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Heteronuclear transition metal-alkyne clusters

III *. Alkyne assisted formation of molybdenum–iron bonds. Synthesis and crystal structure of [MoFe(μ-C_4Ph_4)$(CO)_4$-$(S_2CNEt_2)_2$] **

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Abstract

Reactions between [Mo$(R^1C_2R^2)_2(S_2CNR_2)_2$] ($R^1 = R^2 =$ Ph, R = Et; $R^1 = R^2 =$ Et, R = Me or Et; $R^1 =$ Me, $R^2 =$ Ph, R = Et) and [$Fe_2(CO)_9$] afford the complexes [MoFe(μ-$C_4R^1_2R^2_2$)$(CO)_4(S_2CNR_2)_2$], which have been characterised by analytical and spectroscopic methods. The molecular structure of [MoFe(μ-C_4Ph_4)$(CO)_4$-$(S_2CNEt_2)_2$] has been established by an X-ray diffraction study. The molybdenum–iron bond [2.690(2) Å] is bridged by a C_4Ph_4 moiety, formed by the coupling of two diphenylacetylene ligands, π-bound to molybdenum and σ-bound to iron. Three carbonyl ligands are bonded to the iron atom while the fourth semi-bridges the metal–metal bond. The molybdenum atom is also ligated by the dithiocarbamate groups. Spectroscopic data for the new compounds are reported and discussed.

Introduction

Recently we reported examples of tungsten-alkyne complexes acting as precursors to heteronuclear clusters [1,2]. The two synthetic routes investigated were based on the tris-alkyne tungsten complexes [W(CO)(alkyne)$_3$], in which the alkynes are formally each donating $3\frac{1}{3}$ electrons to the tungsten centre, and on the

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* For Part II see Ref. 1

** Dedicated to Gordon Stone on the occasion of his 65th birthday.

four-electron donor mono-alkyne complexes [W(CO)(alkyne)(S_2CNR_2)$_2$]. Both of these types of complex react with [$Co_2(CO)_8$] to form dicobalt–tungsten systems, the formation of which is presumably facilitated by the ability of a monodentate alkyne to coordinate to an incoming metal-ligand fragment. This paper reports the extension of this synthetic methodology to the bis-alkyne complexes [Mo(alkyne)$_2$(S_2CNR_2)$_2$] in which the alkynes are acting as three-electron donors [3].

Results and discussion

Reaction of equimolar amounts of [Mo(C_2Ph_2)$_2$(S_2CNMe_2)$_2$] and [$Co_2(CO)_8$] in CH_2Cl_2 at room temperature affords [Co_2(μ-C_2Ph_2)(CO)$_6$] as the only identifiable product in ca. 40% yield [4]. Complete transfer of an alkyne ligand from the molybdenum to the dicobalt centre appears to be the dominant process. The possibility of alkyne transfer, as opposed to metal–metal bond formation, to afford the stable molecule [Co_2(μ-C_2Ph_2)(CO)$_6$] has been noted previously [5]. Interestingly, it has been shown that the complexes [MCl($CF_3C_2CF_3$)$_2$(η-C_5H_5)] (M = Mo or W) react with [$Co_2(CO)_8$] to form the bis(μ-alkyne) complexes [MCo(μ-$CF_3C_2CF_3$)$_2$(CO)$_3$(η-C_5H_5)], with no evidence of dicobalt alkyne complex formation [6].

In contrast, reaction of [Mo(C_2Ph_2)$_2$(S_2CNMe_2)$_2$] with [$Fe_2(CO)_9$], known to be a source of Fe(CO)$_4$ fragments, affords a new compound characterised by its solution (CH_2Cl_2) infrared spectrum, ν_{max}(CO) 2040s, 1975m, 1960m and 1860w cm^{-1}. The absorption at 1860 cm^{-1} indicates that a bridging carbonyl ligand is present, but further characterisation was hampered by low solubility and good NMR data could not be obtained, although it was apparent that the phenyl and methyl groups were still present. The use of diethyldithiocarbamate and alkynes containing alkyl substituents afforded analogous complexes (**1**–**4**) of higher solubility (see Experimental) that were characterised by analytical and spectroscopic data (see Tables 1 and 2), and in the case of **1** a single-crystal X-ray diffraction study was performed.

