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REACTIVITY OF THE METAL–METAL-MULTIPLE BOND IN METAL CARBONYL DERIVATIVES

VI *. INVESTIGATION INTO THE REACTION OF $[C_5Me_5(CO)_2Mo]_2$ WITH $CH_3N{=}C{=}S$: FORMATION AND CRYSTAL STRUCTURE OF $(C_5Me_5)_2(CO)_4Mo_2(\mu,\eta^2\text{-}C{\equiv}NCH_3)$

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Summary

The reaction of $[C_5Me_5(CO)_2Mo]_2$ with $CH_3N{=}C{=}S$ gives $C_5Me_5(CO)_2$-MoS_2CNHCH_3 and $[C_5Me_5(CO)_2Mo]_2(\mu,\eta^2\text{-}CNCH_3)$. The latter compound contains a bridging isonitrile ligand of fluxionial behavior as proved by high temperature ^1H NMR spectroscopy. The $\sigma + \pi$ bonded character of the isonitrile bridge was established by X-ray crystallography.

Much attention has been paid to the addition of small donor molecules to the formally unsaturated $[C_5H_5Mo(CO)_2]_2$ [2]. Careful investigations have been carried out recently on the symmetrical addition of allenes [3]. In contrast, relatively little is known about the reaction with heteroallenes, X=C=Y. As a first example we showed that $(C_5R_5)_2Mo_2(CO)_4$ ($R = H, CH_3$) reacts with boiling S=C=S to give complexes of the type $C_5R_5(CO)_3Mo\text{–}Mo(CO)_2(\eta^2\text{-}CS_2)C_5R_5$, containing a nonbridging, terminal η^2-CS_2 ligand [4]. Stimulated by this result, we are using other heteroallenes, e.g. isothiocyanates and carbodiimides. In this paper we report on the reaction of $[C_5Me_5(CO)_2Mo]_2$ with $CH_3N{=}C{=}S$.

Experimental section

All procedures were carried out under nitrogen with solvents freshly distilled under nitrogen from appropriate drying agents. I was prepared according to a published procedure [5], CH_3NCS was obtained from Fluka.

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* For part V see ref. 1.

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Preparation of $[C_5Me_5(CO)_2Mo]_2(\mu,\eta^2\text{-}CNCH_3)$

A mixture of 287 mg (0.50 mmol) of I with 36 mg (0.50 mmol) of CH_3NCS and 100 ml of toluene was stirred at 100°C for 15 h. After concentration of the solvent to 20 ml the solution was transferred to a column (30 × 3 cm, SiO_2). Using toluene a red band was first eluted, containing 70% of unreacted starting material. A second red-brown band was chromatographed again with toluene/pentane (3/1) to remove the remaining starting material. The red-brown complex III was isolated in 25% yield with respect to reacted material; it was recrystallized from ether at −35°C. A third red-brown band contained complex II.

Analysis of $[C_5Me_5(CO)_2Mo]_2(\mu,\eta^2\text{-}CNCH_3)$, (III): Found: C, 50.73; H, 5.47; N, 1.98%; Molweight (field desorption mass spectroscopy from toluene), 607 (related to ^{92}Mo). $C_{26}H_{33}Mo_2NO_4$ calcd.: C, 50.70; H, 5.40; N, 2.28%; Molweight 615.4.

Properties: Darkred crystals, mp. 168°C (dec.).

X-Ray data collection, solution and refinement

Data were collected as previously described [6]. The instrument was a CAD-4 diffractometer operating on the OS/4 mode. Important crystallographic data collection and processing parameters are listed in Table 1. From the crystals available only one of marginal quality was found and used but since even proof of structure was desirable given the nature of the reaction studied, a data set was collected.

