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A Linear Cluster with Mixed Ligands, $[WCu_2S_4(tpt)_2(PPh_3)_2]$

QUANMING WANG, XINTAO WU, QUN HUANG AND TIANLU SHENG

State Key Laboratory of Structural Chemistry, Fujian Institute of Research on Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, People's Republic of China. E-mail: wxt@ms.fjirsm.ac.cn

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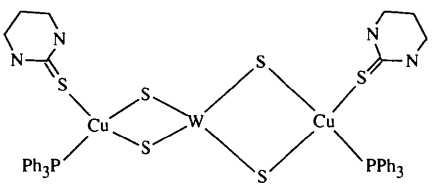
Abstract

The structure determination of tetra-μ-sulfido-1:2κ^4S;-1:3κ^4S-bis(tetrahydropyrimidine-2-thione)-2κS,3κS-bis-(triphenylphosphine)-2κP,3κP-dicoppertungsten, $[Cu_2WS_4(C_4H_8N_2S)_2(C_{18}H_{15}P)_2]$, is reported. The compound contains a linear cluster core $[CuS_2WS_2Cu]$. Each Cu atom has a distorted tetrahedral coordination, from two S atoms of a tetradentate WS_4^{2-} moiety, one S atom of tetrahydropyrimidine-2-thione (tpt) and one P atom of PPh_3.

Comment

Several linear clusters $MS_4M'_2(PPh_3)_3.0.8CH_2Cl_2$ (M = Mo, W; M' = Cu, Ag) have been prepared over the last two decades (Müller, Bögge & Schimanski, 1983). Additionally, the linear heterometallic trinuclear clusters $[Et_4N][(PPh_3)_2AgS_2MS_2Cu(CN)]$ (M = Mo, W) have been synthesized in recent years (Du, Zhu, Chen, Wu & Lu, 1992*a*,*b*). The title compound, (I), also has a linear core $[CuS_2W_2Cu]$, but in which both Cu atoms are tetrahedrally coordinated by mixed ligands.



(I)

As shown in Fig. 1, the W atom has tetrahedral coordination, WS_4^{2-}. Furthermore, each Cu atom is coordinated by a distorted tetrahedron of two S atoms of the tetradentate WS_4^{2-} moiety, one S atom of tpt and one P atom of PPh_3. The average W—Cu, W—μ-S and Cu—μ-S distances of 2.7525 (8), 2.201 (2) and 2.321 (2) Å, respectively, are comparable with the corresponding values of 2.740 (3), 2.214 (8) and 2.284 (8) Å found in $(PPh_3)_3WS_4Cu_2.0.8CH_2Cl_2$. The Cu—$S_{tpt}$ bond length of 2.363 (3) Å is longer than that of 2.206 (2) Å in $[Cu(tpt)_2Cl]$ (Bret, Castan & Jugie, 1983).

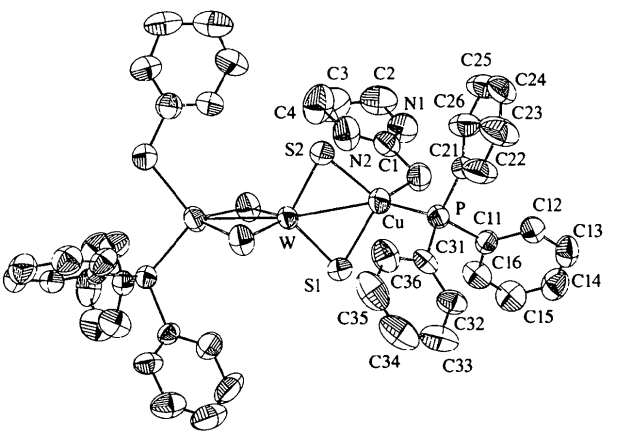


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

The title compound was synthesized by reaction of $(PPh_3)_3WS_4Cu_2.0.8CH_2Cl_2$ with excess tpt in CH_2Cl_2. Red–orange air-stable crystals were crystallized by allowing the filtrate to stand for several days after addition of 2-propanol.

