

Synthesis and structural characterisation of *trans*-Cp₂Mo₂O₂(μ-O)(μ-Te) and *cis*-Cp₂Mo₂O₂(μ-O)(μ-S)

Pradeep Mathur^{a,*}, Sanjukta Ghose^a, Md. Munkir Hossain^a, Peter B. Hitchcock^b,
John F. Nixon^b

^a Chemistry Department, Indian Institute of Technology, Bombay 400 076, India

^b School of Chemistry and Molecular Sciences, University of Sussex, Brighton BN1 9QJ, UK

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Abstract

The two compounds, *trans*-Cp₂Mo₂O₂(μ-O)(μ-Te) and *cis*-Cp₂Mo₂O₂(μ-O)(μ-S), were isolated in 31% and 36% yields respectively, when a benzene solution of Cp₂Mo₂Fe₂(CO)₇(μ₃-S)(μ₃-Te) was refluxed for 3 h in the presence of air. Characterisation was carried out by IR, ¹H NMR and ¹²⁵Te NMR spectroscopy. The structures, established by single crystal X-ray diffraction analysis, consist of an oxo-bridged dimolybdenum unit containing a μ₂-Te atom in one case and a μ₂-S atom in the other. Also attached to each Mo atom are a Cp ligand and a terminally bonded oxo group. © 1997 Elsevier Science S.A.

Keywords: Molybdenum; Tellurium; Sulphur; Oxo

1. Introduction

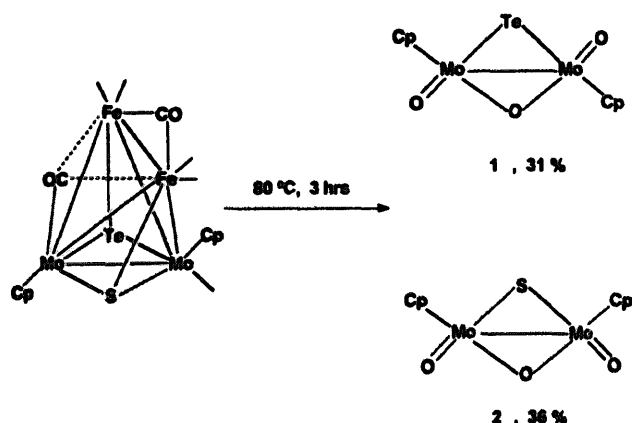
Transition metal clusters containing single atom ligands derived from several main group elements of the periodic table have been the centre of considerable attention in recent years [1]. The use of Group 16 elements for the stabilization of transition metal carbonyl complexes is now well established [2]. Recently, we have prepared several types of mixed-metal, mixed-chalcogenide cluster complexes under facile reaction conditions, and have identified the class of compounds, Cp₂Mo₂Fe₂(CO)₇(μ₃-E)(μ₃-E') (E, E' = S, Se, Te) as a starting material for obtaining other chalcogen-bridged cluster compounds [3]. Metal-oxo compounds are interesting because of their potential to serve as structural models for metalloproteins involved in oxygen transport and oxygen metabolism in living systems [4]. Apart from this, metal-oxo derivatives have long been used successfully as oxidants in organic synthesis and also in industrial processes involving homogeneous as well as heterogeneous catalysts [5]. Recently, we described the synthesis and structure of a novel imido-bridged complex, Cp₂Mo₂O₂(μ-Te)(μ-NC₆H₅), isolated from the

thermolytic reaction between Cp₂Mo₂Fe₂(CO)₇(μ₃-Te)₂ and PhN₃ [6]. In continuation of our interest in metal-oxo complexes, we report here on the thermolysis of Cp₂Mo₂Fe₂(CO)₇(μ₃-S)(μ₃-Te) in the presence of air to form two oxo-bridged dimolybdenum complexes, containing a μ₂-Te atom in one case and a μ₂-S atom in the other, and their complete spectroscopic and structural characterisation.

