

Synthesis and structural characterization of a novel dimolybdenum(I) compound with mixed-tribridging ligands: $[\text{Bu}_4\text{N}][\text{Mo}_2(\mu\text{-SPh})_2(\mu\text{-Cl})(\text{CO})_6]$

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A novel mixed-tribridged dimolybdenum(I) compound $[\text{Bu}_4\text{N}][\text{Mo}_2(\mu\text{-SPh})_2(\mu\text{-Cl})(\text{CO})_6]$ (**1**) has been synthesized from the reaction of $\text{Mo}_2(\text{CO})_8(\text{SPh})_2$ with Bu_4NCl . Compound **1** was characterized by IR, UV-Vis and ^1H , ^{13}C , ^{95}Mo NMR spectroscopic analyses. The electrochemical behavior was measured by cyclic voltammetry, indicating a quasi-reversible two-electron transfer in one step. The crystal structure determined by X-ray crystallography shows that **1** contains a $[\text{Mo}_2(\mu\text{-S})_2(\mu\text{-Cl})]^-$ core with a planar Mo_2S_2 unit and a Cl bridge. The Mo—Mo distance is 0.28709(7) nm, and the Mo—Cl—Mo angle is 66.44(4)°. A new face-sharing bioctahedral structure is discussed.

Keywords Molybdenum(I) compound, crystal structure, Mo_2S_2 unit, cofacial bioctahedral structure, IR, NMR, CV

Introduction

The study of bimetallic complexes has been an active research area as metal-metal interaction generally results in unique physical and chemical properties.¹ In this respect, dinuclear molybdenum complexes have been extensively investigated.² To our knowledge, however, so far a few Mo(I) complexes have been systematically investigated in the literature except $[\text{CpMo}(\text{CO})_3]_2$,³ $[\text{CpMo}(\text{CO})_2]_2$ ⁴ and $[\text{Mo}_2\text{Cp}_2(\text{CO})_4(\mu\text{-Ph}_2\text{PCH}_2\text{PPh}_2)]$.⁵ Recently, an attempt to develop novel Mo(I) complexes has been carried out by introducing a variety of function-

al ligands into the parent compound $\text{Mo}_2(\mu\text{-SPh})_2(\text{CO})_8$ (**2**). We describe here synthesis and characterization of a mixed tri-bridged dimolybdenum(I) compound **1**, where chloro ligand was introduced into **2** by a carbonyl substitution reaction. Compound **1** exhibits a unique face-sharing bioctahedral structure, which could be viewed as one dodecahedron with upper-half splitting.

Experimental

Materials and methods

All the work described here was carried out under an atmosphere of N_2 . All solvents used were dried and deoxygenated prior to use. $\text{Mo}_2(\mu\text{-SPh})_2(\text{CO})_8$ (**2**) was prepared according to published method.⁶ Bu_4NCl was purchased from Shanghai Chemical Reagent Co. Elemental analyses were performed on a Carlo Erba MOD 1106 analyzer. Infrared spectra (KBr/CsI pellet) were recorded on a Magna 750 spectrometer. UV-Vis spectra were recorded in acetone solutions in a quartz cell on a Shimadzu UV-3000 UV-Vis spectrometer. NMR spectra of the sample in acetone were measured on Varian Unity-500 NMR spectrometer operated at 499.864 MHz for ^1H , 125.708 MHz for ^{13}C , 32.544 MHz for ^{95}Mo . Proton and carbon chemical shifts are relative to internal acetone solvent, and molybdenum's is relative to external aqueous 2 mol/L Na_2MoO_4 solution. Cyclic voltam-

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metry (CV) measurement was carried out on CV-1B from BAS (Bioanalytical Systems), using 0.10 mol/L Et_4NBF_4 as the supporting electrolyte and acetone as solvent. The working electrode was a glassy carbon (area 0.0804 cm^2), the auxiliary electrode was a platinum wire and the reference electrode was an aqueous SCE separated from the sample solution by a salt bridge containing 0.1 mol/L Et_4NBF_4 in the solvent. Solutions were deoxygenated and blanketed with nitrogen. As a comparison, compound **2** and ferrocene were measured under the identical condition. $E_{1/2} = +0.48 \text{ V}$ ($+0.54/0.42 \text{ V vs. SCE}$) for the ferrocene-ferrocenium couple and $E_{1/2} = -0.35 \text{ V}$ ($-0.41/-0.30 \text{ V vs. SCE}$) for compound **2**. The concentration of the compounds in these measurements was 0.0013 mol/L, the scan rate was 100 mV/s.

