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Coordination of 4-mercapto-1,2-dithiole-3-thione heterocycles to ruthenium(II) and molybdenum(VI) centres

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ABSTRACT

Deprotonation of 4-mercapto-1,2-dithiole-3-thiones with NEt_3 followed by reaction with $[Ru(H)(Cl)(CO)(PPh_3)_3]$ affords virtually quantitative yields of turquoise $[Ru(H)(RC_3S_4)(CO)(PPh_3)_2]$ (R = Ph, Mes) in which the heterocycle is bound as a bidentate uninegative ligand through the two exocyclic sulfur atoms. The presence of both possible isomers in each case is indicated by NMR spectroscopy. Reaction of the 4-mercapto-1,2-dithiole-3-thiones with $[MoO_2(acac)_2]$ results in displacement of the acac ligands and formation of $[MoO_2(RC_3S_4)_2]$. The crystal structures of $[Ru(H)(MesC_3S_4)(CO)(PPh_3)_2]$ and $[MoO_2(MesC_3S_4)_2]$ have been determined.

1. Introduction

Compounds which contain the 1,2-dithiole-3-thione ring system are currently attracting attention as chemopreventive agents against various types of cancer. One such compound, Oltipraz (4-methyl-5-(2-pyrazinyl)-1,2-dithiole-3-thione), shown in Fig. 1, which was originally developed as an anti-schistosomiasis treatment, is currently undergoing a large scale clinical trial in the Qidong region of China, an area associated with a high incidence of aflatoxin-induced liver cancer [1]. Other compounds containing the 1,2-dithiole-3-thione ring are known to have chemopreventive properties which are at least as good as, if not better than, Oltipraz [2].

One of the drawbacks of such compounds is that their synthesis often involves toxic and malodorous sulfurising reagents such as P_2S_5, Lawesson's reagent, or H_2S [3]. We recently reported a novel route to 4-mercapto substituted 1,2,-dithiole-3-thiones **4** by sequential treatment of alkynyl lithiums with carbon disulfide and elemental sulfur (Scheme 1) [4]. We also showed that the first intermediates in this reaction, the alkynyl dithiocarboxylate anions $RC{\equiv}CCS_2^-$ **2**, were sufficiently stable in solution at room temperature to be coordinated to ruthenium(II) centres by reaction with $[RuH(Cl)(CO)(PPh_3)_3]$ or $[Ru(CPh{=}CHPh)(Cl)(CO)(PPh_3)_2]$ [5].

In this paper we demonstrate that a further intermediate in this synthesis, the 4-mercapto-1,2-dithiole-3-thione anions **3**, can also be isolated by coordination to ruthenium, and that they coordinate as bidentate ligands through the 4-mercapto substituent and the sulfur atom of the C=S group. In addition we show that deprotonation of the heterocyclic thiol products also provides an effective route to complexes of this type. Previous studies of the coordination chemistry of the 1,2-dithiole-3-thione ring system have largely concentrated on the 4,5-dithiolate derivative (often known as dmt), which can be prepared by thermal isomerisation of the readily prepared dmit dianion (Steimecke rearrangement) as shown in Scheme 2 [6]. Bis-chelate complexes of the type $[NBu_4]_2[M(dmt)_2]$ (M = Ni, Pd, Pt, Cu) can then be prepared in which the dianionic ligand bonds through the exocyclic 4,5-dithiolate unit, in the same manner as in the dmit isomers [7].

