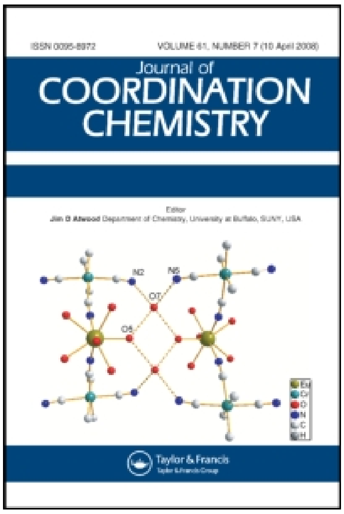

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Syntheses and Reactions of Dimolybdenum Alkyne Compounds Containing Functionally Substituted Ligands. The Crystal Structure of $[Co_2Mo_2(\mu_4\text{-}CHCH)(\mu\text{-}CO)_4(CO)_4\text{-}(\eta^5\text{-}C_5H_4C(O)Me)_2]$

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SYNTHESES AND REACTIONS OF DIMOLYBDENUM ALKYNE COMPOUNDS CONTAINING FUNCTIONALLY SUBSTITUTED LIGANDS. THE CRYSTAL STRUCTURE OF $[Co_2Mo_2(\mu_4\text{-CHCH})(\mu\text{-CO})_4(CO)_4\text{-}(\eta^5\text{-}C_5H_4C(O)Me)_2]$

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Three dimolybdenum alkyne complexes containing functionally substituted ligands $[Mo_2(\mu\text{-CHCH})(CO)_4(\eta^5\text{-}C_5H_4C(O)R)_2]$ [R = OEt, (**1a**); R = Me, (**1b**); R = Ph, (**1c**)] were synthesized by reactions of acetylene with *in situ* generated metal–metal triply bonded complexes $[Mo(CO)_2(\eta^5\text{-}C_5H_4C(O)R)]_2$ (R = OEt, Me, Ph). Further reaction of (**1a**), (**1b**) or (**1c**) with $Co_2(CO)_8$ in refluxing toluene gave another three new butterfly compounds $[Co_2Mo_2\text{-}(\mu_4\text{-CHCH})(\mu\text{-CO})_4(CO)_4(\eta^5\text{-}C_5H_4C(O)R)_2]$ [R = OEt, (**2a**); R = Me, (**2b**); R = Ph, (**2c**)]. The resulting compounds were characterized by elemental analyses, IR, ^{1}H NMR and MS. The crystal structure of (**2b**) was determined by single-crystal X-ray analysis. The results indicate that the existence of functional groups on the cyclopentadienyl ring has an influence on the reactivity of this type of complex.

Keywords: Alkyne complexes; cluster; molybdenum; cobalt; crystal structure

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INTRODUCTION

Dimolybdenum alkyne complexes $[Mo_2(\mu\text{-}R^IC{\equiv}CR^{II})(CO)_4(\eta^5\text{-}C_5H_5)_2]$ can react with a variety of reagents such as phosphines, thiols, elemental sulfur, $Ru_3(CO)_{12}$ and $Co_2(CO)_8$.[1-5] In the products the alkyne ligand is usually retained. Sometimes, alkyne scission occurs.[4] However, the reactivity of this type of complex may be influenced by the existence of a functional group on the cyclopentadienyl ring or on the alkyne ligand, just as it is influenced by the identity of the alkyne substituents R^I and R^{II} in some reactions.[4,5] Recently, we reported the reactivity of dimolybdenum asymmetry alkyne (phenylacetylene) complexes containing functionally substituted ligands on the cyclopentadienyl ring $[Mo_2(\mu\text{-}HC{\equiv}CPh)(CO)_4(\eta^5\text{-}C_5H_4C(O)R)_2]$ (R = OEt, Me, Ph).[6] Here, we report the reactivity of dimolybdenum symmetry alkyne (acetylene) complexes containing functionally substituted ligands on the cyclopentadienyl ring $[Mo_2(\mu\text{-}HC{\equiv}CH)(CO)_4(\eta^5\text{-}C_5H_4C(O)R)_2]$ (R = OEt, Me, Ph) and the crystal structure of $[Co_2Mo_2(\mu_4\text{-}HC{\equiv}CH)(\mu\text{-}CO)_4(CO)_4(\eta^5\text{-}C_5H_4C(O)Me)_2]$.

