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Synthesis and characterization of $[\text{Mo}(\mu\text{-EPh})(\text{CO})_3(\text{CH}_3\text{CN})]_2$ (E = Se, Te), including the X-ray structure of the tellurium derivative

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Dedicated to Professor Bernard Gautheron on the occasion of his retirement.

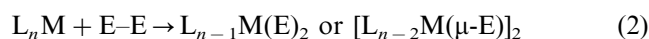
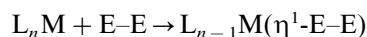
Abstract

The reaction of $\text{Mo}(\text{CO})_3(\text{MeCN})_3$ and E_2Ph_2 (E = Se, Te) yields the edge-sharing bioctahedral, metal–metal bonded Mo(I) products $[\text{Mo}(\text{CO})_3(\text{MeCN})(\mu\text{-EPh})]_2$. The structure of the telluroolato derivative was confirmed by X-ray crystallography: triclinic, space group $P\bar{1}$, $a = 7.3149(17)$, $b = 9.6959(16)$, $c = 9.7090(10)$ Å, $\alpha = 80.366(10)$, $\beta = 76.563(13)$, $\gamma = 72.877(16)^\circ$, $V = 636.43(19)$ Å³, $D_{\text{calc}} = 2.222$ Mg m⁻³, $\mu = 3.271$ mm⁻¹, $R_1 = 0.0418$, $wR_2 = 0.0689$ for 163 parameters and 2238 data with $I > 2\sigma(I)$. The interaction of these compounds with excess E_2Ph_2 as a possible entry to homoleptic $\text{Mo}(\text{EPh})_3$ has been investigated. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Crystal structures; Molybdenum complexes; Tellurium complexes

1. Introduction

The reaction of carbonyl compounds with reagents containing a single E–E bond between elements of Groups 17 (halogens), 16 (chalcogens) and 15 (pnictogens) presents an interesting dichotomy between two fundamental reaction pathways in organometallic chemistry, namely coordinative addition (Eq. (1)) or oxidative addition (Eq. (2)). The outcome of the reaction is dictated by the oxidative power of E–E and by the susceptibility of the carbonyl compound to oxidation [1].



For instance, while S_2Me_2 oxidatively adds to $[\text{Cp-Mo}(\text{CO})_3]_2$ to afford $[\text{CpMo}(\mu\text{-SMe})_2]_2$ [2], compounds E_2Ph_2 (E = S, Se, Te) only coordinatively add to $\text{MnX}(\text{CO})_5$ and $\text{ReX}(\text{CO})_5(\text{THF})_2$ (X = Br, I), to afford $[\text{M}(\mu\text{-X})(\mu\text{-E}_2\text{Ph}_2)(\text{CO})_3]_2$ (M = Mn or Re, respectively) [3,4]. The stronger oxidant I_2 , on the other hand, is able to oxidize the same Re(I) starting material to $\text{ReI}_3\text{-(CO)}_3$ or to mixed-valence Re(I)–Re(III) or Re(I)–Re(IV) products [5,6].

The addition of I_2 to $\text{Mo}(\text{CO})_6$ generates MoI_3 and represents a clean method for the formation of anhydrous Mo(III) iodide [7]. Homoleptic alkyl- and aryl-sulfido Mo(III) materials have also been obtained by thermal interaction of S_2R_2 and $\text{Mo}(\text{CO})_6$ under forcing conditions [8,9]. It would be interesting to establish whether an analogous approach could lead to homoleptic alkylselenides and tellurides, which are a relatively unexplored class of compounds [10] with potential use as precursors to semiconducting metal selenides and tellurides. The reaction between $\text{Mo}(\text{CO})_6$ and the heavier dichalcogenide Te_2Ph_2 has been studied by two

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different groups under different conditions (in refluxing toluene, or upon UV irradiation in refluxing THF) [11,12]. In both cases, a Mo(I) complex, $[\text{Mo}(\mu\text{-TePh})(\text{CO})_4]_2$, was isolated and structurally characterized. Since the elimination of the tightly bonded CO ligand is a difficult process for low-valent molybdenum, a more promising route is the use of partially substituted Mo(0) carbonyl derivatives, such as $(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{Mo}(\text{CO})_3$ or $\text{Mo}(\text{CO})_3(\text{MeCN})_3$. The room temperature (r.t.) reaction of $\text{Mo}(\text{CO})_3(\text{MeCN})_3$ and S_2Ph_2 in MeCN affords a Mo(I) product, $[\text{Mo}(\mu\text{-SPh})(\text{CO})_3(\text{MeCN})]_2$, whose X-ray structure has been determined [13]. We have investigated the same reactions with the heavier dichalcogenides Se_2Ph_2 and Te_2Ph_2 and report here the results of these studies.