Table 1

Analytical [a] and physical data for the molybdenum–iron complexes

Compound	Colour	Yield (%)	ν_{max}(CO) (cm^{-1})	Analysis (found(calcd.)(%))		
				C	H	N
[MoFe(μ-C_4Ph_4)-(CO)$_4$(S_2CNEt_2)$_2$] (**1**)	orange	85	2040s, 1981m, 1958m,1869w [a]	54.2 (55.0)	4.6 (4.4)	2.9 (3.1)
[MoFe(μ-C_4Et_4)-(CO)$_4$(S_2CNEt_2)$_2$] (**2**)	orange	21	2039s, 1974m 1963m,1867w [b]	42.7 (43.1)	5.5 (5.5)	3.8 (3.9)
[MoFe(μ-C_4Et_4)-(CO)$_4$(S_2CNMe_2)$_2$] (**3**)	orange	43	2040s, 1976m 1965m,1867w [b]	40.1 (39.5)	5.2 (4.8)	3.7 (4.1)
[MoFe(μ-$C_4Me_2Ph_2$)-(CO)$_4$(S_2CNEt_2)$_2$] (**4**)	orange	30	2046s, 1988m 1965m,1877w 1858w [b]	49.0 (48.5)	4.9 (4.5)	3.7 (3.5)

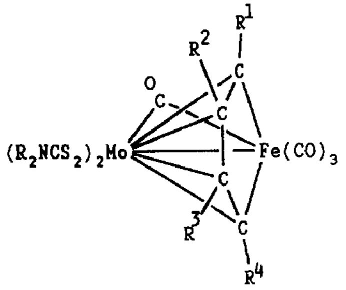
[a] In toluene. [b] In light petroleum.

Table 2

^1H and ^{13}C NMR data [a] for the molybdenum–iron complexes

Compound	$^1H(\delta)$	$^{13}C(\delta)$ [c]
1	7.6–6.6 (m, 20H, Ph), 3.9–3.2 (m, 8H, CH_2), 1.4–0.9 (m, 12H, Me)	241.8 (μ-CO), 215.3, 209.6, 208.1, 205.2, 203.6 (3×CO and 2× $S_2\mathit{C}NEt_2$), 170.8, 156.9 (2×C_α), 150–124 (m, Ph and C_β), 45.1, 44.5, 44.0, 43.9 (CH_2), 12.9, 12.8, 12.7, 12.6 (Me)
2	3.9–3.5 [m, 8H, $S_2CN(C\mathit{H}_2Me)_2$], 2.5–2.1 [m, 8H, $C_4(C\mathit{H}_2Me)_4$], 1.6×0.8 (m, 24H, CH_3)	240.0 (μ-CO), 213.4, 211.5, 208.9, 207.0, 203.7 (3×CO and 2× $S_2\mathit{C}NEt_2$), 185.6, 164.4, (2×C_α), 141.4, 130.5 (2×C_β), 44.8, 44.3, 43.8, 43.7 [$S_2CN(\mathit{C}H_2Me)_2$], 35.6, 33.6, 24.1, 21.4 [$C_4(\mathit{C}H_2Me)_4$], 19.8, 18.8, 15.5, 15.4 [$C_4(CH_2\mathit{C}H_3)_4$], 12.7, 12.6, 12.3 [$S_2CN(CH_2\mathit{C}H_3)_2$]
3	3.21 (s, 3H, S_2CNMe_2), 3.20 (s, 3H, S_2CNMe_2), 3.18 (s, 3H, S_2CNMe_2), 3.12 (s, 3H, S_2CNMe_2), 2.5–1.9 [m, 8H, $C_4(C\mathit{H}_2Me)_4$], 1.5–1.0 [m, 12H, $C_4(CH_2C\mathit{H}_3)_4$]	239.3 (μ-CO), 212.5, 211.8, 208.1, 206.5, 203.9 (3×CO and 2× S_2CNMe_2), 184.7, 164.8 (2×C_α), 140.4, 130.3 (2×C_β), 38.7, 38.3, 37.7, 37.6 ($S_2CN\mathit{Me}_2$), 34.8, 33.1, 23.2, 20.6 [$C_4(\mathit{CH}_2Me)_4$], 18.8, 17.9, 14.5, 14.4 [$C_4(CH_2\mathit{Me})_4$]
4a	[d]7.4–6.9 (m, Ph), 4.1–3.3 (m, CH_2), 2.19 (s, Me), 1.60 (s, Me), 1.4–0.8 (m, $CH_2C\mathit{H}_3$)	[d]241.4 (μ-CO), 214.6, 210.9, 208.1, 205.3, 204.4 (3×CO and 2× $S_2\mathit{C}NEt_2$), 174.8, 156.5 (2×C_αPh), 151–120 (m, Ph and C_β), 45–41 (m, CH_2), 23–18 (m, Me), 13–12 (m, $CH_2\mathit{C}H_3$)
4b	[d]7.4–6.9 (m, Ph), 4.1–3.3 (m, CH_2), 2.20 (s, Me) 1.80 (s, Me), 1.4–0.8 (m, $CH_2C\mathit{H}_3$)	[d]243.9 (μ–CO), 212.2, 209.8, 206.0, 205.5, 203.7 (3×CO and 2×$S_2\mathit{C}NEt_2$), 186.3 (C_αMe), 159.6 (C_αPh), 151–120 (m, Ph and C_β), 45–41 (m, CH_2), 23–18 (m, Me), 13–12 (m, $CH_2\mathit{C}H_3$)