The Niggli matrix [7] showed the lattice to be end-centered and the distribution of unitary structure factors was that of an acentric space group. The centrosymmetric choice, $C2/c$, with $Z = 4$ would require the molecule to lie at a special position (with molecular symmetry of either an inversion center or a two-fold axis) neither one of

TABLE 1

SUMMARY OF DATA COLLECTION AND PROCESSING PARAMETERS

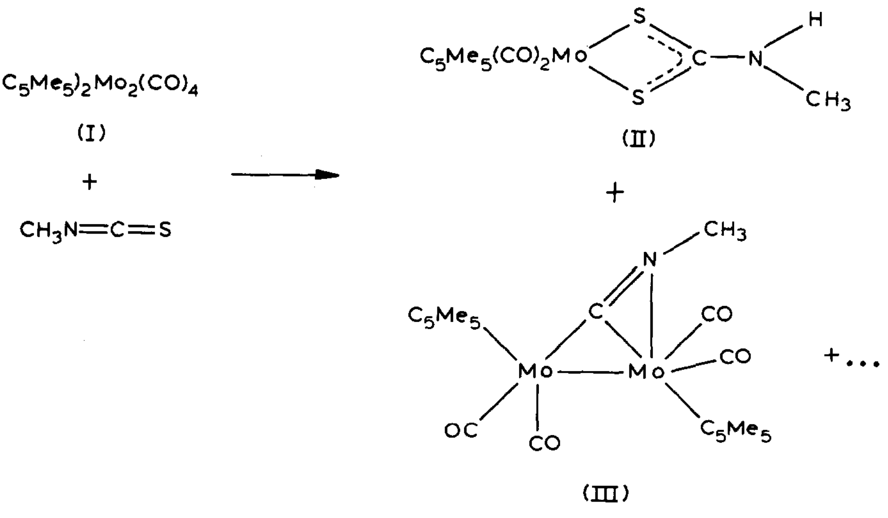
Space group	C_c
Cell constants	a 16.517(5) Å b 8.754(14) Å c 21.599(6) Å β 121.70(2)°
Cell volume	V 2657.041 Å^3
Molecular formula	$C_{26}H_{33}Mo_2NO_4$
Molecular weight	615.444
Density (calcd.)	ρ 1.5383 g cm^{-3}
Radiation	Mo-K_α (λ 0.71609 Å)
Absorption coefficient	μ 9.50 cm^{-1}
Data collection range	$4° \leq 2\theta \leq 50°$
Scan width	$\Delta\theta = (1.00+0.35 \tan\theta)°$
Maximum scan time	240 sec
Scan speed range	0.38 to 5.03° min^{-1}
Total data collected	2560
Data with $I > 3\sigma(I)$	971
Total variables	100
$R = \Sigma\|\|F_0\|-\|F_c\|\|/\Sigma\|F_0\|$	0.0604
$R_w = [\Sigma w(\|F_0\|-\|F_c\|)^2/\Sigma w\|F_0\|^2]^{1/2}$	0.0586
Weights	$w = 0.9787/\sigma^2(F)$
Goodness of fit	1.66

which is possible for this substance unless there is crystallographic disorder in the lattice. Since we found no evidence of such disorder, the completely consistent assignment of space group C_c was made. The two independent Mo atoms were found in a Patterson map and the rest of the non-hydrogen atoms in subsequent Fourier maps. Given the large number of atoms for the number of observed data, the C_5Me_5 ring atoms were refined as rigid bodies using DFIX in SHELX-76 [8] (see Table 4). Under these conditions, the refinement proceeded smoothly to the values listed in Table 1.

Results and discussion

Whereas the reaction of $[C_5H_5(CO)_2Mo]_2$ with CH_3NCS gives as the only isolable product traces of the dithiocarbamato complex $C_5H_5(CO)_2MoS_2CNHCH_3$ [9], $[C_5Me_5(CO)_2Mo]_2$ gives several products, depending on the reaction conditions. With a tenfold excess of CH_3NCS in toluene at 45°C, the only product is $C_5Me_5(CO)_2MoS_2CNHCH_3$ (II), which can be characterized by means of IR (ν(NH) 3370, ν(CO) 1925, 1810 cm^{-1}) and ^1H NMR spectroscopy ($\delta(CH_3)$ ^{1}1.83, $\delta(NCH_3)$ ^{2}2.98 ppm). This product may arise from sulfur loss of another ligand, followed by sulfur insertion. The resulting dithiocarbonimidato complex [10,11] formally requires protonation in order to stabilize the $Cp(CO)_2Mo$ fragment as a three electron donor.

