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Di-μ-iodido-bis[bis(cyclohexyldiphenylphosphine-κP)silver(I)]

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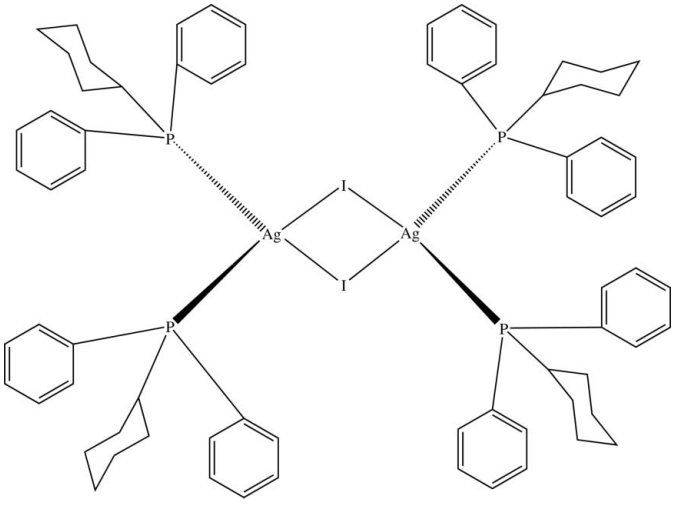
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.007 Å; R factor = 0.039; wR factor = 0.090; data-to-parameter ratio = 15.9.

The title compound, $[Ag_2I_2(C_{18}H_{21}P)_4]$, has a dimeric structure located on a twofold rotation axis with each tetrahedral Ag^I ion coordinated by two terminal phosphines and bridged by iodide ligands. Although this structural motif has been reported in bromide– and chloride–silver complexes, this is the first reported dimeric iodide–silver complex with monodentate phosphine.

Related literature

Background information on monodentate phosphine–AgX (X = Br, Cl) adducts can be found in Attar *et al.* (1991), Bowmaker *et al.* (1993), Cassel (1979) and Teo & Calabrese (1976).



Experimental

Crystal data

$[Ag_2I_2(C_{18}H_{21}P)_4]$
M_r = 1542.81
Monoclinic, $C2/c$
a = 27.805 (4) Å
b = 13.3327 (17) Å
c = 17.598 (4) Å
β = 93.474 (13)°
V = 6511.8 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.69 mm^{-1}
T = 120 (2) K
0.38 × 0.24 × 0.16 mm

Data collection

Bruker APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.556, T_{max} = 0.763
30202 measured reflections
5724 independent reflections
4448 reflections with $I > 2\sigma(I)$
R_{int} = 0.099

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.090
S = 1.05
5724 reflections
361 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.94 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

We thank Professor Klaus H. Theopold, Director of the Center for Catalytic Science and Technology, for synthetic assistance and the Department of Chemistry and Biochemistry for the purchase of reagents.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2047).

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supplementary materials

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Di-μ-iodido-bis[bis(cyclohexyldiphenylphosphine-κP)silver(I)]

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Comment

The 1:2 AgI:$PCyPh_2$ dimeric complex is similar to the reported triphenyphosphine complexes with chlorine or bromine (Cassel, 1979; Bowmaker *et al.*,1993; Teo & Calabrese, 1976) and the 5-phenyldibenzophosphine complex with chloride (Attar *et al.*, 1991). The 1:2 AgI:$PCyPh_2$ dimeric complex is the first reported complex of this type with bridging iodine and monodentate phosphine. The molecule is located on a twofold axis.

Experimental

Synthesis of $[Ag(PCyPh_2)_2I]_2$: 1 equivalent of AgI and 2.1 equivalents of diphenylcyclohexylphosphine were added in a vial containing anhydrous methylenechloride·The mixture was allowed to stir for 3 h at room temperature in darkness. Colorless crystals were grown by slow cooling of a saturated methylenechloride solution from ambient to 273 K in the dark. (yield 54%).

Refinement

H atoms were assigned calculated positions with U_{iso} restrained to be $0.2U_{eq}$ of the bonded C atom and a C—H distance of 0.95–0.99 Å.