2. Results and discussion

When a benzene solution of Cp₂Mo₂Fe₂(CO)₇(μ₃-S)(μ₃-Te) was refluxed in the presence of air for 3 h, the following two compounds were isolated: Cp₂Mo₂O₂(μ-O)(μ-Te) (**1**) (31%) and Cp₂Mo₂O₂(μ-O)(μ-S) (**2**) (36%) (Scheme 1). Compound **2** has previously been prepared by the reaction of Cp₂Mo₂(CO)₄ and As₄S₄ and characterised by IR and ¹H NMR spectroscopy [7]. We have identified **1** and **2** on the basis of comparison of their IR and ¹H NMR spectroscopic features with that reported for **2** earlier and with that of the related compound Cp₂Mo₂O₂(μ-O)(μ-Se) [7]. The infrared spectra for compounds **1** and **2** show intense bands at 907 cm⁻¹ and 910 cm⁻¹ respectively due to the presence of terminally bonded oxygen atoms

* Corresponding author.



Scheme 1.

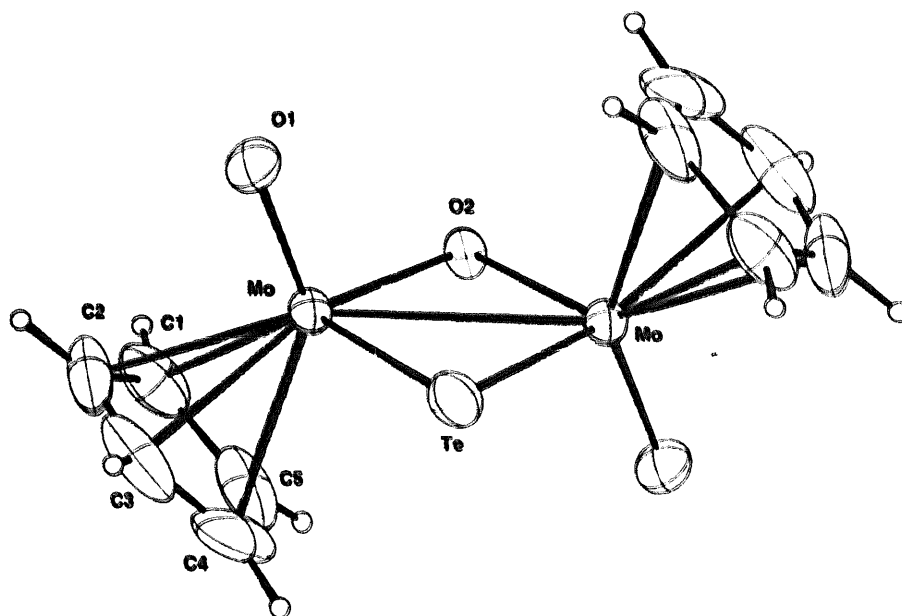
[8,9]. There is also the presence of medium intensity bands in the range $810\text{--}845\text{ cm}^{-1}$ due to the C–H groups [7,10]. The ^1H NMR spectra for 1 and 2 show a single sharp peak each at $\delta = 6.09\text{ ppm}$ and $\delta = 6.21\text{ ppm}$ respectively, which can be attributed to the two equivalent Cp ligands in each case [11]. These chemical shifts are comparable to the chemical shift of $\delta = 6.11\text{ ppm}$ observed for the Cp ligands in the compound $\text{trans-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-Se})(\mu\text{-O})$ [7] and the chemical shift of $\delta = 6.28\text{ ppm}$ observed for the Cp ligand in $\text{Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-Te})(\mu\text{-NC}_6\text{H}_5)$ [6]. The ^{125}Te NMR spectrum of compound 1 shows a single sharp signal at $\delta = 804\text{ ppm}$, which is 105 ppm upfield of the signal observed for $\text{Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-Te})(\mu\text{-NC}_6\text{H}_5)$ [6].

