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TRIPLE BONDS BETWEEN MOLYBDENUM AND TUNGSTEN ATOMS SUPPORTED BY THIOLATE LIGANDS: $(RS)_3M{\equiv}M(SR)_3$

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Abstract—Toluene solutions of $M_2(NMe_2)_6$ and mesitylene thiol react at 80°C to yield $M_2(S\text{-mes})_6$ compounds (M = Mo and W) with the liberation of $HNMe_2$. Similarly $M_2(OBu^t)_6$ in toluene reacts with mesitylene thiol under reflux to yield $M_2(S\text{-mes})_6$ compounds. In all cases the reactions also yield some decomposition products. The compounds $M_2(SBu^t)_2(NMe_2)_4$ react with mesitylene thiol in hexane at room temperature to give $M_2(S\text{-mes})_6$ compounds without significant side reactions. The $M_2(S\text{-mes})_6$ compounds are the first examples of compounds containing triple bonds between molybdenum and tungsten atoms supported exclusively by thiolate ligands. Pertinent bond distances (Å) and angles (°) are: M—M = 2.228(1) (Mo), 2.312(2) (W); M—S = 2.325(2) (Mo), 2.32(1) (W); M—M—S = 96.6(1) (Mo), 97.2(6) (W). The preparations of the new compounds $W_2(SBu^t)_2(NMe_2)_4$ and $W_2(OBu^t)_2(S\text{-mes})_4$ are reported.

Homoleptic compounds of formula $X_3M{\equiv}MX_3$ are known for X = bulky β-elimination stabilized alkyls[1,2] (e.g. CH_2CMe_3 and CH_2SiMe_3), NMe_2[3,4] and OR,[5,6] where R = a bulky alkyl or trialkylsilyl group (e.g. *t*-Bu, *i*-Pr, CH_2-*t*-Bu, $SiMe_3$ and $SiEt_3$). We have wondered for some time whether this series could be extended to include thiolato (mercaptide), SR, ligands and phosphido, PR_2, ligands. Though there was no reason to believe that such ligands could not exist our initial attempts to prepare $(RS)_3Mo{\equiv}Mo(SR)_3$ compounds were thwarted by problems arising from molybdenum's high affinity for sulphur, facile C—S bond cleavages, polymerizations by μ-SR formation and oxidation of the Mo_2^{6+} unit.[7] We were able, however, to attach two SR ligands to the $(Mo{\equiv}Mo)^{6+}$ unit in the preparation of the compounds 1,2-$Mo_2(SR)_2(NMe_2)_4$ [eqn (1)]:[8]

$$1,2\text{-}Mo_2Cl_2(NMe_2)_4 + 2LiSR \rightarrow 1,2\text{-}Mo_2(SR)_2(NMe_2)_4 + 2LiCl \quad (1)$$

where R = Me and Bu^t.

Subsequently, by the use of the bulky aromatic thiol mesitylene thiol we were able to prepare compounds of formula $Mo_2X_2(S\text{-mes})_4$ (mes = mesitylene, 2,4,6-trimethylphenyl) according to eqn (2):[9]

$$Mo_2X_6 + 6HS\text{-mes} \rightarrow 1,2\text{-}Mo_2X_2(S\text{-mes})_4 + 4XH \quad (2)$$

where X = NMe_2, OBu^t, OPr^i and OCH_2Bu^t.

Here the use of an aromatic thiol suppresses the facile C—S bond cleavage reaction and the 2,6-dimethyl substituents on the aromatic ring suppress oligomerization reactions by μ-SR formation. The use of only one methyl substituent in the 2-position (*ortho*) led to polymeric materials, but the use of two methyl groups, one in each of the *ortho* positions in the mesitylene group, led to only partial substitution. It was not clear why only partial substitution occurred in eqn (2). Steric or electronic factors, or both, could be involved. However, we were encouraged in our quest for a successful synthesis of $M_2(SR)_6$ compounds since it seemed that by relatively subtle changes in the steric properties of the aromatic thiol or the leaving group [X in eqn (2)] we could achieve a synthesis of the target compounds. We describe here the fulfillment of this goal.[10]