Figures

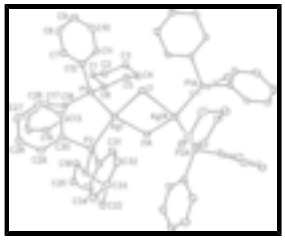


Fig. 1. Molecular diagram of the 1:2 AgI:$PCyPh_2$ dimeric complex with ellipsoids at 30% probability. Hydrogen atoms are omitted for clarity.

Di-μ-iodido-bis[bis(cyclohexyldiphenylphosphine-κ*P*)silver(I)]

Crystal data

$[Ag_2I_2(C_{18}H_{21}P)_4]$	F_{000} = 3104
M_r = 1542.81	D_x = 1.574 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 934 reflections
a = 27.805 (4) Å	θ = 2.7–22.1°

b = 13.3327 (17) Å	μ = 1.69 mm^{-1}
c = 17.598 (4) Å	T = 120 (2) K
β = 93.474 (13)º	Tabular, colourless
V = 6511.8 (19) Å^3	0.38 × 0.24 × 0.16 mm
Z = 4	

Data collection

Bruker APEX diffractometer	5724 independent reflections
Radiation source: fine-focus sealed tube	4448 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.099
Detector resolution: 836.6 pixels mm^{-1}	θ_{max} = 25.0º
T = 120(2) K	θ_{min} = 2.0º
ω scans	h = −32→32
Absorption correction: multi-scan (SADABS; Bruker, 2002)	k = −15→15
T_{min} = 0.556, T_{max} = 0.763	l = −20→20
30202 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H-atom parameters constrained
$wR(F^2)$ = 0.090	$w = 1/[\sigma^2(F_o^2) + (0.0207P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.005
5724 reflections	$\Delta\rho_{max}$ = 0.94 e Å^{-3}
361 parameters	$\Delta\rho_{min}$ = −0.50 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. Data collection is performed with four batch runs at φ = 0.00 ° (600 frames), at φ = 90.00 ° (600 frames), at φ = 180 ° (600 frames) and at φ = 270 ° (600 frames). Frame width = 0.30 ° in ω. Data is merged, corrected for decay, and treated with multi-scan absorption corrections.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-

factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.936332 (13)	0.23521 (3)	0.22857 (2)	0.02529 (11)
I1	1.011583 (11)	0.21772 (2)	0.122587 (17)	0.02443 (10)
P1	0.87855 (4)	0.08676 (9)	0.22706 (7)	0.0218 (3)
P2	0.89485 (4)	0.40206 (9)	0.21388 (7)	0.0238 (3)
C1	0.86182 (16)	−0.0960 (3)	0.3073 (3)	0.0258 (11)
H1A	0.8616	−0.1352	0.2595	0.031*
H1B	0.8286	−0.0719	0.3137	0.031*
C2	0.87775 (17)	−0.1632 (4)	0.3743 (3)	0.0345 (13)
H2A	0.8736	−0.1265	0.4223	0.041*
H2B	0.8567	−0.2231	0.3739	0.041*
C3	0.92953 (17)	−0.1967 (3)	0.3723 (3)	0.0347 (13)
H3A	0.9325	−0.2442	0.3296	0.042*
H3B	0.9391	−0.2324	0.4202	0.042*
C4	0.96339 (17)	−0.1084 (3)	0.3626 (3)	0.0314 (12)
H4A	0.9652	−0.0679	0.4099	0.038*
H4B	0.9961	−0.1340	0.3547	0.038*
C5	0.94707 (15)	−0.0418 (3)	0.2958 (3)	0.0241 (11)
H5A	0.9496	−0.0793	0.2477	0.029*
H5B	0.9685	0.0175	0.2945	0.029*
C6	0.89525 (15)	−0.0071 (3)	0.3020 (3)	0.0236 (11)
H6A	0.8945	0.0291	0.3517	0.028*
C7	0.82980 (17)	−0.0480 (4)	0.1225 (3)	0.0313 (12)
H7A	0.8026	−0.0438	0.1526	0.038*
C8	0.82872 (19)	−0.1112 (4)	0.0600 (3)	0.0399 (14)
H8A	0.8002	−0.1479	0.0462	0.048*
C9	0.86880 (19)	−0.1215 (4)	0.0171 (3)	0.0345 (13)
H9A	0.8681	−0.1672	−0.0244	0.041*
C10	0.90939 (19)	−0.0653 (4)	0.0349 (3)	0.0331 (12)
H10A	0.9367	−0.0712	0.0052	0.040*
C11	0.91036 (17)	0.0005 (3)	0.0967 (3)	0.0264 (11)
H11A	0.9384	0.0395	0.1086	0.032*
C12	0.87082 (17)	0.0097 (3)	0.1414 (3)	0.0257 (11)
C13	0.80198 (16)	0.1262 (3)	0.3217 (3)	0.0258 (11)
H13A	0.8223	0.1006	0.3625	0.031*
C14	0.75725 (17)	0.1661 (3)	0.3368 (3)	0.0322 (12)
H14A	0.7472	0.1675	0.3875	0.039*
C15	0.72758 (18)	0.2036 (4)	0.2778 (3)	0.0385 (14)
H15A	0.6973	0.2323	0.2879	0.046*
C16	0.74172 (18)	0.1995 (4)	0.2052 (3)	0.0398 (14)
H16A	0.7207	0.2232	0.1646	0.048*
C17	0.78624 (16)	0.1612 (4)	0.1898 (3)	0.0325 (12)
H17A	0.7958	0.1607	0.1388	0.039*

