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Silver Acetate–Triphenylphosphine Complexes. Acetatobis(triphenylphosphine)silver(I) and its Sesquihydrate, and Bis[acetato(triphenylphosphine)silver(I)] Hydrate and its Hemihydrate

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Abstract

The Ag atom in both independent molecules of acetatobis(triphenylphosphine)silver(I), $[Ag(C_2H_3O_2)(C_{18}H_{15}P)_2]$, is bonded to the triphenylphospine and chelating acetato ligands in a distorted tetrahedral environment. Acetatobis(triphenylphosphine)silver(I) sesquihydrate, $[Ag(C_2H_3O_2)(C_{18}H_{15}P)_2].1.5H_2O$, also crystallizes as two independent molecules, the Ag atoms of which adopt a similar geometry. One $[Ag(C_2H_3O_2)(C_{18}H_{15}P)_2]$ molecule is linked through a water molecule to another complex molecule, which in turn is linked to the symmetry-related first molecule through the other two water molecules, one of which is disordered, affording a chain structure parallel to the *b* axis. Bis(μ-acetato)-*O,O′:O;O:O*-bis[(triphenylphosphine)silver(I)] hydrate, $[Ag_2(C_2H_3O_2)_2(C_{18}H_{15}P)_2].-H_2O$, comprises two $[Ag(C_2H_3O_2)(C_{18}H_{15}P)]$ moieties; the acetato ligand behaves as a bidentate chelating anion in one complex moiety but as a monodentate anion in the other. The two moieties link through the singly bonded ester O atom into a dinuclear entity. Neighboring dinuclear entities are linked by hydrogen bonds involving their doubly-bonded carboxyl ends with the disordered water molecule to form a zigzag chain along the *c* axis. In bis-(μ-acetato-*O:O*)bis[(triphenylphosphine)silver(I)] hemihydrate, $[Ag_2(C_2H_3O_2)_2(C_{18}H_{15}P)_2].0.5H_2O$, the two Ag—$O_{carboxyl}$ distances are long enough for the geome-

try of the Ag atoms to be regarded as being essentially trigonal planar in the two complex units. The dinuclear molecule uses its doubly-bonded O atoms to link with the disordered water molecule to afford a hydrogen-bonded tetranuclear entity.

Comment

Acetatobis(triphenylphosphine)silver(I), (I), has been synthesized by reacting silver ketenide with excess triphenylphosphine in refluxing toluene (Femi-Onadeko, 1980), but it can be conveniently prepared by treating silver acetate with two molar equivalents of triphenylphosphine in boiling toluene. This complex is the silver analog of acetatobis(triphenylphosphine)copper(I) (Drew, Othman, Edwards & Richards, 1975), which exists as a monomeric acetato-chelated tetrahedral compound. Both compound (I) and its sesquihydrate, (II), show similar four-coordinate tetrahedral geometries for their Ag atoms. However, the anhydrous compound (I) is packed ($d = 1.392\,g\,cm^{-3}$) such that there are no solvent-accessible voids in its unit cell, as calculated by *PLATON* (Spek, 1990). The sesquihydrate, (II), is more loosely packed ($d = 1.345\,g\,cm^{-3}$) and there are four $72\,Å^3$ voids in the unit cell.

(I)

.$1.5H_2O$

(II)

(III)

