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## Solid channel structure and nanoscale drum-like Ag<sub>6</sub> cluster constructed with pentafluorobenzenethiolate and triphenylphosphine ligands: The use of water-soluble silver(I) carboxylate as silver(I) source

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## Abstract

A nanoscale drum-like hexanuclear silver(I) cluster  $[Ag(pfbt)(PPh_3)]_6 \mathbf{1}$  (Hpfbt = pentafluorobenzenethiol), which showed the arrays of channels based on its self-assembly in the solid, was obtained by the 1:1 molar-ratio reaction of the insoluble polymeric precursor  $\infty$ [Ag(pfbt)]  $\mathbf{2}$  and PPh<sub>3</sub> in chloroform. Synthetic yield and purity of  $\mathbf{1}$  were strongly dependent on the purity of  $\mathbf{2}$ . Compound  $\mathbf{2}$  with higher purity was prepared in good yield using light-stable and water-soluble silver(I) carboxylate [Ag(Hpyrrld)]<sub>2</sub> (H<sub>2</sub>pyrrld = 2-pyrrol-idone-5-carboxylic acid) as the silver(I) source.

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There is currently considerable interest in the coordination chemistry of coinage metals such as silver(I) and gold(I) with biological and/or medicinal activities [1-3]. In the structural viewpoint, silver(I) complexes with thiol ligands have shown a tendency to form cluster and polymer structures [1,4], whereas the corresponding gold(I) complexes have shown supramolecular arrangements of 2-coordinate linear units [1,5-7].

It is known that the cooperation with electron-deficient fluorinated aromatics and electron-rich aromatics can induce electrostatic quadrupole stacking interaction and contribute to the architecture of extended structures in the crystal [8–13]. For example, the supramolecular dimer of the triphenylphosphinegold(I) complex  $[Au(pfbt)(PPh_3)]_2$ (Hpfbt = pentafluorobenzenethiol; Chart 1) through intermolecular quadrupole interactions between the fluorinated phenyl ring (pfbt<sup>-</sup>) and the phenyl ring of the neighboring PPh<sub>3</sub> molecule was realized in the solid state and in solution, the molecular structure of which contained intramolecular Au $\cdots$ F and H(phenyl of PPh<sub>3</sub>) $\cdots$ F interactions as well as 2-coordinate P-Au-S bonding [14]. The related gold(I) com $plex [Au(SPh)(PPh_3)]$  with the unfluorinated aromatic thiolate has shown a dimeric arrangement through the aurophilic interaction [15]. On the other hand, the triphenylphosphinesilver(I) complex with flurorine-free aromatic ligand,  $[Ag(SPh)(PPh_3)]_4$  (SPh<sup>-</sup> = benzenethiolate), has been reported as a tetranuclear silver(I) cluster with highly distorted chair structure [16]. However, the structure of the pfbt<sup>-</sup> analogs of triphenylphosphinesilver(I) complex has not been reported so far. Thus, we have aimed at preparing such a complex and examining the effect of fluorinated and unfluorinated aromatic thiolate ligands on the molecular structure of the triphenylphosphinesilver(I) complexes.

Recently, we have found that the chiral and achiral silver(I) carboxylates,  $[Ag(R- \text{ or } S-\text{Hpyrrld})]_2$  and  $[Ag(R,S-\text{Hpyrrld})]_2$  (H<sub>2</sub>pyrrld = 2-pyrrolidone-5-carboxylic acid) [17,18] are light-stable and water-soluble Ag–O bonding

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Chart 1. Hpfbt (pentafluorobenzenethiol).

