A Novel Compound Containing Coupled Mo_3S_4 Clusters, $[Mo_3S_4(HBpz_3)_2]_2(\mu\text{-}O)(\mu\text{-}C_3H_3N_2)_2$

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Abstract

Treatment of a methanol solution of $Mo_3S_4^{4+}$ ion with pyrazole, $C_3H_4N_2$(Hpz), and potassium tris-pyrazolylborate, $KBH(C_3H_3N_2)_3$($KHBpz_3$) with molar ratios of approximately 1:1:4 allows the isolation of the crystalline, red–brown title compound in about 80% yield. The compound has been identified and structurally characterized by X-ray methods. The crystals, which contain two molecules of interstitial THF per hexanuclear molecule, belong to space group $P2_1/c$ with the following unit cell dimensions: a = 12.890(7), b = 26.807(8), c = 24.07(1) Å, β = 96.16(4)°, V = 8269(11) Å^3, Z = 4. The title molecule has no imposed symmetry, but for practical purposes there is a two-fold symmetry axis passing through the μ-O atom and bisecting the line between the centers of the two Mo_3 clusters.

Introduction

A growing literature exists on compounds containing the $Mo_3S_4^{4+}$ core [1–4]. A major synthetic route to these is by way of the aqua ion, $[Mo_3S_4(H_2O)_9]^{4+}$, which can be treated with ligands that displace some or all of the water molecules. In this laboratory we have previously employed the 1,4,7-triazacyclononane ligand, [9]aneN$_3$, in an attempt to displace all of the water molecules from a cuboidal $Mo_4S_4^{6+}$ (aq) ion. Instead, fragmentation occurred to give the $[Mo_3S_4([9]aneN_3)_3]^{4+}$ ion which was isolated and characterized [1] in the crystalline compound $[Mo_3S_4([9]aneN_3)_3][ZnCl_4][ZnCl_3H_2O)]_2 \cdot 3H_2O$.

The work reported here began with an attempt to treat an aqueous solution of the cuboidal $Mo_4S_4^{4+}$ (aq) ion, prepared by reducing the $M_4S_4^{5+}$ with Zn amalgam in MeOH under argon with $KHBpz_3$. The objective was to replace the water molecules from it, thereby obtaining an $HBpz_3$ complex of a cuboidal Mo_4S_4 species, or, possibly, as in the [9]aneN$_3$ case, obtaining the $[Mo_3S_4(HBpz_3)_3]^+$ ion, comparable to the $[Mo_3S_4([9]aneN_3)_3]^{4+}$ ion, as a result of cluster degradation.

In practice, we have not actually achieved either result, because, unbeknownst to us, the $KHBpz_3$ that we used was contaminated with a small amount of pyrazole. This led to the isolation of the title compound, instead of the intended product, although in very low yield. However, once we realized what was actually going on, a procedure was devised in which a stoichiometric quantity of pyrazole is deliberately introduced and we begin with the $Mo_3S_4^{4+}$ aqua ion. This procedure, which is given in detail below, allows the preparation of the title compound in about 80% yield.

Experimental

Preparation

A green solution containing the $Mo_3S_4^{4+}$ aqua ion was prepared as previously described and evaporated to dryness under vacuum. To a methanol solution approximately 1.4×10^{-3} M in $Mo_3S_4^{4+}$ (concentration estimated spectroscopically), pyrazole (pz) and K[$HBpz_3$] were added in molar ratio 1:1:4 in an argon atmosphere. The color of the solution changed from green to brown over a period of 3–5 min. Stirring was continued for 3 h and the solution was then evaporated to dryness. The brown solid was partially soluble in CH_2Cl_2 and THF. A solution in THF was filtered and layered with hexane. In this way red–brown crystals, suitable for X-ray analysis, were obtained in about 80% yield. UV–Vis spectrum (THF): λ_{max} = 465 nm.

