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μ_3-(*O,O'*-Diethyl dithiophosphato-2κS,-3:4$\kappa^2 S'$)-di-μ_3-sulfido-1:2:3$\kappa^3 S$;1:2:4$\kappa^3 S$-μ-sulfido-1:3$\kappa^2 S$-sulfido-1κS-tris(triphenylphosphine)-2κP;3κP;4κP-molybdenumtrisilver

SHAOWU DU AND XINTAO WU*

State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Science, Fuzhou, Fujian 350002, People's Republic of China

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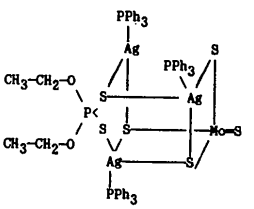
Abstract

The structure of the title compound, $[MoAg_3S_4(C_4H_{10}O_2PS_2)(C_{18}H_{15}P)_3]$, contains an incomplete cubane-like cluster core, $[MoAg_3(C_4H_{10}O_2PS_2)S_3]^{2+}$, in which a diethyl dithiophosphate group acts as a triply bridging ligand to coordinate three Ag atoms with bond lengths of 2.626 (4) [Ag(1)—S(6)], 2.690 (4) [Ag(2)—S(5)] and 2.698 (4) Å [Ag(3)—S(5)].

Comment

Several complexes of the *M*—Ag—S series (*M* = Mo, W) have been found in recent years (Gheller *et al.*, 1984; Müller & Menge, 1972; Müller, Bögge, Königer-Ahlborn & Hellman, 1979) but research into the incomplete cubane-like structures in such a series is just beginning (Nianyong, Yifan & Xintao, 1990). The title compound (I) $[MoAg_3(C_4H_{10}O_2PS_2)(C_{18}H_{15}P)_3S_4]$ is an incomplete cubane-like cluster in which one Ag—S distance is too long for effective bonding [Ag(3)···S(3) 2.971 Å]. The structure of the title compound is similar to that of other cubane-like clusters $[MoAg_3S_3Cl](X)(Ph_3P)_3$ (X = O or S) (Nianyong, Jianhui, Shaowu & Xintao, 1992; Jianhui, Nianyong, Shaowu & Xintao, 1992) except that the Cl^- anion is replaced by a bidentate diethyl dithiophosphate ligand.



(I)

The mean interatomic distances are Mo—Ag 3.046 (2), Ag—(μ_3-S) 2.577 (4), Mo—(μ-S) 2.233 (4) and Mo=S 2.131 (4) Å. The Mo atom is tetrahedrally coordinated by four S atoms and the Ph_3P ligands complete the tetrahedral geometry at each Ag atom.

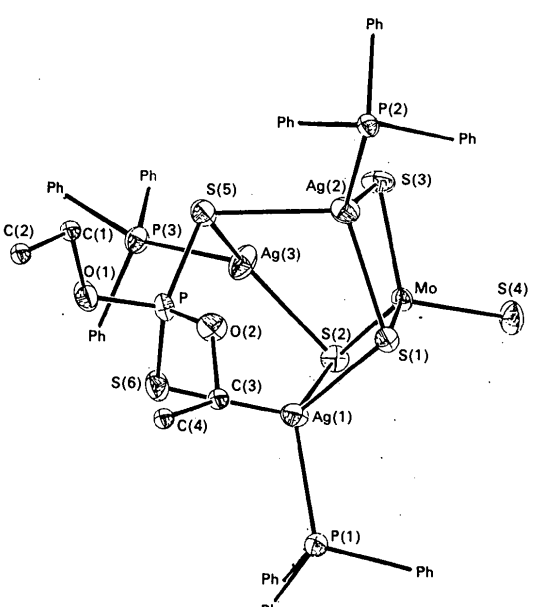


Fig. 1. View of the title compound. Displacement ellipsoids are shown at the 30% probability level.