Crystal data

$[Cu_2WS_4(C_4H_8N_2S)_2$-$(C_{18}H_{15}P)_2]$
M_r = 1196.15
Monoclinic
$C2/c$
a = 17.427 (2) Å
b = 10.021 (5) Å
c = 28.639 (3) Å
β = 106.71 (1)°
V = 4790 (1) Å^3
Z = 4
D_x = 1.66 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 20 reflections
θ = 6.0–27.5°
μ = 3.68 mm^{-1}
T = 293 K
Block
0.40 × 0.40 × 0.20 mm
Red–orange

Data collection

Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (Fair, 1990)
T_{min} = 0.196, T_{max} = 0.479
8944 measured reflections
4477 independent reflections

2511 reflections with $I > 3\sigma(I)$
R_{int} = 0.039
θ_{max} = 25°
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 12$
$l = -33 \rightarrow 33$
3 standard reflections
every 300 reflections
intensity decay: none

Refinement

Refinement on F
R = 0.037
wR = 0.037
S = 1.04
2511 reflections
267 parameters
H atoms not refined
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.08 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U^{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
W	1/2	0.05247 (5)	1/4	3.565 (9)
Cu	0.37341 (6)	0.0787 (1)	0.16695 (3)	4.51 (2)
S	0.2629 (1)	0.2243 (3)	0.15577 (8)	5.50 (6)
S1	0.3919 (1)	−0.0711 (2)	0.23179 (7)	5.07 (5)
S2	0.4978 (1)	0.1784 (2)	0.18617 (7)	4.94 (5)
P	0.3360 (1)	−0.0457 (2)	0.09829 (7)	3.74 (4)
N1	0.2350 (4)	0.4622 (8)	0.1859 (3)	6.6 (2)
N2	0.3615 (5)	0.3916 (8)	0.2147 (3)	7.1 (2)
C1	0.2885 (5)	0.3682 (9)	0.1883 (3)	5.0 (2)
C2	0.2506 (6)	0.5901 (9)	0.2123 (3)	7.7 (3)
C3	0.3240 (6)	0.581 (1)	0.2536 (4)	9.0 (3)
C4	0.3879 (6)	0.516 (1)	0.2422 (4)	9.1 (3)
C11	0.2290 (4)	−0.0808 (7)	0.0806 (3)	3.9 (2)
C12	0.1789 (4)	−0.0749 (8)	0.0324 (3)	4.4 (2)
C13	0.0986 (5)	−0.1065 (9)	0.0219 (3)	5.5 (2)
C14	0.0678 (5)	−0.145 (1)	0.0588 (3)	6.3 (3)
C15	0.1150 (5)	−0.150 (1)	0.1059 (3)	6.1 (2)
C16	0.1941 (4)	−0.1163 (9)	0.1166 (3)	5.1 (2)
C21	0.3551 (4)	0.0222 (8)	0.0442 (2)	3.8 (2)
C22	0.3765 (6)	−0.055 (1)	0.0106 (3)	6.5 (2)
C23	0.3900 (6)	0.004 (1)	−0.0306 (3)	8.2 (3)
C24	0.3813 (5)	0.137 (1)	−0.0378 (3)	6.9 (3)
C25	0.3610 (5)	0.2152 (9)	−0.0048 (3)	7.0 (3)
C26	0.3484 (5)	0.1585 (9)	0.0365 (3)	5.8 (2)
C31	0.3783 (4)	−0.2138 (7)	0.1048 (2)	3.7 (2)
C32	0.3320 (5)	−0.3278 (8)	0.0946 (3)	4.8 (2)
C33	0.3657 (5)	−0.4534 (9)	0.1033 (3)	6.4 (2)
C34	0.4479 (5)	−0.464 (1)	0.1211 (3)	7.5 (3)
C35	0.4963 (5)	−0.353 (1)	0.1300 (3)	7.4 (3)
C36	0.4611 (5)	−0.226 (1)	0.1228 (3)	6.0 (2)

Table 2. *Selected geometric parameters (Å, °)*

W—Cu	2.7525 (8)	Cu—S1	2.337 (2)
W—S1	2.188 (2)	Cu—S2	2.305 (2)
W—S2	2.213 (2)	Cu—P	2.261 (2)
Cu—S	2.363 (3)	S—C1	1.705 (9)
Cu—W—Cu^i	169.03 (4)	W—Cu—S2	50.95 (5)
Cu—W—S1	55.03 (6)	W—Cu—P	131.15 (7)
Cu—W—S1^i	132.94 (6)	S—Cu—S1	115.2 (1)
Cu—W—S2	54.00 (6)	S—Cu—S2	115.99 (9)
Cu—W—S2^i	118.61 (6)	S—Cu—P	101.70 (8)
S1—W—S1^i	111.08 (9)	S1—Cu—S2	100.56 (8)
S1—W—S2	108.44 (7)	S1—Cu—P	106.03 (9)
S1—W—S2^i	109.22 (9)	S2—Cu—P	117.59 (9)
S2—W—S2^i	110.44 (9)	Cu—S—C1	111.9 (3)
W—Cu—S	126.55 (7)	W—S1—Cu	74.85 (8)
W—Cu—S1	50.12 (5)	W—S2—Cu	75.05 (8)

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

The title structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for non-H atoms. H atoms were located 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.

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