X-ray Crystallography

A red–brown crystal was sealed inside a Lindemann glass capillary with some mother liquor. Systematic absences uniquely identified the space group as $P2_1/c$ and data collection proceeded routinely, with no indication of crystal deterioration. The crystallographic characteristics and data collection parameters are listed in Table 1. The positions of the Mo and S atoms were found by the direct

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TABLE 1. Crystal Data for $[Mo_3S_4(HB(pz)_3)_2]_2(\mu\text{-}O)(\mu\text{-}pz)_2 \cdot 2THF$ (2)

Formula	$Mo_6S_8O_3N_{28}C_{50}B_4H_{106}$
Formula weight	2020.97
Space group	$P2_1/c$
Systematic absences	$(h0l)$: $l = 2n + 1$; $(0k0)$: $k = 2n + 1$
a (Å)	12.890(7)
b (Å)	26.807(8)
c (Å)	24.07(1)
β (°)	96.16(4)
V (Å^3)	8269(11)
Z	4
D_{calc} (g/cm^3)	1.62
Crystal size (mm)	0.3 × 0.3 × 0.2
μ(Mo K$_\alpha$) (cm^{-1})	11.10
Data collection instrument	Syntex P$\bar{1}$
Radiation (monochromated in incident beam)	Mo Kα (λ_α = 0.71073 Å)
Orientation reflections, no.	15
range (2θ)	16–39°
Temperature (°C)	10
Scan method	ω–2θ
Data col. range, 2θ (°)	4–45
No. unique data, total with $F_o^2 > 3\sigma(F_o^2)$	5556
	3821
No. parameters refined	572
Transmission factors, max., min.	0.999, 0.900
R[a]	0.068
R_w[b]	0.091
Quality-of-fit indicator[c]	1.66
Largest shift/e.s.d., final cycle	0.72
Largest peak (e/Å^3)	1.37

[a] $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [b] $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$; $w = 1/\sigma^2(|F_o|)$. [c] Quality-of-fit = $[\Sigma w(|F_o| - |F_c|)^2/(N_{obs} - N_{parameters})]^{1/2}$.

TABLE 2. Atomic Positional Parameters and Equivalent Isotropic Displacement Parameters (Å^2) for $[Mo_3S_4(HB(pz)_3)_2]_2(\mu\text{-}O)(\mu\text{-}pz)_2 \cdot 2THF$[a]