complexes. Their triphenylphosphine derivatives, e.g.,  $[Ag_2(R- \text{ or } S-\text{Hpyrrld})_2(\text{H}_2\text{O})(\text{PPh}_3)_2] \cdot \text{H}_2\text{O}, [Ag(R- \text{ or } S-\text{Hpyrrld})(\text{PPh}_3)_2]_2$ ,  $[Ag(R,S-\text{Hpyrrld})(\text{PPh}_3)]_2$  and  $[Ag(R,S-\text{Hpyrrld})(\text{PPh}_3)_2]$ , showed different Ag–O bonding modes depending on the number of PPh<sub>3</sub> ligands and the chirality of the Hpyrrld<sup>-</sup> ligand [19]. The Ag–O bonding complexes and the triphenylphosphinesilver(I) derivatives have been recently used as useful precursors for formation of novel silver(I) clusters such as  $[Ag(2-\text{Hmba})(\text{PPh}_3)]_4$  (2-H<sub>2</sub>mba = 2- mercaptobenzoic acid) with a three-leaves propeller (C<sub>3</sub> symmetry)[20]and [Ag<sub>2</sub>(Himdc)(PPh\_3)\_2]\_2(H\_3imdc = imidaz-ole-4,5-dicarboxylic acid) with a "bivalve"-like skeleton [21].

In this work, a nanoscale, S<sub>6</sub> symmetry drum-like hexanuclear silver(I) cluster  $[Ag(pfbt)(PPh_3)]_6$  1 was successfully obtained in the 1:1 molar-ratio reaction of the insoluble polymeric precursor  $_{\infty}[Ag(pfbt)]$  2 and PPh<sub>3</sub> in chloroform [22,23]. Complex 1 showed the arrays of channels resulting from its self-assembly in the solid state. The water-soluble silver(I) carboxylate  $[Ag(Hpyrrld)]_2$  was used as the silver(I) source for preparation of 2 with higher purity. Herein, we report the synthesis of 1 and 2, and the unequivocal characterization of 1 with elemental analysis, TG/DTA, FTIR, solution (<sup>1</sup>H and <sup>31</sup>P) NMR and solid-state <sup>31</sup>P CPMAS NMR, and single-crystal X-ray crystallography.

Compound 1 as colorless plate crystals was obtained in 79.0% (0.45 g scale) yield by the 1:1 molar-ratio reaction of 2 and PPh<sub>3</sub> in chloroform. Pale yellow powder of 2 was obtained in 95.3% (1.17 g scale) yield by the 1:2 molar-ratio reaction of  $[Ag(R,S-Hpyrrld)]_2$  and Hpfbt in a 1:1 mixed H<sub>2</sub>O/EtOH solvent. Instead of  $[Ag(R,S-Hpyrrld)]_2$ , the silver(I) sources such as Ag<sub>2</sub>O and AgNO<sub>3</sub> gave the impure solid 2 contaminated with unreacted Ag<sub>2</sub>O and NO<sub>3</sub><sup>-</sup> ion [22]. The synthetic reactions of 2 and 1 are shown in Eqs. (1) and (2):

$$1/2[\operatorname{Ag}(R, S-\operatorname{Hpyrrld})]_{2} + \operatorname{Hpfbt} \to {}_{\infty}[\operatorname{Ag}(\operatorname{pfbt})]\mathbf{2} + R, S-\operatorname{H}_{2}\operatorname{pyrrld}$$
(1)  
$$6_{\infty}[\operatorname{Ag}(\operatorname{pfbt})]\mathbf{2} + 6\operatorname{PPh}_{3} \to [\operatorname{Ag}(\operatorname{pfbt})(\operatorname{PPh}_{3})]_{6}\mathbf{1}$$
(2)

The composition and molecular formula of 1 were consistent with elemental analysis, TG/DTA, FTIR, solution (<sup>1</sup>H and <sup>31</sup>P) NMR and solid-state <sup>31</sup>P CPMAS NMR.

The solid-state <sup>31</sup>P CPMAS NMR spectra of 1 showed phosphorus resonance of doublet peaks (two lines) due to  ${}^{1}J(Ag-P)$  coupling for the PPh<sub>3</sub> ligand coordinating to the six equivalent silver(I) atoms. The solid-state  ${}^{31}P$ 