(IV)
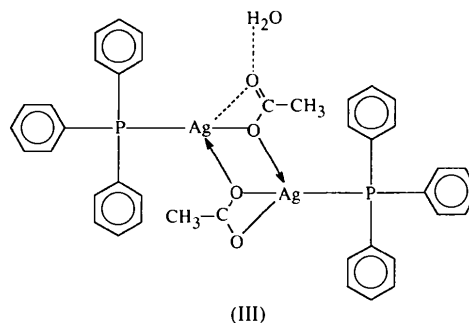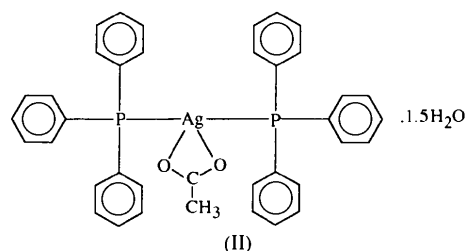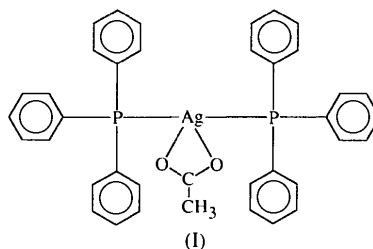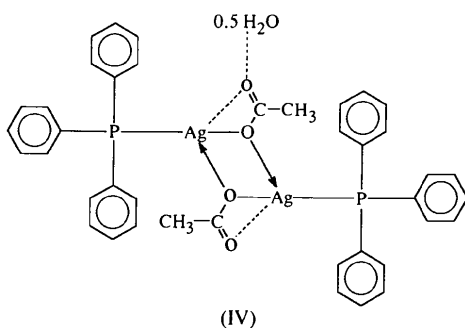
The reaction between equimolar quantities of silver acetate and triphenylphosphine in toluene yields more than one product. Tetrameric tetrakis(acetatotri-
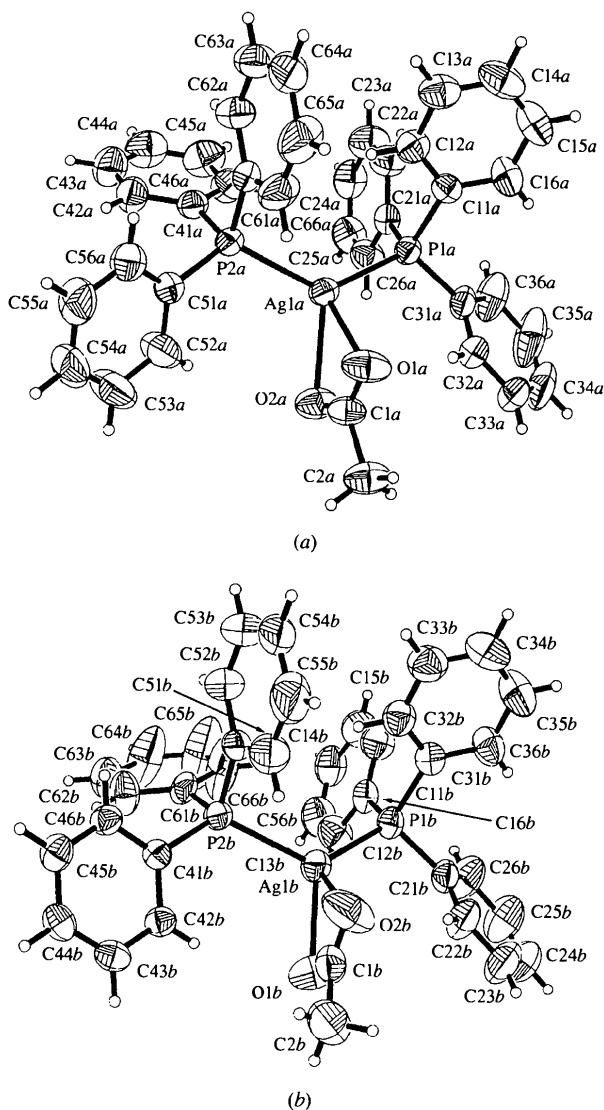

(a)

(b)