Synthesis of $[\text{Bu}_4\text{N}][\text{Mo}_2(\mu\text{-SPh})_2(\mu\text{-Cl})(\text{CO})_6]$ (**1**)

To a stirred green solution of $\text{Mo}_2(\mu\text{-SPh})_2(\text{CO})_8$ (**2**) (0.634 g, 1.0 mmol) in acetone (30 mL) was added Bu_4NCl (0.278g, 1.0 mmol) at room temperature, the solution color instantly turned from green to brown, accompanied by a vigorous CO evolution. After being stirred for 2 h, the solution was reduced to ca. 5 mL under vacuum and 15 mL *i*-PrOH was added dropwise. The resulting brown solution was filtered, then the filtrate was allowed to stay at -4°C for several days to afford black crystals. Compound **1** as a crystalline product was obtained by filtration, washed with *i*-PrOH and dried in vacuum. The rest products were collected from the mother liquid by repeating above procedure till no crystalline products appeared (total 0.75 g, 87%). ν_{max} (KBr): 2962m, 2935w, 2875w, 1994s, 1955s, 1940s, 1903s, 1846s, 1576m, 1475m, 14560w, 1437m, 1375m, 1300w, 1277w, 1176w, 1151w, 1105w, 1065w, 1022m, 999w, 879m, 802w, 750s, 744s, 688s, 621m, 590m, 530w, 515w, 503m, 486w, 476m, 437w, 426m cm^{-1} . ν_{max} (CsI): 590, 530, 515, 503, 484, 478, 436, 426, 359, 345, 284, 247, 224, 191, 174, 152 cm^{-1} . λ_{max} (CH_3COCH_3): 444, 402, 330, 315 nm. δ_{H} (CD_3COCD_3): 7.21—7.61 (m, 10 H, SPh), 3.44 (t, Hz, 8^{d} H, C^{d} $\text{H}_2\text{CH}_2\text{-CH}_2\text{CH}_3$), 1.82 (m, 8^{c} H, $\text{CH}_2\text{C}^{\text{c}}$ $\text{H}_2\text{CH}_2\text{CH}_3$), 1.46 (m, 8^{b} H, $\text{CH}_2\text{CH}_2\text{C}^{\text{b}}$ H_2CH_3), 1.01 (t, 12^{a} H, $\text{CH}_2\text{CH}_2\text{CH}_2\text{C}^{\text{a}}$ H_3). δ_{C} (CD_3COCD_3): 231.66 (CO

trans to Cl), 222.63 (CO *cis* to Cl), 145.31 (Ph), 135.73 (Ph), 133.53 (Ph), 128.73 (Ph), 59.96 (Bu_4N), 24.96 (Bu_4N), 20.86 (Bu_4N), 14.38 (Bu_4N). δ_{Mo} (CD_3COCD_3): -739.5 . Anal. $\text{C}_{34}\text{H}_{46}\text{-ClMo}_2\text{NO}_6\text{S}_2$, Calcd: C, 47.70; H, 5.42; N, 1.64. Found: C, 47.61; H, 5.40; N, 1.65.

X-ray crystal structure determination

X-ray quality crystals of $1 \cdot \text{CH}_3\text{COCH}_3$ were grown in acetone/isopropanol parent liquid. Data were collected on a Siemens SMART area detector and using graphite - monochromated Mo K_{α} X - radiation ($\lambda = 0.0071073 \text{ nm}$) over a hemisphere of reciprocal space by a combination of three sets of exposures. Each set had a different φ angle for the crystal and each exposure of 10 s covered 0.3° in ω . The crystal-to-detector distance was 4.95 cm. Coverage of the unique set was over 99% complete to at least 23° in θ . Details of the crystal parameters, data collection and refinement are given in Table 1. Data were corrected for Lorentz and polarisation