EXPERIMENTAL

All reactions and manipulations were performed under an atmosphere of pure nitrogen by using standard Schlenk or vacuum line techniques. The solvents were treated by the usual method for preparing anhydrous and deoxygenated solvents. Column chromatography was carried out by using silica gel of 160–200 mesh. Tank acetylene was used as purchased without further purification. $Co_2(CO)_8$,[7] $[Mo(CO)_3(\eta^5\text{-}C_5H_4C(O)R)]_2$ (R = Me, OEt, Ph)[8,9] were prepared by literature methods or slight modifications thereof. Infrared spectra were recorded on a Nicolet FT-IR 10 DX spectrophotometer; ^1H NMR spectra were recorded on a Bruker AM-300 MHz spectrometer; elemental analyses and MS determination were performed on a Carlo Erba 1106-type analyzer and Finnigan MAT 8430 (FAB) or HP 5988 (EI), respectively.

Preparation of (1a)–(1c)

Into an 100 cm^3 two-necked flask with a magnetic stir-bar, a rubber septum and a reflux condenser topped with a nitrogen inlet, $[Mo(CO)_3(\eta^5\text{-}C_5H_4C(O)OEt)]_2$ (0.634 g, 1 mmol) and toluene (50 cm^3) were added.

The mixture was refluxed with stirring for 3 h. Upon cooling to room temperature, ethyne was bubbled through the solution for 30 min. After stirring the solution for an additional 2 h, the solvent was removed at reduced pressure and the residue was extracted by CH_2Cl_2. The extracts were concentrated and separated on silica gel column. Elution with CH_2Cl_2 developed a large red band from which 0.400 g of complex (**1a**) as a red solid was obtained, 66.2% yield. Calcd. for $C_{22}H_{20}Mo_2O_8$ (%): C, 43.71; H, 3.31. Found: C, 43.91; H, 3.39. IR (KBr disc): 2002s, 1935vs, 1848w, 1811s, 1715m, 1701m cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.27–1.31 (t, 6H, $2CH_3$), 4.20–4.24 (q, 4H, $2CH_2$), 5.02 (s, 2H, 2CH), 5.42 (s, 4H, $2(H^3, H^4)$), 5.84 (s, 4H, $2(H^2, H^5)$).

The manipulations for the preparation of compounds (**1b**) and (**1c**) were similar to that of compound (**1a**). Compound (**1b**) was previously obtained by Song *et al.*, using a somewhat different method.[10]

Compound (**1b**), 65.1% yield. Calcd. for $C_{20}H_{16}Mo_2O_6$ (%): C, 44.14; H, 2.96. Found: C, 44.25; H, 3.01. IR (KBr disc): 1985s, 1936s, 1909vs, 1843s, 1685m, 1666m cm^{-1}. ^1H NMR ($CDCl_3$): δ 2.28 (s, 6H, $2CH_3$), 5.01 (s, 2H, 2CH), 5.46 (s, 4H, $2(H^3, H^4)$), 5.79 (s, 4H, $2(H^2, H^5)$).

Compound (**1c**), 52.2% yield. Calcd. for $C_{30}H_{20}Mo_2O_6$ (%): C, 53.89; H, 2.99. Found: C, 53.83; H, 3.06. IK (KBr disc): 1997s, 1948s, 1917vs, 1848s, 1829s, 1647m, 1630m cm^{-1}. ^1H NMR ($CDCl_3$): δ 4.97 (s, 2H, 2CH), 5.46 (s, 4H, $2(H^3, H^4)$), 5.83 (s, 4H, $2(H^2, H^5)$), 7.35–7.64 (m, 10H, $2C_6H_5$).

