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Self-Assembled Three-Dimensional Coordination Polymers with Unusual Ligand-Unsupported Ag–Ag Bonds: Syntheses, Structures, and Luminescent Properties[**]

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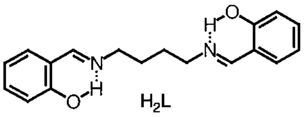
Self-assembled supramolecular architectures are currently of great interest due to their intriguing network topologies and their potential in microelectronics, nonlinear optics, porous materials, and other applications.[1–10] Since the self-assembly of these architectures is highly influenced by factors such as the solvent system,[11] templates,[12a,b] and counterion,[12c, 13] the exploration of synthetic routes is a long-term challenge.

Neutral monodentate phenol groups are notoriously poor donors for transition metal ions. To the best of our knowledge, no supramolecular coordination aggregate self-assembled by neutral bis-monodentate phenol groups has been documented, although a number of deprotonated dicatechol ligands have been successfully applied to self-assemble helicate-type coordination polymers.[14] We report here on the preparation, X-ray crystal structures, and luminescent properties of novel three-dimensional, noninterpenetrating networks in which bis-monodentate phenol groups of a Schiff base ligand serve as bridges, namely, $[Ag_2(H_2L)_3]_n(NO_3)_{2n}$ (**1**) and $[Ag_2(H_2L)_3]_n(ClO_4)_{2n}$ (**2**); H_2L = *N,N'*-bis(salicylidene)-1,4-diaminobutane.



H_2L

X-ray crystallography shows that **1** is made up of a three-dimensional, noninterpenetrating coordination network and nitrate ions (Figure 1). Each Ag^I atom is coordinated by three phenol groups from three H_2L ligands in an unusual, perfect trigonal arrangement (Ag–O 2.190(2) Å, O-Ag-O 119.62(2)°). Trigonal coordination arrangements have recent precedence in two Ag^I complexes with tricyanobenzene and tris(4-ethynylbenzonitrile)benzene ligands, in which the Ag^I atom is not in idealized trigonal geometry.[15] In **1** each pair of the adjacent, centrosymmetrically related Ag^I atoms are joined by a ligand-unsupported metal–metal bond (2.934(2) Å), the Ag–Ag bond lies on a crystallographic threefold axis, and all six H_2L ligands are symmetrically equivalent. The six phenol groups ligated to the pair of Ag^I atoms are in a staggered arrangement structurally similar to that of the hydrogen atoms in an ethane molecule (**I** in Scheme 1). Within the H_2L

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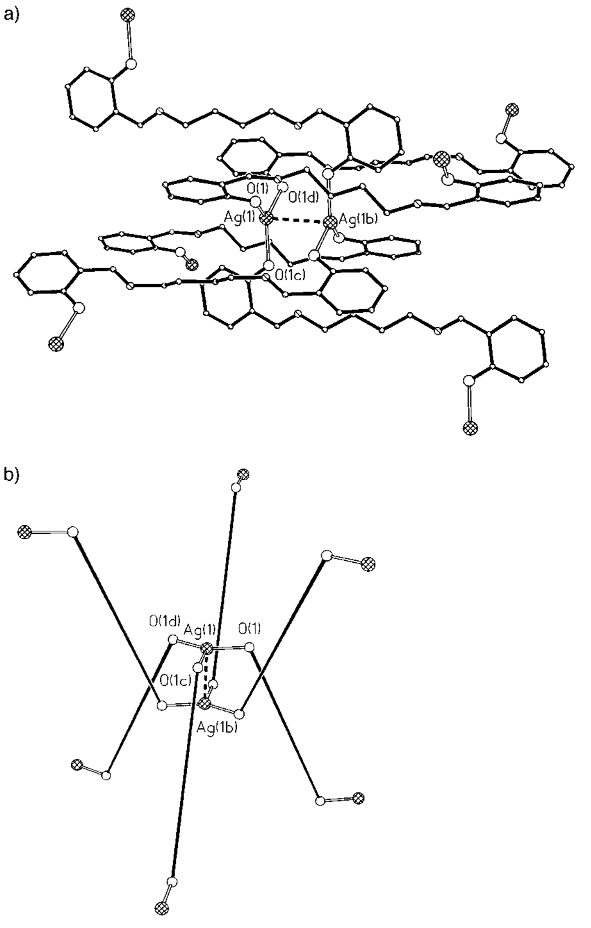
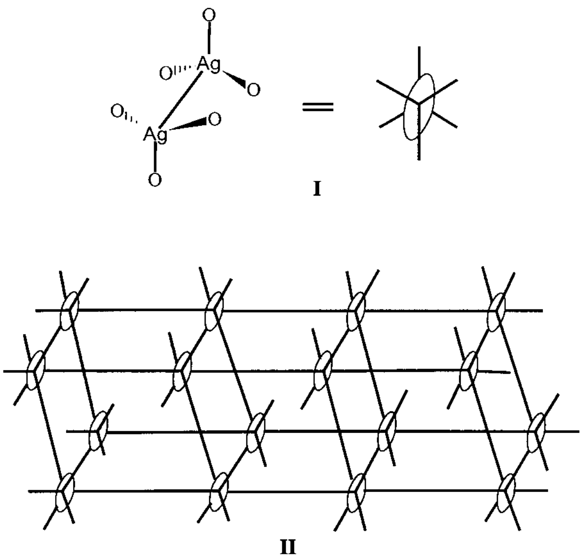