The same product is formed in a similar experiment at 100°C (ratio metal/ligand 1/10), accompanied by several by-products of general composition $(C_5Me_5)_2Mo_2(CH_3NCS)_2S_2$, the structures of which are still unclear [12]. If the same reaction is carried out with a metal/ligand ratio of 1/1, in addition to complex II the red-brown complex III can be isolated (Scheme 1). Its composition is confirmed by elemental analysis and mass spectrum. From the IR spectrum there is evidence for a bridging isonitrile ligand (ν(CN) 1725 cm^{-1}), whereas the terminal CO region shows four absorptions. The ^1H NMR spectrum (Table 2) exhibits one signal for the



SCHEME 1

TABLE 2

IR (KBr) AND ^{1}H NMR SPECTRA OF COMPOUNDS II AND III

IR (cm^{-1})					^{1}H NMR (ppm)^a		
	ν(NH)	ν(CO)	ν(C–N)	ν(N–C–S)	δ(NCH$_3$) (J(NH–CH$_3$))^b	δ(CH$_3$)	Solvent
II	3370m	1925s, 1810vs		1515m	^{2}2.98 (6.0)	^{1}1.83	CDCl$_3$
III		1947s, 1900vs 1863vs, 1820s	1725m		^{1}3.65	^{1}1.94, ^{1}1.84	CDCl$_3$
					^{1}3.0	^{1}1.55, ^{1}1.66	Toluene-d_8

^{a} i-(CH$_3$)$_4$Si; Varian T-60 instrument. ^{b} The NH proton is not detectable.

N–CH$_3$ group, but two resonances for the methyl groups of the cyclopentadienyl ring. The inequivalent character of these groups is a function of the temperature: if the sample is warmed up in toluene-d_8, broadening of the signals occurs and coalescence is observed at about 82°C. Above 90°C there is only one peak. This process is reversible, but superceded by partial decomposition which prevents quantitative analysis.

The spectroscopic data are consistent with a formulation as a bridged isonitrile complex, in which asymmetry is caused by a $\sigma + \pi$ coordination of the bridging ligand [13,14]. However, the coalescence temperature is considerably higher (about 100°C) than in complexes incorporating the semibridging cyanide or cyanoamide ligands [13], which are bonded in the same $\sigma + \pi$ fashion.

In order to confirm our structural proposal and to study the influence of the pentamethyl groups on the geometry of the molecule we decided to do an X-ray examination of the structure. While this study was being completed, Adams and coworkers [14] published a different synthesis and a structural study of the related isonitrile derivative $(C_5H_5)_2Mo_2(CO)_4(\mu,\eta^2\text{-}CNC_6H_5)$ (IV), which differs from III in having C_5H_5 and CN–C_6H_5 groups instead of C_5Me_5 and CN–Me. Since these crystals were, obviously, much better and the quality of refinement superior, we were pleased to note that our results are in good agreement with theirs.

Description of the molecule

The atomic parameters, bond distances and bond angles are given in Tables 3–5. Figures 1–3 show an ORTEP, a stereoplot and a packing diagram of III.

If we look first at the core of the molecule, we find that the distances and angles associated with the (Cp$_2$Mo(1)Mo(2)CN–C) core of III and IV (Table 6) are of the same order. Therefore, despite our much larger e.s.d.'s a consistent picture of the core of these two molecules emerges from the above comparisons of bond distances and angles and the accurate and precise determination of Adams et al. [14] lends credibility to our, otherwise, low-precision determination. Clearly evident in both studies is the dual $\sigma + \pi$ role played by the asymmetric bridging isonitrile with σ Mo–C(N) distances of 1.93(4) (III) and 1.942(2) Å (IV) and π Mo–C(N) distances of 2.21(4) (III) and 2.247(2) Å (IV). Given these broad areas of agreement, the discussion of the meaning of these parameters in the report of Adams [14] makes further comments by us superfluous.