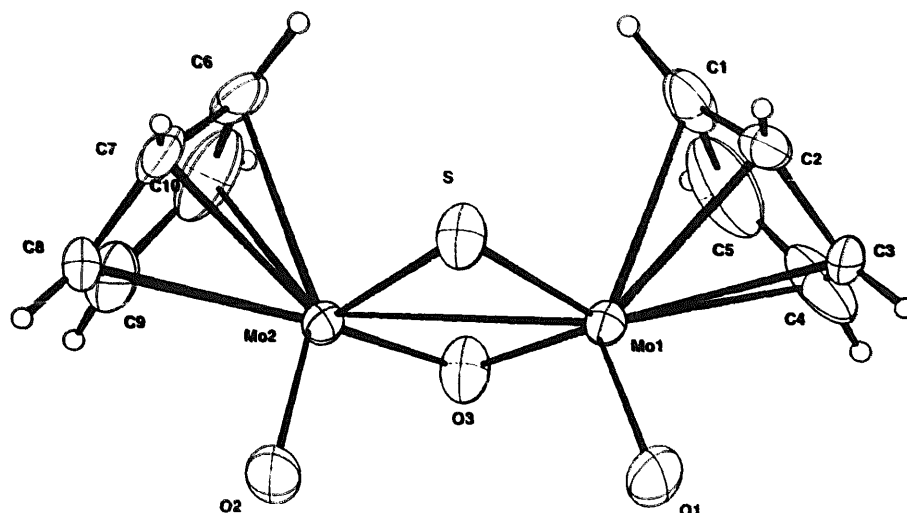
Orange crystals of $\text{trans-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-Te})$ (1) and reddish-orange crystals of $\text{cis-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-S})$ (2) were grown from dichloromethane–hexane solvent mixtures at 0°C and their structures were eluci-

dated by single crystal X-ray diffraction analysis. The molecular structures of 1 and 2 are depicted in Figs. 1 and 2 respectively. The crystal data and details of measurement are given in Table 1. Fractional atomic coordinates of 1 and 2 are given in Tables 2 and 3 respectively. Selected bond distances and bond angles for compounds 1 and 2 are given in Tables 4 and 5 respectively.

The structure of $\text{trans-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-Te})$ (1) consists of a dimolybdenum unit bridged by a $\mu_2\text{-Te}$ atom on one side and by a $\mu_2\text{-O}$ atom on the other. In addition, each Mo atom has a Cp ligand and a terminally bonded oxo group attached to it. The terminal oxo groups exhibit a trans orientation along the Mo–Mo bond. The structure of $\text{cis-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-S})$ (2) also consists of a dimolybdenum core which is bridged by a $\mu_2\text{-S}$ atom on one side and by a $\mu_2\text{-O}$ atom on the other. The terminal oxo groups, as well as the cyclopentadienyl ligands, are in cis orientation along the Mo–Mo axis.

The average Mo–Te bond distance in $\text{trans-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-Te})$ (1) is 2.574 \AA . This is comparable to the average Mo–Te bond length (2.634 \AA) in the related cluster $\text{Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-Te})(\mu\text{-NC}_6\text{H}_5)$ reported earlier by us [6]. The average Mo–S bond length in $\text{cis-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-S})$ (2) is 2.216 \AA . On comparison of these values with other average Mo–E bond lengths in related clusters reported in literature, $\text{trans-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-Se})$ (2.451 \AA) and $\text{trans-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-S})$ (2.319 \AA) [7], it is observed that there is a gradual increase in the Mo–E bond lengths with replacement of the smaller chalcogen atoms by the larger ones. The Mo–Mo bond length in $\text{trans-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-Te})$ (1) ($2.964(1)\text{ \AA}$) is greater than

Fig. 1. Molecular structure of $\text{trans-Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-Te})$.

Fig. 2. Molecular structure of *cis*-Cp₂Mo₂O₂(μ-O)(μ-S).

the metal–metal bond distances in the μ-Se-containing cluster *trans*-Cp₂Mo₂O₂(μ-O)(μ-Se) (2.760(1) Å) or the μ-S-containing clusters *trans*-Cp₂Mo₂O₂(μ-O)(μ-S) (2.732(1) Å), *trans*-Cp₂Mo₂O₂(μ-S)₂ (2.885(1) Å) or [(MoS(μ-S)(S₂CNEt₂))₂] (2.801(2) Å) [7,12]. The

Mo–Te–Mo bond angle in **1** (70.32(4)°) is less than the Mo–Se–Mo angle (75.2(1)°) or the Mo–S–Mo angle (77.2(1)°) in *trans*-Cp₂Mo₂O₂(μ-Se)₂ and *trans*-Cp₂Mo₂O₂(μ-S)₂ respectively [7,12].