C18	0.81737 (15)	0.1233 (3)	0.2479 (3)	0.0248 (11)
C19	0.83838 (17)	0.3677 (3)	0.3350 (3)	0.0268 (11)
H19A	0.8584	0.3071	0.3447	0.032*
H19B	0.8114	0.3496	0.2982	0.032*
C20	0.81820 (17)	0.4027 (4)	0.4094 (3)	0.0324 (12)
H20A	0.7939	0.4558	0.3982	0.039*
H20B	0.8019	0.3457	0.4331	0.039*
C21	0.85711 (19)	0.4429 (4)	0.4649 (3)	0.0354 (13)
H21A	0.8420	0.4715	0.5096	0.042*
H21B	0.8783	0.3870	0.4830	0.042*
C22	0.88707 (18)	0.5224 (4)	0.4294 (3)	0.0355 (13)
H22A	0.8668	0.5822	0.4180	0.043*
H22B	0.9136	0.5425	0.4662	0.043*
C23	0.90791 (16)	0.4870 (3)	0.3574 (3)	0.0274 (11)
H23A	0.9313	0.4325	0.3695	0.033*
H23B	0.9254	0.5430	0.3344	0.033*
C24	0.86880 (16)	0.4490 (3)	0.3005 (3)	0.0270 (11)
H24A	0.8472	0.5066	0.2858	0.032*
C25	0.85027 (17)	0.3252 (3)	0.0829 (3)	0.0277 (11)
H25A	0.8797	0.2895	0.0800	0.033*
C26	0.81439 (19)	0.3152 (4)	0.0253 (3)	0.0346 (13)
H26A	0.8192	0.2722	−0.0166	0.042*
C27	0.77145 (18)	0.3678 (4)	0.0283 (3)	0.0341 (13)
H27A	0.7471	0.3621	−0.0117	0.041*
C28	0.76458 (17)	0.4282 (4)	0.0900 (3)	0.0302 (12)
H28A	0.7351	0.4637	0.0930	0.036*
C29	0.80011 (17)	0.4378 (3)	0.1477 (3)	0.0281 (12)
H29A	0.7948	0.4798	0.1900	0.034*
C30	0.84357 (16)	0.3867 (3)	0.1447 (3)	0.0241 (11)
C31	0.95789 (16)	0.5014 (3)	0.1230 (3)	0.0276 (11)
H31A	0.9705	0.4364	0.1145	0.033*
C32	0.97607 (17)	0.5842 (4)	0.0841 (3)	0.0335 (12)
H32A	1.0020	0.5757	0.0519	0.040*
C33	0.95618 (17)	0.6771 (4)	0.0929 (3)	0.0306 (12)
H33A	0.9666	0.7320	0.0636	0.037*
C34	0.92163 (19)	0.6915 (4)	0.1434 (3)	0.0402 (14)
H34A	0.9100	0.7573	0.1521	0.048*
C35	0.90309 (17)	0.6105 (3)	0.1825 (3)	0.0327 (12)
H35A	0.8777	0.6207	0.2153	0.039*
C36	0.92213 (16)	0.5132 (3)	0.1732 (3)	0.0263 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0247 (2)	0.0233 (2)	0.0284 (2)	−0.00002 (15)	0.00537 (16)	0.00078 (16)
I1	0.02476 (19)	0.02417 (18)	0.02487 (19)	0.00026 (13)	0.00564 (14)	−0.00068 (13)
P1	0.0205 (6)	0.0209 (7)	0.0238 (7)	−0.0006 (5)	0.0011 (5)	0.0009 (5)
P2	0.0269 (7)	0.0186 (6)	0.0265 (7)	0.0007 (5)	0.0061 (6)	−0.0029 (5)