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RESULTS AND DISCUSSION

Syntheses

Previously we had found that the $Mo_2(SR)_2(NMe_2)_4$ compounds, where R = Me and Bu^t, reacted with alcohols (Bu^tOH and Pr^iOH) in hydrocarbon solvents to give dimolybdenum hexaalkoxides[8] and that these do not react with bulky alkyl thiols such as Bu^tSH. However, from eqn (2) it was clear that aromatic thiols would replace alkoxide ligands but it was not clear why the reaction stopped with only partial substitution. Are the $M_2X_2(S\text{-mes})_4$ compounds kinetically or thermodynamically favoured in reaction (2)? If one considers that either an equilibrium $MoOR + R'SH \rightleftharpoons MoSR' + ROH$, or a higher activation energy for the last replacements of the two OR groups on the Mo_2^{6+} centre is at fault then merely increasing the temperature of the reaction and, if necessary, the selective removal of the alcohol by azeotrope distillation, would drive the reaction to the desired $Mo_2(SR')_6$ compounds. Indeed we find that in refluxing toluene, $M_2X_2(S\text{-mes})_4$ compounds do react further with excess mesitylene thiol to give $M_2(S\text{-mes})_6$ compounds. However, under these more forcing conditions some thermal decomposition occurs. Little can be said concerning these decomposition products, though based on a crystallographic study[11] a very minor product, *ca* 2–5% of the sample, obtained in the reaction between $W_2(NMe_2)_6$ and mesitylene thiol, is formulated as the salt $[Me_2\overline{NCH_2CH_2CH_2CH}S\text{-mes}]^+$ $[WO(S\text{-mes})_4]^-$. Crystallographically there can be little if any doubt concerning the authenticity of the oxo tungsten tetrakismesitylenethiolato moiety and the origin of the oxo group must be questioned. A plausible source is from THF and the cation $[Me_2\overline{NCH_2CH_2CH_2CH}S\text{-mes}]^+$ would appear to also involve the C_4-fragment of THF coupled with the S-mes and Me_2N units.

A cleaner route, one involving milder conditions, involves the reaction shown in eqn (3). The aliphatic thiolate ligands are readily

$$M_2(SBu^t)_2(NMe_2)_4 + 6mesSH \rightarrow M_2(S\text{-mes})_6 + 4HNMe_2 + 2Bu^tSH \quad (3)$$

replaced, along with the NMe_2 groups, by the arylthiolato ligands.

Physicochemical properties

The new $M_2(S\text{-mes})_6$ compounds are orange-red crystalline compounds that are soluble in aromatic hydrocarbon solvents. The compounds are air sensitive and thermally unstable above 80°C. They cannot be purified by sublimation. They are diamagnetic and show ^1H NMR data consistent with the observed $X_3M{\equiv}MX_3$ structure. Analytical, IR and ^1H NMR data are given in Experimental.

Solid state and molecular structures

The sample of $Mo_2(S\text{-mes})_6$ that was examined was crystallized from toluene/hexane solutions and, in the space group $R\bar{3}$, hexane molecules are contained in the unit cell. The dimolybdenum compound has crystallographically imposed S_6 symmetry. An ORTEP view of the molecule giving the atom number scheme used in the tables is shown in Fig. 1 and a view down the Mo—Mo bond is given in Fig. 2.

The ditungsten compound crystallized from toluene/hexane in the space group $P\bar{1}$. Molecules of $W_2(S\text{-mes})_6$ have a crystallographically imposed centre of inversion. A view of the $W_2(S\text{-mes})_6$ molecule showing the atom number scheme is given in Fig. 3.

As would be expected the Mo_2 and W_2 containing compounds are very similar and this is shown by a comparison of selected pertinent structural parameters in Table 1. A comparison of the structural parameters associated with the central $X_3M{\equiv}MX_3$ units in structurally characterized homoleptic alkyls, amides, alkoxides and thiolates is given in Table 2. The following points are worthy of note.

(1) For related pairs of molecules the W—W distance is longer by 0.08 or 0.09 Å than the Mo—Mo distance.

(2) The M—X distance is, within the 3σ criteria, the same for Mo—X and W—X.