2. Results and discussion

2.1. Ruthenium complexes

Addition of one equivalent of $[RuH(Cl)(CO)(PPh_3)_3]$ to solutions of the anions **3** (R = Ph or Mes) prepared *in situ* from the appropriate lithium acetylide, carbon disulfide and sulfur, either at room temperature or at −78 °C, rapidly afforded a dark turquoise solution from which the new highly coloured blue-turquoise complexes $[Ru(H)(RC_3S_4)(CO)(PPh_3)_2]$ (**5** R = Ph; **6** R = Mes) could be isolated by column chromatography (Scheme 3). The yields of both compounds are high (88% for **5** and 72% for **6**). The only other product detected was triphenylphosphine sulfide (identified by ^{31}P NMR and mass spectroscopy), formed by the liberated PPh_3 reacting with the excess of sulfur involved in the synthesis of the heterocycle. Deprotonation of the preformed 4-mercapto-1,2-dithiole-3-thione derivatives with triethylamine followed by addition of $[RuH(Cl)(CO)(PPh_3)_3]$ produced slightly improved yields of **5**

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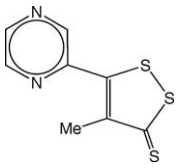
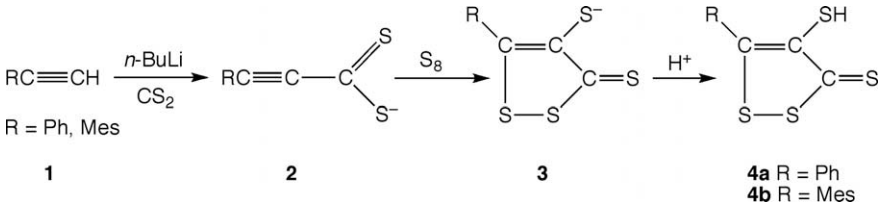


Fig. 1. Structural formula of Oltipraz.

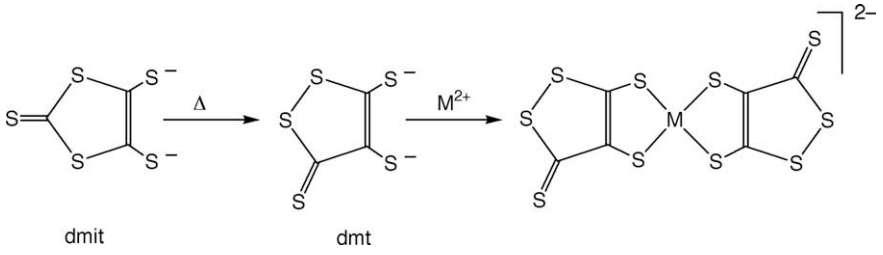
(93%) and **6** (89%). However since the isolated yields of the thiones **4** are only about 50%, the *in situ* reaction gives a better overall yield.

Characterisation of **5** and **6** followed from their spectroscopic data. Each complex shows one strong ν(CO) absorption in the IR spectrum (at 1930 cm^{-1} for **5**, 1932 cm^{-1} for **6**). Apart from phenyl peaks the ^{1}H NMR spectrum of **5** contains two triplet hydride resonances at δ −9.54 (J_{HP} = 18.0 Hz) and δ −9.86 (J_{HP} = 17.5 Hz), indicating the presence of two isomers in a ratio of 97.7:2.3. Similarly the ^{1}H NMR spectrum of **6** also contains two hydride signals, at δ −9.47 (J_{HP} = 19.5 Hz) and −10.43 (J_{HP} = 18.5 Hz) in a ratio of 56:44. The ^{31}P NMR spectra of both compounds also indicate the presence of two isomers. The origin of the large difference in isomer ratios is unknown, but seems likely to be due to steric factors. The FAB mass spectra of both complexes showed appropriate molecular ion envelopes.