C1	0.020 (3)	0.025 (3)	0.032 (3)	0.000 (2)	−0.002 (2)	0.005 (2)
C2	0.030 (3)	0.029 (3)	0.044 (3)	−0.003 (2)	0.004 (2)	0.011 (2)
C3	0.030 (3)	0.026 (3)	0.048 (3)	0.002 (2)	0.002 (3)	0.011 (2)
C4	0.029 (3)	0.022 (3)	0.044 (3)	0.001 (2)	0.002 (2)	0.004 (2)
C5	0.023 (3)	0.018 (2)	0.031 (3)	−0.002 (2)	0.004 (2)	0.000 (2)
C6	0.019 (2)	0.023 (3)	0.029 (3)	−0.001 (2)	0.002 (2)	0.003 (2)
C7	0.030 (3)	0.034 (3)	0.030 (3)	−0.001 (2)	0.002 (2)	−0.001 (2)
C8	0.037 (3)	0.044 (3)	0.038 (3)	−0.013 (3)	−0.010 (3)	−0.002 (3)
C9	0.054 (4)	0.028 (3)	0.019 (3)	0.002 (3)	−0.007 (3)	−0.005 (2)
C10	0.043 (3)	0.030 (3)	0.027 (3)	0.008 (2)	0.009 (2)	0.004 (2)
C11	0.027 (3)	0.027 (3)	0.026 (3)	−0.005 (2)	−0.001 (2)	−0.005 (2)
C12	0.031 (3)	0.021 (3)	0.025 (3)	−0.002 (2)	−0.002 (2)	0.000 (2)
C13	0.023 (3)	0.025 (3)	0.029 (3)	−0.001 (2)	0.002 (2)	−0.002 (2)
C14	0.031 (3)	0.024 (3)	0.043 (3)	−0.004 (2)	0.013 (3)	−0.004 (2)
C15	0.019 (3)	0.027 (3)	0.069 (4)	0.005 (2)	0.006 (3)	0.005 (3)
C16	0.024 (3)	0.036 (3)	0.058 (4)	−0.001 (2)	−0.007 (3)	0.018 (3)
C17	0.024 (3)	0.033 (3)	0.040 (3)	−0.006 (2)	−0.002 (2)	0.009 (2)
C18	0.017 (2)	0.019 (2)	0.038 (3)	−0.0030 (19)	−0.002 (2)	0.003 (2)
C19	0.030 (3)	0.024 (3)	0.026 (3)	0.000 (2)	0.005 (2)	0.001 (2)
C20	0.029 (3)	0.037 (3)	0.032 (3)	0.002 (2)	0.008 (2)	−0.002 (2)
C21	0.048 (3)	0.036 (3)	0.023 (3)	0.010 (3)	0.005 (2)	−0.002 (2)
C22	0.034 (3)	0.042 (3)	0.030 (3)	0.003 (3)	0.000 (2)	−0.011 (2)
C23	0.024 (3)	0.028 (3)	0.029 (3)	0.006 (2)	−0.001 (2)	−0.004 (2)
C24	0.027 (3)	0.025 (3)	0.030 (3)	0.003 (2)	0.004 (2)	−0.004 (2)
C25	0.036 (3)	0.023 (3)	0.026 (3)	−0.004 (2)	0.010 (2)	−0.002 (2)
C26	0.047 (3)	0.027 (3)	0.030 (3)	−0.010 (3)	0.009 (3)	−0.008 (2)
C27	0.035 (3)	0.037 (3)	0.030 (3)	−0.015 (3)	0.002 (2)	−0.005 (2)
C28	0.029 (3)	0.030 (3)	0.032 (3)	0.001 (2)	0.005 (2)	0.001 (2)
C29	0.036 (3)	0.029 (3)	0.021 (3)	−0.001 (2)	0.009 (2)	0.000 (2)
C30	0.027 (3)	0.021 (3)	0.025 (3)	−0.002 (2)	0.008 (2)	0.003 (2)
C31	0.027 (3)	0.023 (3)	0.034 (3)	0.002 (2)	0.009 (2)	−0.001 (2)
C32	0.027 (3)	0.032 (3)	0.043 (3)	−0.001 (2)	0.011 (2)	0.004 (2)
C33	0.036 (3)	0.026 (3)	0.030 (3)	−0.009 (2)	0.000 (2)	0.005 (2)
C34	0.048 (4)	0.018 (3)	0.055 (4)	0.001 (2)	0.012 (3)	0.000 (3)
C35	0.034 (3)	0.025 (3)	0.041 (3)	0.004 (2)	0.012 (2)	0.001 (2)
C36	0.023 (3)	0.021 (3)	0.035 (3)	−0.001 (2)	0.006 (2)	−0.002 (2)