(3) The M—M—X angles span a small range, 96.6–103.7°. The smaller angles seen here for the thiolates are easy to understand in terms of the arrangement of all six mesityl groups which are distal with respect to the central $(M{\equiv}M)^{6+}$ core. In the alkoxide molecule $Mo_2(OCH_2Bu^t)_6$ we see four distal and two proximal arrangements of the $—CH_2Bu^t$ groups leading, respectively, to larger and smaller M—M—O angles.

(4) The M—S distances are essentially the same as were found[9] in the structure of $Mo_2(OPr^i)_2(S\text{-mes})_4$. From the arguments previously presented, we conclude that they are shorter by *ca* 0.10–0.15 Å than those expected for a pure MoS σ-bond and this shortening is due to S to M π-bonding.

Concluding remarks

The use of bulky arylthiolato ligands has recently been shown to lead to unusual monomeric compounds. For example, Koch and co-workers[12]

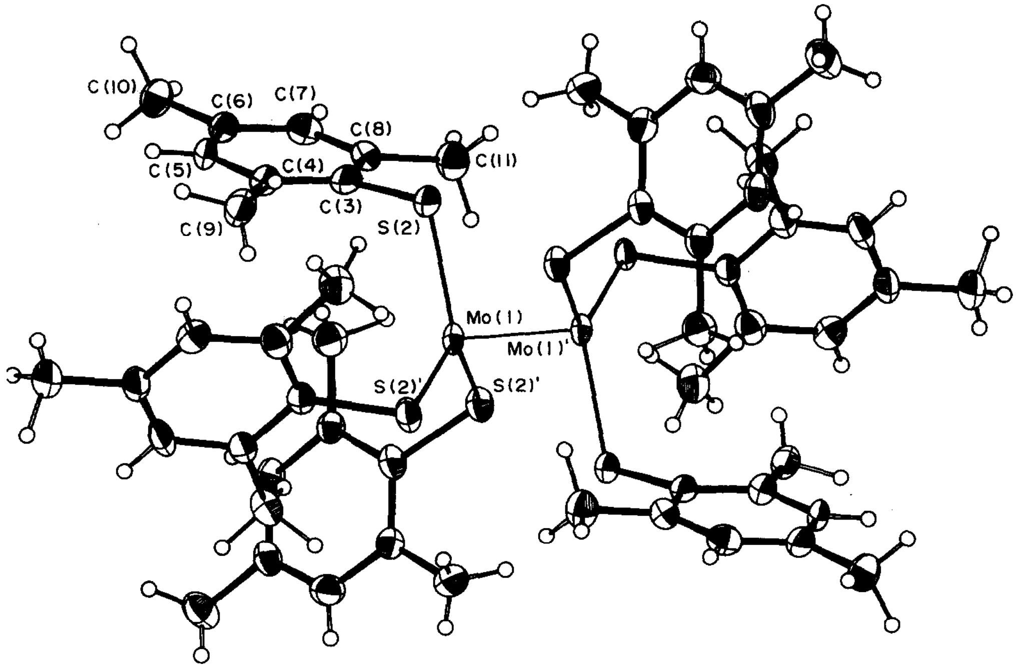

Fig. 1. An ORTEP view of the $Mo_2(S\text{-}mes)_6$ molecule giving the atom number scheme used in the tables.

were able to prepare the first example of a stable Fe(III) tetrathiolate using the metathetic exchange reaction:

$$FeCl_3 + 4LiSC_{10}H_{13} + Et_4NBr \rightarrow [Et_4N][Fe(SC_{10}H_{13})_4] + 3LiCl + LiBr,$$

where $SC_{10}H_{13}$ is the anion derived from 2,3,5,6-tetramethylphenylthiol. Attempts to extend this to Ru and Os gave rise to the first examples of the five-coordinate ruthenium state:

$$M(SC_{10}H_{13})_4(CH_3CN).^{13}$$

Dilworth and co-workers[14] have used the 2,4,6-triisopropylphenylthiolate ligand to obtain some novel carbonyl and nitrosyl mononuclear anionic