2. Experimental

All operations were carried in a protective atmosphere of dinitrogen by using standard Schlenk-line and glove box techniques. Solvents were dried by conventional methods (THF over Na/K; heptane over Na; MeCN over type 4 Å molecular sieves (Fischer) and then reflux over CaH_2) before being distilled. Solution IR spectra were taken on a Perkin–Elmer 1600 series FT spectrophotometer with KBr optics. Elemental analyses were by Atlantic Microlab, Inc., Norcross, GA. $(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{Mo}(\text{CO})_3$ was prepared according to the literature method [14]. Ph_2Te_2 and Ph_2Se_2 (Aldrich) were used as received.

2.1. Preparation of $\text{Mo}_2(\text{TeC}_6\text{H}_5)_2(\text{CO})_6(\text{CH}_3\text{CN})_2$

The compound $(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{Mo}(\text{CO})_3$ (651 mg, 2.39 mmol) was placed in a Schlenk flask equipped with a magnetic stirrer bar. CH_3CN (50 ml) was added with stirring. A clear yellow solution immediately formed. An IR spectrum showed CO stretching bands at 1920 and 1797 cm^{-1} , which were assigned to $\text{Mo}(\text{CO})_3\text{-}(\text{MeCN})_3$ by comparison with the literature [15]. To this solution was added Ph_2Te_2 (980 mg, 2.39 mmol), inducing an instant color change to dark red. An IR spectrum of the resulting solution recorded immediately showed the prominence of the bands of the starting material and new bands, which can be assigned to the final product. The reaction was allowed to continue for 24 h at r.t., resulting in the formation of a dark green microcrystalline solid. The precipitate was isolated by filtration, washed twice with 5 ml portions of heptane, and dried in vacuo (10^{-2} mmHg) for 1 h. Yield 317 mg (30.1%). *Anal. Calc.*: C, 31.03; H, 1.89; N, 3.29. *Found*: C, 29.68; H, 1.82; N, 3.06%. IR (cm^{-1} , CH_3CN): 1961 (s), 1926 (w), 1873 (m). X-ray quality crystals were grown by recrystallizing 61.8 mg of the product from 50 ml of hot MeCN.

2.2. Attempted reaction between $\text{Mo}_2(\text{TeC}_6\text{H}_5)_2(\text{CO})_6(\text{CH}_3\text{CN})_2$ and Te_2Ph_2

Compounds $\text{Mo}_2(\text{TeC}_6\text{H}_5)_2(\text{CO})_6(\text{CH}_3\text{CN})_2$ (100 mg, 0.225 mmol) and Te_2Ph_2 (100 mg, 0.244 mmol) were mixed together in CH_3CN (80 ml), giving a red solution with a green precipitate. The mixture was refluxed for 10 h, with no apparent change.

2.3. Preparation of $\text{Mo}_2(\text{SeC}_6\text{H}_5)_2(\text{CO})_6(\text{CH}_3\text{CN})_2$

The compound $(\eta^6\text{-C}_6\text{H}_5\text{CH}_3)\text{Mo}(\text{CO})_3$ (889 mg, 3.27 mmol) was placed in a Schlenk flask equipped with a magnetic stirrer bar. CH_3CN (40 ml) was added with stirring. A clear yellow solution immediately formed. To this solution was added Ph_2Se_2 (1.019 g, 3.27 mmol), inducing an instant color change to dark red. The reaction was allowed to continue for 24 h at r.t., resulting in the formation of a dark green microcrystalline solid. The mixture was cooled to -20°C for an additional 24 h, then filtered. The precipitate was washed with heptane and dried in vacuo (10^{-2} mmHg) for 1 h. Yield 403 mg (32.7%). *Anal. Calc.*: C, 35.04; H, 2.14; N, 3.71. *Found*: C, 34.72; H, 2.08; N, 3.57%. IR (cm^{-1} , CH_3CN): 1967 (s), 1932 (m), 1873 (m), 1830 (w). IR (cm^{-1} , Nujol mull): 1980 (s), 1940 (vs), 1891 (s), 1861 (vs).

