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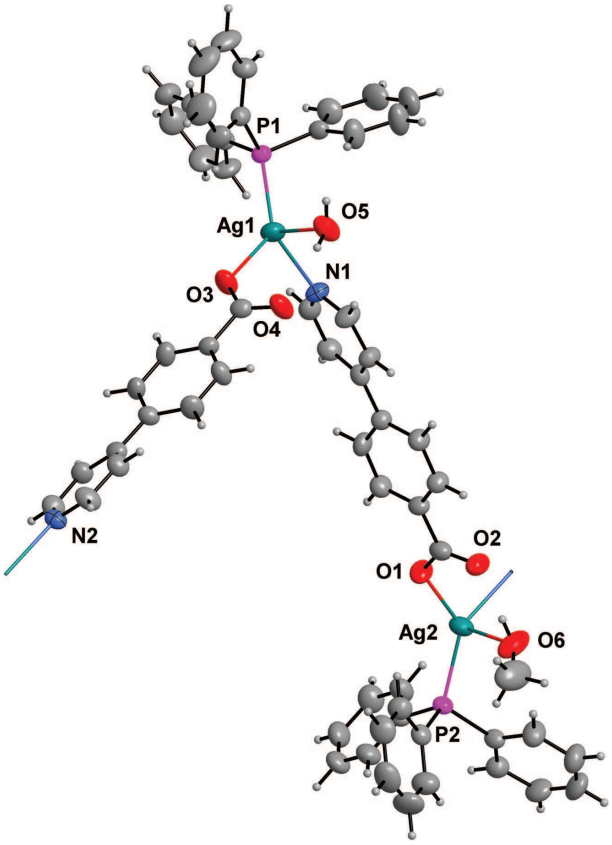
Crystal structure of *catena*-poly[aqua-(methanol-κO)-bis(μ$_2$-4-(pyridin-4-yl)benzoato-κ^2*N*:*O*)-bis(triphenylphospine-κP)disilver(I)], $C_{61}H_{52}Ag_2N_2O_6P_2$



Abstract

$C_{61}H_{52}Ag_2N_2O_6P_2$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 14.4634(11)$ Å, $b = 17.7866(18)$ Å, $c = 20.5509(19)$ Å, $V = 5286.8(8)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0424$, $wR_{ref}(F^2) = 0.0997$, $T = 298(2)$ K.

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A part of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.40 × 0.32 × 0.21 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.86 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	26.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	29489, 10668, 0.065
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6633
$N(param)_{refined}$:	660
Programs:	Bruker programs [1], SHELX [2]

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Source of material

The title compound was obtained by the reaction of silver(I) sulfate (0.0624 g, 0.2 mmol) and triphenylphosphine (PPh_3) (0.0525 g, 0.2 mmol) in the presence of 4-pyridin-4-yl-benzoic acid (0.0398 g, 0.2 mmol) in the mixed solution of CH_3OH (5 mL) and CH_2Cl_2 (5 mL) for 6 h at ambient temperature. The insoluble residues were removed by filtration, and the filtrate was evaporated slowly at room temperature for about 1 week to yield transparent and colorless crystals. Crystals suitable for single-crystal X-ray diffraction were selected directly from the sample as prepared.

Experimental details

Multi-scan absorption corrections were applied using SADABS program. All the structures were solved by direct methods using SHELXS program of the SHELX-2014/6 package and refined with SHELXL-2014/6 [2]. The final