Fig. 1. (a) Molecular structure of 1 (symmetry operation i = y + 2, -x + y + 1, -z + 2; ii = -x + y + 2, -x + 1, z; iii = -x + 2, -y, -z + 2; iv = -y + 1, x - y - 1, z; v = x - y, x - 1, -z + 2) and (b) its skeleton representation with Ag, S, and P atoms. Selected interatomic distances (Å) and angles (°): Ag1–P1 2.4137(5), Ag1–S1 2.6062(5), Ag1–S1<sup>iv</sup> 2.6084(5), Ag1–S1<sup>v</sup> 2.6930(5), Ag1···Ag1<sup>i</sup> separation 3.875 Å; P1–Ag1–S1 119.564(16), P1–Ag1–S1<sup>iv</sup> 115.210(16), S1–Ag1–S1<sup>iv</sup> 109.92(2), P1–Ag1– S1<sup>v</sup> 134.069(17), S1–Ag1–S1<sup>v</sup> 85.798(15), S1<sup>iv</sup>–Ag1–S1<sup>v</sup> 85.751(15)°.

NMR are consistent with the solid-state structure revealed by X-ray crystallography.

Solution <sup>31</sup>P NMR in CDCl<sub>3</sub> of **1** showed one <sup>31</sup>P resonance at 9.30 ppm on the basis of coordination to the six equivalent silver(I) atoms. The <sup>1</sup>H NMR spectra of **1** showed multiplet peaks for aryl protons of the PPh<sub>3</sub> ligand.

X-ray structure analysis revealed that an  $S_6$  symmetry hexanuclear silver(I) cluster 1 was a micelle-like nanoscale object with an external diameter ca. 18 Å and an internal diameter ca. 5 Å (Fig. 1(a) and (b)), and it constructed nanoporous channel structures based on its self-assembly in the solid state (Fig. 2) [24].

The molecular structure of **1**, constructed with six 4coordinated  $Ag(\mu_3-S)_3P$  units (Ag1, Ag1<sup>i</sup>, Ag1<sup>ii</sup>, Ag1<sup>iii</sup>, Ag1<sup>iv</sup> and Ag1<sup>v</sup>), was stabilized with many Ag-( $\mu_3$ -S) and Ag-P bondings as well as several non-covalent, weak



Fig. 2. The solid-state packing of the  $Ag_6$  cluster 1, composed of Ag (blue), P (red), S (yellow), F (green) and C (gray) atoms, along the crystallographic *c* axis. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

and multiple intramolecular interactions, i.e.,  $F \cdots H$  interaction ( $F6 \cdots H112^i 2.574 \text{ Å}$ ,  $F2 \cdots H122 2.353 \text{ Å}$ ),  $F \cdots C$ interaction ( $F2 \cdots C122^{ii} 3.169 \text{ Å}$ ,  $F3 \cdots C124^{ii} 3.102 \text{ Å}$ ) and C(pfbt) $\cdots C$ (Ph) interaction ( $C1 \cdots C112 3.488 \text{ Å}$ ,  $C2 \cdots C111 3.487 \text{ Å}$ ,  $C2 \cdots C112 3.367 \text{ Å}$ ,  $C3 \cdots C113$ 3.481 Å,  $C3 \cdots C114 3.419 \text{ Å}$ ,  $C2 \cdots C122^{ii} 3.320 \text{ Å}$ ), the distances of which were close to or slightly shorter than the sum of the van der Waals radii (H 1.20 Å, F 1.47 Å, C 1.70 Å). In **1**, there was neither Ag–Ag interaction (3.857 Å) nor interaction between Ag and F atoms (3.328 Å).

On the other hand, the external interactions among the Ag<sub>6</sub> clusters resulted in the self-assembly: one Ag<sub>6</sub> unit was linked to the neighboring six Ag<sub>6</sub> units each other through a dual mode of the intermolecular F5(F–Ph)···H115(Ph) interaction (2.470 Å) between the two Ag<sub>6</sub> cluster units. Thus, the aryl embraces through the dual F···H interaction significantly contribute to the packing and the arrays of channels [25,26]. The unique pfbt<sup>-</sup> ligand undoubtedly contributes to the intramolecular and intermolecular, weak multiple interactions which stabilize and drive the formation of the Ag<sub>6</sub> cluster **1** and its self-assembly.

