Crystal structure of bis(μ^2-1,3-bis(diphenylphosphino)propane-κ^2*P,P′*)-bis(acetato-κ^2*O,O′*)disilver(I)-methanol (1:4), $[Ag_2(CH_3COO)_2(C_{27}H_{26}P_2)_2]\cdot 4CH_3OH$, $C_{62}H_{74}Ag_2O_8P_4$

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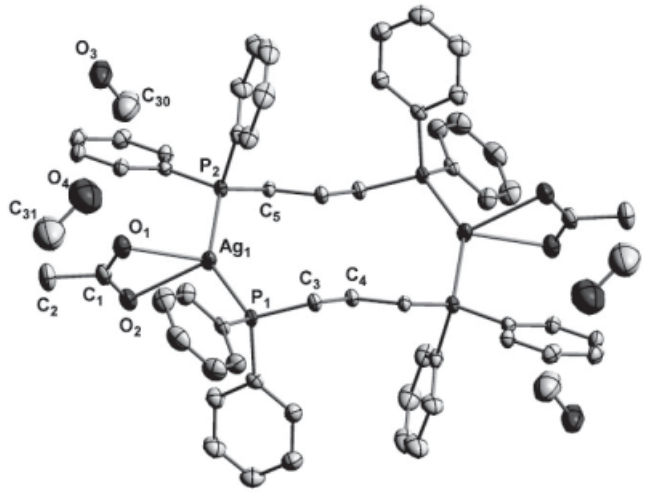

Abstract

$C_{62}H_{74}Ag_2O_8P_4$, monoclinic, $P2_1/n$ (no. 14), a = 10.024(1) Å, b = 17.888(1) Å, c = 17.730(2) Å, β = 106.233(2)°, V = 3052.3 Å^3, Z = 2, $R_{gt}(F)$ = 0.0531, $wR_{ref}(F^2)$ = 0.1483, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.30×0.38×0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.98 cm^{-1}
Diffractometer, scan mode:	Bruker Smart CCD area detector, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15063, 5364
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3592
$N(param)_{refined}$:	346
Programs:	SHELX [10]

Source of material

A mixture of CH_3COOAg (0.2 mmol), 1,3-bis(diphenylphosphino)propane (*dppp*, 0.2 mmol) and isoquinoline (0.5 ml) in the mixed solution of MeOH and CH_2Cl_2 (10 mL, *v/v* = 1:1) was stirred for 6h at room temperature. The insoluble residues were removed by filtration, and the filtrate was allowed to be evaporated slowly at room temperature for a week to yield colourless crystalline products.

Experimental details

The oxygen atoms O3 and O4 belong to two uncoordinated methanol molecules which lead to easy dispersion, leading to high U values, including the involved hydrogen atoms.

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Discussion

The coordination chemistry of silver(I) complexes with diphosphine ligands has been extensively studied [1-5]. We are interested in synthesizing new Ag(I) complexes containing diphosphine ligands and isoquinoline. Recently, we reported the first silver(I) complex $[Ag_2(dppm)_2(C_9H_7N)_2](CF_3SO_3)_2\cdot C_9H_7N$, which contains isoquinoline and bis(diphenylphosphine)methane [6]. The title complex $[Ag_2(CH_3COO)_2(C_{27}H_{26}P_2)_2]\cdot 4CH_3OH$, is unexpectedly obtained in the course of synthesizing Ag(I) complexes containing 1,3-bis(diphenylphosphino)propane ligands and isoquinoline. In the title structure, each silver atom is coordinated by two phosphorus atoms from two *dppp* ligands, and each of the two CH_3COO^- anions chelates an Ag atom with Ag–O bond distances of 2.520(4) and 2.550(5) Å to form a twelve-membered ring. Such a coordination mode is not common because CH_3COO^- in oligonuclear Ag(I) complexes containing diphosphine ligands usually acts as a bridging ligand [7-9]. The Ag–P bond lengths (2.424(2) and 2.439(2) Å) agree with those in $[Ag(CH_3OO)dppp]_2\cdot 2MeCN$ [7]. The coordination sphere around the Ag(I) atoms is a distorted tetrahedron, which is confirmed by the angles P–Ag–P and O–Ag–P (P(1)–Ag(1)–P(2) = 143.5(1)°, P(1)–Ag(1)–O(2) = 108.1(1)°, O(1)–Ag(1)–O(2) = 51.3(2)°, O(1)–Ag(1)–P(2) = 102.8(1)°). Moreover, intramolecular O–H···O hydrogen bonds between methanol molecules and the coordinated acetate ligand is observed.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	0.6464	0.8294	0.1765	0.161
H(4)	4*e*	0.7662	0.8558	0.4088	0.264
H(2A)	4*e*	1.0835	0.8378	0.5245	0.136
H(2B)	4*e*	1.1699	0.7640	0.5299	0.136
H(2C)	4*e*	1.1535	0.8018	0.6066	0.136
H(3A)	4*e*	0.3677	0.6655	0.6067	0.053
H(3B)	4*e*	0.3713	0.6638	0.5190	0.053
H(4A)	4*e*	0.4194	0.5399	0.5231	0.052
H(4B)	4*e*	0.4725	0.5389	0.6153	0.052
H(5A)	4*e*	0.7597	0.4368	0.3807	0.048
H(5B)	4*e*	0.8089	0.4435	0.4724	0.048
H(7)	4*e*	0.6382	0.8092	0.5143	0.082
H(8)	4*e*	0.5960	0.9373	0.5177	0.110
H(9)	4*e*	0.4967	0.9843	0.6100	0.102
H(10)	4*e*	0.4300	0.9068	0.6937	0.102
H(11)	4*e*	0.4757	0.7806	0.6929	0.082
H(13)	4*e*	0.4832	0.6352	0.7308	0.075
H(14)	4*e*	0.5794	0.6131	0.8626	0.095
H(15)	4*e*	0.8150	0.6213	0.9162	0.105
H(16)	4*e*	0.9555	0.6509	0.8401	0.103
H(17)	4*e*	0.8619	0.6714	0.7067	0.080