Atom	x	y	z	B (Å^2)
Mo(1)	0.8536(2)	0.11455(9)	0.2461(1)	3.02(6)
Mo(2)	0.8581(2)	0.06210(9)	0.3474(1)	2.98(6)
Mo(3)	0.8253(2)	0.16769(9)	0.3441(1)	2.87(6)
Mo(4)	0.4777(2)	0.29814(9)	0.2906(1)	3.25(6)
Mo(5)	0.6567(2)	0.26951(9)	0.3603(1)	3.04(6)
Mo(6)	0.6150(2)	0.37271(9)	0.3375(1)	3.29(6)
S(1)	0.9731(6)	0.0568(3)	0.2815(3)	4.1(2)
S(2)	0.9438(6)	0.1209(3)	0.4022(3)	3.7(2)
S(3)	0.7146(6)	0.1055(3)	0.3016(3)	3.2(2)
S(5)	0.4863(6)	0.2481(3)	0.3675(3)	4.0(2)
S(6)	0.6509(6)	0.3365(3)	0.4223(3)	3.8(2)
S(7)	0.9410(6)	0.1840(3)	0.2802(3)	3.6(2)
S(8)	0.4366(6)	0.3705(3)	0.3337(3)	3.7(2)
S(9)	0.6503(6)	0.3105(3)	0.2733(3)	3.7(2)
O(1)	0.715(1)	0.2137(6)	0.3244(6)	2.7(4)
N(11)	0.766(2)	0.1691(9)	0.185(1)	4.5(6)
N(12)	0.955(2)	0.1155(9)	0.177(1)	4.6(6)
N(13)	0.753(2)	0.0661(9)	0.1857(9)	4.3(6)
N(14)	0.756(2)	0.1594(9)	0.130(1)	4.6(6)*
N(15)	0.918(2)	0.1120(9)	0.124(1)	5.0(6)*
N(16)	0.750(2)	0.0692(9)	0.131(1)	5.4(6)*
N(21)	0.963(2)	0.0115(7)	0.4026(9)	3.2(5)
N(22)	0.811(2)	−0.0193(8)	0.313(1)	4.6(6)
N(23)	0.737(2)	0.0379(7)	0.4005(8)	2.7(5)
N(24)	0.927(2)	−0.0305(9)	0.424(1)	5.1(6)*
N(25)	0.797(2)	−0.0574(8)	0.3489(9)	3.8(5)*
N(26)	0.738(2)	−0.0081(9)	0.426(1)	4.8(6)*
N(31)	0.747(2)	0.1752(7)	0.4237(9)	3.4(5)
N(32)	0.892(2)	0.2378(7)	0.3820(9)	3.1(5)
N(41)	0.462(2)	0.2241(8)	0.2448(9)	3.9(6)
N(42)	0.304(2)	0.2880(8)	0.285(1)	4.9(7)
N(43)	0.446(2)	0.3195(7)	0.1990(9)	3.6(6)
N(44)	0.380(2)	0.2143(9)	0.206(1)	4.4(6)*
N(45)	0.248(2)	0.2702(9)	0.236(1)	5.3(6)*
N(46)	0.359(2)	0.2986(9)	0.164(1)	4.7(6)*
N(51)	0.830(2)	0.2773(8)	0.3873(9)	3.5(6)
N(52)	0.682(2)	0.2143(9)	0.4289(9)	4.2(6)
N(61)	0.618(2)	0.4428(8)	0.391(1)	4.2(6)
N(62)	0.569(2)	0.4381(8)	0.2700(9)	4.3(6)
N(63)	0.773(2)	0.4001(8)	0.320(1)	4.7(6)
N(64)	0.668(2)	0.4857(9)	0.377(1)	4.4(6)*
N(65)	0.629(2)	0.4801(9)	0.273(1)	4.6(6)*
N(66)	0.797(2)	0.450(1)	0.318(1)	6.1(7)*
C(1)	0.276(5)	0.350(2)	0.942(3)	17(2)*
C(2)	0.242(5)	0.399(2)	0.884(2)	15(2)*
C(3)	0.355(5)	0.384(2)	0.949(3)	16(2)*
C(4)	0.272(5)	0.379(2)	0.837(3)	15(2)*
C(5)	0.351(6)	0.393(3)	0.890(3)	20(3)*
C(6)	0.418(4)	0.359(2)	0.530(2)	14(2)*
C(7)	0.311(3)	0.359(2)	0.551(2)	8(1)*
C(8)	0.268(7)	0.338(4)	0.502(4)	28(4)*
C(9)	0.309(4)	0.292(2)	0.500(2)	13(2)*
C(10)	0.421(5)	0.303(2)	0.512(3)	16(2)*
C(11)	0.718(2)	0.212(1)	0.195(1)	4.5(7)*
C(12)	0.678(3)	0.232(1)	0.141(2)	7(1)*
C(13)	0.705(2)	0.197(1)	0.103(1)	5.4(8)*
C(14)	1.060(2)	0.119(1)	0.181(1)	4.7(7)*
C(15)	1.087(3)	0.119(1)	0.126(1)	6.9(9)*
C(16)	0.999(2)	0.115(1)	0.092(1)	5.3(8)*
C(17)	0.675(2)	0.033(1)	0.197(1)	4.6(7)*
C(18)	0.622(2)	0.018(1)	0.145(1)	5.1(8)*
C(19)	0.672(3)	0.042(1)	0.106(1)	6.4(9)*
C(21)	1.065(2)	0.016(1)	0.418(1)	5.8(8)*
C(22)	1.100(3)	−0.024(1)	0.452(1)	5.7(8)*
C(23)	1.015(3)	−0.051(1)	0.456(1)	5.8(8)*
C(24)	0.816(2)	−0.039(1)	0.258(1)	4.4(7)*
C(25)	0.798(3)	−0.092(1)	0.264(1)	5.6(8)*
C(26)	0.791(2)	−0.103(1)	0.322(1)	4.5(7)*
C(27)	0.657(3)	−0.014(1)	0.456(1)	6.6(9)*
C(28)	0.601(3)	0.030(1)	0.452(1)	5.9(8)*
C(29)	0.650(2)	0.061(1)	0.414(1)	3.3(6)*
C(41)	0.519(2)	0.180(1)	0.253(1)	4.2(7)*
C(42)	0.474(3)	0.144(1)	0.216(1)	5.9(8)*

(continued)

(continued)