Figure 1. Coordination environment of the Ag^I atoms in **1**. a) H_2L ligands are shown explicitly. b) H_2L ligands are represented by long rods.



Scheme 1. Formula **I** depicts the staggered arrangement of phenol groups about the Ag^I atoms in **1**. The three-dimensional network of **1** is represented by **II**.

ligand, each phenol group forms a strong intramolecular hydrogen bond with the adjacent N atom (N ··· O 2.539(4) Å).

The three-dimensional network of **1** consists of rhombohedral building blocks (**II** in Scheme 1, Figure 2a). In the rhombohedron, each top site is occupied by a pair of ligand-unsupported Ag^I atoms, which are surrounded by six phenol groups. The phenol groups at another ends of the ligands bridge the rhombohedrons to form a novel, noninterpenetrating, three-dimensional network (**II** in Scheme 1), in which rhombic channels (Ag ··· Ag 13.33 Å) run along the *a* and *b* axes, as illustrated in Figure 2b. The NO_3^- counterions lie on crystallographic threefold axes and occupy these channels. Network **2** is isomorphous to **1**, and the structural parameters are very similar.

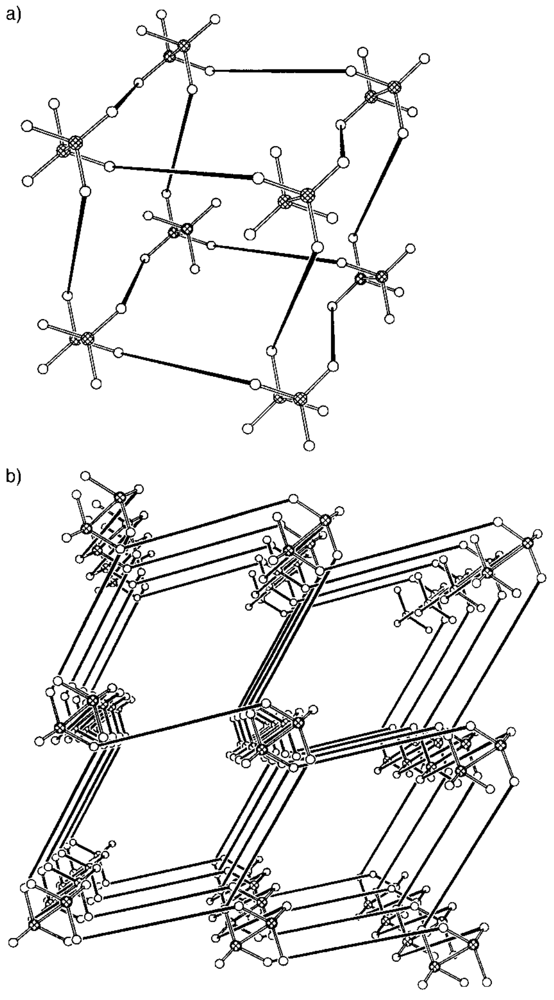


Figure 2. a) The rhombohedron in **1**. b) The three-dimensional network of **1** viewed along the *a* or *b* axis.