(Continued on p. 253)

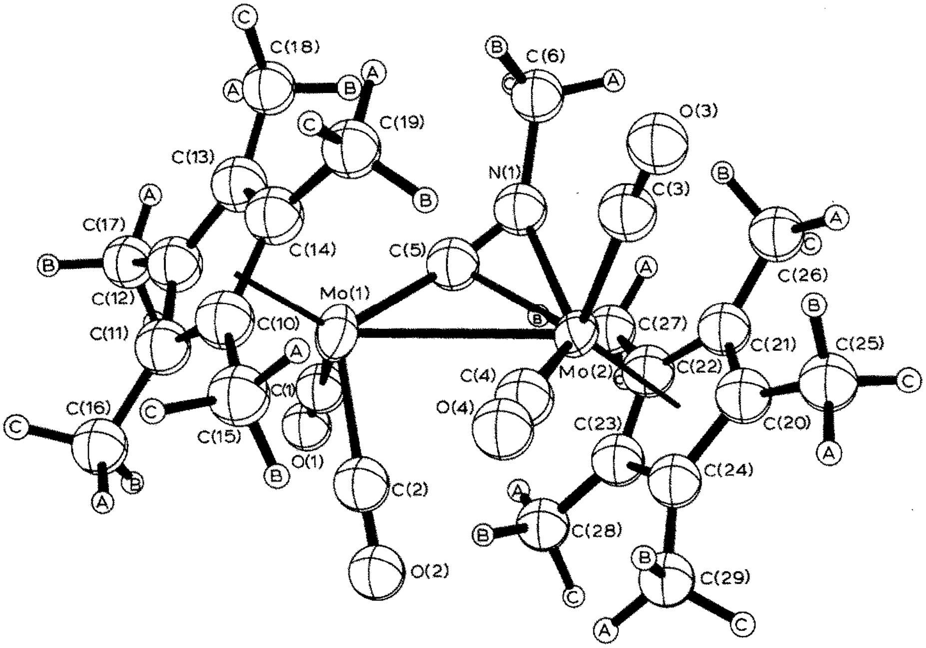


Fig. 1. ORTEP of III showing the numbering system used.

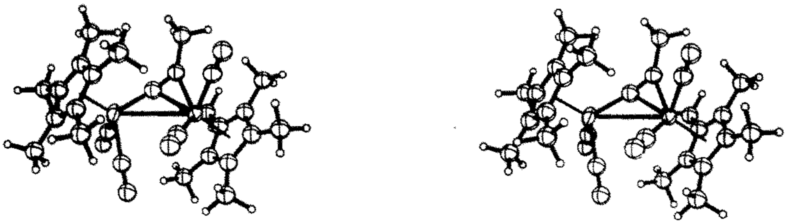


Fig. 2. Stereo pair of III.

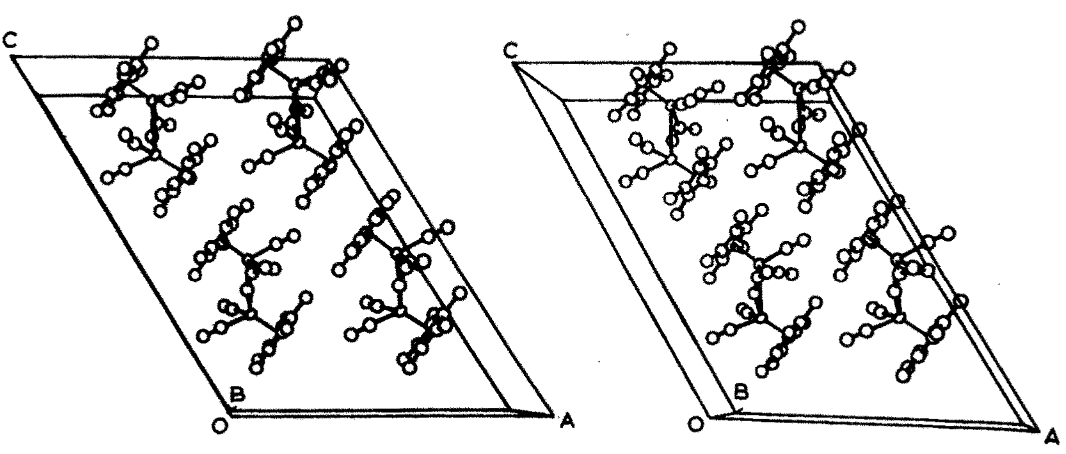


Fig. 3. Packing diagram of III.