[a] Chemical shifts (δ) in ppm. [b] Measured in $CDCl_3$. [c] Measured in $CDCl_3$, hydrogen-1 decoupled. [d] Mixture of isomers, see text for details.



	R	R^1	R^2	R^3	R^4
1	Et	Ph	Ph	Ph	Ph
2	Et	Et	Et	Et	Et
3	Me	Et	Et	Et	Et
(4a)	Et	Ph	Me	Me	Ph
(4b)	Et	Ph	Me	Ph	Me

The molecular structure of **1** is shown in Fig. 1 and bond lengths and angles are given in Table 3. The molecule consists of a molybdenum and an iron atom singly bonded to each other [2.690(2) Å] and bridged by a C_4Ph_4 fragment formed by the linkage of two diphenylacetylene ligands. The C_4 fragment is σ-bonded to the iron atom through C(30) and C(60), with bond lengths of 2.030(9) and 2.001(8) Å, respectively, and π-bonded to the molybdenum atom through all four carbon atoms.

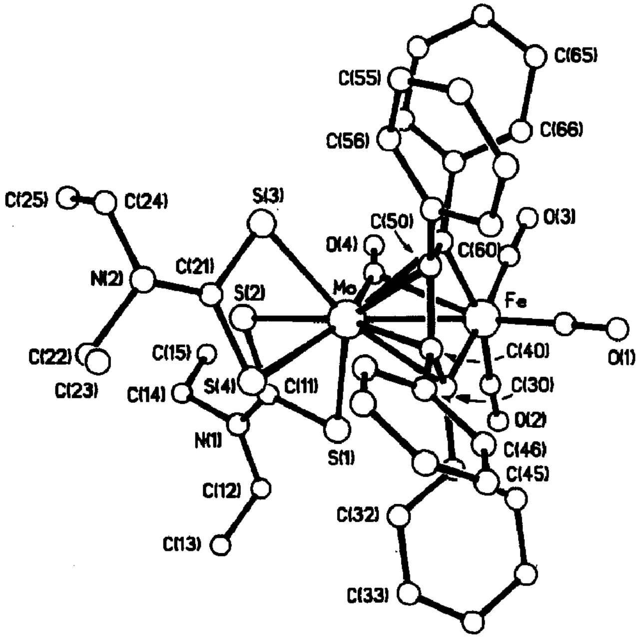


Fig. 1. Molecular structure of $[MoFe(\mu\text{-}C_4Ph_4)(CO)_4(S_2CNEt_2)_2]$ (**1**).