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ag1	0.74447(4)	0.52780(4)	0.60076(3)	0.04366(18)
Ag2	0.01417(4)	0.02818(4)	0.57824(3)	0.04327(18)
N1	0.6348(4)	0.4342(4)	0.6128(3)	0.0399(18)
N2	0.1310(4)	0.9452(3)	0.5934(3)	0.0372(17)
O1	0.1115(4)	0.1341(3)	0.5870(3)	0.0502(16)
O2	0.1299(4)	0.1272(3)	0.6947(3)	0.0551(17)
O3	0.6351(4)	0.6245(3)	0.6041(3)	0.0518(17)
O4	0.5913(4)	0.6091(3)	0.7064(3)	0.0550(18)
O5	0.7537(4)	0.5421(3)	0.7235(2)	0.0568(16)
H5B	0.7131	0.5732	0.7365	0.068*
H5C	0.8067	0.5588	0.7341	0.068*
O6	−0.0199(5)	0.0454(4)	0.7039(3)	0.072(2)
H6C	0.0273	0.0706	0.7007	0.086*
P1	0.89828(12)	0.52371(13)	0.56126(9)	0.0327(5)
P2	−0.14252(13)	0.02221(13)	0.54702(10)	0.0342(5)
C1	0.1533(6)	0.1497(5)	0.6383(5)	0.043(2)
C2	0.2369(6)	0.2008(4)	0.6348(4)	0.036(2)
C3	0.2828(5)	0.2229(4)	0.6903(4)	0.040(2)
H3	0.2604	0.2085	0.7309	0.048*
C4	0.3623(6)	0.2663(5)	0.6861(4)	0.042(2)
H4	0.3923	0.2805	0.7242	0.051*
C5	0.3978(5)	0.2889(4)	0.6274(4)	0.034(2)
C6	0.3503(6)	0.2665(4)	0.5715(4)	0.043(2)
H6	0.3725	0.2811	0.5310	0.052*
C7	0.2714(6)	0.2236(4)	0.5749(4)	0.044(2)
H7	0.2410	0.2097	0.5369	0.053*
C8	0.5901(6)	0.4241(5)	0.6691(4)	0.047(2)
H8	0.6107	0.4502	0.7056	0.057*
C9	0.5140(6)	0.3763(4)	0.6758(4)	0.041(2)
H9	0.4854	0.3707	0.7160	0.049*
C10	0.4813(6)	0.3374(4)	0.6220(4)	0.035(2)
C11	0.5297(6)	0.3466(5)	0.5644(4)	0.043(2)
H11	0.5115	0.3208	0.5272	0.052*
C12	0.6051(6)	0.3941(5)	0.5624(4)	0.046(2)
H12	0.6371	0.3983	0.5233	0.055*
C13	0.5830(6)	0.6372(5)	0.6509(5)	0.036(2)
C14	0.5024(6)	0.6910(4)	0.6407(4)	0.034(2)
C15	0.4480(6)	0.7130(5)	0.6913(4)	0.046(2)
H15	0.4606	0.6947	0.7328	0.055*
C16	0.3750(6)	0.7614(5)	0.6826(4)	0.048(2)
H16	0.3395	0.7760	0.7182	0.058*
C17	0.3539(5)	0.7888(4)	0.6208(4)	0.035(2)
C18	0.4087(5)	0.7662(4)	0.5695(4)	0.040(2)
H18	0.3961	0.7841	0.5279	0.048*
C19	0.4820(6)	0.7173(4)	0.5786(4)	0.044(2)
H19	0.5176	0.7020	0.5433	0.053*
C20	0.1743(6)	0.9389(5)	0.6505(4)	0.047(2)
H20	0.1550	0.9690	0.6849	0.056*
C21	0.2462(6)	0.8898(4)	0.6607(4)	0.042(2)
H21	0.2745	0.8880	0.7013	0.050*
C22	0.2772(5)	0.8429(4)	0.6114(4)	0.037(2)
C23	0.2320(6)	0.8512(4)	0.5520(4)	0.041(2)
H23	0.2508	0.8227	0.5165	0.049*
C24	0.1598(6)	0.9009(5)	0.5451(4)	0.042(2)
H24	0.1298	0.9038	0.5052	0.051*
C25	0.9656(5)	0.6043(4)	0.5886(4)	0.035(2)
C26	0.9186(6)	0.6682(5)	0.6068(5)	0.052(2)
H26	0.8544	0.6690	0.6063	0.062*
C27	0.9674(9)	0.7312(5)	0.6259(5)	0.073(3)
H27	0.9355	0.7739	0.6391	0.087*
C28	1.0623(9)	0.7317(6)	0.6256(6)	0.077(4)
H28	1.0944	0.7748	0.6377	0.092*