Fig. 1. ORTEPII (Johnson, 1976) plot of (a) molecule a and (b) molecule b of acetatobis(triphenylphosphine)silver(I) at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.
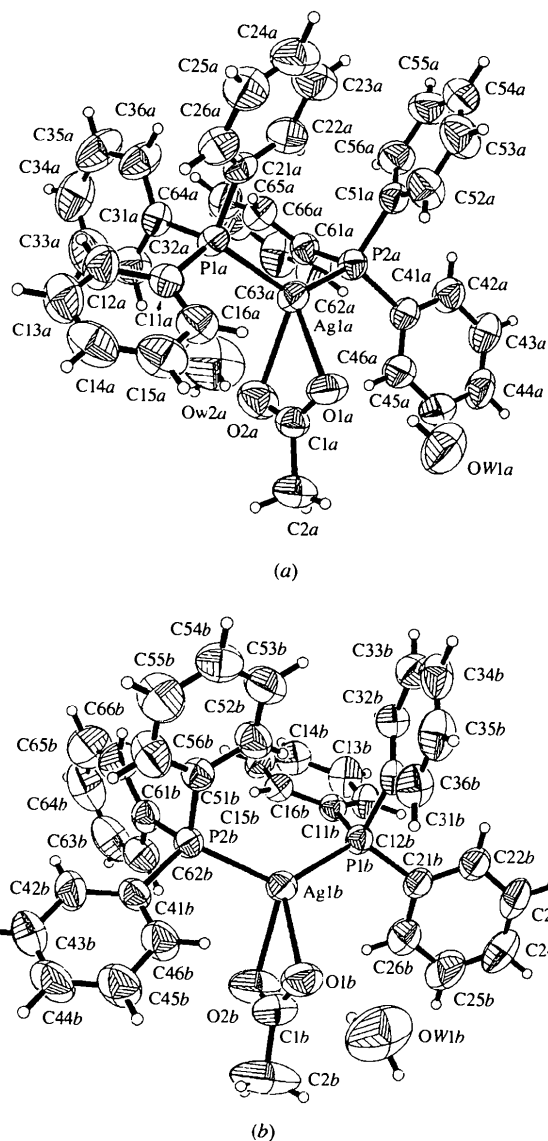

(a)

(b)

Fig. 2. ORTEPII (Johnson, 1976) plot of (a) molecule a and (b) molecule b of acetatobis(triphenylphosphine)silver(I) sesquihydrate at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. The disordered water molecule is shown as one molecule.

phenylphosphinesilver) was obtained when the mixture was recrystallized from the same solvent (Blues, Drew & Femi-Onadeko, 1977). In the present study, bis-[acetato(triphenylphosphine)silver(I)] hydrate, (III), and the hemihydrate, (IV), were isolated in separate reactions using the same method and solvent. The structure of (IV) has been reported previously; the water molecule (Ng & Othman, 1995) has been shown by the new diffraction data to be disordered over the twofold axis.

In mononuclear compound (I), the acetato group chelates to the Ag atom, the chelation being more

isobidentate in one molecule than the other [in molecule a, Ag—O = 2.420 (2) and 2.438 (2) Å; in molecule b, Ag—O = 2.379 (3) and 2.510 (3) Å]. The compound also exists as a mononuclear sesquihydrate, (II), the acetato group of which shows a similar bonding feature [in molecule a, Ag—O = 2.395 (3) and 2.492 (4) Å; in molecule b, Ag—O = 2.423 (4) and 2.425 (5) Å]. Complexation of silver(I) acetate with only one molecule of triphenylphosphine affords dinuclear (III), which contains one molecule of water, as well as compound (IV), which contains only half a molecule of water. In (IV), both acetato groups are regarded as being essentially unidentate [Ag1a—O1a = 2.204 (3) and Ag1a··O2a = 2.706 (3) Å; Ag1b—O1b = 2.197 (3) and Ag1b··O2b = 2.773 (4) Å] as the Ag atoms display trigonal planar geometries (sum of angles = 360°). The doubly-bonded O atoms, which form hydrogen

bonds to the water molecule, distort the geometry of the Ag atoms towards square planar. The two acetato-(triphenylphosphine)silver(I) halves of (IV) are related by a non-crystallographic center of inversion. On the other hand, in (III), only one Ag atom is chelated by the acetato group [Ag1b—O1b = 2.355 (3) and Ag1b—O2b = 2.481 (3) Å] but its geometry is distorted tetrahedral rather than distorted square planar. The other Ag atom of (III) shows a P—Ag—O angle of 167.40 (8)°, the magnitude of which is similar to those in (IV), which argues for trigonal planar coordination for this Ag atom [P1a—Ag1a—O1a = 167.40 (8), P1a—Ag1a—O1b = 115.27 (7) and O1a—Ag1a—O1b = 77.2 (1)°; sum of angles at Ag = 359.9 (3)°].

