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Oxo-1κO-μ_3-(pyridine-2-thiolato-2κN,3:4$\kappa^2 S$)-tri-μ_3-sulfido-1:2:3$\kappa^3 S$;1:2:4$\kappa^3 S$;2:3:4$\kappa^3 S$-tris(triphenylphosphine)-2κP;3κP;4κP-tricoppermolybdenum

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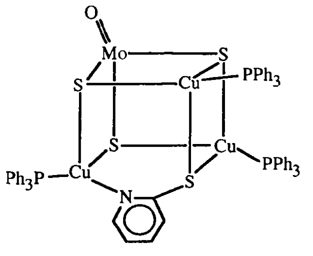
Abstract

The title compound, [MoO{μ_3-Cu($C_{18}H_{15}P$)}$_3$(μ_3-S)$_3$(μ_3-C_5H_4NS)], contains an incomplete cubane-like cluster core, [$MoCu_3S_3(C_5H_4NS)]^{2+}$, in which the S and N atoms of a pyridine-2-thiolato ligand bridge three Cu atoms.

Comment

Recently, some incomplete cubane-like heterometallic clusters belonging to the *M*/Cu/S (*M* = Mo or W) system have been synthesized in our laboratory. For example, clusters containing the cores [$M_2CuS_4]^{3+}$ (Zhu, Zheng & Wu, 1990), [$MoCu_3S_3(S_2COEt)]^{2+}$ (Zhu, Du, Chen & Wu, 1992) and [WCu_3S_3{$S_2P(COEt)_2$}]$^{2+}$ (Du & Wu, 1994) have been prepared. The structure of the title compound, (I), is similar to that of [$MoCu_3S_3(S_2COEt)](O)(PPh_3)_3$, except that S_2COEt^- is replaced by a bidentate pyridine-2-thiolato ligand.



(I)

As shown in Fig. 1, the Mo atom in (I) has tetrahedral coordination, $MoOS_3^{2-}$; furthermore, each Cu atom is coordinated by a distorted tetrahedron of two S atoms of tetradentate $MoOS_3^{2-}$, one P atom of PPh_3 and one S (or N) atom of the pyridine-2-thiolato ligand. The average Mo···Cu, μ_3-S—Mo and μ_3-S—Cu distances of 2.738 (2), 2.255 (3) and 2.310 (3) Å, respectively, are comparable with the corresponding values of 2.735 (1), 2.255 (2) and 2.302 (2) Å found in [$MoCu_3S_3(S_2COEt)](O)(PPh_3)_3$. The mean Cu—S bond length (of $C_5H_4NS^-$) is 2.458(3) Å and the Cu—N bond length is 2.103 (9) Å.

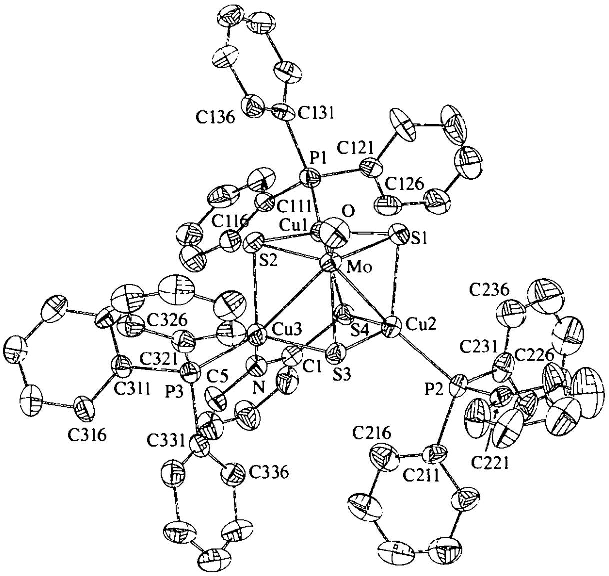


Fig. 1. The molecular structure of (I) showing 40% probability displacement ellipsoids. For clarity, H atoms have been omitted. The phenyl C atoms are numbered sequentially around each ring.

Experimental

The title compound was synthesized by reaction of [$MoCu_2S_3](O)(PPh_3)_3$ with C_5H_4NSCu in CH_2Cl_2. Black crystals were obtained by evaporating the filtrate for several days after addition of 2-propanol.