Experimental

Crystal data

$[MoAg_3S_4(C_4H_{10}O_2PS_2)(C_{18}H_{15}P)_3]$
M_r = 1519.87
Triclinic
$P\bar{1}$
a = 13.576 (2) Å
b = 20.603 (3) Å
c = 11.675 (2) Å
α = 101.03 (1)°
β = 107.20 (1)°
γ = 77.25 (1)°
V = 3014.6 (9) Å^3
Z = 2
D_x = 1.67 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 20 reflections
θ = 15-27.5°
μ = 1.494 mm^{-1}
T = 296 K
Cubic
0.55 × 0.50 × 0.20 mm
Dark red
Crystal source: from 2-propanol, dichloromethane and ethanol

Data collection

Rigaku AFC-5R diffractometer
ω-2θ scans
Absorption correction: empirical
T_{min} = 0.71, T_{max} = 1.13
11 094 measured reflections
10 603 independent reflections
5691 observed reflections [$I > 5\sigma(I)$]

R_{int} = 0.032
θ_{max} = 25°
$h = 0 \rightarrow 16$
$k = -25 \rightarrow 25$
$l = -14 \rightarrow 14$
3 standard reflections monitored every 250 reflections
intensity variation: none

Refinement

Refinement on F
R = 0.054
wR = 0.087
S = 2.17
5691 reflections
377 parameters
H-atom parameters not refined

$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{max}$ = 0.34
$\Delta\rho_{max}$ = 1.06 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 e Å^{-3}
Extinction correction: none
Atomic scattering factors from Cromer & Waber (1974)