Experimental

Acetatobis(triphenylphosphine)silver(I), (I), was obtained by reacting silver(I) acetate with two molar equivalents of triphenylphosphine in a small volume of toluene. The mixture was briefly heated until the reactants had dissolved; slow cooling of the filtered solution gave colorless crystals of the compound. Another attempt at the synthesis yielded compound (II) and it could not be ascertained if either impurities in the solvent or the slight excess of triphenylphosphine used in the reaction had catalyzed its formation. The hydrate, (III), was obtained when a 1:1 silver acetate/triphenylphosphine ratio was used. The hemihydrate, (IV), was adventitiously obtained in a separate synthesis employing this stoichiometry.

Fig. 3. ORTEPII (Johnson, 1976) plot of bis[acetato(triphenylphosphine)silver(I)] hydrate at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. The disordered water molecule is shown as one molecule.
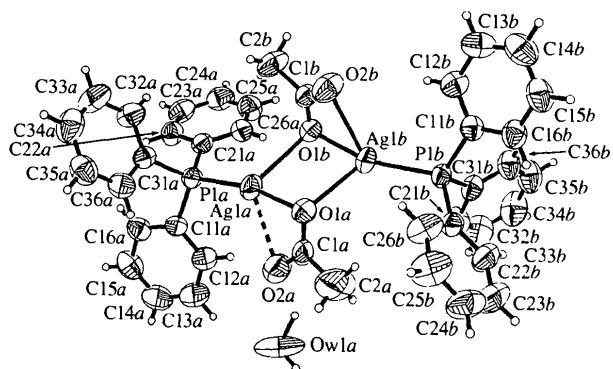

Fig. 4. ORTEPII (Johnson, 1976) plot of bis[acetato(triphenylphosphine)silver(I)] hemihydrate at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. The disordered water molecule is shown as one molecule.

Compound (I)

Crystal data

[Ag(C$_2$H$_3$O$_2$)(C$_{18}$H$_{15}$P)$_2$]	Mo $K\alpha$ radiation
M_r = 691.45	λ = 0.71073 Å
Triclinic	Cell parameters from 25
$P\bar{1}$	reflections
a = 10.078 (1) Å	θ = 14.5–15.0°
b = 13.7748 (9) Å	μ = 0.740 mm^{-1}
c = 24.167 (3) Å	T = 298 (2) K
α = 92.048 (8)°	Cube
β = 90.67 (1)°	0.58 × 0.58 × 0.58 mm
γ = 100.186 (9)°	Colorless
V = 3299.5 (6) Å^3	
Z = 4	
D_x = 1.392 Mg m^{-3}	
D_m not measured	

Data collection

Enraf–Nonius CAD-4	9658 reflections with
diffractometer	$I > 2\sigma(I)$
ω scan	R_{int} = 0.011
Absorption correction:	θ_{max} = 24.97°
ψ scans (North, Phillips	$h = 0 \rightarrow 11$
& Mathews, 1968)	$k = -16 \rightarrow 16$
T_{min} = 0.614, T_{max} = 0.651	$l = -28 \rightarrow 28$
12264 measured reflections	3 standard reflections
11596 independent	frequency: 60 min
reflections	intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.101$
$S = 1.071$
11594 reflections
673 parameters
H atoms: riding model,
$\quad U(H) = 1.5U_{eq}(C)$

$w = 1/[\sigma^2(F_o^2) + (0.0586P)^2 + 1.2901P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.003$
$\Delta\rho_{max} = 0.443$ e Å^{-3}
$\Delta\rho_{min} = -0.420$ e Å^{-3}
Extinction correction: none
Scattering factors from
$\quad$ International Tables for
$\quad$ Crystallography (Vol. C)

Table 1. Selected geometric parameters ($\mathring{A}$, °) for (I)