Crystal data

[$Cu_3Mo(O)S_3(C_5H_4NS)$-
($C_{18}H_{15}P)_3$]
M_r = 1295.79
Triclinic
$P\bar{1}$
a = 10.131 (3) Å
b = 12.711 (5) Å
c = 23.594 (7) Å
α = 87.27 (3)°
β = 101.59 (3)°
γ = 109.05 (3)°
V = 2813 (2) Å^3
Z = 2
D_x = 1.53 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 19 reflections
θ = 9–11°
μ = 1.60 mm^{-1}
T = 293 K
Rectangular
0.22 × 0.20 × 0.12 mm
Black

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (Fair, 1990)
$T_{\min} = 0.716$, $T_{\max} = 0.825$
9841 measured reflections
9841 independent reflections
4834 reflections with $I > 3\sigma(I)$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = -28 \rightarrow 28$
3 standard reflections every 300 reflections intensity decay: 2.3%

Refinement

Refinement on F
$R = 0.054$
$wR = 0.060$
$S = 1.16$
4834 reflections
649 parameters
H atoms not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.020$
$\Delta\rho_{\max} = 0.60$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Mo—Cu1	2.709 (2)	Cu1—P1	2.219 (3)
Mo—Cu2	2.729 (2)	Cu2—S1	2.348 (3)
Mo—Cu3	2.777 (2)	Cu2—S3	2.278 (3)
Mo—S1	2.255 (3)	Cu2—S4	2.457 (3)
Mo—S2	2.257 (3)	Cu2—P2	2.220 (3)
Mo—S3	2.252 (3)	Cu3—S2	2.297 (3)
Mo—O	1.716 (9)	Cu3—S3	2.301 (3)
Cu1—S1	2.343 (4)	Cu3—P3	2.305 (3)
Cu1—S2	2.293 (3)	Cu3—N	2.103 (9)
Cu1—S4	2.459 (3)	S4—C1	1.741 (9)
S1—Mo—S2	108.15 (9)	S2—Cu3—P3	112.7 (2)
S1—Mo—S3	107.2 (1)	S2—Cu3—N	111.5 (2)
S1—Mo—O	110.3 (2)	S3—Cu3—P3	104.2 (1)
S2—Mo—S3	106.3 (1)	S3—Cu3—N	112.3 (2)
S2—Mo—O	112.4 (3)	P3—Cu3—N	112.2 (2)
S3—Mo—O	112.2 (2)	Mo—S1—Cu1	72.16 (8)
S1—Cu1—S2	104.0 (2)	Mo—S1—Cu2	72.68 (7)
S1—Cu1—S4	92.0 (2)	Cu1—S1—Cu2	83.5 (2)
S1—Cu1—P1	123.0 (1)	Mo—S2—Cu1	73.08 (8)
S2—Cu1—S4	112.87 (9)	Cu1—S2—Cu3	96.5 (2)
S2—Cu1—P1	119.2 (2)	Mo—S3—Cu2	74.08 (7)
S4—Cu1—P1	102.5 (2)	Mo—S3—Cu3	75.16 (9)
S1—Cu2—S3	103.3 (2)	Cu2—S3—Cu3	97.1 (2)
S1—Cu2—S4	91.9 (1)	Cu1—S4—Cu2	78.9 (1)
S1—Cu2—P2	126.7 (2)	Cu1—S4—C1	112.5 (3)
S3—Cu2—S4	112.8 (1)	Cu2—S4—C1	113.6 (4)
S3—Cu2—P2	110.4 (1)	Cu3—N—C1	126.5 (6)
S4—Cu2—P2	110.5 (1)	Cu3—N—C5	115.0 (6)
S2—Cu3—S3	103.4 (2)		

The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for non-H atoms. H atoms were placed in calculated positions and not refined. Structure solution and refinement were carried out on a COMPAQ PROLINEA 4/50 computer using the *MolEN* (Fair, 1990) program package. Other programs used included *ORTEP*II (Johnson, 1976) for the molecular graphics.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1304). Services for accessing these data are described at the back of the journal.

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trans-Tetracarbonylbis[tris(4-chlorophenyl)-phosphine-*P*]molybdenum(0)

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Abstract

As a result of its *trans* geometry, the title compound, $[Mo\{(C_6H_4Cl)_3P\}_2(CO)_4]$, has short Mo—P bonds [mean value 2.483 (1) Å] in spite of the presence of bulky chloro-substituted phenyl rings. The Mo atom displays nearly perfect octahedral coordination. Average Mo—C and C—O distances are 2.021 (5) and 1.141 (6) Å, respectively.

Comment

The unit cell of the title compound, (I), contains two independent molecules with their Mo atoms lying on inversion centres (Fig. 1). Each Mo atom displays octahedral coordination. The two chloro-substituted phosphine ligands are *trans* with respect to each other, with an average Mo—P distance of 2.483 (1) Å, which is shorter than comparable values in $[Mo(CO)_5PPh_3]$ [2.560 (1) Å; Cotton, Darensbourg & Ilsley, 1981], $[Mo(CO)_5\{P(4-$

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