Complex 1 is in contrast to the triphenylphosphinegold(I) complex with the pfbt<sup>-</sup> ligand,  $[Au(pfbt)(PPh_3)]_2$ , which is a new class of supramolecular aggregation through the intermolecular electrostatic quadrupole interactions in the solid state and in solution [14]. Complex 1 can be also compared with the Ag<sub>4</sub> cluster with the highly distorted chair structure of  $[Ag(SPh)(PPh_3)]_4$  with the fluorine-free SPh<sup>-</sup> ligand [16]: the Ag–S distances (2.6062(5), 2.6084(5), 2.6930(5) Å) in **1** are longer than those (2.5245(7), 2.5147(8), 2.4925(7), 2.5593(8) Å) in  $[Ag(SPh)(PPh_3)]_4$ .

The molecular structure of **1** is similar to that of the previously reported Ag<sub>6</sub> cluster with a "drum"-like skeleton,  $[Ag{S_2P(OPr^i)_2}]_6$  (S<sub>2</sub>P(OPr<sup>i</sup>)<sub>2</sub> = disopropyldithiophosphine) [27], while that of **1** is quite different from those of the previously reported Ag<sub>6</sub> clusters formed with the Sdonor atom [28–33].

In summary, the nanoscale micelle-like Ag cluster 1 with an S<sub>6</sub> symmetry (an external diameter ca. 18 Å and an internal diameter ca. 5 Å), which showed solid channel structure, was obtained using the separately prepared, insoluble polymeric precursor 2 and PPh<sub>3</sub>. Using the water-soluble silver(I) source [Ag(Hpyrrld)]<sub>2</sub>, compound 2 with higher purity was obtained in good yield. The present silver(I) source would be useful for formation of novel silver(I) clusters, in the place of the traditional silver(I) sources such as AgNO<sub>3</sub>, Ag<sub>2</sub>O, silver(I) acetate and so on [20,21]. Also, novel nanoporous structures based on the silver(I) complexes would be built by using some fluorinated aromatic thiolates (F–SPh<sup>-</sup>) with the changed number and site of F atom and changing the molar ratios of the precursor  $\infty$ [Ag(F–SPh)] and PPh<sub>3</sub>.

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- [22] Compound 2: To 0.944 g (2.00 mmol) of [Ag(R,S-Hpyrrld)]<sub>2</sub> dissolved in 40 mL of 1:1 (v/v) solvent mixture of H<sub>2</sub>O and EtOH was added 432 µL (4.00 mmol) of Hpfbt, followed by stirring for 1 h. Pale yellow powder formed was collected on a membrane filter (JG  $0.2 \,\mu\text{m}$ ), washed with water (100 mL  $\times$  2), EtOH (100 mL  $\times$  2) and ether (100 mL × 2), and thoroughly dried in vacuo for 2 h. The pale yellow powder, obtained in 95.3% (1.17 g scale) yield, was sparingly soluble in acetone, EtOH, CH2Cl2, CHCl3 and DMSO, and insoluble in water and ether. Anal. Found: C, 23.48. Calcd for C<sub>6</sub>F<sub>5</sub>SAg or [Ag(pfbt)]: C, 23.47%. TG/DTA data: no weight loss was observed before decomposition. Decomposition gradually began around 249 °C with an exothermic peak at 367 °C. Prominent IR bands at 1600-400 cm<sup>-1</sup> region (KBr disk): 1522s, 1479vs, 1098m, 966vs, 867m cm<sup>-1</sup>. Synthesis of **2** using  $Ag_2O$  instead of  $[Ag(R,S-Hpyrrld)]_2$ : To a suspension of 0.232 g (1.00 mmol) of Ag<sub>2</sub>O in 20 mL of 1:1 (v/v) solvent mixture of H<sub>2</sub>O and EtOH was added 266 µL (2.00 mmol) of Hpfbt, followed by stirring for one day. The material obtained here was a mixture of black powder (unreacted Ag<sub>2</sub>O) and pale yellow powder (compound 2). Synthesis of 2 using AgNO<sub>3</sub> instead of [Ag(R,S-Hpyrrld)]<sub>2</sub>: To a solution of 0.169 g (1.00 mmol) of AgNO<sub>3</sub> in 20 mL of 1:1 (v/v) solvent mixture of H2O and EtOH was added