2.4. Reaction between $\text{Mo}_2(\text{SeC}_6\text{H}_5)_2(\text{CO})_6(\text{CH}_3\text{CN})_2$ and Se_2Ph_2

2.4.1. In a 1:2 ratio

The compound $\text{Mo}_2(\text{SeC}_6\text{H}_5)_2(\text{CO})_6(\text{CH}_3\text{CN})_2$ (48 mg, 0.064 mmol) was placed in a Schlenk flask equipped with a magnetic stirrer bar. Toluene (20 ml) was added with stirring, giving a green solution. To this solution was added Ph_2Se_2 (38 mg, 0.123 mmol), inducing a color change to green–brown. After 1 h at r.t., no new IR bands were observed in the CO stretching region. The reaction was heated to reflux for 1 h, resulting in the formation of a dark brown precipitate. The mixture was cooled to r.t., then filtered. The precipitate was washed with toluene (5 ml) and dried in vacuo (10^{-2} mmHg) for 30 min. Yield 47 mg (74%). *Anal. Calc.* for $\text{C}_{31}\text{H}_{23}\text{Mo}_2\text{NO}_5\text{Se}_4$ [$\text{Mo}_2(\text{SePh})_4(\text{CO})_5\text{-}(\text{MeCN})$]: C, 37.34; H, 2.32; N, 1.40. *Found*: C, 37.84; H, 2.58; N, 1.01%. IR (cm^{-1} , Nujol mull): 2012 (s), 1959 (vs), 1937 (vs), 1878 (s).

2.5. In a 1:1 ratio

The compound $\text{Mo}_2(\text{SeC}_6\text{H}_5)_2(\text{CO})_6(\text{CH}_3\text{CN})_2$ (135 mg, 0.18 mmol) was placed in a Schlenk flask equipped with a magnetic stirrer bar. Toluene (40 ml) was added with stirring, giving a green solution. To this solution was added Ph_2Se_2 (57 mg, 0.18 mmol), inducing a color

change to green–brown. After 90 min at r.t., the reaction was heated to reflux for 1 h, resulting in the formation of a dark brown precipitate. The mixture was cooled to r.t., then filtered. The precipitate was washed with toluene (4 × 5 ml) and dried in vacuo (10⁻² mmHg) for 1 h. Yield 143 mg (80%). The IR spectrum (Nujol mull) of this product was identical to that obtained from the reaction of Mo₂(SeC₆H₅)₂(CO)₆(CH₃CN)₂ with 2 equiv. of Ph₂Se₂.

2.6. X-ray crystallography for the compound

Mo₂(TeC₆H₅)₂(CO)₆(CH₃CN)₂

A dark green plate with dimensions 0.250 × 0.125 × 0.013 mm was placed on the Enraf–Nonius CAD-4 diffractometer. The crystal final cell parameters and orientation matrix were determined from 25 reflections in the range 8.9 < θ < 17.7° and confirmed with axial photographs. Relevant crystal and data collection parameters are listed in Table 1. All data (± h ± k ± l) were collected, resulting in the measurement of 4473 reflections, of which 2238 were unique [*R*_{int} = 0.0468]. Three periodically monitored standard reflections re-

Table 1
Crystal data for the compound Mo₂(TeC₆H₅)₂(CO)₆(CH₃CN)₂

Formula weight	851.45
Temperature (K)	153(2)
Wavelength (Å)	0.71073
Crystal system	triclinic
Space group	<i>P</i> 1
Unit cell dimensions	
<i>a</i> (Å)	7.3149(17)
<i>b</i> (Å)	9.6959(16)
<i>c</i> (Å)	9.7090(10)
<i>α</i> (°)	80.366(10)
<i>β</i> (°)	76.563(13)
<i>γ</i> (°)	72.877(16)
<i>V</i> (Å ³)	636.43(19)
<i>Z</i>	1
<i>D</i> _{calc} (Mg m ⁻³)	2.222
Absorption coefficient (mm ⁻¹)	3.271
<i>F</i> (000)	398
Crystal size (mm ³)	0.2500 × 0.1250 × 0.0125
θ Range for data collection (°)	2.93–24.97
Index ranges	−8 ≤ <i>h</i> ≤ 8, −11 ≤ <i>k</i> ≤ 11, −11 ≤ <i>l</i> ≤ 11
Reflections collected	4473
Independent reflections	2238 [<i>R</i> _{int} = 0.0468]
Completeness to θ = 24.97° (%)	99.9
Absorption correction	integration
Max/min transmission	0.9599, 0.7098
Refinement method	full-matrix least-squares on <i>F</i> ²
Data/restraints/parameters	2238/0/163
Goodness-of-fit on <i>F</i> ²	1.126
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> ₁ = 0.0418, <i>wR</i> ₂ = 0.0689 [1719 data]
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0676, <i>wR</i> ₂ = 0.0747
Largest difference peak and hole (e Å ⁻³)	1.332 and −1.253