Reactions of (1a)–(1c) with $Co_2(CO)_8$

To the flask described above, $Co_2(CO)_8$ (0.137 g, 0.4 mmol), compound (**1a**) (0.242 g, 0.4 mmol) and toluene (30 cm^3) were added. The mixture was refluxed with stirring for 50 mins; the colour changed from red to deep green. After addition of 5 g of silica gel the solvent was removed and the residue chromatographed. Elution with CH_2Cl_2 produced: (i) a small quantity of $Co_2(CO)_8$, (ii) unreacted (**1a**) (0.069 g), (iii) dark green solid (**2a**) (0.084 g), 25.2% yield. Calcd. for $C_{26}H_{20}Co_2Mo_2O_{12}$ (%): C, 37.41; H, 2.40. Found: C, 37.50; H, 2.42. IR (KBr disc): 2052s, 2006vs, 1937w, 1844s, 1798s, 1725s cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.30–1.35 (t, 6H, $2CH_3$), 4.24–4.29 (q, 4H, $2CH_2$), 5.60 (s, 4H, $2(H^3, H^4)$), 5.89 (s, 4H, $2(H^2, H^5)$), 8.02 (s, 2H, 2CH). MS (FAB): 835 (M^+), 723 (M^+-4CO), 696 (M^+-5CO), 668 (M^+-6CO), 638 (M^+-7CO), 611 (M^+-8CO), 475 ($Co_2Mo_2C_2H_2C_5H_4C(O)OEt^+$), 340 ($Co_2Mo_2C_2H_2^+$), 311 ($Co_2Mo_2^+$).

The manipulations for the reactions of compounds (**1b**) and (**1c**) with $Co_2(CO)_8$ were similar to that of compound (**1a**).

Compound (**2b**), 21.0% yield. Calcd. for $C_{24}H_{16}Co_2Mo_2O_{10}$ (%): C, 37.21; H, 2.07. Found: C, 37.02; H, 2.05. IR (KBr disc): 2054s, 2015vs, 1990s, 1979s, 1853vs, 1811s, 1678s cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 2.43 (s, 6H, $2CH_3$), 5.66 (s, 4H, 2(H^3, H^4)), 5.86 (s, 4H, 2(H^2, H^5)), 7.93 (s, 2H, 2CH). MS (FAB): 775 (M^+), 663 (M^+–4CO), 608 (M^+–6CO), 579 (M^+–7CO), 550 (M^+–8CO), 442 ($Co_2Mo_2C_2H_2C_5H_4C(O)Me^+$), 340 ($Co_2Mo_2C_2H_2^+$), 311 ($Co_2Mo_2^+$).

Compound (**2c**), 19.5% yield. Calcd. for $C_{34}H_{20}Co_2Mo_2O_{10}$ (%): C, 45.43; H, 2.23. Found: C, 45.43; H, 2.17. IR (KBr disc): 2062s, 2033vs, 1933w, 1838s, 1796s, 1644s cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 5.70 (s, 4H, 2(H^3, H^4)), 5.97 (s, 4H, 2(H^2, H^5)), 7.49–7.79 (m, 10H, $2C_6H_5$), 8.12 (s, 2H, 2CH). MS (FAB): 899 (M^+), 787 (M^+–4CO), 732 (M^+–6CO), 674 (M^+–8CO), 507 ($Co_2Mo_2C_2H_2C_5H_4C(O)Ph^+$), 340 ($Co_2Mo_2C_2H_2^+$), 311 ($Co_2Mo_2^+$).

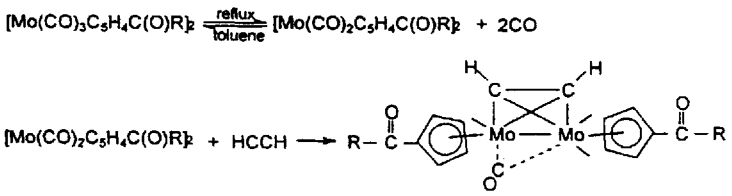
Crystallography of (2b)

Crystals of (**2b**) suitable for diffraction analysis were grown from petroleum ether/CH_2Cl_2 (1 : 1) solution at −20°C. Preliminary examination and data collection were performed with MoK_α radiation ($\lambda = 0.71069$ Å) on a Rigaku AFC7R diffractometer equipped with a graphite monochromator.

