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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1040). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Iodotris(triphenylphosphine)silver(I), [Ag(PPh₃)₃I]

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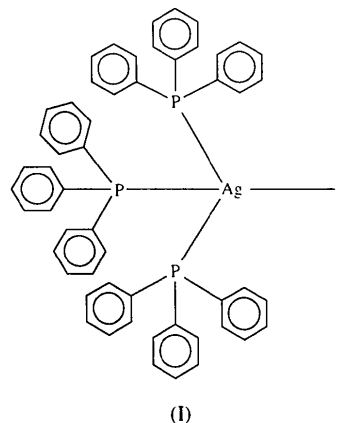
Abstract

The geometry about the Ag atom in the title complex, [AgI(C₁₈H₁₅P)₃], is distorted tetrahedral with the Ag—I bond length 2.8660 (11) Å and Ag—P distances 2.5335 (14), 2.5589 (12) and 2.6814 (12) Å. The P—Ag—P/I angles show large variations [104.54 (3)–116.10 (4)°] from the ideal tetrahedral value (109.45°).

These distortions, as well as those around the P atoms in the three triphenylphosphine ligands, may be attributed to several intramolecular (H···I and H···C/H) short contacts.

Comment

A number of tris(triphenylphosphine)silver(I) derivatives of the type [Ag(PPh₃)₃X] have been characterized by X-ray crystallography: X = Cl (Cassel, 1981), X = Cl, Br or I (Englehardt, Healy, Patrick & White, 1987), X = I, BF₄ (Camalli & Caruso, 1987), and X = NO₃ (Barron *et al.*, 1986; Bruce & Duffy, 1986). All these complexes are molecular species, in each of which the Ag atom has distorted tetrahedral geometry. The nitrate derivative is monoclinic and isostructural with the room-temperature (RT) iodide structure reported by Englehardt *et al.* (1987), with the N atom of the bidentate nitrate group occupying almost the same position as the I atom. In the BF₄ complex there is one Ag—F interaction which is long [2.82 (1) Å], but it definitely indicates a bonding (albeit weak) interaction. The RT iodide structure reported



by Englehardt *et al.* (1987) is monoclinic with one long and two short Ag—P bonds [2.780 (3), 2.544 (2) and 2.573 (3) Å], while the structure reported by Camalli & Caruso (1987) is triclinic with the Ag—P bonds falling within a narrow range [2.572 (4)–2.616 (3) Å]. We undertook an X-ray study of the complex at 140 K (low temperature, LT) to obtain more accurate data and confirm the unusual variations in the reported molecular geometry parameters. The LT structure reported here (Fig. 1) is isostructural with the RT monoclinic form reported by Englehardt *et al.* (1987).

The Ag^I centre is tetrahedral with one Ag—P bond [2.6814 (12) Å] much longer than the other two [2.5335 (14) and 2.5589 (12) Å]. These values follow the trend observed in the RT monoclinic structure (Englehardt *et al.*, 1987), but in the latter case the longest Ag—P bond is *ca* 0.10 Å longer than the corresponding value in our structure. The situation is somewhat dif-

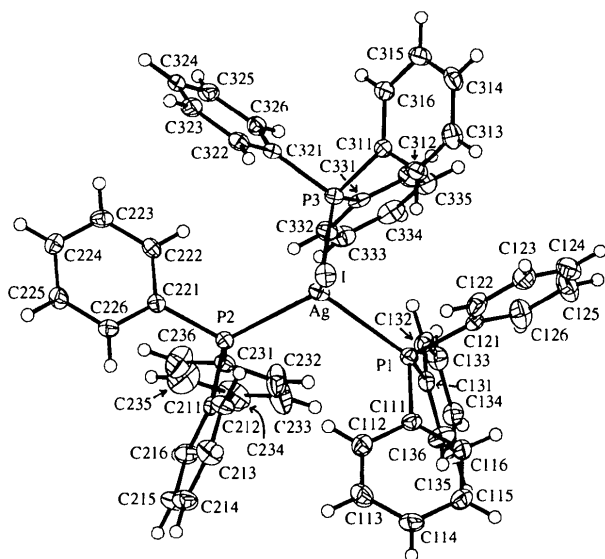


Fig. 1. Structure of $[\text{Ag}(\text{PPh}_3)\text{I}]$ showing the atom-numbering scheme (C336 is obscured by C312). Displacement ellipsoids are drawn at the 50% probability level. H atoms, represented by small circles of arbitrary radii, have the same numbers as their parents.