Compounds **1** and **2** are evidently products of cluster

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

	Compound 1	Compound 2
Empirical formula	C ₁₀ H ₁₀ Mo ₂ O ₄ Te	C ₁₀ H ₁₀ Mo ₂ O ₄ S
Formula weight	497.6	402.1
Crystal system	Monoclinic	Orthorhombic
Space group	<i>P</i> 2 ₁ / <i>c</i> (No. 14)	<i>Pnma</i> (No. 62)
<i>a</i> (Å)	6.746(5)	18.952(4)
<i>b</i> (Å)	11.056(5)	9.344(2)
<i>c</i> (Å)	8.893(3)	6.688(4)
<i>α</i> (deg)	90	90
<i>β</i> (deg)	100.16(4)	90
<i>γ</i> (deg)	90	90
<i>V</i> (Å ³)	652.9(6)	1184.4(8)
<i>Z</i>	2	4
<i>D</i> (calc) (mg m ^{−3})	2.53	2.26
Temperature (K)	293(2)	293(2)
<i>hkl</i> ranges	<i>h</i> = 0 to 9, <i>k</i> = 0 to 15, <i>l</i> = −12 to +12	<i>h</i> = 0 to 26, <i>k</i> = 0 to 13, <i>l</i> = 0 to 9
<i>F</i> (000)	460	776
Crystal size (mm)	0.15 × 0.15 × 0.10	0.25 × 0.25 × 0.08
Absorption coefficient (mm ^{−1})	4.10	2.28
<i>θ</i> range for data collection (deg)	2 to 30	2 to 30
No. of reflections collected	2035	1815
No. of independent reflections	1898 (<i>R</i> (int) = 0.0602)	1815 (<i>R</i> (int) = 0.0000)
Reflections with <i>I</i> > 2σ(<i>I</i>)	1749	1628
Structure solution	Direct methods	Direct methods
Refinement methods	Full-matrix least squares on all <i>F</i> ²	Full-matrix least squares on all <i>F</i> ²
Data/restraints/parameters	1898/0/83	1815/0/106
Goodness-of-fit on <i>F</i> ²	1.246	1.145
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> ₁ = 0.039, <i>wR</i> ₂ = 0.096	<i>R</i> ₁ = 0.065, <i>wR</i> ₂ = 0.156
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.043, <i>wR</i> ₂ = 0.098	<i>R</i> ₁ = 0.069, <i>wR</i> ₂ = 0.161
Largest diff. peak and hole (eÅ ^{−3})	0.88 and −0.64	2.06 and −3.31
Maximum shift/e.s.d.	0.001	0.001

Table 2

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for compound 1, where U_{eq} is defined as one-third of the trace of the orthogonalised U_{ij} tensor

Atom	x	y	z	U_{eq}
Mo	581.7(5)	86.2(3)	1683.1(4)	33(1)
Te	1397(2)	1679(1)	−204(1)	49(1) ^a
O(1)	−1222(5)	788(4)	2466(4)	53(1)
O(2)	−1094(14)	−1306(6)	217(11)	24(2) ^a
C(1)	3653(10)	512(7)	3250(9)	76(2)
C(2)	2550(10)	−102(10)	4116(7)	81(3)
C(3)	2177(12)	−1186(11)	3613(11)	95(3)
C(4)	3209(14)	−1396(9)	2432(13)	101(4)
C(5)	4171(9)	−352(13)	2168(8)	101(4)

^a Occupancy 0.5.