The structure was refined by full-matrix least-squares methods with anisotropic thermal parameters for the non-hydrogen atoms. All calculations were performed using the TEXSAN program system. Crystal data and experimental details are collected in Table I. Full lists of crystallographic data are available from the authors upon request.

RESULTS AND DISCUSSION

The formations of compounds (**1a**)–(**1c**) are shown in Scheme 1. The first step is a reversible reaction. According to the literature procedure,[4,10] 10–24 h is necessary. In our attempt to shorten the reaction time, we reduced the concentration of the reaction system (1 mmol/50 cm^3). This favoured the decarbonylation process greatly. In most cases, 3–5 h was

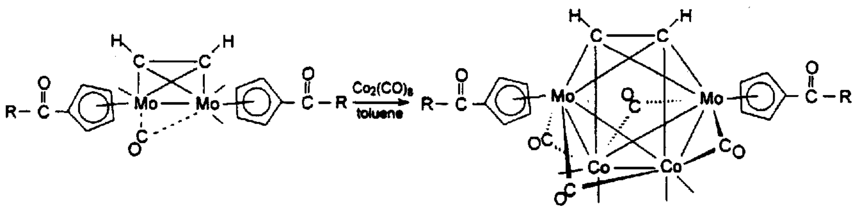


SCHEME 1 Formation of compounds (**1a**)–(**1c**) [R = OEt, **1a**; Me, **1b**; Ph, **1c**].

TABLE I Crystal and refinement data for (**2b**)

Empirical formula	$C_{24}H_{16}Co_2Mo_2O_{10}$
Formula weight	774.13
Crystal system	Orthorhombic
Space group	*Pbcn*
$F(000)$	1512.00
Structure solution	Direct method
Lattice parameters	
a (Å)	11.050(2)
b (Å)	11.135(3)
c (Å)	20.904(5)
V (Å^3)	2571(1)
Z	4
D_c (g cm^{-3})	1.999
Absorption coefficient (cm^{-1})	22.74
Temperature (°C)	20
$2\theta_{max}$(°)	55.0
No. observations	2386 ($I > 3.00\sigma(I)$)
No. variable	205
R^a	0.025
R_w^b	0.033
Goodness of fit indicator	1.21
Max. shift in final cycle	0.04
Max. residual (e Å^{-3})	0.57

[a] $R = \sum\|F_o| - |F_c\|/\sum|F_o|$; [b] $R_w = [\sum\omega(|F_o| - |F_c|)^2/\sum\omega F_o^2]^{1/2}$.



SCHEME 2 Formation of compounds (**2a**)–(**2c**) [R = OEt, **2a**; Me, **2b**; Ph, **2c**].

sufficient for this step. The second step could be achieved instantly, for there was no difference in yield in letting the second step react for 1 h or for 10 h. Compounds (**1a**)–(**1c**) are air-stable red solids. They are soluble in polar solvents like CH_2Cl_2 and THF.

Complexes (**1a**)–(**1c**) react with $Co_2(CO)_8$ in refluxing toluene. The products (**2a**)–(**2c**) were cluster expansions with butterfly configuration which could be readily isolated by column chromatography as dark green or dark blue solids (Scheme 2).

Compared with the complex $[Mo_2(\mu\text{-}R^IC{\equiv}CR^{II})(CO)_4(\eta^5\text{-}C_5H_5)_2]$, which contains no functionally substituted ligand in the molecule,[5] complexes (**1a**)–(**1c**) required a longer time to react with $Co_2(CO)_8$ and a quantity of unreacted starting materials could always be isolated.

This indicates that the existence of functional groups on the cyclopentadienyl ring reduce the reactivity of dimolybdenum alkyne complexes.