TABLE 2. *(continued)*

Atom	x	y	z	B (Å^2)
C(43)	0.384(2)	0.165(1)	0.187(1)	4.9(7)*
C(44)	0.235(2)	0.297(1)	0.324(1)	4.0(7)*
C(45)	0.131(3)	0.285(1)	0.300(1)	6.4(9)*
C(46)	0.143(2)	0.268(1)	0.247(1)	5.3(8)*
C(47)	0.507(2)	0.346(1)	0.168(1)	4.4(7)*
C(48)	0.448(3)	0.343(1)	0.111(1)	6.2(9)*
C(49)	0.364(2)	0.314(1)	0.110(1)	5.2(8)*
C(52)	0.891(2)	0.312(1)	0.420(1)	5.3(8)*
C(53)	0.988(2)	0.293(1)	0.433(1)	4.1(7)*
C(54)	0.991(2)	0.246(1)	0.410(1)	4.3(7)*
C(55)	0.757(2)	0.152(1)	0.473(1)	3.8(7)*
C(56)	0.695(2)	0.174(1)	0.511(1)	4.0(7)*
C(57)	0.653(2)	0.215(1)	0.482(1)	4.0(7)*
C(61)	0.660(2)	0.519(1)	0.416(1)	5.0(8)*
C(62)	0.607(3)	0.501(1)	0.458(1)	6.0(9)*
C(63)	0.583(2)	0.452(1)	0.440(1)	3.6(6)*
C(64)	0.580(2)	0.514(1)	0.235(1)	5.0(8)*
C(65)	0.494(2)	0.492(1)	0.211(1)	4.8(7)*
C(66)	0.482(2)	0.442(1)	0.230(1)	4.2(7)*
C(67)	0.897(3)	0.454(2)	0.304(2)	9(1)*
C(68)	0.939(3)	0.407(2)	0.297(2)	8(1)*
C(69)	0.857(2)	0.373(1)	0.306(1)	5.4(8)*
C(81)	0.192(4)	0.629(2)	0.845(2)	11(1)*
C(82)	0.252(3)	0.568(2)	0.846(2)	9(1)*
C(83)	0.336(3)	0.558(1)	0.895(2)	7(1)*
C(91)	−0.069(2)	0.689(1)	0.949(1)	3.7(6)*
C(93)	0.026(3)	0.756(1)	0.895(1)	6.6(9)*
B(1)	0.805(3)	0.114(1)	0.103(2)	5.3(9)*
B(2)	0.813(3)	−0.050(1)	0.410(1)	3.7(8)*
B(4)	0.296(3)	0.255(2)	0.183(2)	6(1)*
B(6)	0.717(3)	0.491(1)	0.319(2)	4.7(9)*

[a]Starred atoms were refined isotropically. Numbers in parentheses are e.s.d.s in the least significant digits. Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as: $4/3[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab(\cos\gamma)\beta_{12} + ac(\cos\beta)\beta_{13} + bc(\cos\alpha)\beta_{23}]$.

methods program MULTAN and the rest of the molecule was located and refined by a series of alternating difference electron density maps and least-squares refinements. After all atoms in the main cluster species were established and refined, a difference map showed two THF molecules per asymmetric unit as well as some additional peaks for which no chemically sensible model has been found. The THF molecules were refined isotropically with all five atoms treated as carbon atoms. Of the remaining peaks (with up to 3e/Å^3 density) those with the highest density were refined as carbon atoms. For the main cluster species only the Mo, S and coordinated N and O atoms were refined anisotropically in order to preserve a satisfactory parameter-to-data ratio.