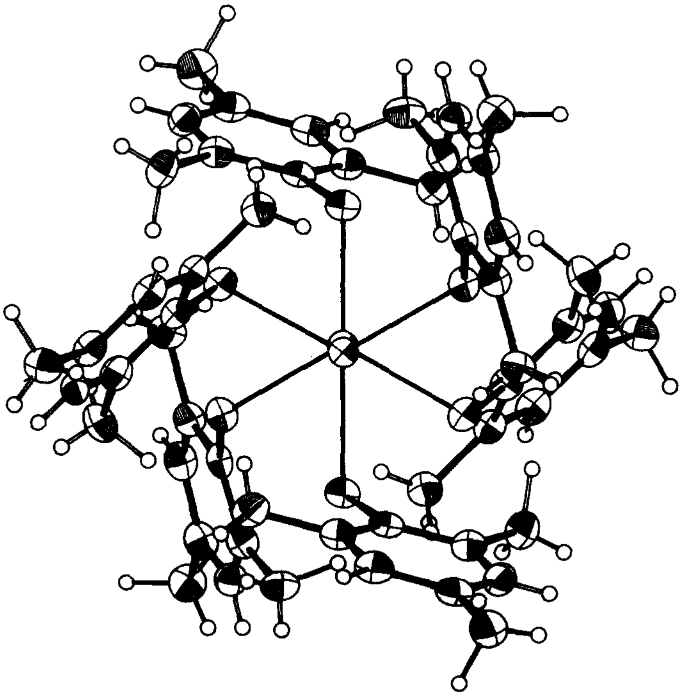

Fig. 2. An ORTEP view of the $Mo_2(S\text{-}mes)_6$ molecule looking down the Mo—Mo bond.

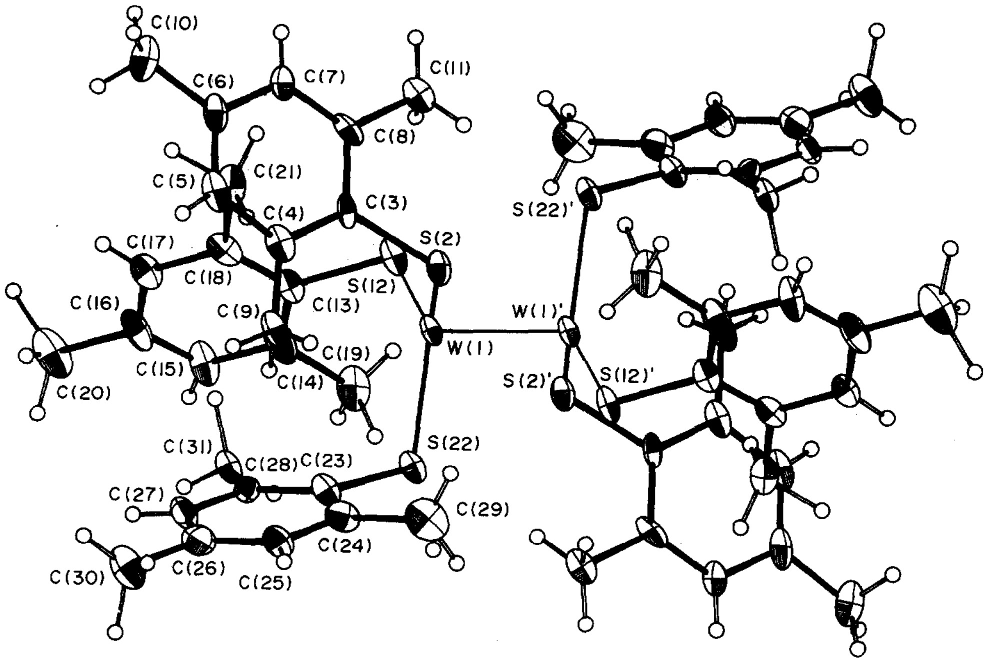

Fig. 3. An ORTEP view of the $W_2(S\text{-mes})_6$ molecule giving the atom number scheme used in the tables.