The compounds were well characterized by elemental analysis, IR, ^{1}H NMR and MS. In the IR spectra of (**1a**)–(**1c**), the absorption bands around 2000 cm^{-1} are caused by terminal carbonyls, the absorption bands in the range 1811–1864 cm^{-1} are caused by semi-bridging carbonyls and the ones in the range 1612–1718 cm^{-1} are caused by the carbonyls (C=O) on the substituted cyclopentadienyl rings. IR spectra of (**2a**)–(**2c**) contain peaks attributable to terminal carbonyls in the range 1933–2065 cm^{-1}, bridging carbonyls in the range 1778–1859 cm^{-1} and the carbonyls (C=O) on the substituted cyclopentadienyl rings in the range 1644–1725 cm^{-1}.

For the terminal protons on the intact alkyne ligand, (**1a**), (**1b**) and (**1c**) show one singlet around 5.00 ppm, while (**2a**), (**2b**) and (**2c**) show one singlet around 8.00 ppm in their ^{1}H NMR spectra. For the eight protons on the two substituted cyclopentadienyl rings (C_5H_4), all compounds show two symmetric singlets in the range 5.42–5.94 ppm; the downfield singlet should be assigned to the H^2 and H^5 protons which are close to the substituent and the upfield one to the H^3 and H^4 protons, being remote from the substituent.

X-ray single-crystal structure analyses of (**2b**) were undertaken. Final atomic coordinates and thermal parameters of the non-hydrogen atoms are given in Table II. Selected bond lengths and angles are listed in Table III. The molecular structure is presented in Figure 1.

TABLE II Positional parameters and *B*(*eq*) for compound (**2b**)

Atom	*x*/*a*	*y*/*b*	*z*/*c*	*B*(*eq*)
Mo	0.01588(2)	0.33253(2)	0.34620(1)	2.205(6)
Co	0.11459(3)	0.22038(4)	0.24505(2)	2.543(8)
O(1)	−0.0960(3)	0.0846(2)	0.3817(1)	4.53(6)
O(2)	0.2679(2)	0.2169(3)	0.3689(1)	5.42(7)
O(3)	0.1372(3)	−0.0317(2)	0.2814(2)	6.05(8)
O(4)	0.3635(2)	0.2434(3)	0.2006(2)	6.99(9)
O(5)	0.1960(3)	0.2908(3)	0.5126(1)	5.79(8)
C(1)	0.0971(3)	0.4595(3)	0.4226(2)	3.09(6)
C(2)	0.0127(3)	0.5277(3)	0.3882(2)	3.26(7)
C(3)	−0.1031(3)	0.4770(3)	0.3986(2)	3.13(7)
C(4)	−0.0920(3)	0.3775(3)	0.4386(1)	3.00(6)
C(5)	0.0342(3)	0.3643(3)	0.4541(1)	2.86(6)
C(6)	0.0902(3)	0.2784(3)	0.4988(2)	3.78(8)
C(7)	0.0137(5)	0.1804(5)	0.5269(2)	5.7(1)
C(8)	−0.0622(3)	0.1719(3)	0.3575(1)	3.19(7)
C(9)	0.1762(3)	0.2534(3)	0.3506(2)	3.44(7)
C(10)	0.1259(3)	0.0643(3)	0.2650(2)	3.61(8)
C(11)	0.2681(3)	0.2381(3)	0.2189(2)	3.78(8)
C(12)	0.0653(2)	0.3880(3)	0.2468(1)	2.35(5)

$B(eq) = (8/3)\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha)$.

TABLE III Selected bond lengths (Å) and angles (°) for compound (**2b**)