Ag1a—P1a	2.4483 (8)	Ag1b—P1b	2.4264 (8)
Ag1a—P2a	2.4332 (8)	Ag1b—P2b	2.4608 (8)
Ag1a—O1a	2.420 (2)	Ag1b—O1b	2.379 (3)
Ag1a—O2a	2.438 (2)	Ag1b—O2b	2.510 (3)
P1a—Ag1a—P2a	129.62 (3)	P1b—Ag1b—O1b	125.61 (8)
P1a—Ag1a—O1a	106.03 (7)	P1b—Ag1b—O2b	118.59 (9)
P1a—Ag1a—O2a	114.83 (7)	P2b—Ag1b—O1b	109.18 (8)
P2a—Ag1a—O1a	114.83 (7)	P2b—Ag1b—O2b	102.0 (1)
P2a·Ag1a—O2a	112.87 (7)	O1b—Ag1b—O2b	52.3 (1)
O1a—Ag1a—O2a	53.33 (7)	C21b—P1b—Ag1b	112.19 (8)
C11a—P1a—Ag1a	112.81 (7)	C31b—P1b—Ag1b	113.11 (7)
C21a—P1a—Ag1a	114.10 (8)	C11b—P1b—Ag1b	117.44 (8)
C31a—P1a—Ag1a	116.09 (8)	C41b—P2b—Ag1b	120.57 (6)
C41a—P2a—Ag1a	115.72 (7)	C51b—P2b—Ag1b	104.76 (8)
C51a—P2a—Ag1a	118.46 (8)	C61b—P2b—Ag1b	115.5 (2)
C61a—P2a—Ag1a	107.72 (8)	C61—P2b—Ag1b	115.0 (2)
P1b—Ag1b—P2b	124.13 (3)		

Compound (II)

Crystal data
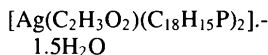
[Ag(C$_2$H$_3$O$_2$)(C$_{18}$H$_{15}$P)$_2$].-
$\quad$ 1.5H$_2$O
$M_r = 718.48$
Monoclinic
$C2/c$
$a = 44.318$ (6) Å
$b = 13.2622$ (8) Å
$c = 25.060$ (3) Å
$\beta = 105.492$ (5)°
$V = 14194$ (3) Å^3
$Z = 16$
$D_x = 1.345$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25
$\quad$ reflections
$\theta = 14.5$–$15.0°$
$\mu = 0.694$ mm^{-1}
$T = 298$ (2) K
Cube
$0.44 \times 0.44 \times 0.44$ mm
Colorless

Data collection

Enraf–Nonius CAD-4
$\quad$ diffractometer
ω scans
Absorption correction:
$\quad \psi$ scans (North, Phillips
$\quad$ & Mathews, 1968)
$\quad T_{min} = 0.700$, $T_{max} = 0.737$
11926 measured reflections
11605 independent
$\quad$ reflections

7914 reflections with
$\quad I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 24.99°$
$h = -52 \rightarrow 50$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 29$
3 standard reflections
$\quad$ frequency: 60 min
$\quad$ intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.135$

$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 8.9548P]$
where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.059$
11605 reflections
661 parameters
H atoms: riding model,
$\quad U(H) = 1.5U_{eq}(C)$;
$\quad$ water H atoms placed at
$\quad$ calculated positions with
$\quad U = 0.1$ Å^2

$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 0.508$ e Å^{-3}
$\Delta\rho_{min} = -0.328$ e Å^{-3}
Extinction correction: none
Scattering factors from
$\quad$ International Tables for
$\quad$ Crystallography (Vol. C)

Table 2. Selected geometric parameters ($\mathring{A}$, °) for (II)