Table 1 Crystal and structure refinement data for $1 \cdot \text{CH}_3\text{COCH}_3$

Empirical formula	$\text{C}_{37}\text{H}_{52}\text{ClMo}_2\text{NO}_7\text{S}_2$
F_w	914.25
Temperature (K)	293(2)
Wavelength	0.71073
Space group	Monoclinic
Crystal system	P2(1)/n
a (nm)	0.96470(1)
b (nm)	2.38437(4)
c (nm)	1.94845(3)
B ($^\circ$)	103.2890(10)
V ($\text{nm}^3 \times 10^3$)	4361.91(10)
Z	4
D_c (g/cm^3)	1.392
μ (mm^{-1})	0.775
$F(000)$	1880
size	$0.35 \times 0.15 \times 0.07$
η ($^\circ$)	$2.02-23.27$
reflns collected	16893
independent reflns	6266
data/restraints/params	6266/0/452
Gof	0.717
$R1, wR2(I > 2(\rho I))$	0.0415, 0.1434
$R1, wR2$ (all data)	0.0565, 0.1677
$(\Delta/\sigma)_{\text{max}}$	0.029
$\Delta\rho_{\text{max}}$	0.632
$\Delta\rho_{\text{min}}$	-0.414
$R1 = \sum(F_o - F_c)/\sum F_o, \quad wR2 = [\sum w(F_o^2 - F_c^2)/\sum w(F_o^2)]^{1/2}$	

Table 2 Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{nm}^2 \times 10^5$) for $1 \cdot \text{CH}_3\text{COCH}_3$.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (eq)
Mo(1)	1945(1)	730(1)	7398(1)	56(1)
Mo(2)	1088(1)	1652(1)	8155(1)	55(1)
Cl	1075(2)	1693(1)	6808(1)	77(1)
S(1)	-471(2)	873(1)	7603(1)	59(1)
S(2)	3557(2)	1447(1)	8075(1)	59(1)
C(1)	2668(6)	109(3)	8010(3)	60(1)
C(2)	940(8)	161(3)	6703(4)	82(2)
C(3)	3546(7)	608(3)	6916(4)	73(2)
C(4)	1117(6)	1431(2)	9112(3)	58(1)
C(5)	-777(7)	2042(3)	8123(3)	71(2)
C(6)	1963(8)	2376(3)	8556(4)	80(2)
C(11)	-1064(6)	410(2)	8197(3)	58(1)
C(12)	-2515(7)	348(3)	8105(4)	76(2)
C(13)	-3055(8)	-12(4)	8545(4)	90(2)
C(14)	-2181(8)	-317(3)	9040(4)	86(2)
C(15)	-734(8)	-264(3)	9137(4)	86(2)
C(16)	-178(6)	108(3)	8719(3)	76(2)
C(21)	4749(6)	1227(2)	8873(3)	58(1)
C(22)	4398(6)	880(2)	9367(3)	63(1)
C(23)	5409(7)	739(3)	9968(4)	71(2)
C(24)	6780(8)	929(3)	10078(4)	80(2)
C(25)	7149(7)	1273(4)	9580(4)	92(2)
C(26)	6151(6)	1434(3)	8984(4)	76(2)
O(1)	3151(5)	-265(2)	8362(2)	79(1)
O(2)	408(7)	-188(3)	6334(3)	125(2)
O(3)	4438(6)	528(3)	6639(3)	108(2)
O(4)	1064(5)	1298(2)	9680(2)	77(1)
O(5)	-1822(6)	2267(2)	8115(3)	107(2)
O(6)	2444(7)	2783(3)	8812(4)	120(2)
N	-622(5)	-2847(2)	9117(3)	73(1)
C(31)	-1665(7)	-2433(3)	9326(4)	90(2)
C(32)	-3024(8)	-2337(4)	8778(5)	101(2)
C(33)	-4103(15)	-2015(7)	9211(11)	246(13)
C(34)	-5091(28)	-1848(7)	8728(14)	297(13)
C(41)	693(7)	-2849(3)	9727(3)	76(2)
C(42)	1858(7)	-3248(4)	9672(4)	92(2)
C(43)	3181(9)	-3148(5)	10263(5)	120(3)
C(44)	4327(11)	-3564(5)	10250(6)	144(4)
C(51)	-1289(7)	-3423(3)	8999(4)	76(2)
C(52)	-1775(10)	-3682(3)	9598(4)	100(2)
C(53)	-2192(13)	-4293(4)	9436(5)	132(4)
C(54)	-2561(16)	-4602(6)	10019(6)	170(5)
C(61)	-244(8)	-2674(3)	8431(4)	81(2)
C(62)	403(9)	-2095(4)	8427(5)	98(2)
C(63)	589(13)	-1966(4)	7684(6)	132(4)
C(64)	1160(16)	-1393(6)	7624(8)	179(6)
O	-656(14)	3521(7)	8752(8)	264(7)
C(71)	-2058(26)	4221(10)	8207(16)	364(21)
C(72)	-854(18)	3906(6)	8345(8)	156(5)
C(73)	289(23)	3996(8)	7991(10)	237(9)