Mo–Co	2.6870(5)	Co–C(12)	1.945(3)	Mo–Co*	2.6973(5)
O(1)–C(8)	1.158(4)	Mo–C$_p$	2.325	O(2)–C(9)	1.158(4)
O(3)–C(10)	1.130(4)	O(4)–C(11)	1.123(4)	Mo–C(8)	2.000(3)
Mo–C(9)	1.981(3)	Mo–C(12)	2.235(3)	Mo–C(12)*	2.228(3)
Co–Co*	2.5409(9)	Co–C(8)*	2.285(3)	Co–C(10)	1.791(4)
C(12)–C(12)*	1.448(6)	Co–C(11)	1.794(3)		
Co–Mo–Co*	56.32(2)	Co–Mo–C(1)	132.05(8)		
Co–Mo–C(8)	81.51(9)	Co–Mo–C(9)	57.78(9)		
Co–Mo–C(12)	45.41(7)	Co–Mo–C(12)*	66.78(7)		
Co*–Mo–C(1)	168.00(8)	Co*–Mo–C(8)	55.86(9)		
Co*–Mo–C(9)	107.8(1)	Co*–Mo–C(12)	66.49(7)		
Co*–Mo–C(12)*	45.29(8)	C(8)–Mo–C(9)	89.0(1)		
C(8)–Mo–C(12)	117.5(1)	C(1)–Mo–C(12)	112.2(1)		
C(8)–Mo–C(12)*	100.2(1)	C(9)–Mo–C(12)	87.0(1)		
C(9)–Mo–C(12)*	121.6(1)	C(12)–Mo–C(12)*	37.9(1)		
Mo–Co–Mo*	97.14(2)	Mo–Co–Co*	62.05(2)		
Mo–Co–C(8)*	138.17(9)	Mo–Co–C(10)	107.2(1)		
Mo–Co–C(11)	125.0(1)	Mo–Co–C(12)	54.93(8)		
Mo–Co*–Co	61.64(2)	Mo–Co*–C(8)	46.42(8)		
Mo–Co*–C(10)*	130.6(1)	Mo–Co*–C(11)*	103.8(1)		
Mo–C(5)–C(6)	124.1(2)	Mo–Co*–C(12)*	54.49(8)		
Co–Co*–C(8)	79.86(9)	C(1)–C(5)–C(6)	124.8(3)		
Co–Co*–C(10)*	92.9(1)	Co–Co*–C(11)*	165.4(1)		
Co–Co*–C(12)*	73.69(8)	C(8)*–Co–C(10)	90.4(1)		
C(8)*–Co–C(11)	88.8(1)	Mo–C(8)–Co*	77.7(1)		
C(8)*–Co–C(12)	100.0(1)	Mo–C(8)–O(1)	160.3(3)		
C(10)–Co–C(11)	96.4(2)	Co*–C(8)–O(1)	121.8(3)		
C(10)–Co–C(12)	161.1(1)	Mo–C(9)–O(2)	162.7(3)		
C(11)–Co–C(12)	99.5(1)	Co–C(10)–O(3)	175.0(3)		
Co–C(11)–O(4)	176.2(3)	Mo–C(12)–Mo*	129.5(1)		
Mo–C(12)–Co	79.7(1)	Mo–C(12)–C(12)*	70.8(2)		
Mo–C(12)*–Co*	80.2(1)	Mo–C(12)*–C(12)	71.3(2)		
Co*–C(12)*–C(12)	106.31(8)				

*Symmetry operator: $(-x, y, 0.5 - z)$.

As seen in Figure 1, compound (**2b**) is a butterfly cluster with a bridging acetylene ligand. The butterfly consists of two cobalt atoms that form the hinge and two molybdenum atoms that act as the wingtips. Each molybdenum atom bears an acetylcyclopentadienyl ring and each cobalt atom has two terminal carbonyl ligands. In addition, each Co–Mo bond is spanned by an asymmetrically bridging carbonyl ligand that is more closely bonded to molybdenum than to cobalt. The acetylene ligand lies parallel to the Co–Co bond and is bonded to all four metal atoms. The whole molecule is symmetric under the $(-x, y, 0.5-z)$ symmetry operator. In the butterfly core, the Co–Co bond length (2.5409(9) Å), the average Co–Mo bond length (2.6922(5) Å) and the average Co–C bond length (1.945(3) Å) are close to the corresponding values observed in the related cluster.[5,6] The Mo···Mo distance is 3.92(4) Å, well beyond the range of possible bonding. The average C–Mo bond length is 2.232(3) Å, the C(12)–C(12)* bond length