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(19)	4*e*	0.5318	0.6594	0.3653	0.094
H(20)	4*e*	0.3663	0.6823	0.2467	0.121
H(21)	4*e*	0.3717	0.6190	0.1382	0.130
H(22)	4*e*	0.5439	0.5342	0.1397	0.134
H(23)	4*e*	0.7171	0.5131	0.2572	0.097
H(25)	4*e*	0.9738	0.4587	0.3835	0.062
H(26)	4*e*	1.1808	0.4745	0.3514	0.076
H(27)	4*e*	1.2675	0.5929	0.3443	0.078

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(28)	4*e*	1.1475	0.6950	0.3676	0.075
H(29)	4*e*	0.9401	0.6809	0.3977	0.062
H(30A)	4*e*	0.6923	0.7355	0.2426	0.263
H(30B)	4*e*	0.6803	0.7946	0.3058	0.263
H(30C)	4*e*	0.8263	0.7745	0.2952	0.263
H(31A)	4*e*	0.8472	0.9835	0.4705	0.368
H(31B)	4*e*	0.9592	0.9359	0.4456	0.368
H(31C)	4*e*	0.8894	0.9046	0.5079	0.368

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ag(1)	4*e*	0.74586(5)	0.64598(3)	0.52125(3)	0.0479(3)	0.0468(3)	0.0683(4)	–0.0052(2)	0.0222(2)	–0.0096(2)
O(1)	4*e*	0.8701(5)	0.7634(3)	0.4959(3)	0.062(3)	0.065(3)	0.097(4)	–0.002(3)	0.026(3)	–0.005(3)
O(2)	4*e*	0.9879(5)	0.6904(3)	0.5899(3)	0.059(3)	0.067(3)	0.083(4)	–0.012(2)	0.013(3)	–0.011(3)
O(3)	4*e*	0.7152(7)	0.8375(4)	0.2133(5)	0.102(5)	0.149(7)	0.137(6)	–0.023(5)	0.006(5)	0.051(5)
O(4)	4*e*	0.769(1)	0.9007(7)	0.4004(7)	0.20(1)	0.22(1)	0.21(1)	–0.007(9)	0.006(9)	0.03(1)
P(1)	4*e*	0.5948(2)	0.68101(8)	0.60084(9)	0.0369(8)	0.0348(7)	0.0523(9)	–0.0057(6)	0.0125(7)	–0.0071(7)
P(2)	4*e*	0.7676(1)	0.56212(8)	0.41686(9)	0.0326(7)	0.0361(7)	0.0489(9)	–0.0026(6)	0.0095(7)	–0.0008(7)
C(1)	4*e*	0.9776(8)	0.7458(4)	0.5459(5)	0.060(5)	0.055(4)	0.084(6)	–0.012(4)	0.025(4)	–0.023(4)
C(2)	4*e*	1.1081(8)	0.7916(5)	0.5523(6)	0.065(5)	0.074(5)	0.133(8)	–0.021(4)	0.028(5)	–0.010(5)
C(3)	4*e*	0.4170(6)	0.6453(3)	0.5713(4)	0.037(3)	0.038(3)	0.057(4)	–0.005(3)	0.012(3)	–0.007(3)
C(4)	4*e*	0.4034(5)	0.5599(3)	0.5707(4)	0.035(3)	0.039(3)	0.056(4)	–0.005(2)	0.014(3)	–0.006(3)
C(5)	4*e*	0.7415(5)	0.4617(3)	0.4254(3)	0.032(3)	0.040(3)	0.046(3)	–0.005(2)	0.008(3)	0.003(3)
C(6)	4*e*	0.5629(6)	0.7814(3)	0.6037(4)	0.045(3)	0.038(3)	0.058(4)	–0.005(3)	0.005(3)	–0.004(3)
C(7)	4*e*	0.5975(7)	0.8284(4)	0.5514(5)	0.065(5)	0.056(4)	0.083(5)	0.006(3)	0.018(4)	0.011(4)
C(8)	4*e*	0.572(1)	0.9053(5)	0.5532(6)	0.098(7)	0.061(5)	0.106(7)	0.009(5)	0.011(6)	0.029(5)