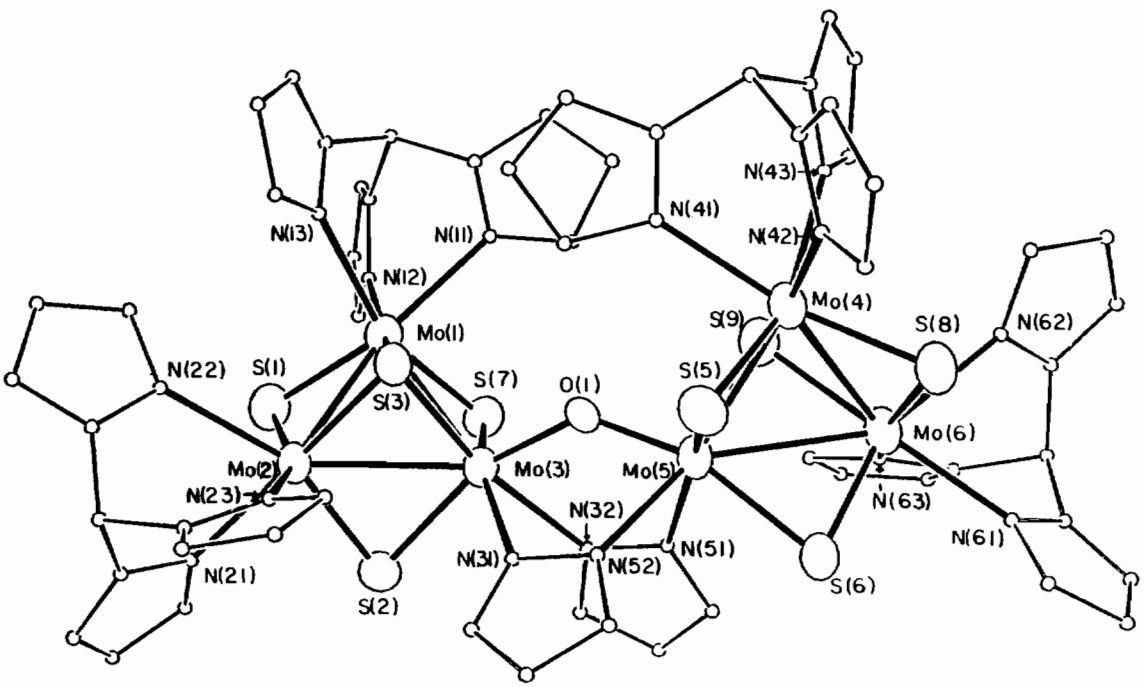

Fig. 1. ORTEP drawing of $[Mo_3S_4(HB(pz)_3)_2]_2(\mu\text{-}O)(\mu\text{-}pz)_2$.

Final atomic positional parameters and isotropic (or equivalent isotropic) displacement parameters are listed in Table 2. See also 'Supplementary Material'.

Results and Discussion

The $[Mo_3S_4(HBpz_3)_2]_2(\mu\text{-}O)(\mu\text{-}pz)_2$ molecule hereafter called, simply, the molecule) is depicted in Fig. 1. No cyrstallographic symmetry is imposed on the molecule, but it comes close to having a two-fold symmetry axis passing through the bridging oxygen atom and bisecting the Mo(3)–Mo(5) bond. The dihedral angle between the two Mo_3 planes is 116.7(1)°.

Each of the essentially equivalent halves of the molecule consists of an Mo_3S_4 cluster with one μ_3S atom and three μ_2-S atoms. On each of the two outer Mo atoms there is a tridentate $HBpz_3^-$ ligand. The two halves are then joined by the bridging oxygen atom (with an Mo–O–Mo angle of 135.2(8)°) and two bridging bidentate (μ_2, η^2) pyrazole anions. Tables 3 and 4 give lists of bond distances and angles that pertain to the Mo_3S_4 cores and their adjacent ligand atoms. The most surprising features of the structure are the Mo–Mo distances, which deviate

TABLE 3. Selected Bond Distances (Å) for $[Mo_3S_4(HB(pz)_3)_2]_2(\mu\text{-}O)(\mu\text{-}pz)_2$[a]

Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
Mo(1)	(Mo(2)	2.811(3)	Mo(4)	Mo(5)	2.811(3)
Mo(1)	Mo(3)	2.813(4)	Mo(4)	Mo(6)	2.823(3)
Mo(1)	S(1)	2.285(8)	Mo(4)	S(5)	2.278(8)
Mo(1)	S(3)	2.360(8)	Mo(4)	S(8)	2.290(8)
Mo(1)	S(7)	2.284(8)	Mo(4)	S(9)	2.331(8)
Mo(1)	N(11)	2.29(2)	Mo(4)	N(41)	2.27(2)
Mo(1)	N(12)	2.22(2)	Mo(4)	N(42)	2.25(3)
Mo(1)	N(13)	2.25(2)	Mo(4)	N(43)	2.27(2)
Mo(2)	Mo(3)	2.862(3)	Mo(5)	Mo(6)	2.860(3)
Mo(2)	S(1)	2.290(9)	Mo(5)	S(5)	2.295(8)
Mo(2)	S(2)	2.264(8)	Mo(5)	S(6)	2.342(8)
Mo(2)	S(3)	2.357(7)	Mo(5)	S(9)	2.359(8)
Mo(2)	N(21)	2.24(2)	Mo(5)	O(1)	1.92(2)
Mo(2)	N(22)	2.39(2)	Mo(5)	N(51)	2.26(2)
Mo(2)	N(23)	2.21(2)	Mo(5)	N(52)	2.22(2)
Mo(3)	S(2)	2.325(7)	Mo(6)	S(6)	2.263(8)
Mo(3)	S(3)	2.355(7)	Mo(6)	S(8)	2.292(8)
Mo(3)	S(7)	2.296(8)	Mo(6)	S(9)	2.351(8)
Mo(3)	O(1)	1.907(15)	Mo(6)	N(61)	2.28(2)
Mo(3)	N(31)	2.27(2)	Mo(6)	N(62)	2.42(2)
Mo(3)	N(32)	2.22(2)	Mo(6)	N(63)	2.24(2)