C29	1.1088(7)	0.6689(6)	0.6076(4)	0.060(3)
H29	1.1731	0.6690	0.6074	0.071*
C30	1.0611(6)	0.6045(5)	0.5895(4)	0.045(2)
H30	1.0936	0.5614	0.5779	0.054*
C31	0.9557(5)	0.4425(4)	0.5987(4)	0.034(2)
C32	0.9506(7)	0.4358(5)	0.6649(4)	0.058(3)
H32	0.9220	0.4737	0.6888	0.070*
C33	0.9863(8)	0.3751(6)	0.6971(5)	0.070(3)
H33	0.9829	0.3723	0.7423	0.084*
C34	1.0272(8)	0.3183(6)	0.6624(6)	0.068(3)
H34	1.0506	0.2765	0.6840	0.082*
C35	1.0336(7)	0.3229(5)	0.5971(6)	0.062(3)
H35	1.0616	0.2845	0.5736	0.074*
C36	0.9977(6)	0.3860(5)	0.5648(4)	0.052(2)
H36	1.0027	0.3894	0.5198	0.063*
C37	0.9250(5)	0.5144(4)	0.4748(4)	0.0339(19)
C38	0.9926(6)	0.5550(4)	0.4436(4)	0.044(2)
H38	1.0268	0.5905	0.4665	0.052*
C39	1.0098(6)	0.5431(5)	0.3783(4)	0.055(3)
H39	1.0552	0.5713	0.3575	0.066*
C40	0.9613(7)	0.4907(5)	0.3438(4)	0.058(3)
H40	0.9755	0.4815	0.3004	0.070*
C41	0.8918(7)	0.4520(5)	0.3738(4)	0.057(3)
H41	0.8576	0.4169	0.3505	0.069*
C42	0.8720(5)	0.4651(5)	0.4387(4)	0.050(2)
H42	0.8225	0.4404	0.4582	0.061*
C43	−0.2084(5)	0.1004(4)	0.5806(4)	0.035(2)
C44	−0.1624(6)	0.1663(5)	0.5949(4)	0.047(2)
H44	−0.0991	0.1698	0.5876	0.057*
C45	−0.2099(7)	0.2270(5)	0.6203(5)	0.058(3)
H45	−0.1788	0.2715	0.6293	0.070*
C46	−0.3036(8)	0.2214(6)	0.6320(5)	0.069(3)
H46	−0.3355	0.2624	0.6490	0.082*
C47	−0.3491(7)	0.1571(6)	0.6192(5)	0.069(3)
H47	−0.4121	0.1540	0.6279	0.083*
C48	−0.3038(6)	0.0958(5)	0.5932(5)	0.059(3)
H48	−0.3361	0.0518	0.5841	0.071*
C49	−0.1995(5)	−0.0607(4)	0.5804(4)	0.035(2)
C50	−0.1862(7)	−0.0758(5)	0.6459(5)	0.059(3)
H50	−0.1494	−0.0441	0.6709	0.070*
C51	−0.2276(7)	−0.1381(5)	0.6745(5)	0.064(3)
H51	−0.2206	−0.1469	0.7188	0.076*
C52	−0.2790(8)	−0.1870(5)	0.6369(6)	0.061(3)
H52	−0.3061	−0.2290	0.6559	0.074*
C53	−0.2901(6)	−0.1741(5)	0.5722(6)	0.057(3)
H53	−0.3239	−0.2077	0.5469	0.069*
C54	−0.2512(6)	−0.1106(4)	0.5435(4)	0.046(2)
H54	−0.2599	−0.1017	0.4994	0.055*
C55	−0.1698(5)	0.0195(5)	0.4605(4)	0.0356(19)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C56	−0.2466(6)	0.0555(4)	0.4336(4)	0.049(2)
H56	−0.2862	0.0829	0.4602	0.059*
C57	−0.2640(6)	0.0506(5)	0.3679(4)	0.053(3)
H57	−0.3155	0.0745	0.3504	0.064*
C58	−0.2055(7)	0.0104(5)	0.3279(4)	0.058(3)
H58	−0.2170	0.0078	0.2834	0.069*
C59	−0.1304(6)	−0.0254(6)	0.3541(4)	0.060(2)
H59	−0.0918	−0.0537	0.3274	0.072*
C60	−0.1116(6)	−0.0200(5)	0.4200(4)	0.051(2)
H60	−0.0594	−0.0432	0.4371	0.061*
C61	−0.0840(8)	0.0833(8)	0.7429(5)	0.097(4)
H61A	−0.0599	0.1318	0.7543	0.146*
H61B	−0.0950	0.0547	0.7818	0.146*
H61C	−0.1409	0.0892	0.7195	0.146*