C(9)	4*e*	0.5117(9)	0.9331(4)	0.6079(6)	0.096(6)	0.043(4)	0.100(7)	0.011(4)	–0.001(5)	–0.002(5)
C(10)	4*e*	0.4739(9)	0.8875(4)	0.6582(5)	0.105(7)	0.055(5)	0.092(6)	0.016(4)	0.024(5)	–0.011(5)
C(11)	4*e*	0.5004(8)	0.8118(4)	0.6571(4)	0.085(5)	0.048(4)	0.075(5)	0.003(4)	0.028(4)	–0.006(4)
C(12)	4*e*	0.6620(6)	0.6569(3)	0.7043(4)	0.050(4)	0.045(3)	0.051(4)	0.001(3)	0.008(3)	–0.004(3)
C(13)	4*e*	0.5789(7)	0.6386(4)	0.7522(4)	0.057(4)	0.070(4)	0.060(4)	–0.004(3)	0.015(4)	–0.001(4)
C(14)	4*e*	0.6362(9)	0.6253(5)	0.8310(5)	0.084(6)	0.088(6)	0.066(5)	–0.006(4)	0.022(5)	0.007(4)
C(15)	4*e*	0.777(1)	0.6301(5)	0.8629(5)	0.090(7)	0.094(6)	0.069(5)	0.015(5)	0.006(5)	0.018(5)
C(16)	4*e*	0.8600(9)	0.6475(5)	0.8178(5)	0.065(5)	0.098(6)	0.080(6)	0.006(5)	–0.004(5)	0.012(5)
C(17)	4*e*	0.8036(7)	0.6604(4)	0.7378(5)	0.052(4)	0.071(5)	0.072(5)	0.002(3)	0.010(4)	0.003(4)
C(18)	4*e*	0.6423(6)	0.5833(3)	0.3247(4)	0.036(3)	0.052(4)	0.063(4)	–0.002(3)	0.008(3)	0.014(3)
C(19)	4*e*	0.5364(8)	0.6340(4)	0.3203(5)	0.056(4)	0.085(6)	0.092(6)	0.010(4)	0.016(4)	0.032(5)
C(20)	4*e*	0.4360(9)	0.6474(6)	0.2491(7)	0.059(5)	0.116(8)	0.117(8)	0.019(5)	0.008(6)	0.049(7)
C(21)	4*e*	0.440(1)	0.6102(7)	0.1848(7)	0.073(6)	0.124(9)	0.104(8)	–0.012(6)	–0.016(6)	0.049(7)
C(22)	4*e*	0.543(1)	0.5596(6)	0.1853(6)	0.103(8)	0.113(8)	0.092(7)	–0.012(6)	–0.017(6)	0.027(6)
C(23)	4*e*	0.6457(8)	0.5466(5)	0.2561(5)	0.071(5)	0.084(6)	0.071(5)	0.004(4)	–0.005(4)	0.017(5)
C(24)	4*e*	0.9341(6)	0.5687(3)	0.3927(3)	0.037(3)	0.045(3)	0.042(3)	–0.002(3)	0.010(3)	0.001(3)
C(25)	4*e*	1.0078(6)	0.5065(4)	0.3797(4)	0.041(3)	0.056(4)	0.059(4)	0.002(3)	0.015(3)	0.001(3)
C(26)	4*e*	1.1322(7)	0.5160(4)	0.3610(4)	0.050(4)	0.076(5)	0.067(5)	0.014(4)	0.023(3)	0.008(4)
C(27)	4*e*	1.1839(7)	0.5867(4)	0.3566(4)	0.045(4)	0.084(5)	0.069(5)	–0.005(4)	0.023(4)	0.008(4)
C(28)	4*e*	1.1122(7)	0.6473(4)	0.3704(4)	0.056(4)	0.066(4)	0.068(5)	–0.018(4)	0.021(4)	0.007(4)
C(29)	4*e*	0.9879(6)	0.6389(4)	0.3884(4)	0.044(3)	0.054(4)	0.058(4)	–0.005(3)	0.016(3)	0.002(3)
C(30)	4*e*	0.730(1)	0.7812(9)	0.2685(8)	0.15(1)	0.21(2)	0.16(1)	–0.02(1)	0.03(1)	0.07(1)
C(31)	4*e*	0.875(2)	0.934(1)	0.461(1)	0.23(2)	0.28(2)	0.20(2)	–0.07(2)	0.01(2)	0.07(2)