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

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Mo	0.81383 (9)	0.21819 (6)	0.77215 (10)	2.24 (5)
Ag(1)	0.69957 (10)	0.13586 (6)	0.86264 (10)	3.35 (5)
Ag(2)	0.87526 (10)	0.28773 (6)	1.02438 (10)	3.46 (5)
Ag(3)	0.60769 (10)	0.29711 (7)	0.83435 (12)	4.48 (6)
S(1)	0.8855 (3)	0.1653 (2)	0.9365 (3)	2.7 (1)
S(2)	0.6536 (3)	0.1975 (2)	0.6752 (3)	3.2 (2)
S(3)	0.8042 (4)	0.3290 (2)	0.8136 (3)	3.9 (2)
S(4)	0.9113 (4)	0.1796 (2)	0.6524 (4)	4.4 (2)
S(5)	0.6884 (3)	0.3258 (2)	1.0751 (3)	3.4 (2)
S(6)	0.5620 (3)	0.1927 (2)	0.9856 (4)	3.7 (2)
P	0.6515 (3)	0.2465 (2)	1.1173 (3)	2.8 (2)
P(1)	0.7062 (3)	0.0138 (2)	0.8216 (3)	2.8 (2)
P(2)	1.0056 (3)	0.3388 (2)	1.1850 (3)	2.5 (1)
P(3)	0.4260 (3)	0.3570 (2)	0.7995 (3)	3.0 (2)
O(1)	0.5903 (8)	0.2706 (5)	1.2183 (9)	3.8 (5)
O(2)	0.7593 (8)	0.2048 (5)	1.1791 (9)	3.8 (5)
C(1)	0.6277 (15)	0.3224 (9)	1.3201 (16)	5.2 (4)
C(2)	0.6150 (18)	0.3039 (11)	1.4307 (20)	7.2 (5)
C(3)	0.7741 (14)	0.1354 (9)	1.1895 (16)	4.9 (4)
C(4)	0.7509 (25)	0.1221 (16)	1.2940 (28)	11.0 (8)
C(111)	0.5865 (11)	−0.0122 (7)	0.7251 (11)	2.7 (3)
C(112)	0.5777 (15)	−0.0777 (9)	0.6878 (16)	4.9 (4)
C(113)	0.4873 (16)	−0.0990 (10)	0.6116 (17)	5.5 (4)
C(114)	0.4042 (13)	−0.0512 (8)	0.5714 (14)	4.3 (3)
C(115)	0.4084 (14)	0.0153 (8)	0.6037 (15)	4.4 (3)
C(116)	0.5008 (12)	0.0352 (8)	0.6808 (13)	3.7 (3)
C(121)	0.8048 (12)	−0.0305 (7)	0.7452 (12)	3.2 (3)
C(122)	0.8377 (14)	0.0054 (9)	0.6788 (15)	4.5 (4)
C(123)	0.9100 (16)	−0.0281 (10)	0.6108 (18)	6.2 (5)
C(124)	0.9469 (16)	−0.0967 (10)	0.6143 (18)	6.0 (4)
C(125)	0.9169 (17)	−0.1300 (11)	0.6793 (19)	6.4 (5)
C(126)	0.8463 (15)	−0.0998 (9)	0.7480 (16)	5.1 (4)
C(131)	0.7362 (12)	−0.0252 (7)	0.9583 (13)	3.4 (3)
C(132)	0.6552 (15)	−0.0394 (9)	0.9955 (16)	5.4 (4)
C(133)	0.6786 (22)	−0.0625 (13)	1.1068 (23)	8.5 (6)
C(134)	0.7813 (21)	−0.0775 (12)	1.1713 (21)	7.5 (5)