refinements were performed by full-matrix least-squares methods with anisotropic thermal parameters for non-hydrogen atoms on F^2. The hydrogen atoms were generated geometrically and refined with fixed displacement parameters riding on the concerned atoms. The structure was refined as an inversion twin with a 0.22(4)/0.78(4) ratio of the twin components [2].

Discussion

During the past decades, a large number of silver(I) complexes with interesting structures and properties have been reported. The targeted synthesis of silver(I) complexes with designed structures and properties is still a complex task, considering that the structures and properties of complexes can be influenced by many factors, such as organic ligands, metal ions, solvent systems, and metal-to-ligand ratios [3]. These ligands including nitrogen and oxygen atoms have received widespread attention in the study of coordination chemistry. Meanwhile, heterocyclic ligands containing nitrogen and carboxyl groups are employed in many laboratories in recent years. A great deal of attention has been paid on ligands which have heterocyclic nitrogen atoms and carboxylate groups mainly because of their potential applications [4, 5]. As known to all, silver(I) salts are classified as a kind of soft acid. According to the Hard-Soft-Acid-Base (HSAB) theory, P-donor and N-donor ligands can easily coordinate to Ag(I) salts [6].

The asymmetric unit of the title structure is comprised of two Ag(I) atoms, two PPh_3 ligands, two 4-pyridin-4-yl-benzoate (4,4-pybz) ligands, one water molecule and one methanol molecule. Both silver(I) metal centers are fourfold coordinated (*cf*. the figure) to one oxygen atom from a carboxyl fragment of the 4,4-pybz ligand, one N atom from the pyridine ring fragment of the 4,4-pybz ligand, one P atom from one PPh_3 and one oxygen atom from one water or methanol molecule to establish a distorted tetrahedral geometry in each case. The metal ions of the silver(I) title complex are bridged by two 4-pyridin-4-yl-benzoate ligands to form the infinite chain structure. The two Ag—P distances are 2.3579(14) and 2.3698(13) Å, which are similar to those in $[Ag_2((P(C_6H_5))_3)_2(C_{12}NO_2)_2(H_2O)_2]_n$ (2.3599(9)−2.3686(10) Å). The angles around each Ag atom vary from 86.17(14)° to 136.49(12)°, which are similar to those in $[Ag_2((P(C_6H_5))_3)_2(C_{12}NO_2)_2(H_2O)_2]_n$ (86.84(10)°–136.87(8)°) and different from those in $[Ag_2((P(C_6H_5))_3)_2(C_{12}NO_2)_2]\cdot CH_3OH$ (53.38(8)°–136.69(6)°)) [7]. In summary, a new Ag(I) coordination polymer was synthesized and characterized. The result will give an important information for designing and synthesizing new compounds of Ag(I) complexes using pyridylbenzoate ligands. Ag—N and Ag—O bond lengths are all in the expected ranges for such a coordination polymer [8].

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