[a]Numbers in parentheses are e.s.d.s in the least signifcant digits.

TABLE 4. Selected Bond Angles (°) for $[Mo_3S_4(HB(pz)_3)_2]_2(\mu\text{-}O)(\mu\text{-}pz)_2$[a]

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
Mo(2)	Mo(1)	Mo(3)	61.18(9)	S(7)	Mo(1)	N(11)	84.3(6)
Mo(2)	Mo(1)	S(1)	52.2(2)	S(7)	Mo(1)	N(12)	87.2(6)
Mo(2)	Mo(1)	S(3)	53.4(2)	S(7)	Mo(1)	N(13)	158.4(6)
Mo(2)	Mo(1)	S(7)	97.6(2)	N(11)	Mo(1)	N(12)	78.3(8)
Mo(2)	Mo(1)	N(11)	147.6(6)	N(11)	Mo(1)	N(13)	75.1(8)
Mo(2)	Mo(1)	N(12)	134.0(6)	N(12)	Mo(2)	N(13)	82.4(8)
Mo(2)	Mo(1)	N(13)	103.2(6)	Mo(1)	Mo(2)	Mo(3)	59.46(9)
Mo(3)	Mo(1)	S(1)	100.0(2)	Mo(1)	Mo(2)	S(1)	52.0(2)
Mo(3)	Mo(1)	S(3)	53.3(2)	Mo(1)	Mo(2)	S(2)	96.9(2)
Mo(3)	Mo(1)	S(7)	52.3(2)	Mo(1)	Mo(2)	S(3)	53.5(2)
Mo(3)	Mo(1)	N(11)	96.9(6)	Mo(1)	Mo(2)	N(21)	140.5(6)
Mo(3)	Mo(1)	N(12)	139.5(6)	Mo(1)	Mo(2)	N(22)	99.9(6)
Mo(3)	Mo(1)	N(13)	135.7(6)	Mo(1)	Mo(2)	N(23)	134.1(5)
S(1)	Mo(1)	S(3)	104.0(3)	Mo(3)	Mo(2)	S(1)	98.5(2)
S(1)	Mo(1)	S(7)	97.6(3)	Mo(3)	Mo(2)	S(2)	52.4(2)
S(1)	Mo(1)	N(11)	160.0(7)	Mo(3)	Mo(2)	S(3)	52.6(2)
S(1)	Mo(1)	N(12)	81.9(6)	Mo(3)	Mo(2)	N(21)	133.8(5)
S(1)	Mo(1)	N(13)	99.7(6)	Mo(3)	Mo(2)	N(22)	149.5(5)
S(3)	Mo(1)	S(7)	104.8(3)	Mo(3)	Mo(2)	N(23)	101.2(5)
S(3)	Mo(1)	N(11)	94.7(6)	S(1)	Mo(2)	S(2)	97.8(3)
S(3)	Mo(1)	N(12)	165.5(6)	S(1)	Mo(2)	S(3)	103.9(3)
S(3)	Mo(1)	N(13)	83.5(6)	S(1)	Mo(2)	N(21)	88.9(6)
S(1)	Mo(2)	N(22)	81.8(6)	Mo(2)	Mo(3)	O(1)	138.7(5)
S(1)	Mo(2)	N(23)	158.3(5)	Mo(2)	Mo(3)	N(31)	98.2(5)
S(2)	Mo(2)	S(3)	103.8(3)	Mo(2)	Mo(3)	N(32)	140.9(5)
S(2)	Mo(2)	N(21)	81.5(5)	S(2)	Mo(3)	S(3)	102.0(3)

(continued)