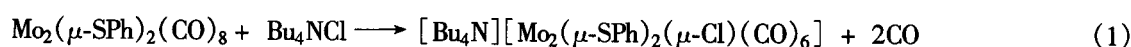
effects and for absorption effects by SADABS.⁷ The structure was solved by conventional direct methods (SHELXTL) and was refined by the full-matrix least-squares method on all F^2 data using Silicon Graphics Indy computer and local programmes.⁸ All non-hydrogen

atoms were refined anisotropically; hydrogen atoms were located at idealized positions and refined with fixed isotropic thermal parameters. The atomic coordinates and thermal parameters for $1 \cdot \text{CH}_3\text{COCH}_3$ are listed in Table 2.

Results and discussion

Synthesis

Compound **1** was prepared by reacting $\text{Mo}_2(\mu\text{-SPh})_2(\text{CO})_8$ with 1 equiv. of Bu_4NCl in CH_3COCH_3 (Eq. (1))



Decarbonylation reaction of $\text{Mo}_2(\mu\text{-SPh})_2(\text{CO})_8$ resulted in mixed-tribridged product **1**. Attempt to prepare tetra-bridged species as designed formula $[\text{Mo}_2(\mu\text{-SPh})_2(\mu\text{-Cl})_2(\text{CO})_4]^{2-}$ by using excess (> 4fold) Bu_4NCl met with no success. The use of excess Bu_4NCl in the preparation still gave tri-bridged products **1** (confirmed by IR). Excess of Bu_4NCl accelerated the reaction, but had no influence on the yield.

Spectra

The infrared spectrum of **1** exhibited the vibration absorptions of $\nu_{(\text{CO})}$ at 1994(s), 1955(s), 1940(s), 1903(s), 1846(s); $\nu_{(\text{Mo-Cl})}$ at 247; $\nu_{(\text{Mo-S})}$ at 426; $\nu_{(\text{Mo-C})}$ at 359 and 284 cm^{-1} . The CO peaks shift to red relative to those of **2**, owing to the increased metal-to-CO back-donating with Cl replacing CO. A ratio 2:1 of ^{13}C NMR of carbonyl observed indicates only one carbonyl was substituted in the $\text{Mo}(\text{CO})_4$ fragment of **2**. The ^{95}Mo NMR chemical shift for compound **1** is $\delta - 739.5$, which is at more downfield than that of $\text{Mo}_2(\mu\text{-SPh})_2(\text{CO})_8$ ($\delta - 1112.30$), indicating a larger deshielding when chlorine displaces carbonyl group. This downfield shift should be mainly caused by the fact that chlorine is a weaker electron-donating ligand than carbonyl, even though carbonyl is a strong π -acceptor. Only one ^{95}Mo NMR chemical shift was observed in **1**. This is consistent with the X-ray analysis that the geometry of the two Mo atoms in **1** is essentially identical.