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References

1. Fournier, E.; Sicard, S.; Decken, A.; Harvey, P. D.: Organometallic Oligomers Based on 1,8-Diisocyano-p-menthane (dmb):Syntheses and Characterization of the $\{[M(\textit{diphos})(\textit{dmb})]BF_4\}_n$ and $\{[Pd_2(\textit{diphos})_2(\textit{dmb})](ClO_4)_2\}_n$ Materials (M = Cu, Ag; *diphos* = dppe, dppp). Inorg. Chem. **43** (2004) 1491-1501.
2. Brandys, M. C.; Puddephatt, R. J.: Ring, polymer and network structures in silver(I) complexes with dipyridyl and diphosphine ligands. Chem. Commun. (2001) 1508-1509.
3. Cui, L. N.; Zhao, X.; Jiang, Y. H.; Li, Z. F.; Jin, Q. H.; Zhang, C. L.: Crystal structure of catena-{bis(μ-acetato-κ*O,O′:O′*)[μ-bis(diphenylphosphino)-butane-κ*P,P′*]disilver(I)}, $Ag_2(C_2H_3O_2)_2(C_{28}H_{28}P_2)$. Z. Kristallogr. NCS **226** (2011) 627-628.
4. Fournier, E.; Lebrun, F.; Drouin, M.; Decken, A.; Harvey, P. D.: Preparation and Solid-State Characterization of Mixed-Ligand Coordination/Organometallic Oligomers and Polymers of Copper(I) and Silver(I) Using Diphosphine and Mono- and Diisocyanide Ligands. Inorg. Chem. **43** (2004) 3127-3135.
5. Jin, Q. H.; Hu, K. Y.; Chen, L. M.; Sun, J. J.; Yang, L.; Li, P. Z.: Crystal structure of tetrakis(μ-thiocyanato)bis(μ-(bis(diphenylphosphino)methane-*P,P′*))tetrasilver(I), $Ag_4(SCN)_4(C_{25}H_{22}P_2)_2$. Z. Kristallogr. NCS. **223** (2008) 79-81.
6. Huang, X.; Li, J.; Qiu, Q. M.; Liu, M.; Jin, Q. H.: Bis[μ-bis(diphenylphosphanyl)methane-κ^2*P:P′*]bis[(isoquinoline-κ*N*)silver(I)] bis(trifluoromethanesulfonate)-isoquinoline (1/1). Acta Crystallogr. **E68** (2012) m1022-m1023.
7. Effendy; Di Nicola, C.; Fianchini, M.; Pettinari, C.; Skelton, B. W.; Somers, N.; White, A. H.: The structural definition of adducts of stoichiometry MX: dppx (1:1) M = Cu^I, Ag^I, X = simple anion, dppx = $Ph_2P(CH_2)_xPPh_2$, x = 3-6. Inorg. Chim. Acta. **358** (2005) 763-795.
8. Cingolani, A.; Effendy; Pettinari, C.; Skelton, B. W.; White, A. H.:Novel solvates in AgX: dpex (n:1) systems (n = 1 or 2); X = oxyanion; dppx = $Ph_2P(Y)PPh_2$, Y = $(CH_2)_3$, Fc. Inorg. Chim. Acta. **359** (2006) 2170-2177.
9. Ohkouchi, M.; Masui, D.; Yamaguchi, M.; Yamagishi, T.: Mechanism of silver(I)-catalyzed Mukaiyama aldol reaction: active species in solution in $AgPF_6$-(S)-BINAP versus AgOAc-(S)-BINAP systems. J. Molecular Catalysis A: Chemical. **170** (2001) 1-16.
10. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.