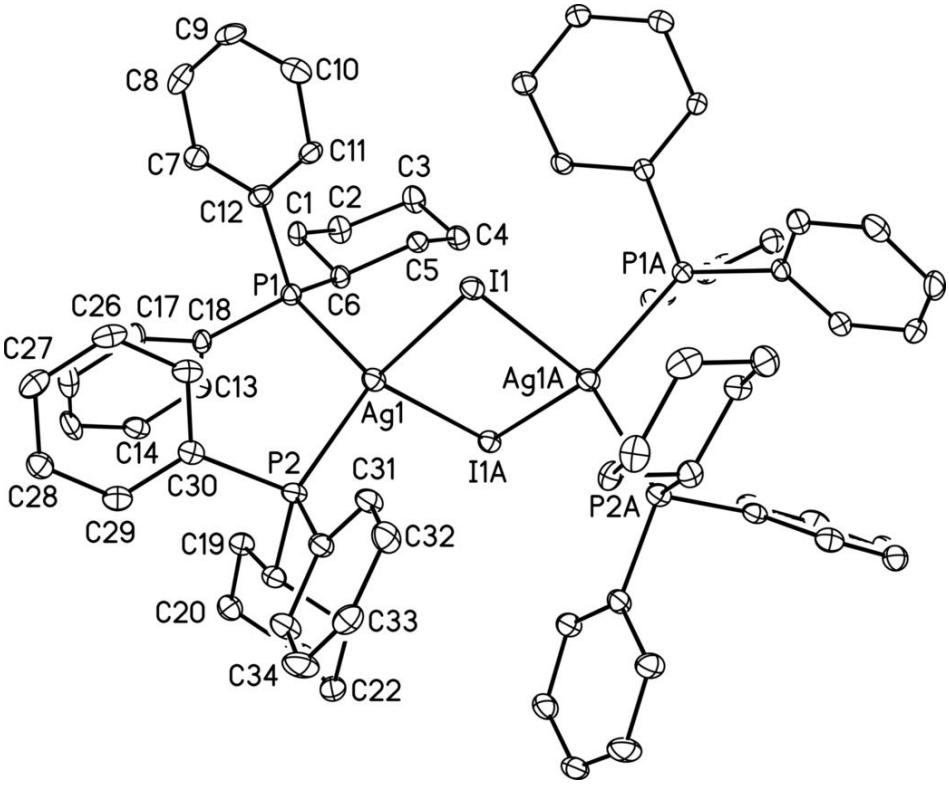
Geometric parameters (Å, °)

Ag1—P2	2.5120 (12)	C13—C14	1.393 (6)
Ag1—P1	2.5485 (12)	C14—C15	1.380 (7)
Ag1—I1	2.8955 (6)	C15—C16	1.360 (7)
Ag1—I1^{i}	2.9245 (7)	C16—C17	1.381 (6)
P1—C12	1.826 (5)	C17—C18	1.395 (6)
P1—C18	1.828 (4)	C19—C24	1.523 (6)
P1—C6	1.856 (4)	C19—C20	1.529 (6)
P2—C36	1.830 (5)	C20—C21	1.511 (6)
P2—C30	1.829 (5)	C21—C22	1.508 (7)
P2—C24	1.837 (5)	C22—C23	1.501 (6)