ferent in the triclinic form (Camalli & Curso, 1987) in which all the Ag—P distances fall within a narrow range [2.572 (4)–2.616 (3) Å]. The average value for the Ag—P bond in the present structure (2.591 Å) compares very well with that in the triclinic form (2.596 Å), but is *ca* 0.04 Å shorter than in the RT monoclinic structure (2.632 Å; Englehardt *et al.*, 1987). It is also noted that the Ag—P distances in all three iodide structures are markedly longer than those in the nitrate [2.525 (1)–2.630 (2), average 2.567 Å (Barron *et al.*, 1986); 2.522 (3)–2.630 (3), average 2.568 Å (Bruce & Duffy, 1986)], chloride [2.520 (1)–2.552 (1), average 2.543 Å (Cassel, 1981); 2.558 (5)–2.582 (4), average 2.572 Å (Englehardt *et al.*, 1987)] and bromide [2.528 (3)–2.549 (7), average 2.536 Å (Englehardt *et al.*, 1987)] derivatives. The Ag—I distance of 2.8660 (11) Å is very similar to those in the RT monoclinic [2.856 (1) Å (Englehardt *et al.*, 1987)] and triclinic [2.655 (1) and 2.864 (1) Å (Camalli & Curso, 1987)] forms.

The P—Ag—I and P—Ag—P angles lie in the ranges 104.54 (3)–107.86 (3)° (average 106.0°) and 107.91 (4)–116.10 (4)° (average 112.6°), respectively. These values are comparable with the corresponding angles [104.03 (6)–109.00 (6), average 106.7°; 107.00 (7)–116.93 (7), average 112.0°] in the RT monoclinic structure (Englehardt *et al.*, 1987), and in both cases the two shorter Ag—P bonds subtend the largest P—Ag—P angle. In the triclinic form, the P—Ag—I and P—Ag—P angles lie in the ranges 103.8 (1)–109.0 (1)° (average 105.6°) and 110.8 (1)–114.1 (1)° (average 112.9°), respectively, the latter showing less variation than those in both the monoclinic structures.

The variations from ideal values (109.4°) are consistent with the 'tetrahedral' coordination about the Ag^I centre being trigonally distorted, with the iodo ligand 'axial', and may be explained by the presence of several intramolecular I···H short contacts [$\text{I}\cdots\text{H}122 = 3.17$, $\text{I}\cdots\text{H}212 = 3.14$, $\text{I}\cdots\text{H}312 = 3.24$ Å]. The P—C bond lengths vary from 1.814 (4) to 1.846 (4) Å (average 1.831 Å), which are comparable with those in other related structures. The bond angles at the three P atoms display wide variations with C—P—C angles ranging from 100.6 (2) to 105.8 (2)° and Ag—P—C angles ranging from 108.7 (1) to 123.6 (1)°. The large variations in the Ag—P—C angles are explained by several intramolecular short contacts between different PPh₃ ligands, the most important of these interactions being $\text{C}122\cdots\text{H}312 = 2.70$, $\text{H}132\cdots\text{C}332 = 2.73$ and $\text{H}222\cdots\text{C}322 = 2.78$ Å. The orientations of the three PPh₃ ligands are the same as in the RT structure with one ligand adopting a skew and two adopting staggered conformations (Englehardt *et al.*, 1987).

The present structure is also very similar to those reported for the triclinic and trigonal forms of $\text{CuI}(\text{PPh}_3)_3$, in which the average P—Cu—P angles are 115.0 and 110.3°, respectively, and the Cu—X (X = I, P) distances are *ca* 0.20 Å shorter (Barron *et al.*, 1987). It was suggested that the overall geometry of the tetrahedral $(\text{Ph}_3\text{P})_3\text{CuX}$ core including the unequal Cu—P distances [2.327 (2)–2.362 (3) Å] is significantly influenced by several intramolecular H···C/H and H···X short contacts, which also affect the conformational changes within the PPh₃ ligands. This explanation seems equally suited to the Ag case where close I···H and H···C/H contacts are again apparent.

Experimental

The compound was prepared as colourless crystals, m.p. 446–447 K (lit. m.p. 438–443 K; Englehardt *et al.*, 1987) in a manner described for the chloride analogue (Cassel, 1981) by substituting [ⁿBu₄N]I for [Et₄N]Cl. Suitable single crystals were grown from a chloroform solution of the complex layered with hexane and left undisturbed overnight. IR bands (KBr, cm^{-1}) 3048, 1584, 1478(*m*), 1310, 1155, 1091(*m*), 1025, 997, 854, 743(*s*), 694(*s*), 512(*s*). ³¹P NMR (RT, CDCl_3): $\delta = -1.18$ p.p.m., relative to H_3PO_4 .