133  $\mu$ L (1.00 mmol) of Hpfbt. To it, 1.0 mL of 1.0 M NaOH aqueous solution (1.00 mmol) was added dropwise and the stirring continued for 1 h. Pale yellow powder formed was collected on a membrane filter (JG 0.2  $\mu$ m), washed with water (50 mL × 2), EtOH (50 mL × 2) and ether (50 mL × 2), and thoroughly dried in vacuo for 2 h. The pale yellow powder obtained in 91.2% (0.28 g scale) yield was contaminated with NO<sub>3</sub><sup>-</sup> ion. Prominent IR bands at 1600–400 cm<sup>-1</sup> region (KBr disk): 1521m, 1479s, 1384vs (NO<sub>3</sub><sup>-</sup>), 1097m, 966s, 867m cm<sup>-1</sup>.

- [23] Compound 1 : To 0.307 g (1.00 mmol) of 2 suspended in 20 mL of CHCl<sub>3</sub> was added 0.262 g (1.00 mmol) of PPh<sub>3</sub>, followed by stirring for 1 h. After filtering the colorless clear solution through a folded filter paper (Whatman #5), a vapor diffusion using the filtrate as an inner solution and ether as an external solvent was performed at room temperature. After one day, colorless plate crystals were formed, which were collected on a membrane filter (JG 0.2 µm), washed with ether (50 mL  $\times$  2), and thoroughly dried in vacuo for 2 h. Colorless plate crystals, obtained in 79.0% (0.45 g scale) yield, were soluble in CH2Cl2, CHCl3, sparingly soluble in acetone, DMSO and DMF, and insoluble in water, EtOH and ether. This complex is thermally and light-stable both in solution and in the solid state. Anal. Found: C, 50.61; H, 2.51. Calcd for C144H90S6P6F30Ag6 or [Ag(pfbt)(PPh3)]6: C, 50.64; H, 2.66%. TG/DTA data: no weight loss was observed before decomposition. Decomposition gradually began around 175 °C with an exothermic peak at 272 °C and with an endothermic peak at 187 °C. Prominent IR bands at 1600–400 cm<sup>-1</sup> region (KBr disk): 1504vs, 1474vs (PPh<sub>3</sub>), 1435s (PPh<sub>3</sub>), 1389w, 1095m (PPh<sub>3</sub>), 1079m, 1003w, 969vs, 853vs, 742s (PPh3), 693vs (PPh3), 515s (PPh3), 503m (PPh<sub>3</sub>), 493m cm<sup>-1</sup>. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 22.7 °C): δ 7.19–7.31 (15H, m, Aryl). <sup>31</sup>P NMR (CDCl<sub>3</sub>, 22.3 °C): δ 9.30. Solid-state <sup>31</sup>P CPMAS NMR (substitution method with (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub>, 25.0 °C ): δ 5.76  $(J_{\rm Ag-P}$ 491 Hz).
- [24] The intensity data were collected at 90 K on a Bruker SMART/APEX CCD diffractometer. The structure was solved by direct methods (SHELXTL version 5.10), and refined by a full-matrix least-squares on  $F^2$ . Crystal data for 1:  $C_{144}H_{90}F_{30}P_6S_6Ag_6$ ; M = 3415.56, rhombohedral, space group R3, a = 27.6806(14), b = 27.6806(14), c = 15.1191(15) Å,  $\gamma = 120^\circ$ , V = 10032.5(12) Å<sup>3</sup>, Z = 3,  $D_{calcd} = 1.696$  g cm<sup>-3</sup>,  $\mu = 1.120$  mm<sup>-1</sup>, 35,831 reflections collected, 5426 independent ( $R_{int} = 0.0434$ ), R1 = 0.0270, wR2 = 0.0638 for  $I > 2\sigma(I)$ , R1 = 0.0271, wR2 = 0.0638, GOF = 1.276 for all data. The details of the crystal data have been deposited with Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 279822 for 1.
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