C(135)	0.8586 (19)	−0.0666 (11)	1.1378 (20)	7.0 (5)
C(136)	0.8377 (16)	−0.0396 (10)	1.0268 (18)	5.8 (4)
C(211)	1.0053 (11)	0.4234 (7)	1.1689 (11)	2.7 (2)
C(212)	1.0932 (12)	0.4543 (7)	1.1972 (12)	3.3 (3)
C(213)	1.0861 (12)	0.5187 (7)	1.1819 (13)	3.3 (3)
C(214)	0.9891 (11)	0.5578 (7)	1.1346 (12)	3.1 (3)
C(215)	0.9019 (13)	0.5292 (8)	1.1050 (14)	4.0 (3)
C(216)	0.9085 (11)	0.4634 (7)	1.1217 (12)	2.9 (3)
C(221)	0.9883 (10)	0.3459 (6)	1.3351 (11)	2.2 (2)
C(222)	0.9317 (11)	0.3023 (7)	1.3536 (12)	3.0 (3)
C(223)	0.9170 (13)	0.3056 (8)	1.4675 (14)	4.1 (3)
C(224)	0.9543 (14)	0.3518 (8)	1.5617 (15)	4.4 (3)
C(225)	1.0112 (13)	0.3953 (8)	1.5442 (14)	3.9 (3)
C(226)	1.0254 (12)	0.3940 (7)	1.4317 (13)	3.4 (3)
C(231)	1.1397 (11)	0.2969 (7)	1.1951 (12)	2.9 (3)
C(232)	1.1652 (14)	0.2655 (8)	1.0881 (14)	4.3 (3)
C(233)	1.2668 (15)	0.2355 (9)	1.0850 (16)	5.3 (4)
C(234)	1.3439 (15)	0.2357 (9)	1.1917 (17)	5.3 (4)
C(235)	1.3217 (15)	0.2644 (9)	1.2971 (16)	5.1 (4)
C(236)	1.2198 (13)	0.2943 (8)	1.2994 (14)	4.1 (3)
C(311)	0.3329 (12)	0.2977 (7)	0.7414 (12)	3.1 (3)
C(312)	0.2469 (14)	0.3013 (9)	0.7818 (15)	4.8 (4)
C(313)	0.1749 (15)	0.2576 (9)	0.7267 (16)	5.4 (4)
C(314)	0.1972 (16)	0.2093 (10)	0.6330 (17)	5.7 (4)
C(315)	0.2830 (16)	0.2044 (10)	0.5919 (17)	5.6 (4)
C(316)	0.3509 (16)	0.2487 (10)	0.6476 (17)	5.7 (4)
C(321)	0.3796 (12)	0.4148 (7)	0.6870 (12)	3.2 (3)
C(322)	0.2838 (15)	0.4530 (9)	0.6690 (16)	4.9 (4)
C(323)	0.2488 (15)	0.4975 (9)	0.5804 (16)	5.1 (4)
C(324)	0.3097 (18)	0.4953 (11)	0.5066 (19)	6.5 (5)
C(325)	0.4048 (19)	0.4546 (12)	0.5185 (20)	7.4 (5)
C(326)	0.4406 (16)	0.4152 (10)	0.6110 (18)	5.8 (4)
C(331)	0.3964 (12)	0.4031 (7)	0.9362 (12)	3.2 (3)
C(332)	0.3851 (12)	0.4740 (7)	0.9589 (12)	3.3 (3)
C(333)	0.3696 (13)	0.5079 (8)	1.0686 (14)	4.1 (3)
C(334)	0.3658 (13)	0.4740 (8)	1.1559 (14)	3.9 (3)
C(335)	0.3784 (13)	0.4056 (8)	1.1354 (14)	4.1 (3)
C(336)	0.3934 (13)	0.3715 (8)	1.0267 (14)	3.8 (3)

