = 15.5 Hz). 3¹P{¹H} NMR (CDCl₃): δ = +44.6 (s). 3¹P{¹H} NMR (CDCl₃): δ = +45.9 (s).

4.6. [(WOCl₄)(dppeO₂)]

Was made similarly to [(WOCl₄)(dpdmO₂)] as a yellow powder. Yield: 31%. Required for C₂₆H₂₄Cl₈O₄P₂W₂ (1137.1): C, 28.04; H, 2.14. Found: C, 27.96; H, 2.12. IR spectrum (Nujol, cm⁻¹): 338s, br W–Cl. 1H NMR (CDCl₃): δ = 8.76 (m, [8H]), 7.70 (m, [4H]), 7.59 (m, [8H]). 3.15 (br, s, [4H]). 3¹P{¹H} NMR (CDCl₃): δ = +53.2 (s).

4.7. [WSCl₄(OOPPh₃)]

OOPPh₃ (0.078 g, 0.28 mmol) in dichloromethane (2 mL) was slowly added to a red solution of WSCl₄ (0.100 g, 0.28 mmol) in dichloromethane (5 mL). The solution was then stirred for 1 h, concentrated in vacuo to ~4 mL. The brown precipitate was filtered off, washed with hexane (2 × 1 mL) and dried in vacuo. Yield: 0.120 g, 69%. Required for C₂₅H₂₂Cl₈O₄P₂S₂W₂ (1154.85): C, 27.25; H, 2.11. Found: C, 27.00; H, 2.02%. IR spectrum (Nujol, cm⁻¹): 1077s P=O, 536s W=S, 330s W–Cl. 1H NMR (CD₂Cl₂): δ = 7.93 (br, [8H]), 7.66 (br, [4H]), 7.59 (br, [8H]). 2.78 (m, [4H]). 3¹P{¹H} NMR (CD₂Cl₂): δ = +49.2 (s).

4.11. [WOCl₄(C₂H₅N)]

Pyridine (0.050 g, 0.58 mmol) in toluene (2 mL) was added to a red suspension of WOCl₄ (0.200 g, 0.58 mmol). The red solution formed was stirred for 1 h and then taken to dryness in vacuo to obtain a yellow powder. Yield: 0.120 g, 49%. Required for C₁₈H₁₅Cl₄OPSW (636.0): C, 33.99; H, 2.38. Found: C, 34.13; H, 2.15; N, 3.31. IR spectrum (Nujol, cm⁻¹): 987m W=O, 338s W–Cl. 1H NMR (CD₂Cl₂): δ = 9.37 (dd, [2H], JHH = 6.6, 1.5 Hz), 8.06 (tt, [H], JHH = 7.7, 1.5 Hz), 7.65 (td, [2H], JHH = 6.6, 1.2 Hz).

4.12. [WSCl₄(C₂H₅N)]

Pyridine (0.050 g, 0.58 mmol) in toluene (2 mL) was added to a red solution of WSCl₄ (0.200 g, 0.58 mmol). The brown solution formed was stirred for 1 h, and then taken to dryness in vacuo to yield a brown/pink powder. Yield: 0.160 g, 87%. Required for C₁₀H₈Cl₄NSW (436.8): C, 13.75; H, 1.15; N, 3.21. IR spectrum (Nujol, cm⁻¹): 533s W=S, 334s br W–Cl. 1H NMR (CD₂Cl₂): δ = 8.78 (br, [2H]), 8.56 (br, [H]), 8.11 (br, [2H]).

4.13. [WOCl₄(2,2’-bipy)]

2,2’-Bipy (0.068 g, 0.44 mmol) in dichloromethane (5 mL) was added slowly to an orange suspension of WOCl₄ (150 mg, 0.44 mmol) in dichloromethane (5 mL). The green solid formed was stirred for 1 h at room temperature and then concentrated to ~10 mL in vacuo. The green solid was isolated by filtration, washed with hexane (2 × 1 mL) and dried in vacuo. Yield: 0.067 g, 30%. Required for C₁₀H₈Cl₄N₄OW (499.85): C, 24.11; H, 1.62; N, 5.63. Found: C, 24.33; H, 1.73; N, 5.84%. IR spectrum (Nujol, cm⁻¹): 1137 m P=O, 329s W–Cl, 310s W–Cl. 1H NMR (CD₂Cl₂): δ = 7.81 (m, [6H]), 7.68 (t, [3H]), 7.55 (m, [6H]). 3¹P{¹H} NMR (CD₂Cl₂): δ = +45.9 (s).

4.14. [WSCl₄(2,2’-bipy)]

2,2’-Bipy (0.065 g, 0.42 mmol) in dichloromethane (5 mL) was added slowly to a red suspension of WSCl₄ (0.150 g, 0.42 mmol) in dichloromethane (5 mL). The purple/red solution was stirred for 1 h, concentrated to ~5 mL and the red/pink powder filtered off, washed with hexane (2 × 1 mL) and dried in vacuo, yielding a red/pink powder. Yield: 0.165 g, 76%. Required for C₁₀H₈Cl₄NSW (513.9): C, 23.37; H, 1.57; N, 5.45. Found: C, 23.36; H, 1.59; N, 5.50%. IR spectrum (Nujol, cm⁻¹): 521s W=S, 316s W–Cl. 1H NMR (CD₂Cl₂): δ = 8.99 (d, [2H], JHH = 8.1 Hz), 8.85 (d, [2H], JHH = 4.4 Hz), 8.22 (t, [2H], JHH = 7.8 Hz), 7.66 (t, [2H], JHH = 4.0 Hz).

4.15. X-ray experimental

Data collections used a Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) Saturn724+ detector mounted at the window of an FR-E+ SuperBright molybdenum (λ = 0.71073) rotating anode generator with VHF Varimax optics (70 micron focus) with the crystal held at 100 K (N₂ cryostream). Crystallographic parameters are in Table 1. Structure solution and refinement were performed using SHELXS97, SHELX-2014/7 [30] and were mostly straightforward, some minor disorder within one OPMe₃ group is noted in the structure of [W₆O₁₃(O₃P)₃Cl₉]+.
Table 1
Crystallographic data.a

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Conflicts of interest

The authors have no conflicts to declare.

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Appendix A. Supplementary data

CCDC 1882209: [WOC14(OPPh3)], 1882210: [WOC142(-dppmO2)], 882211: [WSC142(dppeO2)], 1882212: [WO3(O–O)2C142(OPMe3)2] 2CH2Cl2, 1883854: [WSC14(bipy)] contains the supplementary crystallographic data. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data to this article can be found online at https://doi.org/10.1016/j.poly.2019.01.044.

References


OPMe3)2] 2CH2Cl2, this was modelled satisfactorily using split C atom site occupancies. H atoms were added and refined with a riding model.