TABLE 4. *(continued)*

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
S(2)	Mo(2)	N(22)	158.1(6)	S(2)	Mo(3)	S(7)	94.2(3)
S(2)	Mo(2)	N(23)	101.4(5)	S(2)	Mo(3)	O(1)	157.6(5)
S(3)	Mo(2)	N(21)	165.2(6)	S(2)	Mo(3)	N(31)	82.1(5)
S(3)	Mo(2)	N(22)	97.6(6)	S(2)	Mo(3)	N(32)	90.6(5)
S(3)	Mo(2)	N(23)	81.3(5)	S(3)	Mo(3)	S(7)	104.5(3)
N(21)	Mo(3)	N(22)	76.6(7)	S(3)	Mo(3)	O(1)	87.0(5)
N(21)	Mo(2)	N(23)	84.1(7)	S(3)	Mo(3)	N(31)	97.2(5)
N(22)	Mo(2)	N(23)	76.6(8)	S(3)	Mo(3)	N(32)	165.2(6)
Mo(1)	Mo(3)	Mo(2)	59.36(9)	S(7)	Mo(3)	O(1)	103.4(5)
Mo(1)	Mo(3)	S(2)	95.5(2)	S(7)	Mo(3)	N(31)	158.2(5)
Mo(1)	Mo(3)	S(3)	53.4(2)	S(7)	Mo(3)	N(32)	82.1(6)
Mo(1)	Mo(3)	S(7)	51.9(2)	O(1)	Mo(3)	N(31)	76.5(7)
Mo(1)	Mo(3)	O(1)	106.2(5)	O(1)	Mo(3)	N(32)	78.5(7)
Mo(1)	Mo(3)	N(31)	149.6(5)	N(31)	Mo(3)	N(32)	76.5(7)
Mo(1)	Mo(3)	N(32)	133.9(6)	Mo(5)	Mo(4)	Mo(6)	61.02(8)
Mo(2)	Mo(3)	S(2)	50.5(2)	Mo(5)	Mo(4)	S(5)	52.4(2)
Mo(2)	Mo(3)	S(3)	52.6(2)	Mo(5)	Mo(4)	S(8)	100.1(2)
Mo(2)	Mo(3)	S(7)	95.9(2)	Mo(5)	Mo(4)	S(9)	53.6(2)
Mo(5)	Mo(4)	N(41)	94.4(5)	N(41)	Mo(4)	N(43)	75.6(8)
Mo(5)	Mo(4)	N(42)	138.7(6)	N(42)	Mo(4)	N(43)	84.2(8)
Mo(5)	Mo(4)	N(43)	134.5(6)	Mo(4)	Mo(5)	Mo(6)	59.71(8)
Mo(6)	Mo(4)	S(5)	96.8(2)	Mo(4)	Mo(5)	S(5)	51.8(2)
Mo(6)	Mo(4)	S(8)	52.0(2)	Mo(4)	Mo(5)	S(6)	95.2(2)
Mo(6)	Mo(4)	S(9)	53.2(2)	Mo(4)	Mo(5)	S(9)	52.7(2)
Mo(6)	Mo(4)	N(41)	146.5(6)	Mo(4)	Mo(5)	O(1)	106.2(5)
Mo(6)	Mo(4)	N(42)	133.3(6)	Mo(4)	Mo(5)	N(51)	150.0(6)
Mo(6)	Mo(4)	N(43)	104.5(5)	Mo(4)	Mo(5)	N(52)	132.3(6)
S(5)	Mo(4)	S(8)	97.1(3)	Mo(6)	Mo(5)	S(5)	95.4(2)
S(5)	Mo(4)	S(9)	105.4(3)	Mo(6)	Mo(5)	S(6)	50.4(2)
S(5)	Mo(4)	N(41)	82.8(6)	Mo(6)	Mo(5)	S(9)	52.5(2)
S(5)	Mo(4)	N(42)	86.4(6)	Mo(6)	Mo(5)	O(1)	137.9(5)
S(5)	Mo(4)	N(43)	157.6(6)	Mo(6)	Mo(5)	N(51)	97.2(5)
S(8)	Mo(4)	S(9)	103.4(3)	Mo(6)	Mo(5)	N(52)	143.1(6)
S(8)	Mo(4)	N(41)	161.5(6)	S(5)	Mo(5)	S(6)	92.7(3)
S(8)	Mo(4)	N(42)	81.4(6)	S(5)	Mo(5)	S(9)	103.9(3)
S(8)	Mo(4)	N(43)	101.5(6)	S(5)	Mo(5)	O(1)	105.1(5)
S(9)	Mo(4)	N(41)	94.4(6)	S(5)	Mo(5)	N(51)	157.3(6)
S(9)	Mo(4)	N(42)	166.4(7)	S(5)	Mo(5)	N(52)	81.0(6)
S(9)	Mo(4)	N(43)	82.5(6)	S(6)	Mo(5)	S(9)	102.0(3)
N(41)	Mo(4)	N(42)	80.2(8)	S(6)	Mo(5)	O(1)	157.8(5)
S(6)	Mo(5)	N(51)	81.0(6)	S(6)	Mo(6)	S(9)	104.7(3)
S(6)	Mo(5)	N(52)	92.9(6)	S(6)	Mo(6)	N(61)	81.5(6)
S(9)	Mo(5)	O(1)	86.7(5)	S(6)	Mo(6)	N(62)	158.1(6)
S(9)	Mo(5)	N(51)	98.7(6)	S(6)	Mo(6)	N(63)	101.8(6)
S(9)	Mo(5)	N(52)	164.0(6)	S(8)	Mo(6)	S(9)	102.7(3)
O(1)	Mo(5)	N(51)	77.5(7)	S(8)	Mo(6)	N(61)	89.8(6)
O(1)	Mo(5)	N(52)	77.3(8)	S(8)	Mo(6)	N(62)	79.7(6)
N(51)	Mo(5)	N(52)	77.6(8)	S(8)	Mo(6)	N(63)	158.0(6)
Mo(4)	Mo(6)	Mo(5)	59.27(8)	S(9)	Mo(6)	N(61)	164.9(6)
Mo(4)	Mo(6)	S(6)	96.7(2)	S(9)	Mo(6)	N(62)	97.1(6)
Mo(4)	Mo(6)	S(8)	51.9(2)	S(9)	Mo(6)	N(63)	82.4(6)
Mo(4)	Mo(6)	S(9)	52.6(2)	N(61)	Mo(6)	N(62)	76.7(8)
Mo(4)	Mo(6)	N(61)	141.3(6)	N(61)	Mo(6)	N(63)	82.9(8)
Mo(4)	Mo(6)	N(62)	98.5(5)	N(62)	Mo(6)	N(63)	78.4(8)
Mo(4)	Mo(6)	N(63)	134.4(6)	Mo(1)	S(1)	Mo(2)	75.8(3)
Mo(5)	Mo(6)	S(6)	52.9(2)	Mo(2)	S(2)	Mo(3)	77.1(2)
Mo(5)	Mo(6)	S(8)	98.7(2)	Mo(1)	S(3)	Mo(2)	73.1(2)
Mo(5)	Mo(6)	S(9)	52.7(2)	Mo(1)	S(3)	Mo(3)	73.3(2)