Table 3

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for compound 2, where U_{eq} is defined as one-third of the trace of the orthogonalised U_{ij} tensor

Atom	x	y	z	U_{eq}
Mo(1)	2049.7(3)	2500	850.2(9)	34(1)
Mo(2)	687.9(3)	2500	−411.1(8)	38(1)
S	1394(1)	4360(3)	−103(4)	64(1)
O(3)	1394(1)	4360(3)	−103(4)	64(1)
O(1)	2021(14)	2500	3382(11)	84(3)
O(2)	187(4)	2500	1657(10)	83(3)
C(1)	2818(6)	2881(13)	−1977(11)	63(8)
C(2)	3048(7)	3813(10)	−432(14)	48(4)
C(3)	3293(7)	2960(14)	1182(15)	61(7)
C(4)	3215(6)	1500(12)	633(21)	94(10)
C(5)	2922(9)	1452(11)	−1319(21)	102(11)
C(6)	638(5)	3054(16)	−3919(14)	55(4)
C(7)	118(5)	3894(12)	−2941(18)	58(4)
C(8)	−396(5)	2950(14)	−2132(16)	87(12)
C(9)	−192(7)	1528(12)	−2610(26)	80(7)
C(10)	447(8)	1593(14)	−3714(21)	92(9)

Table 4

Selected bond lengths (\AA) and bond angles (deg) for 1

Mo–O(1)	1.693(4)	C(4)–C(5)	1.364(14)
Mo#1–O(2)	2.236(8)	Mo–C(1)	2.332(6)
Mo–C(2)	2.340(6)	Mo–C(3)	2.330(7)
Mo–C(4)	2.419(7)	Mo–C(5)	2.432(6)
Mo–M(1)	2.068(7)	Mo–Te	2.559(1)
Mo#1–Te	2.588(2)	Mo–Mo#1	2.964(1)
C(1)–C(2)	1.346(11)	C(1)–C(5)	1.441(14)
C(2)–C(3)	1.289(13)	C(3)–C(4)	1.378(14)
O(1)–Mo–O(2)	103.5(3)	O(1)#1–Mo#1–O(2)	104.6(3)
O(1)–Mo–Te	102.0(2)	O(2)–Mo–Te	103.9(2)
O(1)#1–Mo#1–Te	102.2(2)	O(2)–Mo#1–Te	101.9(2)
Mo–Te–Mo#1	70.32(4)	Mo–O(2)–Mo#1	83.9(2)
M(1)–Mo–O(1)	118.5(3)	M(1)–Mo–O(2)	115.6(3)
M(1)–Mo–Te	111.6(2)	C(2)–C(1)–C(5)	105.7(8)
C(3)–C(2)–C(1)	111.3(8)	C(2)–C(3)–C(4)	109.1(8)
C(5)–C(4)–C(3)	107.9(9)	C(4)–C(5)–C(1)	105.5(7)

M(1) is the centroid of the ring C(1) to C(5). Symmetry transformations used to generate equivalent atoms (#1): $-x, -y, -z$.

Table 5

Selected bond lengths (\AA) and bond angles (deg) for 2

Mo(1)–O(1)	1.694(8)	Mo(1)–M(1)	2.08(1)
Mo(1)–S/O(3)	2.230(3)	Mo(1)–C(1)	2.41(1)
Mo(1)–C(2)	2.41(1)	Mo(1)–C(3)	2.41(1)
Mo(1)–C(4)	2.40(1)	Mo(1)–C(5)	2.41(2)
Mo(2)–O(2)	1.678(7)	Mo(2)–M(2)	2.07(1)
Mo(2)–S/O(3)	2.202(3)	Mo(2)–C(6)	2.40(1)
Mo(2)–C(7)	2.39(1)	Mo(2)–C(8)	2.39(1)
Mo(2)–C(9)	2.40(1)	Mo(2)–C(10)	2.41(1)
O(1)–Mo(1)–M(1)	115.2(2)	O(1)–Mo(1)–S/O(3)	105.5(2)
M(1)–Mo(1)–S	113.1(2)	S/O(3)–Mo(1)–S/O(3)′	102.4(1)
O(2)–Mo(2)–M(2)	114.4(3)	O(2)–Mo(2)–S/O(3)	105.5(2)
M(2)–Mo(2)–S/O(3)	110.89(7)	S/O(3)–Mo(2)–S/O(3)′	104.2(1)

Symmetry transformations used to generate equivalent atoms (′): $x, -y + \frac{1}{2}, z$.

fragmentation. In the presence of oxygen there occurs extensive decarbonylation and degradation of the parent mixed-metal dichalcogenide cluster with the retention of the Cp_2Mo_2 unit and the rupture of Mo–Te–Mo bonds or Mo–S–Mo bonds in favour of a bridging oxo group linking the two Mo atoms, together with the incorporation of terminal oxo groups. The fate of the Fe-containing fragments is not known at present. Since no other products were observed in the reaction, it can be assumed that the Fe fragments end up as decomposition material which is observed on chromatographic work-up.