TABLE 3

ATOMIC COORDINATES AND THERMAL PARAMETERS (× 1000)

Atom	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo(1)	0.2890(0)	0.0370(5)	0.3070(0)	62(4)	92(4)	43(4)	5(3)	36(3)	1(3)
Mo(2)	0.3971(2)	0.0219(5)	0.4828(2)	42(3)	71(3)	44(3)	14(3)	26(3)	5(3)
O(1)	0.071(1)	−0.007(4)	0.238(2)	169(18)					
O(2)	0.219(3)	0.338(3)	0.342(2)	139(16)					
O(3)	0.613(1)	0.060(3)	0.555(2)	92(11)					
O(4)	0.486(2)	0.320(2)	0.464(2)	101(11)					
N(1)	0.373(2)	−0.196(4)	0.431(2)	77(10)					
C(1)	0.153(1)	−0.009(3)	0.266(2)	42(9)					
C(2)	0.227(3)	0.214(5)	0.324(2)	185(28)					
C(3)	0.533(1)	−0.039(6)	0.534(4)	185(32)					
C(4)	0.443(2)	0.217(2)	0.465(2)	50(11)					
C(5)	0.324(3)	−0.115(4)	0.382(2)	80(12)					
C(6)	0.426(3)	−0.347(5)	0.443(3)	128(17)					
C(10)	0.383(1)	0.179(1)	0.271(1)	45(8)					
C(11)	0.290(1)	0.155(1)	0.209(1)	51(9)					
C(12)	0.276(1)	−0.005(1)	0.197(1)	69(10)					
C(13)	0.360(1)	−0.080(1)	0.252(1)	85(11)					
C(14)	0.426(1)	0.034(1)	0.297(1)	37(9)					
C(15)	0.427(1)	0.331(1)	0.302(1)	92(12)					
C(16)	0.220(1)	0.277(1)	0.164(1)	192(24)					
C(17)	0.188(1)	−0.082(1)	0.138(1)	156(19)					
C(18)	0.376(1)	−0.249(1)	0.260(1)	144(19)					
C(19)	0.524(1)	0.006(1)	0.361(1)	81(9)					
C(20)	0.419(1)	0.057(2)	0.597(1)	43(8)					
C(21)	0.383(1)	−0.094(2)	0.577(1)	45(8)					
C(22)	0.290(1)	−0.086(2)	0.515(1)	40(8)					
C(23)	0.267(1)	0.071(2)	0.497(1)	73(14)					
C(24)	0.347(1)	0.159(2)	0.548(1)	47(8)					
C(25)	0.516(1)	0.101(2)	0.659(1)	90(12)					
C(26)	0.436(1)	−0.238(2)	0.615(1)	96(12)					
C(27)	0.225(1)	−0.218(2)	0.476(1)	92(11)					
C(28)	0.175(1)	0.133(2)	0.436(1)	132(15)					
C(29)	0.355(1)	0.330(2)	0.549(1)	122(14)					
H(6A)	0.445(3)	−0.429(5)	0.486(3)	140(0)					
H(6B)	0.485(3)	−0.334(5)	0.435(3)	140(0)					
H(6C)	0.364(3)	−0.387(5)	0.393(3)	140(0)					
H(15A)	0.498(1)	0.314(1)	0.349(1)	140(0)					
H(15B)	0.385(1)	0.391(1)	0.319(1)	140(0)					
H(15C)	0.431(1)	0.397(1)	0.262(1)	140(0)					
H(16A)	0.250(1)	0.387(1)	0.186(1)	140(0)					
H(16B)	0.156(1)	0.262(1)	0.166(1)	140(0)					
H(16C)	0.202(1)	0.268(1)	0.109(1)	140(0)					
H(17A)	0.198(1)	−0.204(1)	0.143(1)	140(0)					
H(17B)	0.174(1)	−0.045(1)	0.086(1)	140(0)					
H(17C)	0.128(1)	−0.052(1)	0.143(1)	140(0)					
H(18A)	0.314(1)	−0.307(1)	0.218(1)	140(0)					
H(18B)	0.390(1)	−0.285(1)	0.313(1)	140(0)					
H(18C)	0.436(1)	−0.278(1)	0.255(1)	140(0)					
H(19A)	0.538(1)	−0.116(1)	0.368(1)	140(0)					
H(19B)	0.530(1)	0.053(1)	0.409(1)	140(0)					
H(19C)	0.576(1)	0.060(1)	0.352(1)	140(0)					
H(25A)	0.523(1)	0.223(2)	0.661(1)	140(0)					