(continued)

TABLE 4. *(continued)*

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
Mo(5)	Mo(6)	N(61)	134.3(6)	Mo(2)	S(3)	Mo(3)	74.8(2)
Mo(5)	Mo(6)	N(62)	149.0(5)	Mo(4)	S(5)	Mo(5)	75.8(3)
Mo(5)	Mo(6)	N(63)	101.3(6)	Mo(5)	S(6)	Mo(6)	76.8(3)
S(6)	Mo(6)	S(8)	97.6(3)	Mo(1)	S(7)	Mo(3)	75.8(3)
Mo(4)	S(8)	Mo(6)	76.1(3)	Mo(5)	S(9)	Mo(6)	74.8(2)
Mo(4)	S(9)	Mo(5)	73.6(3)	Mo(3)	O(1)	Mo(5)	135.2(8)
Mo(4)	S(9)	Mo(6)	74.2(3)				

[a]Numbers in parentheses are e.s.d.s in the least significant digits.

considerably from equality and are all longer than expected. In each half of the molecule the metal atoms form an isosceles triangle in which two Mo–Mo distances are 2.815(6) Å and the other 2.861(3) Å. The weighted average of all six is 2.830(12) Å, whereas Mo–Mo distances in $Mo_3S_4^{4+}$ derivatives are normally about 2.77 Å [1–4].

Supplementary Material

A list of anisotropic thermal parameters as well as the structure factors and more extensive tables of bond angles are available on request from author F.A.C.

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