3. Experimental section

3.1. General procedures

The starting material $\text{Cp}_2\text{Mo}_2\text{Fe}_2(\text{CO})_7(\mu_3\text{-S})(\mu_3\text{-Te})$ was prepared as reported in the literature [3]. All solvents were dried and distilled immediately prior to use. Infrared spectra were recorded on a Nicolet Impact 400 FTIR spectrophotometer as dichloromethane solution in 0.1 mm pathlength NaCl cells. Elemental analyses were performed using a Carlo Erba automatic analyser. ^1H and ^{125}Te NMR spectra were recorded on a Varian VXR-300S spectrometer in CDCl_3 . Operating frequency for ^{125}Te NMR was 94.705 MHz, with a pulse width of 9.5 μs and a delay of 1 s. The spectrum is referenced to Me_2Te ($\delta = 0$).

3.2. Preparation of $\text{Cp}_2\text{Mo}_2\text{O}_2(\mu\text{-O})(\mu\text{-E})$ ($E = \text{Te}, 1$; $E = \text{S}, 2$)

A benzene solution (20 ml) containing $\text{Cp}_2\text{Mo}_2\text{Fe}_2(\text{CO})_7(\mu_3\text{-S})(\mu_3\text{-Te})$ (0.012 mmol, 10 mg) was subjected to reflux with constant stirring in presence of air for 3 h. After evaporation of the solvent in vacuo, the residue was dissolved in dichloromethane

and subjected to chromatographic work-up using silica-gel TLC plates. Elution with 30/70 (v/v) hexane–dichloromethane mixture afforded the following two compounds in order of elution: orange *trans*-Cp₂Mo₂O₂(μ -O)(μ -Te) (1) (yield, 1.85 mg (31%)) and reddish orange *cis*-Cp₂Mo₂O₂(μ -O)(μ -S) (2) (yield, 1.75 mg (36%)). 1: Anal. Found: C, 24.2; H, 2.21. C₅H₅Mo₂O₃Te calc.: C, 24.1; H, 2.01%. 2: Anal. Found: C, 30.0; H, 2.70. C₅H₅Mo₂O₃S calc.: C, 29.9; H, 2.53%.

3.3. Crystallography

Crystals of compounds 1 and 2 suitable for X-ray diffraction analysis were grown from hexane and dichloromethane solvent mixtures by slow evaporation of the solvents at 0°C. Crystallographic data (summarised in Table 1) were measured at 293(2) K on a CAD4 automatic four circle diffractometer, using Enraf–Nonius CAD4 software [13]. All non-H atoms were anisotropic. Hydrogen atoms were included in riding mode with $U_{iso}(H)$ equal to $1.2U_{eq}(C)$. Solutions of the structures were performed using direct methods and the refinement was carried out by Full-Matrix Least Squares procedures. The computing systems used for this were SHELXS-86 and SHELXL-93 respectively [14,15]. The interactive graphics and the final drawings were done by CAMERON [16].

In the case of *trans*-Cp₂Mo₂O₂(μ -O)(μ -Te) (1), the molecule lies on a crystallographic inversion centre with consequent disorder of the bridging Te and O atoms. The crystal system is monoclinic with $P2_1/c$ (No. 14) as the space group. For *cis*-Cp₂Mo₂O₂(μ -O)(μ -S) (2), the molecule lies with Mo(1), Mo(2), O(1) and O(2) on the crystallographic mirror plane. The bridging S and O(3) atoms were disordered across the mirror plane and could not be resolved, so a single site of occupancy 0.5S/0.5C was refined. The Cp groups were both disordered across the mirror plane and in each case a rigid group of idealised geometry and half occupancy was refined. The figure shows only one orientation of the Cp rings. The crystal system is orthorhombic with a $Pnma$ (No. 62) space group.

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