Table 3

Selected internuclear distances (Å) and angles (°) for [MoFe(μ-C_4Ph_4)(CO)$_4$(S_2CNEt_2)$_2$] (1), with estimated standard deviations in parentheses

Mo–Fe	2.690(2)	Mo–S(1)	2.539(3)	Mo–S(2)	2.559(2)
Mo–S(3)	2.502(3)	Mo–S(4)	2.516(3)	Mo–C(4)	2.00(1)
Mo–C(30)	2.302(8)	Mo–C(40)	2.477(8)	Mo–C(50)	2.496(8)
Mo–C(60)	2.304(8)	Fe–C(1)	1.77(1)	Fe–C(2)	1.82(1)
Fe–C(3)	1.76(1)	Fe...C(4)	2.46(1)	Fe–C(30)	2.030(9)
Fe–C(60)	2.001(8)	S(1)–C(11)	1.708(9)	S(2)–C(11)	1.70(1)
S(3)–C(21)	1.71(1)	S(4)–C(21)	1.70(1)	C(11)–N(1)	1.32(1)
N(1)–C(12)	1.50(1)	N(1)–C(14)	1.51(1)	C(21)–N(2)	1.33(1)
N(2)–C(22)	1.65(2)	N(2)–C(24)	1.48(2)	C(1)–O(1)	1.13(1)
C(2)–O(2)	1.15(1)	C(3)–O(3)	1.15(2)	C(4)–O(4)	1.17(1)
C(30)–C(40)	1.41(1)	C(40)–C(50)	1.45(1)	C(50)–C(60)	1.42(1)
S(1)–Mo–S(2)	67.2(1)	S(1)–Mo–S(3)	136.6(1)	S(2)–Mo–S(3)	73.9(1)
S(1)–Mo–S(4)	84.9(1)	S(2)–Mo–S(4)	79.0(1)	S(3)–Mo–S(4)	69.3(1)
Fe–Mo–C(4)	61.2(3)	Fe–Mo–C(30)	47.2(2)	Fe–Mo–C(40)	67.4(2)
C(30)–Mo–C(40)	34.2(3)	Fe–Mo–C(50)	67.0(2)	C(30)–Mo–C(50)	60.4(3)
C(40)–Mo–C(50)	34.0(3)	Fe–Mo–C(60)	46.5(2)	C(30)–Mo–C(60)	69.8(3)
C(40)–Mo–C(60)	60.4(3)	C(50)–Mo–C(60)	34.0(3)	Mo–Fe–C(1)	131.4(3)
Mo–Fe–C(2)	108.1(4)	C(1)–Fe–C(2)	103.2(5)	Mo–Fe–C(3)	120.4(3)
C(1)–Fe–C(3)	95.9(5)	C(2)–Fe–C(3)	89.1(5)	Mo–Fe–C(4)	45.3(2)
C(1)–Fe–C(4)	161.0(4)	C(2)–Fe–C(4)	94.6(4)	C(3)–Fe–C(4)	77.6(4)
Mo–Fe–C(30)	56.3(2)	Mo–Fe–C(60)	56.6(2)	C(30)–Fe–C(60)	81.6(3)
Mo–S(1)–C(11)	90.7(3)	Mo–S(2)–C(11)	90.2(3)	Mo–S(3)–C(21)	88.5(3)
Mo–S(4)–C(21)	88.3(3)	S(1)–C(11)–S(2)	111.7(5)	S(1)–C(11)–N(1)	123.5(7)
S(2)–C(11)–N(1)	124.7(7)	C(11)–N(1)–C(12)	122.7(8)	C(11)–N(1)–C(14)	121.3(8)
C(12)–N(1)–C(14)	115.9((8)	S(3)–C(21)–S(4)	113.5(5)	S(3)–C(21)–N(2)	120.0(8)
S(4)–C(21)–N(2)	126.5(8)	C(21)–N(2)–C(22)	118(1)	C(21)–N(2)–C(24)	125(1)
C(22)–N(2)–C(24)	117(1)	Fe–C(1)–O(1)	175(1)	Fe–C(2)–O(2)	175(1)
Fe–C(3)–O(3)	178(1)	Mo–C(4)–Fe	73.5(3)	Mo–C(4)–O(4)	165.2(8)
Fe–C(4)–O(4)	121.2(7)	Mo–C(30)–Fe	76.5(3)	Mo–C(30)–C(31)	126.8(5)
Fe–C(30)–C(31)	124.3(6)	Mo–C(30)–C(40)	79.7(5)	Fe–C(30)–C(40)	111.7(6)
C(31)–C(30)–C(40)	121.3(7)	Mo–C(40)–C(30)	66.1(4)	Mo–C(40)–C(41)	134.8(5)
C(30)–C(40)–C(41)	124.4(7)	Mo–C(40)–C(50)	73.7(4)	C(30)–C(40)–C(50)	115.2(7)
C(41)–C(40)–C(50)	120.2(7)	Mo–C(50)–C(40)	72.3(4)	Mo–C(50)–C(51)	141.7(5)
C(40)–C(50)–C(51)	123.1(7)	Mo–C(50)–C(60)	65.5(4)	C(40)–C(50)–C(60)	114.2(7)
C(51)–C(50)–C(60)	121.5(7)	Mo–C(60)–Fe	77.0(3)	Mo–C(60)–C(50)	80.4(5)
Fe–C(60)–C(50)	112.8(6)	Mo–C(60)–C(61)	133.5(5)	Fe–C(60)–C(61)	122.0(5)
C(50)–C(60)–C(61)	119.3(7)				