Ag1a—P1a	2.434 (1)	Ag1b—P1b	2.404 (1)
Ag1a—P2a	2.436 (1)	Ag1b—P2b	2.441 (1)
Ag1a—O1a	2.395 (3)	Ag1b—O1b	2.423 (4)
Ag1a—O2a	2.492 (4)	Ag1b—O2b	2.425 (5)
P1a—Ag1a—P2a	129.44 (5)	P1b—Ag1b—P2b	128.53 (5)
P1a—Ag1a—O1a	114.2 (1)	P1b—Ag1b—O1b	119.9 (1)
P1a—Ag1a—O2a	108.9 (1)	P1b—Ag1b—O2b	118.9 (1)
P2a—Ag1a—O1a	115.1 (1)	P2b—Ag1b—O1b	107.2 (1)
P2a—Ag1a—O2a	109.2 (1)	P2b—Ag1b—O2b	104.8 (1)
O1a—Ag1a—O2a	52.5 (1)	O1b—Ag1b—O2b	53.7 (1)
C11a—P1a—Ag1a	119.1 (1)	C11b—P1b—Ag1b	112.6 (1)
C21a—P1a—Ag1a	113.5 (1)	C21b—P1b—Ag1b	117.5 (1)
C31a—P1a—Ag1a	109.5 (1)	C31b—P1b—Ag1b	111.6 (1)
C41a—P2a—Ag1a	118.0 (1)	C41b—P2b—Ag1b	116.2 (1)
C51a—P2a—Ag1a	115.7 (1)	C51b—P2b—Ag1b	114.9 (1)
C61a—P2a—Ag1a	108.4 (1)	C61b—P2b—Ag1b	110.6 (1)

Compound (III)

Crystal data
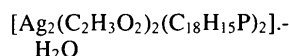
[Ag$_2$(C$_2$H$_3$O$_2$)$_2$(C$_{18}$H$_{15}$P)$_2$].-
$\quad$ H$_2$O
$M_r = 876.38$
Monoclinic
$P2_1/c$
$a = 15.850$ (1) Å
$b = 12.4271$ (6) Å
$c = 19.935$ (2) Å
$\beta = 103.960$ (4)°
$V = 3810.7$ (5) Å^3
$Z = 4$
$D_x = 1.528$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25
$\quad$ reflections
$\theta = 14.5$–$15.0°$
$\mu = 1.153$ mm^{-1}
$T = 298$ (2) K
Cube
$0.65 \times 0.65 \times 0.65$ mm
Colorless

Data collection

Enraf–Nonius CAD-4
$\quad$ diffractometer
ω scans
Absorption correction:
$\quad \psi$ scans (North, Phillips
$\quad$ & Mathews, 1968)
$\quad T_{min} = 0.415$, $T_{max} = 0.473$
6895 measured reflections
6681 independent reflections

5546 reflections with
$\quad I > 2\sigma(I)$
$R_{int} = 0.008$
$\theta_{max} = 24.98°$
$h = -18 \rightarrow 18$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 23$
3 standard reflections
$\quad$ frequency: 60 min
$\quad$ intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.118$
$S = 1.138$
6681 reflections
373 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2 + 1.7144P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max} = 1.060$ e Å^{-3}
$\Delta\rho_{min} = -0.478$ e Å^{-3}

H atoms: riding model,
$U(H) = 1.5U_{eq}(C)$;
water H-atoms placed at
calculated positions with
$U = 0.1$ Å^2

Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)

Table 3. *Selected geometric parameters (Å, °) for (III)*

Ag1a—P1a	2.350 (1)	Ag1b—P1b	2.3428 (9)
Ag1a—O1a	2.258 (3)	Ag1b—O1a	2.451 (3)
Ag1a—O1b	2.565 (3)	Ag1b—O1b	2.355 (3)
Ag1a—O2a	2.622 (4)	Ag1b—O2b	2.481 (3)
P1a—Ag1a—O1a	167.40 (8)	P1b—Ag1b—O1a	124.46 (8)
P1a—Ag1a—O1b	115.27 (7)	P1b—Ag1b—O1b	153.74 (8)
P1a—Ag1a—O2a	117.50 (9)	P1b—Ag1b—O2b	126.06 (9)
O1a—Ag1a—O1b	77.2 (1)	O1a—Ag1b—O1b	77.8 (1)
O1a—Ag1a—O2a	51.9 (1)	O1a—Ag1b—O2b	98.9 (1)
O1b—Ag1a—O2a	116.1 (1)	O1b—Ag1b—O2b	53.2 (1)
C31a—P1a—Ag1a	113.0 (1)	C31b—P1b—Ag1b	114.8 (1)
C21a—P1a—Ag1a	114.47 (9)	C21b—P1b—Ag1b	114.8 (1)
C11a—P1a—Ag1a	112.78 (9)	C11b—P1b—Ag1b	112.76 (9)
Ag1a—O1a—Ag1b	105.5 (1)	Ag1b—O1b—Ag1a	99.2 (1)

