Crystal structure of *catena*-{bis(μ-acetato-*O*,*O*′:*O*′)[μ-bis(diphenylphosphino)butane-*P*,*P*′]disilver(I)}, $Ag_2(C_2H_3O_2)_2(C_{28}H_{28}P_2)$

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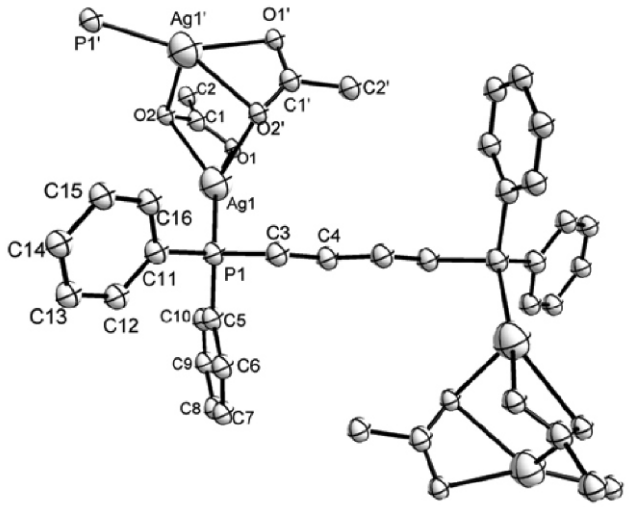

Abstract

$C_{32}H_{34}Ag_2O_4P_2$, monoclinic, *C*12/*c*1 (no. 15), a = 19.718(2) Å, b = 14.107(1) Å, c = 11.258(1) Å, β = 98.308(1)°, V = 3098.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.109, T = 298 K.

Source of material

A new silver complex $Ag_2(CH_3COO)_2$(dppb) has been prepared under the catalysis of phenathroline. Bis(diphenylphosphino)-butane (dppb, 0.2 mmol, 0.0853 g) and phenanthroline (phen, 0.2 mmol, 0.0396 g) were added into the stirred solution of $Ag(CH_3COO)$ (0.2 mmol, 0.0355 g) in a mixture of CH_3CN (5 ml) and CH_3OH (5 ml). Stirring was continued for 2 h at room temperature, then the white precipitate was filtered off. Subsequent slow evaporation of the colorless filtrate at ambient temperature resulted in the formation of colorless crystals of the title complex. Crystals suitable for single crystal X-ray diffraction were selected directly from the sample as prepared.

Discussion

Reports concerned with the structural and kinetic features of silver(I)-phosphine-oligodentate N-based complexes are growing in number because their lunminescence properties and participation of these compounds in biological process are discovered [1-3]. We found that some nitrogen heterocyclic ligands as catalysts play an important role in the formation of products of mixed P- and N-ligands with special structures. For examples, $[Ag_4SCN_4dppm_2]$ and $[AgSCNdppm]_2$ were obtained with quinoline and phenathroline as catalysts, respectively. $[AgClO_4(PPh_3)_3]$ [4], $[AgClO_4(PPh_3)_3(MeOH)]$ [5] and $[Ag(PPh_3)(CH_3COO)]_2 \cdot H_2O \cdot CH_3OH$ [6] were prepared with 2-aminopyrimidine as catalyst.

Each silver atom is coordinated by one P atom from dppb and three O atoms from two CH_3COO^- anions.The coordination sphere of the Ag(I) atom is a distorted tetrahedron, which is confirmed by the angles around the silver atom, ∠P1–Ag1–O1 = 155.96(1)°, ∠P1–Ag1–O2 = 145.36(9)°, ∠P1–Ag1–O2' = 92.65(9)° and ∠O1–Ag1–O2' = 101.65(1)°. In the title complex, CH_3COO bridges two Ag atoms through one μ_2-O atom and chelates the Ag atom through two O atoms. The bond distance d(Ag—P) = 2.3271(1) Å is shorter than those in similar complexes AgX · dppb (X = Br, I, CN, ClO_4, NO_3, CF_3COO) [7]. The bond distances d(Ag—O) = 2.2730(4)-2.6386(4) Å are shorter than those in the similar complex $Ag(CH_3COO)$ · dppp (2.497(3) Å and 2.383(2) Å) [7]. No significant hydrogen bonds are observed in the crystal structure.