Carbon atoms C(30) and C(60), the α carbon atoms, are closer to the molybdenum [Mo–C(30) 2.302(8) and Mo–C(60) 2.304(8) Å] than the β carbon atoms [Mo–C(40) 2.477(8) and Mo–C(50) 2.496(8) Å]. The C–C bond lengths in the C_4 fragment are intermediate between those expected for carbon–carbon double and single bonds, and the four atoms are coplanar, implying some degree of delocalisation. The iron lies 0.49 Å out of this plane. A semi-bridging carbonyl ligand also spans the Mo–Fe bond [Mo–C(4) 1.995(11) and Fe–C(4) 2.457(10) Å]. The iron atom carries three terminal carbonyl ligands while the molybdenum retains the two dithiocarbamate ligands. Linkage of alkyne ligands at a dimetal centre to form complexes containing metallacyclopentadiene groups has been observed previously, and related examples include $[WCo\{\mu\text{-}C_4(CF_3)_4\}(CO)_4Cp]$ [7], $[MnCo(\mu\text{-}C_4Me_2Ph_2)(CO)_6]$[8] and $[MoFe(\mu\text{-}C_4Me_2Ph_2)(CO)_2Cp_2]$ [9].

The fast atom bombardment (FAB) mass spectrum of **1** from 3-nitrobenzyl alcohol displays isotopic envelopes corresponding to $[M-2CO]^+$ and $[M-4CO]^+$. The latter envelope is the most intense, and a MASPAN analysis yields a fit of $R = 4.3\%$.

The ^1H and $^{13}C\{^1H\}$ NMR spectra of **1**–**4** are consistent with the solid structure established for **1**. The ^{13}C–$\{^1H\}$ NMR spectra are most readily interpreted for **2** and **3** for which the C_β resonances are not obscured by the aromatic resonances. Thus for **3** C_α resonances occur at 185.6 and 164.4 ppm while the two C_β atoms resonate at 140.4 and 130.3 ppm. A peak at 239.3 ppm is assigned to the semi-bridging carbonyl ligand, while the five peaks observed in the region 213–203 ppm are assigned to the three terminal carbonyl and two dithiocarbamate ligands. Four distinct $S_2CN\mathit{Me}_2$ resonances indicate that there is no rotation about the C–N bonds on the NMR timescale at room temperature.