Compound (IV)

Crystal data

[Ag$_2$(C$_2$H$_3$O$_2$)$_2$(C$_{18}$H$_{15}$P)$_2$].-
0.5H$_2$O
$M_r = 867.38$
Orthorhombic
$P2_12_12$
$a = 12.1443$ (6) Å
$b = 19.2794$ (8) Å
$c = 15.8815$ (9) Å
$V = 3718.4$ (3) Å^3
$Z = 4$
$D_x = 1.549$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25
reflections
$\theta = 14.5–15.0°$
$\mu = 1.180$ mm^{-1}
$T = 298$ (2) K
Block
$0.58 \times 0.50 \times 0.43$ mm
Colorless

Data collection

Enraf–Nonius CAD-4
diffractometer
ω scan
Absorption correction:
ψ scans (North, Phillips
& Mathews, 1968)
$T_{min} = 0.566$, $T_{max} = 0.602$
7107 measured reflections
6538 independent reflections

5685 reflections with
$I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 24.97°$
$h = 0 \rightarrow 14$
$k = -22 \rightarrow 22$
$l = -18 \rightarrow 0$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.080$
$S = 1.048$
6538 reflections
375 parameters
H atoms: riding model,
$U(H) = 1.5U_{eq}(C)$; water
H-atom located and
refined
$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 0.2894P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.496$ e Å^{-3}
$\Delta\rho_{min} = -0.478$ e Å^{-3}
Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)
Absolute configuration:
Flack & Schwarzenbach
(1988)
Flack parameter = -0.03 (3)

Table 4. *Selected geometric parameters (Å, °) for (IV)*

Ag1a—P1a	2.343 (1)	Ag1b—P1b	2.346 (1)
Ag1a—O1a	2.204 (3)	Ag1b—O1a	2.675 (3)
Ag1a—O1b	2.568 (3)	Ag1b—O1b	2.197 (3)
Ag1a—O2a	2.706 (3)	Ag1b—O2b	2.773 (4)
P1a—Ag1a—O1a	164.64 (8)	P1b—Ag1b—O1a	112.29 (7)
P1a—Ag1a—O1b	114.32 (7)	P1b—Ag1b—O1b	167.73 (8)
P1A—Ag1a—O2a	116.76 (8)	P1b—Ag1b—O2b	118.17 (9)
O1a—Ag1a—O1b	80.8 (1)	O1b—Ag1b—O1a	78.52 (9)
O1a—Ag1a—O2a	51.8 (1)	O1b—Ag1b—O2b	50.0 (1)
O1b—Ag1a—O2a	110.4 (1)	O1a—Ag1b—O2b	114.6 (1)
C11a—P1a—Ag1a	113.5 (1)	C11b—P1b—Ag1b	113.9 (1)
C21a—P1a—Ag1a	113.8 (1)	C21b—P1b—Ag1b	112.9 (1)
C31a—P1a—Ag1a	112.8 (1)	C31b—P1b—Ag1b	114.3 (1)
Ag1a—O1a—Ag1b	98.6 (1)	Ag1b—O1b—Ag1a	102.1 (1)

The phenyl rings in the four structures were refined as rigid hexagons. In (I), one of the rings is disordered over two positions. In (II), one of the water molecules is disordered over two positions; the two half-site-occupancy O atoms were refined with an *EADP* instruction in *SHELXL93* (Sheldrick, 1993). In (III), the water is also disordered over two positions and an *EADP* instruction was also used. In (IV), the water molecule which lies on the twofold axis is disordered over two positions but the *EADP* constraint was not used; the water H atom was located from a difference Fourier map and refined.

For all compounds, data collection: *CAD-4 VAX/PC* (Enraf–Nonius, 1988); cell refinement: *CAD-4 VAX/PC*; data reduction: *Xtal3.0* (Hall & Stewart, 1990); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990a); program(s) used to refine structure: *SHELXL93*; molecular graphics: *ORTEPII* (Johnson, 1976) in *SHELXLTL/PC* (Sheldrick, 1990b); software used to prepare material for publication: *SHELXL93*.

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