Crystal data

$[\text{AgI}(\text{C}_{18}\text{H}_{15}\text{P})_3]$
 $M_r = 1021.58$
 Monoclinic
 $P2_1/n$
 $a = 18.847$ (3) Å
 $b = 13.715$ (2) Å
 $c = 17.543$ (3) Å
 $\beta = 95.93$ (4)°
 $V = 4510.5$ (12) Å³
 $Z = 4$
 $D_x = 1.504$ Mg m⁻³

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å
 Cell parameters from 250 reflections
 $\theta = 1.84$ –25.06°
 $\mu = 1.274$ mm⁻¹
 $T = 140$ (2) K
 Parallelepiped
 0.26 × 0.22 × 0.20 mm
 Colourless

Data collection

Delft Instruments FAST
area-detector diffractometer
Collection method: see Pflugrath & Messerschmidt (1989) and Darr, Drake, Hursthouse & Malik (1993)
Absorption correction: refined from ΔF (DIFABS; Walker & Stuart, 1983)
 $T_{\min} = 0.889$, $T_{\max} = 0.997$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0489$
 $wR(F^2) = 0.1177$
 $S = 1.011$
6910 reflections
532 parameters
H atoms: riding model
 $w = 1/[\sigma^2(F_o^2) + (0.0764P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
 $\Delta\rho_{\max} = 3.236 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.806 \text{ e } \text{Å}^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

	x	y	z	U_{eq}
I	-0.10392 (2)	0.27263 (2)	0.10978 (2)	0.02674 (13)
Ag	-0.00064 (2)	0.19352 (2)	0.22461 (2)	0.02126 (13)
P1	0.00177 (6)	0.31952 (8)	0.34172 (6)	0.0186 (3)
P2	-0.04150 (6)	0.02193 (8)	0.25713 (6)	0.0180 (3)
P3	0.11810 (6)	0.20491 (8)	0.16940 (6)	0.0183 (3)
C111	-0.0865 (2)	0.3395 (3)	0.3727 (2)	0.0201 (10)
C112	-0.1378 (3)	0.2671 (3)	0.3608 (3)	0.0290 (11)
C113	-0.2040 (3)	0.2775 (4)	0.3855 (3)	0.0351 (13)
C114	-0.2220 (3)	0.3628 (4)	0.4203 (3)	0.0298 (11)
C115	-0.1726 (3)	0.4370 (4)	0.4307 (3)	0.0293 (11)
C116	-0.1050 (3)	0.4262 (3)	0.4075 (3)	0.0267 (11)
C121	0.0239 (2)	0.4432 (3)	0.3128 (2)	0.0217 (10)
C122	-0.0174 (3)	0.4806 (3)	0.2492 (3)	0.0313 (12)
C123	-0.0041 (3)	0.5728 (4)	0.2222 (3)	0.0409 (14)
C124	0.0484 (3)	0.6289 (4)	0.2575 (3)	0.0410 (14)
C125	0.0897 (3)	0.5933 (4)	0.3210 (4)	0.048 (2)
C126	0.0769 (3)	0.5009 (3)	0.3495 (3)	0.0358 (13)
C131	0.0594 (3)	0.3047 (3)	0.4327 (2)	0.0209 (10)
C132	0.1292 (3)	0.2807 (3)	0.4301 (3)	0.0262 (11)
C133	0.1763 (3)	0.2706 (3)	0.4965 (3)	0.0275 (11)
C134	0.1507 (3)	0.2836 (3)	0.5674 (3)	0.0281 (12)
C135	0.0805 (3)	0.3065 (4)	0.5711 (3)	0.0384 (13)
C136	0.0342 (3)	0.3165 (4)	0.5042 (3)	0.0338 (12)
C211	-0.1331 (2)	0.0145 (3)	0.2837 (2)	0.0186 (10)
C212	-0.1857 (3)	0.0659 (3)	0.2384 (3)	0.0266 (11)
C213	-0.2566 (3)	0.0577 (4)	0.2530 (3)	0.0327 (12)
C214	-0.2750 (3)	-0.0007 (4)	0.3120 (3)	0.0314 (12)
C215	-0.2228 (3)	-0.0505 (4)	0.3563 (3)	0.0309 (12)
C216	-0.1521 (3)	-0.0439 (3)	0.3422 (2)	0.0251 (10)
C221	-0.0396 (2)	-0.0816 (3)	0.1912 (2)	0.0189 (10)
C222	0.0208 (2)	-0.0948 (3)	0.1524 (2)	0.0213 (10)
C223	0.0246 (3)	-0.1707 (3)	0.1017 (2)	0.0237 (10)
C224	-0.0325 (3)	-0.2360 (3)	0.0888 (2)	0.0220 (10)
C225	-0.0918 (3)	-0.2234 (3)	0.1270 (3)	0.0249 (11)
C226	-0.0961 (2)	-0.1481 (3)	0.1779 (2)	0.0224 (10)
C231	0.0142 (2)	-0.0216 (3)	0.3419 (2)	0.0206 (10)
C232	0.0385 (3)	0.0473 (4)	0.3963 (3)	0.0433 (15)
C233	0.0837 (4)	0.0207 (4)	0.4600 (3)	0.055 (2)