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

Mo—Ag(1)	3.036 (2)	S(5)—P	1.995 (5)
Mo—Ag(2)	2.980 (2)	S(6)—P	1.977 (5)
Mo—Ag(3)	3.122 (2)	P—O(1)	1.58 (1)
Mo—S(1)	2.246 (4)	P—O(2)	1.57 (1)
Mo—S(2)	2.228 (4)	O(1)—C(1)	1.48 (2)
Mo—S(3)	2.225 (4)	O(2)—C(3)	1.42 (2)
Mo—S(4)	2.131 (4)	C(1)—C(2)	1.48 (3)
Ag(1)—S(1)	2.583 (4)	C(3)—C(4)	1.43 (3)
Ag(1)—S(2)	2.588 (4)	P(1)—C(111)	1.81 (1)
Ag(1)—S(6)	2.626 (4)	P(1)—C(121)	1.81 (1)
Ag(2)—S(1)	2.525 (4)	P(1)—C(131)	1.82 (2)
Ag(2)—S(3)	2.598 (4)	P(2)—C(211)	1.79 (1)
Ag(2)—S(5)	2.690 (4)	P(2)—C(221)	1.81 (1)

Ag(3)—S(2)	2.591 (4)	P(2)—C(231)	1.82 (1)
Ag(3)—S(5)	2.698 (4)	P(3)—C(331)	1.80 (1)
Ag(1)—P(1)	2.454 (4)	P(3)—C(321)	1.82 (1)
Ag(2)—P(2)	2.420 (4)	P(3)—C(311)	1.85 (1)
Ag(3)—P(3)	2.454 (4)		
Ag(1)—Mo—Ag(3)	65.33 (4)	C(211)—P(2)—C(231)	104.3 (6)
Ag(2)—Mo—Ag(1)	87.84 (4)	C(221)—P(2)—C(231)	105.8 (6)
Ag(2)—Mo—Ag(3)	73.18 (4)	C(331)—P(3)—C(321)	106.5 (7)
S(2)—Mo—S(1)	112.5 (1)	C(331)—P(3)—C(311)	105.3 (7)
S(3)—Mo—S(1)	113.2 (1)	C(321)—P(3)—C(311)	102.0 (7)
S(3)—Mo—S(2)	107.8 (2)	Mo—S(2)—Ag(1)	77.8 (1)
S(4)—Mo—S(1)	106.6 (2)	Mo—S(2)—Ag(3)	80.4 (1)
S(4)—Mo—S(2)	107.7 (2)	Mo—S(3)—Ag(2)	75.9 (1)
S(4)—Mo—S(3)	108.9 (2)	Ag(1)—S(2)—Ag(3)	79.9 (1)
P(1)—Ag(1)—S(1)	111.2 (1)	Ag(2)—S(1)—Ag(1)	109.6 (1)
P(1)—Ag(1)—S(2)	116.1 (1)	Ag(2)—S(5)—Ag(3)	85.0 (1)
P(1)—Ag(1)—S(6)	110.4 (1)	P—S(5)—Ag(2)	108.2 (2)
S(1)—Ag(1)—S(2)	92.0 (1)	P—S(5)—Ag(3)	95.2 (2)
S(1)—Ag(1)—S(6)	119.3 (1)	P—S(6)—Ag(1)	98.6 (2)
S(2)—Ag(1)—S(6)	106.9 (1)	O(2)—P—O(1)	106.0 (6)
P(2)—Ag(2)—S(1)	127.4 (1)	O(2)—P—S(5)	104.7 (4)
P(2)—Ag(2)—S(3)	121.7 (1)	O(2)—P—S(6)	113.5 (4)
P(2)—Ag(2)—S(5)	109.2 (1)	O(1)—P—S(5)	109.4 (4)
S(1)—Ag(2)—S(3)	93.5 (1)	O(1)—P—S(6)	105.6 (4)
S(1)—Ag(2)—S(5)	105.9 (1)	S(6)—P—S(5)	117.1 (2)
S(3)—Ag(2)—S(5)	93.2 (1)	C(1)—O(1)—P	119 (1)
S(2)—Ag(3)—S(5)	133.0 (1)	C(3)—O(2)—P	124 (1)
P(3)—Ag(3)—S(2)	118.4 (1)	C(2)—C(1)—O(1)	107 (1)
P(3)—Ag(3)—S(5)	103.8 (1)	O(2)—C(3)—C(4)	114 (2)
Mo—S(1)—Ag(1)	77.6 (1)	C(111)—P(1)—Ag(1)	114.5 (5)
Mo—S(1)—Ag(2)	77.1 (1)	C(121)—P(1)—Ag(1)	113.7 (5)
C(321)—P(3)—Ag(3)	118.1 (5)	C(131)—P(1)—Ag(1)	113.2 (5)
C(311)—P(3)—Ag(3)	111.0 (5)	C(211)—P(2)—Ag(2)	112.7 (5)
C(111)—P(1)—C(121)	103.8 (6)	C(221)—P(2)—Ag(2)	114.8 (4)
C(111)—P(1)—C(131)	105.7 (7)	C(231)—P(2)—Ag(2)	114.7 (4)
C(121)—P(1)—C(131)	104.8 (7)	C(331)—P(3)—Ag(3)	112.7 (5)
C(211)—P(2)—C(221)	103.5 (6)		

Data collection was performed using *CONTROL* (Molecular Structure Corporation, 1986) software. The scan speed varied between 2.4 and 8° min^{-1} (in ω) on the basis of *SEARCH* intensity. The scan width was (1.418 + 0.350tanθ)° with maximum (sinθ)/λ = 0.5946 $Å^{-1}$. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983). The heavy atoms, Mo and Ag, were located in the *E* map and the remaining non-H atoms were located using the *DIRDIF* program (Beurskens, 1984). H atoms were placed in geometrically calculated positions (C—H 0.95 Å), but not included in the refinement. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for all Mo, Ag, S, P and O atoms and isotropic displacement parameters for all C atoms. All calculations were performed on a VAX 785 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package. The view of the molecule (Fig. 1) was produced using *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71691 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1055]

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Bis(2,6-di-*tert*-butylphenolato-κO)tin

DAVID M. BARNHART, DAVID L. CLARK* AND JOHN G. WATKIN*

Isotope and Nuclear Chemistry Division, Mail Stop C346, Los Alamos National Laboratory, Los Alamos, NM 87545, USA

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Abstract

The title compound, $[Sn(C_{14}H_{21}O)_2]$ (I), contains a two-coordinate tin metal center with Sn—O distances of 2.003 (3) and 2.044 (3) Å, and an O—Sn—O angle of 88.8 (1)°.

Comment

The related complex $Sn(O\text{-}2,6\text{-}^tBu_2\text{-}4\text{-}Me\text{-}C_6H_2)_2$, prepared from the reaction of tin(II) chloride with lithium 2,6-di-*tert*-butyl-4-methylphenoxide, has