Structure

Fig. 1 shows the structure of dinuclear anion of **1**,

and Table 3 lists selected bond distances and angles. The coordination sphere of each Mo atom consists of two *cis*-CO groups and two *cis*-bridging S atoms from SPh ligands forming the equatorial plane while one CO and one bridging Cl ligand filling the imposed axial positions. Two *fac*- $\text{Mo}(\text{CO})_3$ fragments are linked together by the hetero-tri-bridging ligands forming a $\text{Mo}_2\text{S}_2\text{Cl}$ core,

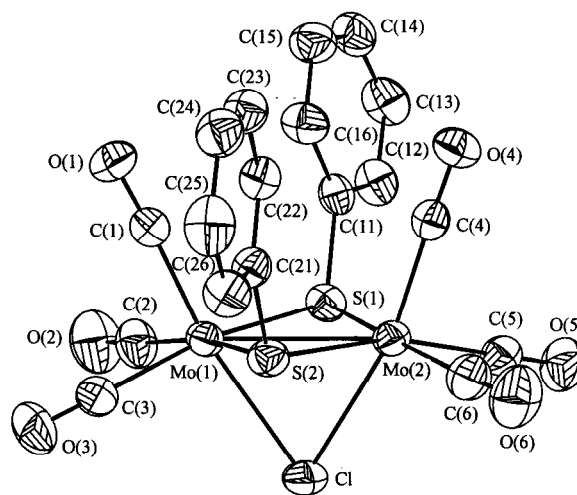
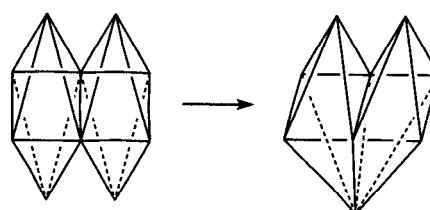


Fig. 1 Structure of the anion of **1**.

Scheme 1



in which Mo₂S₂ unit is nearly planar (within 0.007 nm). Seven atoms, Cl, Mo(1), Mo(2), C(1), O(1), C(4) and O(4) are also approximate coplanar (within 0.0085 nm), with a Mo—Mo bond, to form a “A-frame”.⁹ Both benzene rings from two SPh bridging ligands are in a *syn*-orientation relative to the planar Mo₂S₂ unit, and the chlorine atom bridges two Mo atoms by substituting for two axial CO ligands of the parent compound **2**. Interestingly, the wholly configuration of the anion of **1** can be viewed as one dodecahedron with upper half splitting into two, which can be considered as a result from conversion of an edge-sharing bioctahedron of parent compound **2** to a face-sharing bioctahedral structure (Scheme 1). This interesting change is attributed to the fact that the Mo₂S₂ unit in compound **1** strongly adopts a planar configuration instead of a “butterfly” one which would lead to a cofacial bioctahedral

structure of imposed *D*_{3h} symmetry of the MoS₂ClMo core.

The fact that the Mo—Cl (0.2617(2), 0.2624(2) nm) distances are comparatively long and the Mo—Cl—Mo (66.44(4)°) angle is acute indicates weak Mo—Cl bonding. The structural effect of introducing a bridging chlorine atom replacing an axial CO in compound **2** can be appreciated by comparing the bond lengths and angles in Table 3. The Mo₂S₂ unit contracts slightly resulting in slightly smaller Mo—S—Mo angles and slightly shorter Mo—Mo distance. The Mo—S distances of **1** are essentially equal to those of **2**. It is evident that owing to the *trans* effect of the chlorine bridging ligand, the *trans* Mo—C distances are considerably shorter than those *cis* to chlorine in contrast with **2**, and this might be the reason why tetra-bridged compound could not be obtained by the synthetic reaction.