C234	0.1051 (3)	-0.0727 (4)	0.4707 (3)	0.0426 (14)
C235	0.0814 (3)	-0.1416 (4)	0.4173 (3)	0.051 (2)
C236	0.0369 (3)	-0.1159 (4)	0.3536 (3)	0.0453 (15)
C311	0.1317 (2)	0.3107 (3)	0.1085 (2)	0.0187 (10)
C312	0.0836 (3)	0.3873 (3)	0.1082 (2)	0.0244 (10)
C313	0.0893 (3)	0.4664 (3)	0.0599 (3)	0.0305 (12)
C314	0.1422 (3)	0.4689 (3)	0.0112 (3)	0.0292 (12)
C315	0.1905 (3)	0.3930 (4)	0.0116 (3)	0.0298 (12)
C316	0.1850 (3)	0.3138 (3)	0.0597 (3)	0.0250 (11)
C321	0.1423 (2)	0.1039 (3)	0.1092 (2)	0.0178 (9)
C322	0.0930 (2)	0.0774 (3)	0.0480 (2)	0.0222 (10)
C323	0.1082 (3)	0.0026 (3)	-0.0008 (2)	0.0269 (11)
C324	0.1713 (3)	-0.0473 (3)	0.0115 (2)	0.0242 (11)
C325	0.2198 (3)	-0.0229 (3)	0.0720 (3)	0.0250 (11)
C326	0.2063 (2)	0.0530 (3)	0.1207 (2)	0.0225 (10)
C331	0.1925 (2)	0.2052 (3)	0.2438 (2)	0.0201 (10)
C332	0.1972 (2)	0.1270 (3)	0.2953 (2)	0.0244 (10)
C333	0.2528 (3)	0.1207 (4)	0.3531 (3)	0.0313 (12)
C334	0.3034 (3)	0.1928 (4)	0.3616 (3)	0.0335 (12)
C335	0.2992 (3)	0.2719 (4)	0.3132 (3)	0.0316 (12)
C336	0.2437 (3)	0.2781 (3)	0.2534 (3)	0.0255 (11)

Table 2. Selected geometric parameters (Å , $^\circ$)

I—Ag	2.8660 (11)	P2—C231	1.829 (4)
Ag—P3	2.5335 (14)	P2—C221	1.834 (4)
Ag—P2	2.5589 (12)	P2—C211	1.836 (5)
Ag—P1	2.6814 (12)	P3—C331	1.814 (4)
P1—C111	1.823 (5)	P3—C321	1.829 (4)
P1—C121	1.831 (4)	P3—C311	1.835 (4)
P1—C131	1.846 (4)		
P3—Ag—P2	116.10 (4)	C231—P2—C221	102.3 (2)
P3—Ag—P1	107.91 (4)	C231—P2—C211	104.5 (2)
P2—Ag—P1	113.80 (4)	C221—P2—C211	101.4 (2)
P3—Ag—I	105.69 (3)	C231—P2—Ag	108.73 (14)
P2—Ag—I	107.86 (3)	C221—P2—Ag	123.04 (14)
P1—Ag—I	104.54 (3)	C211—P2—Ag	114.84 (13)
C111—P1—C121	100.6 (2)	C331—P3—C321	101.4 (2)
C111—P1—C131	103.4 (2)	C331—P3—C311	105.8 (2)
C121—P1—C131	102.2 (2)	C321—P3—C311	101.8 (2)
C111—P1—Ag	112.42 (14)	C331—P3—Ag	111.88 (15)
C121—P1—Ag	111.75 (14)	C321—P3—Ag	117.21 (14)
C131—P1—Ag	123.59 (14)	C311—P3—Ag	116.9 (2)

H atoms were included in calculated positions (riding model, C—H = 0.93 Å) with U_{iso} set at $1.2 \times U_{\text{eq}}$ of the parent C atom. Highest residual electron densities in the final difference map which lie close to the Ag and I atoms are devoid of any stereochemical significance.