In three similar compounds $Ag(CH_3COO)$ · dppp, $Ag(CH_3COO)$ · dppb and $Ag(CH_3COO)$ · dppm (dppp = bis(diphenylphosphino)propane and dppm = bis(diphenylphosphino)methane), the CH_3COO^- anion acts as bridging, bridging-chelating and chelating ligand, respectively.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.22 × 0.36 × 0.43 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.02 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7895, 2737
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1885
$N(param)_{refined}$:	182
Programs:	SHELXS-97, SHELXL-97, SHELXTL [8]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	8*f*	0.4540	0.9399	0.4768	0.130
H(2B)	8*f*	0.3930	0.8705	0.4866	0.130
H(2C)	8*f*	0.4428	0.8509	0.3928	0.130
H(3A)	8*f*	0.6647	0.6409	0.9840	0.053
H(3B)	8*f*	0.7334	0.5910	0.9653	0.053
H(4A)	8*f*	0.7732	0.7320	0.8894	0.054
H(4B)	8*f*	0.7036	0.7823	0.9011	0.054
H(6)	8*f*	0.7951	0.5116	0.8269	0.063
H(7)	8*f*	0.8879	0.4982	0.7241	0.073
H(8)	8*f*	0.8836	0.5687	0.5386	0.079
H(9)	8*f*	0.7878	0.6492	0.4556	0.071

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Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(10)	8*f*	0.6946	0.6583	0.5542	0.061
H(12)	8*f*	0.6877	0.4072	0.7147	0.058
H(13)	8*f*	0.6434	0.2628	0.7606	0.073

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(14)	8*f*	0.5562	0.2544	0.8741	0.070
H(15)	8*f*	0.5145	0.3914	0.9468	0.077
H(16)	8*f*	0.5569	0.5358	0.9011	0.067

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)	8*f*	0.58113(2)	0.71529(3)	0.70204(4)	0.0613(3)	0.0676(3)	0.0613(3)	0.0178(2)	0.0047(2)	0.0052(2)
O(1)	8*f*	0.5424(2)	0.8491(3)	0.6020(3)	0.062(3)	0.067(2)	0.075(3)	0.000(2)	0.007(2)	0.017(2)
O(2)	8*f*	0.4645(2)	0.7385(3)	0.5908(3)	0.069(3)	0.070(3)	0.066(2)	–0.003(2)	0.020(2)	0.004(2)
P(1)	8*f*	0.66255(6)	0.60441(8)	0.7819(1)	0.0431(8)	0.0403(7)	0.0364(6)	–0.0002(5)	0.0063(5)	–0.0012(5)
C(1)	8*f*	0.4847(3)	0.8174(4)	0.5631(5)	0.056(4)	0.050(3)	0.064(3)	0.010(3)	0.023(3)	0.000(3)
C(2)	8*f*	0.4396(3)	0.8748(5)	0.4716(6)	0.075(5)	0.080(4)	0.099(5)	0.025(4)	–0.006(4)	0.007(4)
C(3)	8*f*	0.7003(3)	0.6388(3)	0.9331(4)	0.053(3)	0.044(3)	0.036(2)	–0.004(2)	0.009(2)	–0.004(2)
C(4)	8*f*	0.7360(3)	0.7350(3)	0.9369(4)	0.059(3)	0.043(3)	0.033(2)	–0.001(2)	0.006(2)	–0.003(2)
C(5)	8*f*	0.7350(2)	0.5872(3)	0.7029(4)	0.048(3)	0.035(2)	0.034(2)	–0.004(2)	0.004(2)	–0.001(2)
C(6)	8*f*	0.7932(3)	0.5393(4)	0.7515(4)	0.055(4)	0.059(3)	0.044(3)	0.002(3)	0.010(2)	0.003(2)
C(7)	8*f*	0.8491(3)	0.5313(4)	0.6906(5)	0.047(3)	0.067(4)	0.070(4)	–0.003(3)	0.013(3)	–0.012(3)
C(8)	8*f*	0.8462(3)	0.5730(4)	0.5801(5)	0.060(4)	0.067(4)	0.078(4)	–0.022(3)	0.035(3)	–0.024(3)
C(9)	8*f*	0.7891(3)	0.6207(4)	0.5304(5)	0.081(4)	0.052(3)	0.050(3)	–0.016(3)	0.027(3)	–0.007(2)
C(10)	8*f*	0.7338(3)	0.6269(3)	0.5899(4)	0.065(4)	0.044(3)	0.042(3)	–0.005(2)	0.007(2)	0.000(2)
C(11)	8*f*	0.6281(2)	0.4871(3)	0.8064(4)	0.045(3)	0.044(3)	0.034(2)	–0.002(2)	0.006(2)	–0.003(2)
C(12)	8*f*	0.6529(3)	0.4045(3)	0.7624(4)	0.051(3)	0.051(3)	0.044(3)	–0.007(2)	0.012(2)	–0.008(2)
C(13)	8*f*	0.6260(3)	0.3182(4)	0.7892(5)	0.072(4)	0.049(3)	0.062(3)	–0.008(3)	0.012(3)	–0.011(2)
C(14)	8*f*	0.5743(3)	0.3128(4)	0.8570(5)	0.059(4)	0.053(3)	0.064(3)	–0.017(3)	0.011(3)	0.003(3)
C(15)	8*f*	0.5494(3)	0.3944(4)	0.8995(5)	0.057(4)	0.071(4)	0.068(4)	–0.012(3)	0.025(3)	0.001(3)
C(16)	8*f*	0.5753(3)	0.4807(4)	0.8733(4)	0.052(3)	0.055(3)	0.064(3)	–0.004(3)	0.022(3)	–0.009(2)

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