Table 2

Selected bond distances (Å) and angles (°) for Mo₂(TeC₆H₅)₂(CO)₆(CH₃CN)₂

<i>Bond distances</i>			
Mo(1)–Mo(1A)	3.1288(13)	Mo(1)–C(3)	1.988(7)
Mo(1)–Te(1)	2.7447(9)	Te(1)–C(11)	2.153(6)
Mo(1)–Te(1A)	2.7409(9)	N(1)–C(4)	1.148(9)
Mo(1)–N(1)	2.200(6)	C(1)–O(1)	1.159(8)
Mo(1)–C(1)	1.949(7)	C(2)–O(2)	1.144(8)
Mo(1)–C(2)	1.999(8)	C(3)–O(3)	1.164(8)
<i>Bond angles</i>			
Mo(1A)–Mo(1)–Te(1)	55.17(2)	N(1)–Mo(1)–C(1)	175.9(3)
Mo(1A)–Mo(1)–Te(1A)	55.28(2)	N(1)–Mo(1)–C(2)	98.8(3)
Mo(1A)–Mo(1)–N(1)	85.36(16)	N(1)–Mo(1)–C(3)	88.5(3)
Mo(1A)–Mo(1)–C(1)	92.8(2)	C(1)–Mo(1)–C(2)	85.0(3)
Mo(1A)–Mo(1)–C(2)	138.67(19)	C(1)–Mo(1)–C(3)	90.4(3)
Mo(1A)–Mo(1)–C(3)	137.6(2)	C(2)–Mo(1)–C(3)	83.7(3)
Te(1)–Mo(1)–Te(1A)	110.45(3)	Mo(1)–Te(1)–Mo(1A)	69.55(3)
Te(1)–Mo(1)–N(1)	89.11(16)	Mo(1)–Te(1)–C(11)	104.88(19)
Te(1)–Mo(1)–C(1)	86.8(2)	Mo(1A)–Te(1)–C(11)	107.32(19)
Te(1)–Mo(1)–C(2)	164.2(2)	Mo(1)–N(1)–C(4)	169.6(6)
Te(1)–Mo(1)–C(3)	82.9(2)	Mo(1)–C(1)–O(1)	177.6(6)
Te(1A)–Mo(1)–N(1)	85.59(15)	Mo(1)–C(2)–O(2)	174.3(7)
Te(1A)–Mo(1)–C(1)	96.4(2)	Mo(1)–C(3)–O(3)	176.4(6)
Te(1A)–Mo(1)–C(2)	83.9(2)	N(1)–C(4)–C(5)	176.9(8)
Te(1A)–Mo(1)–C(3)	165.3(2)		

vealed minor (< 3%) variations in intensity; the data were not corrected for decay. Data were corrected for Lorentz and polarization factors and for absorption based upon crystal faces with transmission factors ranging from 0.7098 to 0.9599. Intensity statistics clearly favored the centrosymmetric case. The structure was determined by direct methods with the successful location of a majority of the non-hydrogen atoms. The remaining non-hydrogen atoms were found from a series of difference-Fourier maps. The structure was refined to convergence [*Δ*/σ ≤ 0.001] by full-matrix least-squares cycles in SHELXTL [16]. Hydrogen atoms were placed in calculated positions and refined by the riding method with unconstrained isotropic thermal parameters. All non-hydrogen atoms were refined anisotropically. The final difference-Fourier map was featureless, indicating that the structure is both correct and complete. Selected bond distances and angles are listed in Table 2.

3. Results and discussion

The reaction between Mo(CO)₃(MeCN)₃ (generated in situ from (η⁶-C₆H₅CH₃)Mo(CO)₃ in the MeCN solvent) and E₂Ph₂ (E = Se, Te) affords the corresponding metal–metal bonded dinuclear complexes [Mo(CO)₃-(MeCN)(μ-EPh)]₂ (see Eq. (3)) by analogy with the previously reported formation of the phenylthiolato analog [13].