Data collection: *MADNES* (Pflugrath & Messerschmidt, 1989); further details from Darr, Drake, Hursthouse & Malik (1993). Cell refinement: *REFINE* in *MADNES*. Data reduction: *ABSMAD* (Karaulov, 1992). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SNOOPI* (Davies, 1983). Software used to prepare material for publication: *SHELXL93*.

We thank the EPSRC for support of the X-ray Crystallography Service at UWC.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1149). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 887–889

Tetrakis(3-methylpyridine)-1 κ N,2 κ N,-
3 κ N,4 κ N-bis(μ_4 -pentathio)-1:2 κ^2 S¹,2 κ S⁴,-
3 κ S²,3:4 κ^2 S⁵;1 κ S^{4'},1:3 κ^2 S^{1'},2:4 κ^2 S^{5'},-
4 κ S^{2'}-tetracopper(I) Bis(3-methylpyridine)
Solvate

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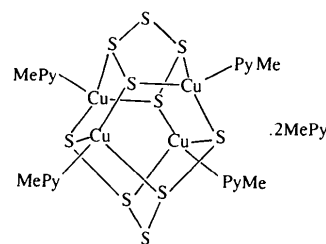
Abstract

The structure of the title compound, [Cu₄(S₅)₂(C₆H₇N)₄].2C₆H₇N, consists of a neutral complex molecule [Cu₄(S₅)₂(CH₃C₅H₄N)₄] and two 3-methylpyridine solvent molecules. In the neutral complex molecule, the four Cu atoms are in a distorted tetrahedral arrangement. The non-bonding Cu···Cu distances in the copper tetrahedron range from 3.281 (1) to 3.948 (1) Å and the Cu—S bond lengths vary from 2.255 (2) to 2.397 (3) Å.

Comment

In recent years, since remarkably rich photoluminescence has been found in tetranuclear complexes (Kevin,

Chong, John & Peter, 1991) and the cage-type structure with μ_3 -S may be potentially used in optical-limiting material (Shi, Ji, Tang, Lang & Xin, 1994), the synthesis of tetranuclear copper(I)-sulfur clusters has attracted attention. To date, a series of tetranuclear copper(I) clusters have been studied (Hathaway, 1987). Herein, a new tetranuclear copper(I) compound, namely, [Cu₄(S₅)₂(CH₃C₅H₄N)₄].2CH₃C₅H₄N, (I), is reported.



(I) (MePy = 2-methylpyridine)

The structure of the neutral complex molecule of the title compound is shown in Fig. 1. The neutral molecule is located on a twofold axis which passes through atoms S(3) and S(6). Each Cu atom has distorted tetrahedral geometry and is coordinated by three μ_3 -S atoms and an N atom from the pyridine ligand. The four Cu atoms are held together by two pentasulfido S₅²⁻ groups acting as bridging and chelating ligands.

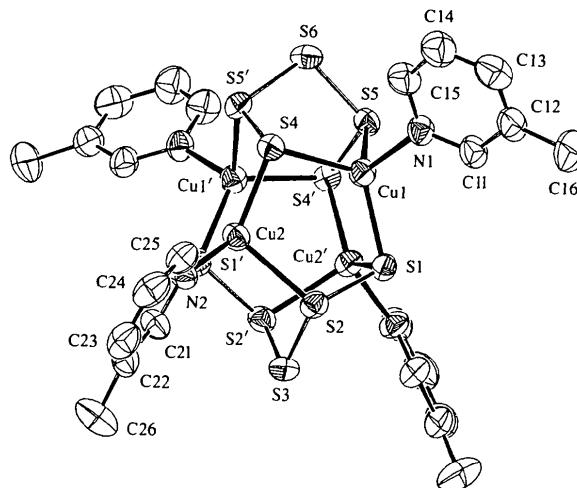


Fig. 1. ORTEP view (Johnson, 1976) of the title compound. Displacement ellipsoids are shown at the 50% probability level.

In the title compound, the four non-bonding Cu···Cu distances are 3.948 (1) [Cu(1)···Cu(1')], 3.312 (2) [Cu(1)···Cu(2)], 3.281 (1) [Cu(2)···Cu(1')] and 3.945 (1) Å [Cu(2)···Cu(2')]. The Cu—S bond lengths vary from 2.255 (2) to 2.397 (3) Å. The S—S bond lengths in the two S₅²⁻ ions vary from 2.062 (3) to 2.076 (5) Å. These distances are close to those found