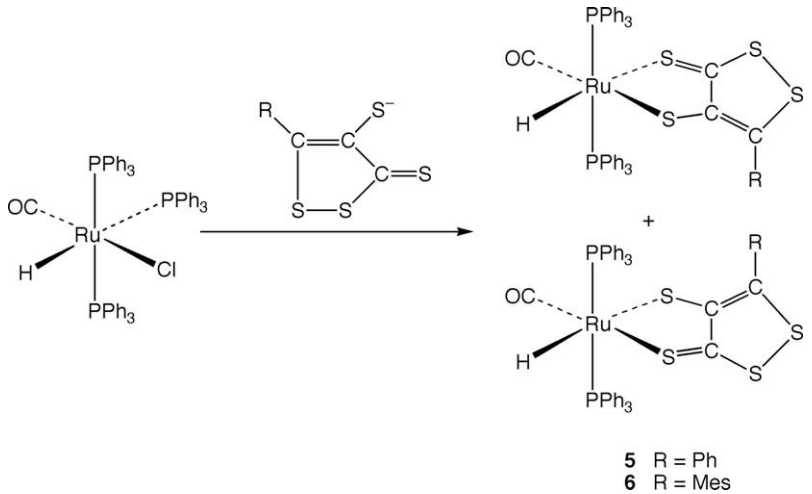
Diffusion of heptane vapour into an ethyl acetate solution of **6** gave large turquoise blocks suitable for X-ray diffraction. The molecular structure is shown in Fig. 2 with selected bond lengths and angles collected in Table 1. As expected the molecule adopts an octahedral geometry with the two PPh_3 ligands in mutually *trans* positions; the P(1)–Ru(1)–P(2) angle is 164.40(5)°. The heterocyclic ligand is coordinated in a bidentate manner through the thione sulfur atom S(1) [Ru(1)–S(1) 2.4341(16) Å] and the thiolate sulfur atom S(2) [Ru(1)–S(2) 2.4585(16) Å]. The C=S bond of the thione unit apparently retains complete double bond character, as shown by the C(2)–S(1) distance of 1.659(6) Å compared to the value of 1.651(3) Å for the corresponding bond in the structure of PhCSSC(=S)C(SMe); this can also be compared with the C(3)–S(2) single bond of the thiolate group in **6** which is 1.740(6) Å. The crystal chosen for study consists exclusively of the isomer in which the thione sulfur is *trans* to the hydride ligand and the thiolate sulfur *trans* to the carbonyl ligand, rather than *vice versa*; however it is not known whether this corresponds to the major or minor isomer in solution.



Scheme 1. Formation of 4-mercapto-1,2-dithiole-3-thiones from alkynes [4].



Scheme 2. Synthesis and coordination of dmt (4,5-dimercapto-1,2-dithiole-3-thione dianion) [6,7].



Scheme 3. Synthesis of ruthenium complexes.

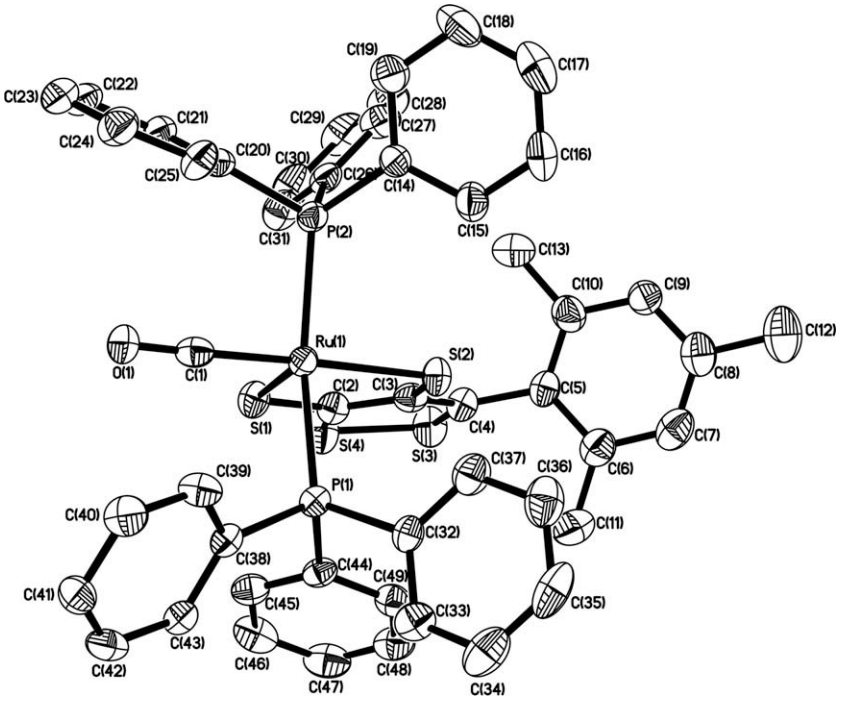


Fig. 2. Molecular structure of complex **6** in the crystal (ORTEP plot, 50% probability ellipsoids). Hydrogen atoms have been omitted for clarity.