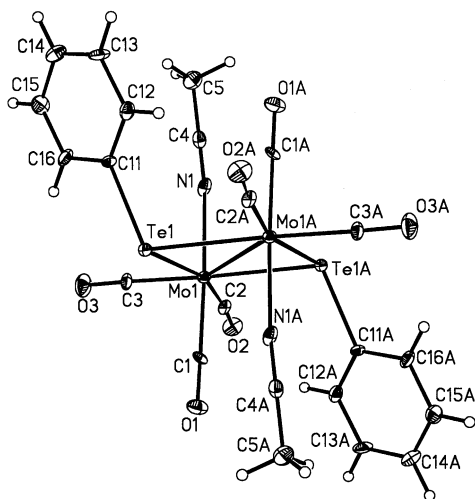
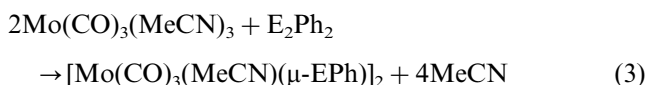


Fig. 1. An ORTEP view of the molecular geometry for the compound $\text{Mo}_2(\text{TeC}_6\text{H}_5)_2(\text{CO})_6(\text{CH}_3\text{CN})_2$. Ellipsoids are drawn at the 30% probability level.



These reactions are therefore new examples of oxidative additions of reagents containing a single E–E bond, i.e. reactions of type (2). No evidence for the formation of a coordinative addition intermediate (Eq. (1)) is shown by IR monitoring. The products exhibit a pattern of CO stretching bands in the IR spectrum similar to that previously reported for the sulfido analog [13], a red-shift being observed on going from S to Se to Te as expected.

The tellurolato derivative was characterized by X-ray crystallography. The molecule sits on an inversion center (see Fig. 1) and can be described as an edge-shared bioctahedron. The geometry is identical in all respects with that of the sulfido analog, $[\text{Mo}(\text{CO})_3(\text{MeCN})(\mu\text{-SPh})]_2$ [13]. The distance between the two metals is 3.1288(13) Å. By comparison, the Mo–Mo separation is 2.982(1) Å in the sulfido analog [13] and 3.116(1) Å in $[\text{Mo}(\text{CO})_4(\mu\text{-TePh})]_2$ [11,12]. Thus, the substitution of a CO ligand with MeCN results in a slight lengthening of the Mo–Mo distance. Although this distance is rather long due to the size of the bridging groups, the presence of a metal–metal bonding interaction is clearly shown by the acute Mo(1)–Te(1)–Mo(1A) angle [69.55(3)°]. This is similar to the value observed in the compound $[\text{Mo}(\text{CO})_4(\mu\text{-TePh})]_2$ [68.8(1)°]. It is well established that the absence of metal–metal bonding in edge-shared bioctahedral compounds results in M–(μ-X)–M angles greater than 90° [17].

An attempt to further oxidize these materials has only met with limited success. The tellurolato derivative

does not react with excess Te_2Ph_2 upon prolonged reflux in MeCN. The oxidizing properties of the E_2Ph_2 ligands should follow the order $\text{Te} < \text{Se} < \text{S}$. Thus, the reaction of the selenolato derivative was investigated in greater detail. No interaction between $[\text{Mo}(\text{CO})_3(\text{MeCN})(\mu\text{-SePh})]_2$ and excess Se_2Ph_2 occurs at r.t., but reflux in toluene with 1 equiv. of Se_2Ph_2 afforded a new compound. The solid state IR spectrum of this product shows the presence of CO ligands and the N elemental analysis shows that the MeCN ligand has been partially retained. The most consistent formulation is $\text{Mo}_2(\text{SePh})_4(\text{CO})_5(\text{MeCN})$. The blue-shift of the CO stretching vibrations relative to the starting Mo(I) complex indicates that oxidation has occurred. Therefore, the product is more likely a $\text{Mo}_2(\text{SePh})_4$ derivative of Mo(II) than a $\text{Mo}_2(\text{SePh})_2(\text{Se}_2\text{Ph}_2)$ derivative of Mo(I). Unfortunately, a more complete characterization of this product was prevented by its sparing solubility in all common solvents. When 2 equiv. of Se_2Ph_2 per Mo(I) dimer were employed, the same product was obtained, indicating that the oxidation state III is not attainable via this route. It can be concluded that this synthetic strategy is not a viable one as an entry to the hitherto unknown $\text{Mo}(\text{EPh})_3$ (E = Se, Te) derivatives.

4. Supplementary material

The crystallographic data have been deposited with the Cambridge Crystallographic Data Centre (CCDC No. 132771) and may be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

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