Table 3 Selected bond lengths (nm) and angles (°) for **1**·CH₃COCH₃

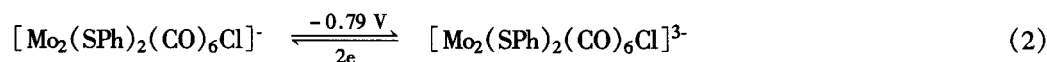
Cl—Mo(1)	0.2617(2)	Mo(1)—Mo(2)	0.28709(7)
Cl—Mo(2)	0.2624(2)	Mo(1)—C(4)	0.1931(6)
Mo(1)—C(1)	0.1929(6)	Mo(1)—C(6)	0.2001(8)
Mo(1)—C(2)	0.2003(8)	Mo(1)—C(5)	0.2013(7)
Mo(1)—C(3)	0.2005(7)	Mo(2)—S(1)	0.2474(2)
Mo(1)—S(1)	0.2476(2)	Mo(2)—S(2)	0.2471(2)
Mo(1)—S(2)	0.2477(2)		
Mo(1)-Cl-Mo(2)	66.44(4)	C(4)-Mo(2)-S(1)	95.0(2)
C(1)-Mo(1)-S(1)	103.1(2)	C(5)-Mo(2)-S(1)	83.3(2)
C(2)-Mo(1)-S(1)	82.8(2)	C(6)-Mo(2)-S(1)	167.7(2)
C(3)-Mo(1)-S(1)	161.8(2)	C(4)-Mo(2)-S(2)	101.9(2)
C(1)-Mo(1)-S(2)	96.1(2)	C(5)-Mo(2)-S(2)	163.1(2)
C(2)-Mo(1)-S(2)	167.0(2)	C(6)-Mo(2)-S(2)	82.5(2)
C(3)-Mo(1)-S(2)	83.8(2)	S(1)-Mo(2)-S(2)	108.82(5)
S(1)-Mo(1)-S(2)	108.58(5)	C(4)-Mo(2)-Cl	166.3(2)
C(1)-Mo(1)-Cl	168.2(2)	C(6)-Mo(2)-Cl	105.3(2)
C(2)-Mo(1)-Cl	103.9(2)	C(5)-Mo(2)-Cl	98.8(2)
C(3)-Mo(1)-Cl	97.2(2)	Mo(1)-S(1)-Mo(2)	70.90(4)
S(1)-Mo(1)-Cl	74.42(5)	Mo(1)-S(2)-Mo(2)	70.93(4)
S(2)-Mo(1)-Cl	74.22(5)	S(1)-Mo(2)-Cl	74.33(5)
S(1)-Mo(1)-Mo(2)	54.51(4)	S(2)-Mo(2)-Cl	74.18(5)
S(2)-Mo(1)-Mo(2)	54.45(4)	S(1)-Mo(2)-Mo(1)	54.59(4)
Cl-Mo(1)-Mo(2)	56.90(4)	S(2)-Mo(2)-Mo(1)	54.62(4)
Cl-Mo(2)-Mo(1)	56.66(4)		

Electrochemical behavior of **1**

The CV of **1** (Fig . 2) shows a redox couple at -0.79(-0.72/ -0.86) V *vs.* SCE with $i_a/i_c \approx 1$. The peak current parameter ($i_p/v^{1/2}$ AC) is 1435

Acms^{1/2} · V^{-1/2} · mol⁻¹, which is twice as that (688 Acms^{1/2} · V^{-1/2} · mol⁻¹) of ferrocene (which has been known as one-electron process) and is comparable with that (1360 Acms^{1/2} · V^{-1/2} · mol⁻¹) of compound **2** (which has been manifested to undergo a chemically and electro-

chemically reversible two-electron reduction in a single step,¹⁰) obtained under identical measurement conditions. This indicates that **1** underwent quasi-reversible



Evidently the CV behavior of **1** is very similar to that of parent compound **2** except the potential is more negative for **1**. This is due to the fact that they have similar plane Mo_2S_2 unit. The two-electron transfer character is attributed to the flexibility of cleavage/creation of Mo—Mo bond in Mo_2S_2 unit.^{6,10,11}

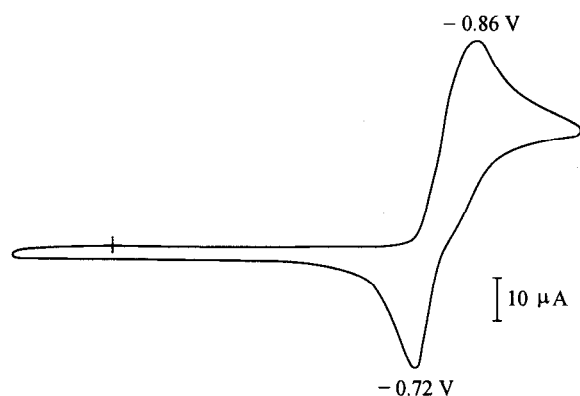


Fig. 2 Cyclic voltammogram of **1** in acetone; Concentration of the sample: 0.0013 mol/L; Scan rate: 100 mV/s.

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