TABLE 3 (continued)

Atom	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
H(25B)	0.570(1)	0.050(2)	0.652(1)	140(0)					
H(25C)	0.524(1)	0.060(2)	0.710(1)	140(0)					
H(26A)	0.506(1)	−0.210(2)	0.660(1)	140(0)					
H(26B)	0.442(1)	−0.309(2)	0.576(1)	140(0)					
H(26C)	0.397(1)	−0.299(2)	0.634(1)	140(0)					
H(27A)	0.261(1)	−0.324(2)	0.502(1)	140(0)					
H(27B)	0.207(1)	−0.219(2)	0.420(1)	140(0)					
H(27C)	0.161(1)	−0.209(2)	0.478(1)	140(0)					
H(28A)	0.127(1)	0.039(2)	0.407(1)	140(0)					
H(28B)	0.188(1)	0.197(2)	0.399(1)	140(0)					
H(28C)	0.143(1)	0.207(2)	0.457(1)	140(0)					
H(29A)	0.289(1)	0.377(2)	0.505(1)	140(0)					
H(29B)	0.413(1)	0.363(2)	0.542(1)	140(0)					
H(29C)	0.367(1)	0.373(2)	0.600(1)	140(0)					

Even though the M–C≡O angles in III (158–168°) are found to be smaller than those given for IV (174–179°) this does not seem to be significant with respect to the e.s.d.'s and should mainly be caused by the poor quality of the crystal. This could be valid, in addition, for the bending of the C=N–CH_3 moiety away from the $C_5(CH_3)_5$ ring C(20) $\rightarrow$ C(29) and out of the plane defined by Mo(1), Mo(2) and C(5) (0.41 and 1.33 Å for N(1) and C(6), respectively). But as on the other hand a position of C(6) coplanar with Mo(1)–C(5)–Mo(2) would lead to unacceptable short contacts (C ··· C ca. 2.50 Å, H ··· H even worse) the deviation from planarity reduces those unfavorable interactions and should be regarded as significant. The same distortion is found in the Adam's molecule but to a smaller degree (0.193 and 0.760 Å for N and C(31)), probably due to the less severe steric interactions, and seems inherent for μ,η^2-isonitrile complexes.

In summary the reaction of $[C_5Me_5(CO)_2Mo]_2$ with S=C=NCH_3 is comparable

TABLE 4

INTRAMOLECULAR DISTANCES (Å) FOR $[\eta\text{-}C_5Me_5(CO)_2Mo]_2(\mu,\eta^2\text{-}CNCH_3)$

Mo(1)–Mo(2)	3.240(3)	Mo(2)–C(3)	1.98(1)
Mo(1)–C(1)	1.97(1)	Mo(2)–C(4)	1.98(1)
Mo(1)–C(2)	2.00(1)	Mo(2)–C(5)	2.21(4)
Mo(1)–C(5)	1.93(4)	Mo(2)–N(1)	2.14(3)
Mo(1)–C(10)	2.42(2)	Mo(2)–C(20)	2.32(2)
Mo(1)–C(11)	2.36(2)	Mo(2)–C(21)	2.39(2)
Mo(1)–C(12)	2.29(2)	Mo(2)–C(22)	2.42(2)
Mo(1)–C(13)	2.31(2)	Mo(2)–C(23)	2.37(2)
Mo(1)–C(14)	2.39(2)	Mo(2)–C(24)	2.31(2)
N(1)–C(5)	1.17(3)	C(3)–O(3)	1.16(1)
N(1)–C(6)	1.53(5)	C(4)–O(4)	1.16(1)
C(1)–O(1)	1.17(1)	Mo(2)–Cen(2)	2.03(2)
C(2)–O(2)	1.18(2)		
Mo(1)–Cen(1)	2.02(2)		

Distances in idealized rigid bodies for pentamethyl-Cp's are d(CC)(ring) 1.420, d(CC)(exo) 1.500, d(CH) 1.08 Å