The preparation of compound **4** from $[Mo(MeC_2Ph)_2(S_2CNEt_2)_2]$ and $[Fe_2(CO)_9]$ produces an inseparable mixture of two major isomers (**4a** and **4b**) and a trace of a third minor isomer. The ratio **4a** : **4b** is approximately 3.5 : 1. Assignment of the structures of the two major isomers was made by comparison of the ^{13}C–$\{^1H\}$ NMR spectrum with those of compounds **1**–**3** (see Table 2); unfortunately the aromatic resonances obscure the C_β resonances making the assignments tentative. Compound **4a** is the isomer formed by the coupling of the CMe ends of the alkynes, while **4b** results from head to tail coupling. The depicted geometry of **4b** is based on the fact that this should involve weaker steric interactions than those in the heat to tail isomer formulated with the identities of R^1, R^2 and R^3, R^4 interchanged.

Experimental

Experiments were carried out by Schlenk-tube techniques under dry oxygen-free nitrogen. Dichloromethane was distilled over calcium hydride and light petroleum (b.p. 40–60 ° C) over sodium. Brockman activity I neutral alumina (150 mesh) was used for chromatography. Analytical and other data for the new compounds are given in Tables 1 and 2. The IR spectra were recorded with a Mattson Alpha Centauri spectrophotometer, and the NMR spectra with a JEOL GX 270 instrument. The FAB mass spectra were recorded by the SERC mass spectrometry service at Swansea using a VG ZAB-E instrument. The compounds $[Mo(alkyne)_2(S_2$-

Table 4

Atomic coordinates ($\times 10^4$), with estimated standard deviations in parentheses, for compound 1

	x	*y*	*z*
Mo	254(1)	6712(1)	7079(1)
Fe	840(1)	5938(1)	6364(1)
S(1)	−641(1)	6717(1)	6217(1)
S(2)	−72(1)	7669(1)	6728(1)
S(3)	632(1)	7330(1)	7967(1)
S(4)	−673(1)	6907(1)	7856(1)
C(11)	−602(5)	7407(4)	6170(4)
N(1)	−969(4)	7698(3)	5768(4)
C(12)	−1442(6)	7448(4)	5295(5)
C(13)	−2128(6)	7459(5)	5570(7)
C(14)	−914(6)	8308(4)	5746(5)
C(15)	−407(7)	8479(5)	5215(6)
C(21)	−161(5)	7339(4)	8254(5)
N(2)	−328(5)	7678(4)	8730(5)
C(22)	−1112(10)	7712(8)	8942(8)
C(23)	−1155(11)	7363(10)	9463(10)
C(24)	122(6)	8079(5)	9029(7)
C(25)	91(7)	8597(5)	8624(7)
C(1)	1047(5)	5260(4)	6547(5)
O(1)	1153(4)	4828(3)	6703(4)
C(2)	394(6)	5881(4)	5600(5)
O(2)	155(5)	5836(4)	5098(4)
C(3)	1566(6)	6082(4)	5924(5)
O(3)	2044(4)	6158(4)	5644(4)
C(4)	854(5)	6933(4)	6358(5)
O(4)	1166(4)	7173(3)	5981(4)
C(30)	50(4)	5802(3)	6947(4)
C(31)	−582(3)	5548(2)	6725(3)
C(32)	−1179	5702	7007
C(33)	−1761	5435	6837
C(34)	−1745	5014	6386
C(35)	−1148	4860	6105
C(36)	−566	5127	6274
C(40)	226(4)	5813(3)	7611(4)
C(41)	−173(3)	5564(2)	8145(3)
C(42)	−271	5827	8737
C(43)	−588	5559	9245
C(44)	−807	5028	9161
C(45)	−709	4765	8570
C(46)	−393	5033	8062
C(50)	873(4)	6040(3)	7750(4)
C(51)	1214(3)	5979(2)	8397(2)
C(52)	1211	5456	8653
C(53)	1569	5341	9214
C(54)	1930	5749	9520
C(55)	1932	6272	9264
C(56)	1574	6387	8702
C(60)	1217(4)	6225(3)	7193(4)
C(61)	1929(3)	6383(3)	7254(3)
C(62)	2144	6919	7234
C(63)	2814	7037	7299
C(64)	3269	6619	7385
C(65)	3053	6083	7405