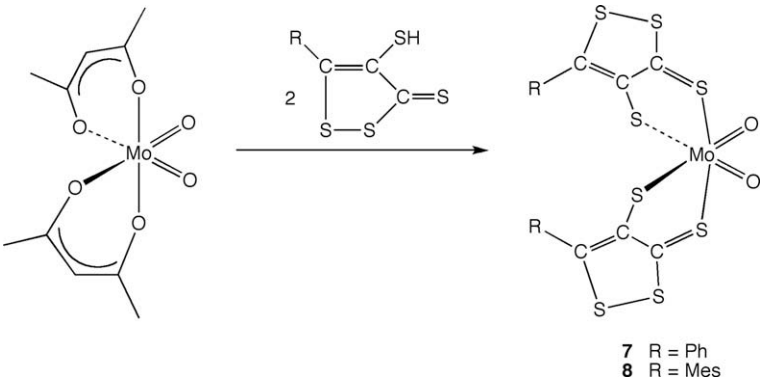
Table 1
Selected bond lengths (Å) and angles (°) for complex **6**.

Ru(1)–C(1)	1.841(7)	Ru(1)–P(1)	2.3500(15)
Ru(1)–P(2)	2.3631(16)	Ru(1)–S(1)	2.4341(16)
Ru(1)–S(2)	2.4585(16)	Ru(1)–H(1RU)	1.5643
S(1)–C(2)	1.659(6)	S(2)–C(3)	1.740(6)
S(3)–C(4)	1.721(6)	S(3)–S(4)	2.054(3)
S(4)–C(2)	1.730(6)	O(1)–C(1)	1.162(7)
C(2)–C(3)	1.421(8)	C(3)–C(4)	1.380(8)
P(1)–Ru(1)–P(2)	164.40(5)	S(1)–Ru(1)–S(2)	84.83(5)
O(1)–C(1)–Ru(1)	177.0(5)		

2.2. Molybdenum complexes

Molybdenum is known to have a high affinity for sulfur and displays an extensive coordination chemistry with sulfur based ligands, some of which is of biological importance in the molybdenum cofactor of oxidoreductase enzymes [8]. We were therefore prompted to investigate the coordination of the thiones to molybdenum centres. It is well known that the acetylacetonate ligands of $[MoO_2(acac)_2]$ can be removed by protonation with mildly acidic ligands such as Schiff's bases to give a wide range of dioxo molybdenum complexes. Stirring $[MoO_2(acac)_2]$ with two equivalents of the thiones **4** for 24 h afforded red-brown precipitates which were collected by filtration and recrystallised (Scheme 4). These complexes, **7** and **8**, were characterised spectroscopically as well as by an X-ray diffraction study of the mesityl derivative. The solid state IR spectrum of **7** (in KBr) showed two strong peaks at 946 and 894 cm^{-1}, characteristic of the *cis*-MoO_2 unit; in **8** these appear at lower frequency, 910 and 875 cm^{-1}. The ^1H NMR spectra displayed only phenyl/mesityl resonances, but the complexes were not sufficiently soluble to obtain ^{13}C NMR spectra. The FAB mass spectra of both compounds showed molecular ion envelopes.

Crystallisation of **8** by diffusion of hexane vapour into a CH_2Cl_2 solution afforded orange-brown crystals of the dichloromethane solvate suitable for structural investigation. The molecular structure is shown in Fig. 3 with selected bond lengths and angles collected in Table 2. Due to the unsymmetrical nature of the bidentate ligand, there are three possible isomers of $[MoO_2(RC_3S_4)_2]$ if the



Scheme 4. Synthesis of molybdenum complexes.