TABLE 5

INTRAMOLECULAR ANGLES (°) FOR $[\eta\text{-}C_5Me_5(CO)_2Mo]_2(\mu,\eta^2\text{-}CNCH_3)$

Mo(2)–Mo(1)–C(1)	108(1)	Mo(1)–Mo(2)–C(3)	116(3)
Mo(2)–Mo(1)–C(2)	80(1)	Mo(1)–Mo(2)–C(4)	77(1)
Mo(2)–Mo(1)–C(5)	42(1)	Mo(1)–Mo(2)–C(5)	36(1)
Mo(2)–Mo(1)–Cen(1)	128(1)	Mo(1)–Mo(2)–N(1)	65(1)
C(1)–Mo(1)–C(2)	71(2)	Mo(1)–Mo(2)–Cen(2)	129(1)
C(1)–Mo(1)–C(5)	90(2)	C(3)–Mo(2)–C(4)	85(2)
C(1)–Mo(1)–Cen(1)	123(2)	C(3)–Mo(2)–C(5)	106(3)
C(2)–Mo(1)–C(5)	110(2)	C(3)–Mo(2)–N(1)	84(2)
C(2)–Mo(1)–Cen(1)	122(2)	C(3)–Mo(2)–Cen(2)	114(3)
C(5)–Mo(1)–Cen(1)	125(2)	C(4)–Mo(2)–C(5)	110(1)
Mo(1)–C(1)–O(1)	167(3)	C(4)–Mo(2)–N(1)	130(1)
Mo(1)–C(2)–O(2)	158(5)	C(4)–Mo(2)–Cen(2)	119(2)
Mo(2)–C(3)–O(3)	168(7)	C(5)–Mo(2)–N(1)	31(1)
Mo(2)–C(4)–O(4)	168(3)	C(5)–Mo(2)–Cen(2)	118(2)
Mo(1)–C(5)–Mo(2)	103(1)	N(1)–Mo(2)–Cen(2)	110(2)
Mo(1)–C(5)–N(1)	158(3)		
Mo(2)–C(5)–N(1)	71(2)		
C(5)–N(1)–Mo(2)	78(2)		
C(5)–N(1)–C(6)	138(3)		
Mo(2)–N(1)–C(6)	139(2)		

to the disproportionation reactions of other heteroallenes X=C=Y [10,15]. However, the resulting X_2CY and CY fragments are in the present case transferred to molecules of completely different character (mononuclear vs. binuclear) with $C_5Me_5(CO)_2MoS_2CNHCH_3$ (II), and $[C_5Me_5(CO)_2Mo]_2(\mu,\eta^2\text{-}CNCH_3)$ (III), as the final products.

TABLE 6

COMPARISON OF BOND DISTANCES (Å) AND ANGLES (°) OF $[C_5Me_5(CO)_2Mo]_2(\mu,\eta^2\text{-}CNCH_3)$ (III) WITH $[C_5H_5(CO)_2Mo]_2(\mu,\eta^2\text{-}CNC_6H_5)$ (IV) [14]

	III	IV
Distances		
Mo–C(N)	2.21(4)	2.247(2)
	1.93(4)	1.942(2)
Mo–N(CN)	2.14(3)	2.207(2)
Mo–C(Cp)	2.29-2.42	2.30-2.40
Angles		
Mo–N–C [a]	139(2)	144.4(2)
Mo–C–N [b]	158(3)	167.7(2)
	71(2)	72.0(1)
Mo–Mo–C [c]	36(1)	36.55(6)
Mo–Mo–N [d]	65(1)	68.69(5)
C–N–C [e]	138(3)	135.2(2)

[a] Mo(2)–N(1)–C(6) (III) vs. Mo(1)–N–C(31) (IV). [b] Mo(1)–C(5)–N(1) (III) vs. Mo(2)–C(5)–N (IV) and Mo(2)–C(5)–N(1) (III) vs. Mo(1)–C(5)–N (IV), respectively. [c] Mo(1)–Mo(2)–C(5) (III) vs. Mo(2)–Mo(1)–C(5) (IV). [d] Mo(1)–Mo(2)–N(1) (III) vs. Mo(2)–Mo(1)–N (IV). [e] C(5)–N(1)–C(6) (III) vs. C(5)–N–C(31) (IV).

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