Table 4 (continued)

	x	y	z
C(66)	2384	5965	7339
C(5)	6376(8)	1486(6)	8555(10)
Cl(1)	6920(4)	1566(2)	7977(3)
Cl(2)	6691(3)	1272(2)	9249(3)
Cl(3)	5772(3)	976(2)	8310(5)
C(6)	7075(11)	1134(8)	6150(11)
Cl(4)	7125(4)	1816(3)	6212(4)
Cl(5)	6802(4)	983(3)	5377(4)
Cl(6)	7827(4)	886(5)	6269(6)

$CNR_2)_2]$ were prepared by published methods [3] and $[Fe_2(CO)_9]$ was purchased from Strem.

Synthesis of the compounds $[MoFe(\mu\text{-}C_4R_4)(CO)_4(S_2CNR_2)_2]$

A solution of $[Mo(C_2Ph_2)_2(S_2CNEt_2)_2]$ (1.08 g, 1.44 mmol) and $[Fe_2(CO)_9]$ (0.79 g, 2.17 mmol) in CH_2Cl_2 (20 cm^3) was stirred for 18 h. The solvent was removed in vacuo and the residue chromatographed on an alumina column (1.5 × 15 cm), with a CH_2Cl_2–light petroleum (1 : 3) mixture as eluant. Trace amounts of unidentified orange and green compounds were eluted before the orange product. Removal of the solvent in vacuo followed by crystallisation from CH_2Cl_2–light petroleum afforded orange-red microcrystals of $[MoFe(\mu\text{-}C_4Ph_4)(CO)_4(S_2CNEt_2)_2]$ (1.12 g, 1.22 mmol). The other derivatives were prepared similarly.

Crystal structure determination of $[MoFe(\mu\text{-}C_4Ph_4)(CO)_4(S_2CNEt_2)_2]$ *(1)*

Crystals of **1** were grown from chloroform–light petroleum as orange prisms. Diffracted intensities were collected on a Nicolet $P2_1$ diffractometer at 293K, from a crystal of dimensions ca. 0.70 × 0.75 × 0.60 mm. Of the 9892 data examined [Wyckoff ω-scans, $2\theta \leqslant 50°$, data with $2\theta \geqslant 40°$ were not collected if a pre-scan showed $I \leqslant$ ca. $3\sigma(I)$], 3983 unique data had $F \geqslant 5\sigma(F)$, and only these were used for structure solution and refinement. The data were corrected for Lorentz, polarisation and X-ray absorption effects, the latter by an empirical method based upon azimuthal scan data.

Crystal data for (1)

$C_{42}H_{40}FeMoN_2O_4S_4 \cdot 2CHCl_3$, $M = 1155.6$, orthorhombic, space group *Pcab* (non-standard setting of *Pbca*, No. 61), a 20.263(8), b 24.695(8), c 20.602(6) Å, U 10309(6) Å^3, $Z = 8$, D_c 1.49 g cm^{-3}, $F(000) = 4688$, Mo-K_α X-radiation (graphite monochromator), $\bar{\lambda}$ 0.71069 Å, μ(Mo-K_α) 10.3 cm^{-1}.

The structure was solved by conventional heavy atom methods and successive difference Fourier syntheses were used to locate all non-hydrogen atoms, which were refined with anisotropic thermal parameters. Hydrogen atoms were not included in the refinement. The asymmetric unit contains two molecules of chloroform which showed substantial thermal motion in the crystal. The presence of these badly behaved solvent molecules is reflected in the comparatively poor final R values. Refinement by blocked-cascade least squares led to $R = 0.062$ ($R' = 0.064$)

with a weighting scheme of the form $w^{-1} = [\sigma^2(F) + 0.001|F|^2]$ giving a satisfactory analysis of variance. The final electron density difference synthesis showed no peaks > 0.72 or < -0.55 e Å^{-3}. Refinement was carried out on a Data General Eclipse computer with the SHELXTL system of programs [10]. Scattering factors with corrections for anomalous dispersion were taken from Ref. 11. Atom coordinates are given in Table 4. A complete list of bond distances and angles, tables of thermal parameters and lists of calculated and observed structure factors are available from the authors.

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