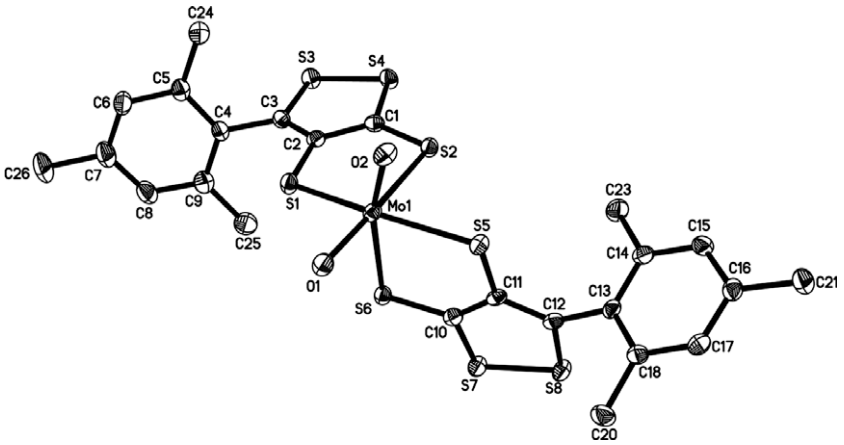


Fig. 3. Molecular structure of complex **8**·CH_2Cl_2 in the crystal (ORTEP plot, 50% probability ellipsoids). Hydrogen atoms and the dichloromethane of crystallisation have been omitted for clarity.

Table 2
Selected bond lengths (Å) and angles (°) for complex **8**·CH_2Cl_2.

Mo(1)–O(1)	1.709(2)	Mo(1)–O(2)	1.714(2)
Mo(1)–S(1)	2.4263(8)	Mo(1)–S(5)	2.4417(8)
Mo(1)–S(2)	2.6838(8)	Mo(1)–S(6)	2.7036(8)
S(1)–C(2)	1.751(3)	S(2)–C(1)	1.689(3)
S(3)–C(3)	1.712(3)	S(3)–S(4)	2.0419(11)
S(4)–C(1)	1.720(3)	S(5)–C(11)	1.751(3)
S(6)–C(10)	1.676(3)	S(7)–C(10)	1.727(3)
S(7)–S(8)	2.0457(11)	S(8)–C(12)	1.716(3)
C(1)–C(2)	1.414(4)	C(2)–C(3)	1.375(4)
C(10)–C(11)	1.424(4)	C(11)–C(12)	1.375(4)
O(1)–Mo(1)–O(2)	105.19(10)	S(1)–Mo(1)–S(5)	154.45(3)
O(1)–Mo(1)–S(2)	163.54(7)	S(1)–Mo(1)–S(2)	80.61(3)
O(2)–Mo(1)–S(6)	165.70(8)	S(5)–Mo(1)–S(6)	80.50(2)

two oxo ligands are assumed to retain their *cis* geometry (as is usually the case). The methyl region of the ^{1}H NMR spectrum of **8** indicates that only one isomer is formed. In the crystal it is the one in which the two thiolate sulfurs are *trans* to each other, and the two thione sulfurs are *trans* to the oxo ligands. A similar arrangement was found in the MoO_2 complex of the dianion of 2,3:8,9-dibenzo-1,4,7,10-tetrathiadecane ($^-SC_6H_4SCH_2CH_2SC_6H_4S^-$); the two thiolate sulfurs were *trans* to each other and the two uncharged thioether sulfur atoms were *trans* to the oxo ligands [9].

It is well known that $[MoO_2(dtc)_2]$, where dtc = $Et_2NCS_2^-$, acts as an oxygen atom transfer reagent towards substrates such as PPh_3 [10]. However attempts to induce a similar reaction by treating **7** with PPh_3, either at room temperature or elevated temperature, showed no evidence of success.