Table 1. Selected bond distances (Å) and angles (°) for the $M_2(SC_6H_2Me_3)_6$ molecules

A	B		Distance Mo	Distance W
M(1)	M(1)'		2.228(1)	2.313(2)
M(1)	S(2)		2.325(2)	2.311(4)
M(1)	S(12)			2.321(4)
M(1)	S(22)			2.303(4)
S(2)	C(3)		1.792(5)	1.79(1)
S(12)	C(13)			1.81(1)
S(22)	C(23)			1.78(1)
C	C(aromatic)*		1.38	1.39
C	C(methyl)*		1.50	1.50

A	B	C	Angle Mo	Angle W
M(1)'	M(1)	S(2)	96.6(2)	96.5(1)
M(1)'	M(1)	S(12)		97.8(1)
M(1)'	M(1)	S(22)		97.2(1)
S(2)	M(1)	S(12)	118.7(1)	119.7(1)
S(2)	M(1)	S(22)		117.0(1)
S(12)	M(1)	S(22)		118.7(1)
M(1)	S(2)	C(3)	110.1(2)	107.4(4)
M(1)	S(12)	C(13)		108.9(4)
M(1)	S(22)	C(23)		108.6(5)

* Averaged distance.

complexes of molybdenum(II), e.g.

$$[Mo(CO)_2(SC_6H_2Pr^i_3)_3]^-.$$

We have shown here that by an appropriate synthetic strategy compounds of formula $(RS)_3M\equiv M(SR)_3$ can be prepared for molybdenum and tungsten. There exists now the opportunity to compare the chemistry of the $(M\equiv M)^{6+}$ unit supported by the closely related alkoxide[15] and thiolate ligands.

Further studies are in progress.

EXPERIMENTAL

General procedures, including the preparations of $M_2(NMe_2)_6$ compounds, where M = Mo and W, $W_2(O\text{-}t\text{-}Bu)_6$ and $Mo_2(S\text{-}t\text{-}Bu)_2(NMe_2)_4$ have been described previously.[9] Mesitylene thiol was prepared from the reaction of mesitylene Grignard reagent and elemental sulphur as described below.

^1H NMR data were obtained from toluene-d_8 solutions using a Nicolet 360 spectrometer. IR spectra were recorded as Nujol mulls using a Perkin–Elmer 283 spectrophotometer. Elemental analyses were obtained from Alfred Bernhard Microanalytical Laboratories (Elbach, F.R.G.).

Preparations

Mesitylene thiol. To a solution of mesitylene magnesium bromide (135 cm³ of a 1 *M* solution in

Table 2. Comparison of selected structural parameters for homoleptic compounds having a central $X_3M{\equiv}MX_3$ unit where M = Mo, W and X = C, N, O or S

Compound	M—M (Å)	M—X av. (Å)	M—M—X (°)	Reference
$Mo_2(CH_2SiMe_3)_6$	2.167(3)	2.13	100.6	1
$W_2(CH_2SiMe_3)_6$	2.255(2)	2.14	101.7	2
$Mo_2(NMe_2)_6$	2.214(3)	1.98	103.7	3
$W_2(NMe_2)_6$	2.294(2)	1.97	103.3	4
$Mo_2(OCH_2CMe_3)_6$	2.222(2)	1.88	103.0	5
$Mo_2(SC_6H_2Me_3)_6$	2.228(1)	2.325	96.6	This work
$W_2(SC_6H_2Me_3)_6$	2.312(2)	2.32	97.1	This work

THF), elemental sulfur (4.32 g, 0.35 g atoms) was added over a period of 20 min. The solution was hydrolyzed by the addition of H_2O (20 cm^3) followed by the slow addition of 6 *N* HCl (30 cm^3). The THF layer which separated was collected and dried over $MgSO_4$ (12 h). Upon removing the THF at reduced pressure a clear yellow oil remained which was distilled at 50°C, 30 torr yielding 2,4,6-trimethylphenyl thiol (mesitylene thiol) 16.74 g (81% yield). This was then dissolved in THF (15 cm^3) to give a 7.33 *M* solution which was stored over molecular sieves and under an atmosphere of N_2.