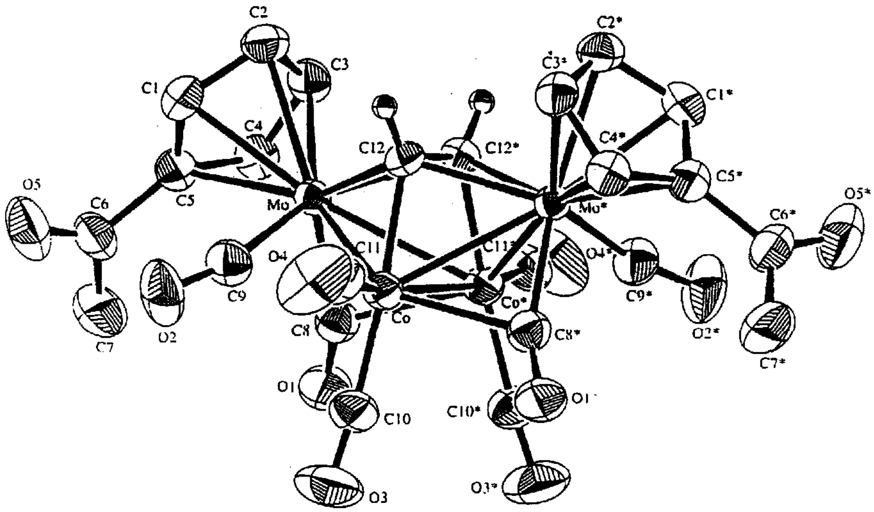


FIGURE 1 The molecular structure of compound **2b**.

is 1.448(6) Å. For the carbonyl C(9)O(2), ∠MoC(9)O(2) = 162.7(3)°, ∠C(9)MoCo = 57.78(9), C(9)–Mo distance is 1.981(3) Å, C(9)–Co distance is 2.338(4) Å, the asymmetric parameter $\alpha = 0.180$. For the carbonyl C(8)O(1), ∠MoC(8)O(1) = 160.3(3)°, ∠C(8)MoCo* = 55.8(6)°, C(8)–Mo distance is 2.000(3) Å, C(8)–Co* distance is 2.285(3) Å, the asymmetric parameter $\alpha = 0.143$. Thus both C(9)O(2) and C(8)O(1) should be classed as semi-bridging carbonyls.[5,11]

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References

[1] G. Conole, K.A. Hill, M. Mcpartlin, M.J. Mays and M.J. Morris, *J. Chem. Soc., Chem. Commun.*, 688 (1989).
[2] G. Conole, M. Mcpartlin, M.J. Mays and M.J. Morris, *J. Chem. Soc., Dalton Trans.*, 2359 (1990).
[3] H. Adams, N.A. Bailey, S.R. Gay, T. Hamilton and M.J. Morris, *J. Organomet. Chem.*, **493**, C25 (1995).
[4] H. Adams, L.J. Gill and M.J. Morris, *Organometallics*, **15**, 4182 (1996).
[5] H. Adams, N.A. Bailey, L.J. Gill, M.J. Morris and F.A. Wildgoose, *J. Chem. Soc., Dalton Trans.*, 1437 (1996).

[6] J. Zhang, X.N. Chen, Y.Q. Yin and X.Y. Huang, *J. Organomet. Chem.*, **579**, 304 (1999).
[7] C.H. Wei and L.F. Dahl, *Inorg Chem.*, **4**, 1 (1965).
[8] L.C. Song and J.Y. Shen, *Chem. J. Chinese Universities*, **13**, 1227 (1992).
[9] X.N. Chen, J. Zhang, Y.Q. Yin and J. Sun, *J. Coord. Chem.*, **49**, 161 (1999).
[10] L.C. Song, J.Y. Shen, Q.M. Hu and X.Y. Huang, *Inorg. Chim. Acta*, **249**, 175 (1996).
[11] M.D. Curtis, K.R. Han and W.M. Butler, *Inorg. Chem.*, **19**, 2096 (1980).