2.3. Conclusion

Complexes containing the anion of 4-mercapto-1,2,-dithiole-3-thiones can be prepared in good yield either *in situ* from terminal alkynes, or by deprotonation of the heterocycle. Coordination occurs through the two exocyclic sulfur atoms, forming a five-membered chelate ring.

3. Experimental

3.1. General

General experimental techniques were as described in a recent paper from this laboratory [11]. Infra-red spectra were recorded either in CH_2Cl_2 solution (0.5 mm NaCl cells) or as KBr discs on a Perkin–Elmer 1600 FT-IR machine. The ^{1}H, ^{13}C and ^{31}P NMR spectra were obtained in $CDCl_3$ solution on a Bruker AC250 machine with automated sample-changer or an AMX400 spectrometer. Chemical shifts are given on the δ scale relative to $SiMe_4$ or 85% H_3PO_4 = 0.0 ppm. The $^{13}C\{^1H\}$ NMR spectra were routinely recorded using an attached proton test technique (JMOD pulse sequence). Mass spectra were recorded on a Fisons/BG Prospec 3000 instrument operating in fast atom bombardment mode with *m*-nitrobenzyl alcohol as matrix. Elemental analyses were carried out by the Microanalytical Service of the Department of Chemistry. The compounds $[Ru(H)(Cl)(CO)(PPh_3)_3]$ [12] and $[MoO_2(acac)_2]$ [13] were prepared by literature methods.

3.2. Synthesis of $[Ru(H)(PhC_3S_4)(CO)(PPh_3)_2]$ (**5**)

(i) One pot reaction

A solution of phenylacetylene (0.29 mL, 2.64 mmol) in THF (17.9 mL) was cooled to −78 °C and treated with ^{n}BuLi (1.64 mL of 1.6 M solution, 2.63 mmol). After stirring at room temperature for 30 min, the solution was re-cooled to −78 °C and carbon disulfide (0.15 mL, 2.5 mmol) was added. The pale yellow solution was allowed to warm to room temperature and stirred for 1 h, causing a colour change to dark red. An excess of elemental sulfur (0.26 g, 8.08 mmol of S) was added, causing an instant colour change to bright red-pink. After stirring for 1 h, 8.75 mL of this solution (i.e. 1.15 mmol of anion) was transferred by syringe to a second Schlenk tube containing a suspension of $[Ru(H)(Cl)(CO)(PPh_3)_3]$ (1.00 g, 1.04 mmol) in THF (20 mL). The resulting blue-turquoise solution was allowed to stir for 24 h. After the addition of a small amount of silica, the solution was evaporated to dryness and the residue loaded onto a chromatography column. Elution with light petroleum and CH_2Cl_2 (7:3), afforded a blue-turquoise band of **5** (0.8295 g, 88%).

(ii) From 4-mercapto-5-phenyl-1,2-dithiole-3-thione

An orange solution of the heterocycle (40 mg, 0.18 mmol) in THF (10 mL) was treated with NEt_3 (0.03 mL, 0.18 mmol), causing a colour change to dark red. The complex $[Ru(H)(Cl)(CO)(PPh_3)_3]$ (0.158 g, 0.166 mmol) was added, and the solution stirred for 1 h. Column chromatography, eluting with light petroleum-dichloromethane (7:3), gave **5** (0.1387 g, 93%).

Data for **5**: IR 1930 cm^{-1}. ^{1}H NMR: δ 7.80–7.10 (m, 35 H, Ph), −9.54 (t, J = 18.0 Hz, 1 H, Ru–H of major isomer), −9.86 (t, J = 17.5 Hz, 1 H, Ru–H of minor isomer). ^{13}C NMR (major isomer): δ 213.1 (s, C═S), 202.2 (t, J = 13 Hz, CO), 163.6 (s, CPh), 151.0 (t, J = 3 Hz, CS), 134.7-127.2 (m, Ph). ^{31}P NMR: δ 43.2 (major isomer), 43.4 (minor isomer). *Anal.* Calc. for $RuC_{46}H_{36}OP_2S_4$: C, 61.68; H,

4.02; S, 14.30. Found: C, 61.94; H, 4.17; S, 14.60%. Mass spectrum: *m/z* 896 $(M+H)^+$.