$W_2(NMe_2)_2(S\text{-mes})_4$. $W_2(NMe_2)_6$ (0.46 g, 0.73 mmol) was dissolved in hexane (20 cm^3) to give a clear yellow solution. Mesitylene thiol (0.7 cm^3 of a 7.3 *M* solution in THF, 7 equiv) was added via syringe. The solution was allowed to stir at room temperature for 12 h. A gradual darkening of the solution occurred and a microcrystalline yellow-orange precipitate formed which was collected and dried *in vacuo*: $W_2(NMe_2)_2$ $(S\text{-mes})_4$ (0.71 g, 0.67 mmol, 92% yield). Found: C, 45.01; H, 5.27; N, 2.81; S, 11.98%. Calc. for $W_2(NMe_2)_2(SC_6H_2Me_3)_4$: C, 45.28; H, 5.28; N, 2.67; S, 12.07%.

IR data: 1595 (m), 1290 (m), 1250 (ms), 1170 (m), 1140 (m), 1050 (s), 1025 (s), 935 (s), 885 (m), 840 (s), 800 (m), 730 (ms), 710 (m), 690 (m), 600 (m), 570 (wm), 550 (wm) cm^{-1}.

$W_2(O\text{-t-Bu})_2(S\text{-mes})_4$. $W_2(O\text{-}t\text{-Bu})_6$ (0.3 g, 0.372 mmol) was dissolved in hexane (15 cm^3). Mesitylene thiol (1.1 cm^3 of a 7.3 *M* solution in THF) was added via syringe at room temperature. The solution was stirred for 24 h. Little colour change was detectable but a reddish precipitate formed which was collected by filtration and dried *in vacuo*. The solids were redissolved in toluene (5 cm^3) warmed to *ca* 50°C. Red-orange crystals were obtained upon cooling to −15°C: $W_2(O\text{-}t\text{-Bu})_2(S\text{-mes})_4$ (0.34 g, 82% yield based on W). Found: C, 47.23; H, 5.55; S, 11.45%. Calc. for $W_2(O\text{-}t\text{-Bu})_2(SC_6H_2Me_3)_4$: C, 47.35; H, 5.72; S, 11.24%.

IR data: 1595 (ms), 1290 (m), 1260 (m), 1235 (m), 1160 (s), 1025 (ms), 965 (vs), 870 (w), 840 (s), 790 (w), 605 (wm), 550 (m), 470 (w), 420 (wm) cm^{-1}.

^1H NMR data at +21°C: O-*t*-Bu, $\delta = 1.90$ (9H); $C_6H_2Me_3$, δ(*p*-methyl) = 2.05 (6H), δ(*o*-methyl) = 2.15 (12H), *meta*-H, $\delta = 6.4$ (δ in ppm relative to Me_4Si).

$W_2(S\text{-t-Bu})_2(NMe_2)_4$. $W_2Cl_2(NMe_2)_4$ (0.35 g, 0.57 mmol) was dissolved in toluene (25 cm^3). LiS-*t*-Bu (0.112 g, 1.2 mmol) was added and the reaction mixture was left to stir at room temperature for 48 h. The colour darkened from a yellow-orange to an orange-brown. The solution was evaporated to dryness *in vacuo* and hexane was added. Solids (LiCl and other insoluble materials) were removed by filtration and the hexane soluble filtrate was collected. Cooling to −15°C gave a yellow-green finely divided microcrystalline precipitate: $W_2(S\text{-}t\text{-Bu})_2(NMe_2)_4$ (0.28 g, 68% yield based on W). Found: C, 26.41; H, 5.79; N, 7.81; S, 8.78%. Calc. for $W_2(S\text{-}t\text{-Bu})_2(NMe_2)_4$: C, 26.59; H, 5.82; N, 7.65; S, 8.86%. $W_2(S\text{-}t\text{-Bu})_2(NMe_2)_4$ sublimes with considerable decomposition (80°C, 10^{-2} torr) but does yield a molecular ion in the mass spectrometer: $M^+ = 722$ based on ^{184}W.

IR data: 1260 (wm), 1245 (ms), 1145 (s), 1039 (m), 1032 (m), 952 (s), 938 (vs), 800 (m), 565 (ms), 535 (m), 340 (wm).