C1—C6	1.513 (6)	C23—C24	1.520 (6)
C1—C2	1.525 (6)	C25—C30	1.384 (6)
C2—C3	1.510 (6)	C25—C26	1.385 (7)
C3—C4	1.524 (6)	C26—C27	1.388 (7)
C4—C5	1.520 (6)	C27—C28	1.375 (6)
C5—C6	1.524 (6)	C28—C29	1.379 (6)
C7—C8	1.384 (7)	C29—C30	1.391 (6)
C7—C12	1.399 (6)	C31—C36	1.379 (6)
C8—C9	1.391 (7)	C31—C32	1.409 (6)
C9—C10	1.374 (7)	C32—C33	1.369 (6)
C10—C11	1.397 (6)	C33—C34	1.361 (7)
C11—C12	1.396 (6)	C34—C35	1.396 (6)
C13—C18	1.392 (6)	C35—C36	1.414 (6)
P2—Ag1—P1	113.67 (4)	C7—C12—P1	124.0 (4)
P2—Ag1—I1	110.50 (3)	C18—C13—C14	121.1 (5)
P1—Ag1—I1	114.23 (3)	C15—C14—C13	119.7 (5)
P2—Ag1—I1^{i}	111.24 (3)	C16—C15—C14	119.9 (5)
P1—Ag1—I1^{i}	103.01 (3)	C15—C16—C17	120.9 (5)
I1—Ag1—I1^{i}	103.45 (2)	C16—C17—C18	120.9 (5)
Ag1—I1—Ag1^{i}	75.796 (19)	C13—C18—C17	117.6 (4)
C12—P1—C18	104.6 (2)	C13—C18—P1	122.5 (3)
C12—P1—C6	102.7 (2)	C17—C18—P1	119.5 (4)
C18—P1—C6	103.4 (2)	C24—C19—C20	111.5 (4)
C12—P1—Ag1	119.22 (15)	C21—C20—C19	112.1 (4)
C18—P1—Ag1	112.61 (15)	C22—C21—C20	111.9 (4)
C6—P1—Ag1	112.67 (14)	C23—C22—C21	112.4 (4)
C36—P2—C30	98.9 (2)	C22—C23—C24	111.3 (4)
C36—P2—C24	104.0 (2)	C23—C24—C19	111.6 (4)
C30—P2—C24	105.1 (2)	C23—C24—P2	111.0 (3)
C36—P2—Ag1	123.97 (15)	C19—C24—P2	110.1 (3)
C30—P2—Ag1	107.71 (15)	C30—C25—C26	120.4 (5)
C24—P2—Ag1	114.71 (16)	C25—C26—C27	120.4 (5)
C6—C1—C2	111.0 (4)	C28—C27—C26	119.2 (5)
C3—C2—C1	112.9 (4)	C27—C28—C29	120.5 (5)
C2—C3—C4	111.8 (4)	C28—C29—C30	120.8 (4)
C5—C4—C3	112.3 (4)	C25—C30—C29	118.6 (4)
C4—C5—C6	111.2 (4)	C25—C30—P2	116.9 (4)
C1—C6—C5	110.7 (4)	C29—C30—P2	124.3 (4)
C1—C6—P1	116.4 (3)	C36—C31—C32	121.1 (4)
C5—C6—P1	110.5 (3)	C33—C32—C31	119.6 (5)
C8—C7—C12	120.1 (5)	C34—C33—C32	120.5 (5)
C7—C8—C9	120.8 (5)	C33—C34—C35	120.6 (5)
C10—C9—C8	119.8 (5)	C34—C35—C36	120.1 (5)
C9—C10—C11	119.8 (5)	C31—C36—C35	117.9 (4)
C12—C11—C10	121.1 (4)	C31—C36—P2	119.4 (3)
C11—C12—C7	118.4 (4)	C35—C36—P2	122.1 (4)
C11—C12—P1	117.2 (3)		

Symmetry codes: (i) −*x*+2, *y*, −*z*+1/2.

Fig. 1


C9
C10
C8
C11
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C3
C12
C1
C2
C4
C5
P1
C6
I1
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C17
C18
C27
C13
Ag1
Ag1A
C14
P2
C31
I1A
C30
C28
P2A
C29
C19
C32
C20
C33
C34
C22