3.3. Synthesis of $[Ru(H)(MesC_3S_4)(CO)(PPh_3)_2]$ (**6**)

(i) One pot reaction

In the same manner as above, a solution of mesityl acetylene (0.25 mL, 2.22 mmol) in THF (18.2 mL) was treated sequentially with ^{n}BuLi (1.40 mL of 1.6 M solution, 2.24 mmol), carbon disulfide (0.15 mL, 2.5 mmol), and sulfur (0.21 g, 6.66 mmol of S). A portion of this solution (10.41 mL, containing 1.16 mmol of anion) was then added to a Schlenk tube containing a suspension of $[Ru(H)(Cl)(CO)(PPh_3)_3]$ (1.00 g, 1.04 mmol) in THF (20 mL). This reaction was stirred for 24 h, producing a blue-black solution. Column chromatography as above, eluting with light petroleum-dichloromethane (3:2) afforded a dark turquoise band of **6** (0.7072 g, 72%).

(ii) From 4-mercapto-5-mesityl-1,2-dithiole-3-thione

The heterocycle (50 mg, 0.19 mmol) in THF (10 mL) was deprotonated with triethylamine (0.03 mL, 0.19 mmol) and $[Ru(H)(Cl)(CO)(PPh_3)_3]$ (0.16 g, 0.17 mmol) was added. Stirring for 72 h produced a turquoise-green solution. Column chromatography as above, eluting with a 13:7 mixture of light petroleum and CH_2Cl_2, gave **6** (0.1414 g, 89%).

Data for **6**: IR: 1932 cm^{-1}. ^1H NMR: Major isomer: δ 7.70–7.28 (m, 30 H, Ph), 6.70 (s, 2 H of Mes), 2.20 (s, 3 H, Me), 1.75 (s, 6 H, 2Me), −9.47 (t, J = 19.5 Hz, 1 H, Ru–H). Minor isomer: δ 7.70–7.28 (m, 30 H, Ph), 6.80 (s, 2 H of Mes), 2.27 (s, 3 H, Me), 1.78 (s, 6 H, 2Me), −10.43 (t, J = 18.5 Hz, 1 H, Ru–H). ^{13}C NMR: Major isomer: δ 213.3 (s, C═S), 201.3 (t, J = 15 Hz, CO), 165.0 (s, CMes), 150.1 (t, J = 2 Hz, CS), 139.1 (s, CMe), 137.2 (s, 2CMe), 133.0–127.0 (m, Ph/Mes), 21.2 (s, Me), 19.6 (s, 2Me). Minor isomer: δ 216.1 (s, C═S), 204.0 (t, J = 15 Hz, CO), 165.5 (s, CMes), 153.0 (t, J = 2 Hz, CS), 139.5 (s, CMe), 137.8 (s, 2CMe), 133.0–127.0 (m, Ph/Mes), 21.3 (s, Me), 21.2 (s, 2Me). *Anal.* Calc. for $RuC_{49}H_{42}OP_2S_4$: C, 62.75; H, 4.48; S, 13.66. Found: C, 62.77; H, 4.56; S, 13.69%. Mass spectrum: *m/z* 938 $(M+H)^+$.

Table 3
Summary of crystallographic data for complexes **6** and **8**·CH_2Cl_2.