^1H NMR data, 34°C in benzene-d_6: S-*t*-Bu, $\delta = 1.40$ (9H); NMe_2, $\delta = 3.35$ (12H) (δ in ppm relative to Me_4Si).

$M_2(S\text{-mes})_6$ *compounds*. As noted previously the reaction between $M_2(NMe_2)_6$ compounds, where M = Mo and W, and mesitylene thiol yields $M_2(NMe_2)_2(S\text{-mes})_4$ compounds when the reaction is carried out in hexane at room temperature. The latter compounds are virtually insoluble in hexane.

However, a similar result is obtained when the reaction is carried out in toluene, for which the M_2(S-mes)$_4$(NMe$_2$)$_2$ compounds are appreciably soluble but when the solution is refluxed for 6–8 h a further reaction with mesitylene thiol occurrs. M_2(S-mes)$_6$ compounds are formed and can be obtained as red crystals by careful recrystallation from toluene. However, the bulk material is contaminated with other products giving poor analytical data which is indicative of the presence of nitrogen containing materials. A better preparative route involves the use of the M_2(S-*t*-Bu)$_2$(NMe$_2$)$_4$ compounds and is given below.

Mo_2(S-*t*-Bu)$_2$(NMe$_2$)$_4$ (0.316 g, 0.58 mmol) was dissolved in hexane (15 cm^3). An excess of mesitylene thiol (2 cm^3 of a 7.33 *M* solution in THF) was added via syringe at room temperature. The solution was stirred for *ca* 10 h during which time some tan-coloured solids precipitated. These were removed by filtration and discarded. The orange-red filtrate was collected and reduced in volume *in vacuo*. The solution was slowly cooled to -5°C yielding a pale-red powder and some larger red crystals: Mo_2(S-mes)$_6$ (0.272 g, 43% yield based on Mo). Found: C, 59.21; H, 5.89; S, 17.61%. Calc. for $Mo_2(SC_6H_2Me_3)_6$: C, 59.01; H, 6.01; S, 17.48%.

IR data: 1598 (m), 1300 (mw), 1260 (ms), 1100 (w, br), 1030 (m, br), 845 (s), 795 (w, br), 610 (w), 550 (m), 415 (wm) cm^{-1}.

^{1}H NMR data, +34°C, benzene-d_6 solvent: *ortho*-methyls, $\delta = 2.15$; *para*-methyl, $\delta = 2.09$; *meta*-H, $\delta = 6.50$ (δ in ppm relative to Me$_4$Si).

W_2(S-mes)$_6$ was prepared in a similar manner, but employing W_2(S-*t*-Bu)$_2$(NMe$_2$)$_4$, and was obtained as a red powder or red crystals from toluene solutions in 35–40% yield based on W. Found: C, 50.53; H, 5.28; S, 15.22%. Calc. for $W_2(S_2C_6H_2Me_3)_6$: C, 50.86; H, 5.18; S, 15.07%.

IR data: 1600 (m), 1300 (m), 1260 (ms), 1180 (wm), 1100 (w, br), 1030 (s), 890 (m), 850 (s), 730 (ms), 610 (w), 555 (m), 470 (mw), 420 (m) cm^{-1}.

^{1}H NMR data, 35°C in benzene-d_6 solvent: *ortho*-methyls, $\delta = 2.18$; *para*-methyls, $\delta = 2.00$; *meta*-H, $\delta = 6.55$ (δ in ppm relative to Me$_4$Si).

Crystallographic studies

General operating procedures and listings of programs have been previously given.[16] Crystal data for the M_2(S-mes)$_6$ compounds are summarized in Table 3.

Mo_2(S-mes)$_6$. A suitable nearly equidimensional fragment was cleaved from a larger cubic shaped crystal which exhibited severe faulting on the faces. Examination of the chosen sample indicated it was crystallographically satisfactory. The sample was transferred to the goniostat using standard inert atmosphere handling techniques and cooled to -162°C for characterization and data collection.

An examination of reciprocal space revealed a trigonal lattice with rhombohedral centring. Subsequent solution and refinement confirmed the space group $R\bar{3}$. The structure was solved by "inspection", in that with $Z = 3$ (yielding a reasonable density), the only permissible location of the molecule was at a $\bar{3}$ site in the lattice. Assuming a Mo–Mo separation of 2.3 Å, a trial Mo was placed along the three-fold axis and a Fourier located the remaining non-hydrogens in the molecule. A difference map phased on the isotropic coordinates of the 11 unique atoms revealed three peaks of intensity 1.6–3.5 e Å^{-3} with all remaining peaks below 0.8 e Å^{-3}. The three larger peaks are apparently due to a disordered hexane solvent along the three-fold axis. When three partial occupancy carbon atoms were placed in these positions, the residual (*R*) dropped from 0.065 to 0.043.

A final difference Fourier after the full-matrix refinement was featureless, the largest peak being 0.4 e Å^{-3}. There was no evidence of hydrogens associated with the hexane. It should be noted that the solvent is poorly defined and the bond distances and angles are quite poor.*

W_2(S-mes)$_6$. A suitable crystal was cleaved to form a nearly equidimensional fragment which was transferred to the goniostat using standard inert atmosphere handling techniques. A systematic search of a limited hemisphere of reciprocal space revealed no systematic absences or symmetry, leading to the assignment of a triclinic space group. Subsequent solution and refinement of the structure (as well as statistical tests) confirmed the centrosymmetric space group $P\bar{1}$.

The location of the unique tungsten atom was achieved by direct methods and all remaining non-hydrogen atoms were located in a difference Fourier phased on the metal parameters. Hydrogen atoms were visible in a difference map phased on the non-hydrogens, and were included in the final stages of refinement (anisotropic thermal parameters for W, S, C; isotropic parameters for H).

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* Final atomic positional and thermal parameters and F_o/F_c values have been deposited as supplementary material with the Editor, from whom copies are available on request. Atomic coordinates have also been submitted to the Cambridge Crystallographic Data Centre. The complete structural reports for Mo_2(S-mes)$_6$ and W_2(S-mes)$_6$, MSC Report Nos. 83046 and 84011, are available in microfiche form only from the Indiana University Library at a cost of $2.50 per report.

Table 3. Summary of crystal data

	I	II
Empirical formula	$Mo_2S_6C_{54}H_{66}\cdot xC_6H_{14}$	$W_2S_6C_{54}H_{66}$
Colour of crystal	Red	Red
Crystal dimensions (mm)	0.25 × 0.24 × 0.22	0.05 × 0.04 × 0.04
Space group	$R\bar{3}$	$P\bar{1}$
Cell dimensions		
Temperature (°C)	−162	−156
a (Å)	15.361(10)	11.019(9)
b (Å)	15.361(10)	11.692(10)
c (Å)	20.929(13)	11.559(10)
α (°)		62.63(4)
β (°)		97.03(4)
γ (°)	120	85.32(5)
Z (molecules/cell)	3	1
Volume (Å^3)	4276.79	1296.39
Calculated density (g cm^{-3})	1.381	1.633
Wavelength (Å)	0.71069	0.71069
Molecular weight	1185.53	1275.18
Linear absorption coefficient (cm^{-1})	6.762	47.950
Detector-to-sample distance (cm)	22.5	22.5
Sample-to-source distance (cm)	23.5	23.5
Average omega scan width at half-height	0.25	0.25
Scan speed (° min^{-1})	4.0	4.0
Scan width (° + dispersion)	2.0	2.0
Individual background (s)	10	10
Aperture size (mm)	3.0 × 4.0	3.0 × 4.0
2θ range (°)	6–45	6–50
Total number of reflections collected	3769	4693
Number of unique intensities	1259	4219
Number with $F > 0.0$	1195	
Number with $F > \sigma(F)$	1136	
Number with $F > 2.33\sigma(F)$	1083	
Number with $F > 3.00\sigma(F)$		3795
$R(F)$	0.0346	0.0620
$R_w(F)$	0.0295	0.0594
Goodness of fit for the last cycle	2.023	1.109
Maximum δ/σ for the last cycle	0.05	0.05

A final difference Fourier was featureless, the largest peak being 0.58 e Å^{-3}. Psi scans were essentially flat, so no absorption correction was performed.

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