	6	**8**·CH_2Cl_2
Empirical formula	$C_{49}H_{42}OP_2RuS_4$	$C_{25}H_{24}Cl_2MoO_2S_8$
Formula weight	938.08	779.76
T/K	150(2)	100(2)
Crystal system	monoclinic	orthorhombic
Space group	$P2_1/c$	$P2_12_12_1$
a (Å)	17.854(2)	8.7520(14)
b (Å)	11.8777(13)	16.527(3)
c (Å)	21.597(2)	21.600(3)
α (°)	90	90
β(°)°	111.263(2)	90
γ (°)	90	90
V (Å^3)	4268.2(8)	3124.3(9)
Z	4	4
Density (calcd)(Mgm^{-3})	1.460	1.658
μ (mm^{-1})	0.676	1.150
F(0 0 0)	1928	1576
Crystal size (mm^3)	0.23 × 0.12 × 0.04	0.43 × 0.27 × 0.27
θ range for data collection (°)	1.22–27.60	1.55–27.56
Reflections collected (°)	36 483	34 990
Independent reflections	9567 [$R_{(int)}$ = 0.1189]	7133 [$R_{(int)}$ = 0.0460]
Data/restraints/parameters	9567/0/514	7133/0/349
Goodness-of-fit on F^2	1.010	1.047
Final R_1, wR_2 [$I > 2\sigma(I)$]	R_1 = 0.0608, wR_2 = 0.1384	R_1 = 0.0284, wR_2 = 0.0686
(all data)	R_1 = 0.1522, wR_2 = 0.1805	R_1 = 0.0311, wR_2 = 0.0698
Largest diff. peak and hole (e Å^{-3})	1.448 and −2.075	1.001 and −0.335

3.4. Synthesis of $[MoO_2(PhC_3S_4)_2]$ (**7**)

The heterocycle **4a** (80 mg, 0.33 mmol), dissolved in THF (2 mL) was added to a solution of $[MoO_2(acac)_2]$ (50 mg, 0.17 mmol) in methanol (10 mL). After stirring for 24 h, the red-brown precipitate was filtered off, washed with methanol, and recrystallised from CH_2Cl_2 and light petroleum. Yield 63.6 mg, 63%.

Data for **7**: IR (KBr): 946s, 894s cm^{-1}(Mo═O). ^1H NMR: δ 7.65–7.20 (m, Ph). *Anal.* Calcd. for $C_{18}H_{10}MoO_2S_8$: C, 35.40; H, 1.64; S, 41.97. Found: C, 35.87; H, 1.97; S, 42.55%. Mass spectrum *m/z* 610 (M^+).

3.5. Synthesis of $[MoO_2(MesC_3S_4)_2]$ (**8**)

A solution of heterocycle **4b** (100 mg, 0.35 mmol) in THF (2 mL) was added to $[MoO_2(acac)_2]$ (60 mg, 0.18 mmol) dissolved in methanol (10 mL). After stirring for 24 h, the orange-brown precipitate was filtered off, washed with methanol, and recrystallised from CH_2Cl_2 and light petroleum. Yield 49.6 mg, 43%.

Data for **8**: IR (KBr) 910s, 875s cm^{-1}. (Mo═O). ^1H NMR: δ 7.25 (s, 4H, CH), 2.15 (s, 6H, Me), 1.60 (s, 12H, 2Me). *Anal.* Calcd. for $C_{24}H_{22}MoO_2S_8$: C, 41.50; H, 3.17; S, 36.89. Found: C, 41.58; H, 3.20; S, 37.22%. Mass spectrum *m/z* 696 $(M)^+$.

3.6. Crystal structure determinations

Details of the data collection and refinement are collected in Table 3. A Bruker Smart CCD area detector with Oxford Cryosystems low temperature system was used for data collection. Complex scattering factors were taken from the program package SHELXTL as implemented on a Viglen Pentium computer [14]. Hydrogen atoms were placed geometrically and refined in riding mode with U_{iso} constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the carrier atom.

Acknowledgements

We thank the University of Sheffield for support. We also thank the many undergraduate students who prepared $[RuH(Cl)(CO)(PPh_3)_3]$ as part of their laboratory course.

Appendix A. Supplementary material

CCDC 731884 (**8**) and 731885 (**6**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/data_request/cif.

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2009.09.015.

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