It is noteworthy that, neglecting the Ag–Ag interactions, the frames of **1** and **2** are formed by two-dimensional, highly undulated simple layers of (6,3) topology[16] that catenate in a parallel fashion to give, for the first time, a three-dimensional overall network, instead of the usual two-dimensional one.[3]

The H_2L ligand, which contains two kinds of possible coordination donors (N and O) to ligate the Ag^I atoms, uses only its O donor, in contrast to the coordination of similar ligands.[17] What is more, the Ag–Ag distances of 2.934(2) Å in **1** and 2.946(2) Å in **2**, which are similar to the ligand-unsupported Ag–Ag distance (2.977(1) Å) in the three-dimensional [Ag(4,4′-bpy)](NO_3) polymer (bpy = bipyridine),[7b, 8a] are well below the sum of the van der Walls radii of two silver atoms (3.44 Å)[18] and very close to the Ag–Ag separation in silver metal (2.89 Å), which suggests significant

Ag–Ag bonding. To our knowledge, the only compound with a shorter ligand-unsupported Ag–Ag bond (2.873(2) Å) is $[NBu_4]_4[Ag_2\{Mo_5O_{13}(OMe)_4(NO)\}_2]$, in which the bridging polyoxoanions most probably play a role in the fixing the Ag–Ag separation.[19] The linear, T-shaped, and distorted tetrahedral geometries about Ag^I atoms in three-dimensional networks are well-documented,[7a, 20] but the arrangement of a pair of ligand-unsupported, trigonally ligated Ag^I atoms in **1** and **2** at such a short Ag–Ag distance and with staggered ligands is unusual and may be attributed to the formation of the polymeric structure.[21] The Raman-active bands at 120 and 83 cm^{-1} for **1** and 121 and 82 cm^{-1} for **2** may be assigned to the Ag–Ag vibration and are comparable to those in related compounds.[22]

Commonly Ag^I complexes may emit weak photoluminescence at low temperature.[5] Conversely at room temperature, **1** and **2** in the solid state and in acetonitrile display virtually identical intense blue photoluminescence with emission maxima at 497 (solid) and 460 nm (in acetonitrile), respectively, upon excitation at 370 nm (Figure 3). This implies that the polymers disaggregate into oligomers in acetonitrile. Further work on this subject is in progress.

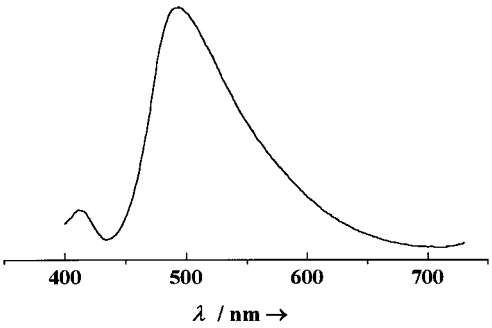


Figure 3. Photo-induced emission spectrum of **1** in the solid state at room temperature with $\lambda_{ex} = 370$ nm.

Experimental Section

H_2L: 1,4-Diaminobutane (0.10 mol) was added gradually to a solution of salicylaldehyde (0.20 mol) in EtOH (100 mL), and the resulting solution was stirred for 2 h at 60°C, and a pale yellow precipitate was collected. Elemental analysis was in accord with the formula H_2L. ^1H NMR (300 MHz, $CDCl_3$): $\delta = 13.64$ (br, 1H, OH), 8.44 (s, 1H, CH=N), 7.38 (t, 1H, arom.), 7.33 (d, 1H, $J = 7.65$ Hz, arom.), 7.05 (d, 1H, $J = 8.22$ Hz, arom.), 6.95 (t, 1H, arom.), 3.72 (t, 2H, $=NCH_2$), 1.90 (t, 2H, CH_2); ^{13}C NMR (75.475 MHz, $CDCl_3$): $\delta = 165.61$, 161.87, 132.85, 131.87, 119.41, 119.21, 117.67, 59.93, 29.20.

1: A solution of H_2L (1.0 mmol) in acetonitrile (10 mL) was added dropwise to a stirred aqueous solution (5 mL) of silver(I) nitrate (1.0 mmol) at 50°C for 15 min. The resulting light yellow solution precipitated yellow-orange block-shaped crystals of **1** (ca. 65% yield based on the ligand) after several days. Elemental analysis calcd for $C_{54}H_{60}Ag_2N_8O_{12}$: C 52.78, H 4.92, N 9.12; found: C 52.63, H 4.92, N 9.14%: IR (KBr): $\tilde{\nu} = 3050$w, 2938w, 2860m, 1630vs, 1609s, 1581m, 1525m, 1496s, 1461m, 1384vs, 1356vs, 1285s, 1236m, 1187s, 1138s, 1110m, 1053m, 1011m, 976m, 927m, 878m, 857m, 751s, 653w, 568w, 547w, 526w cm^{-1}. Crystal data for **1**:[23] $C_{54}H_{60}Ag_2N_8O_{12}$, $M_r = 1228.84$, trigonal, space group $R\bar{3}$ (No. 148), $a = 15.234(4)$, $b = 15.234(4)$, $c = 19.425(11)$ Å, $V = 3904(3)$ Å^3, $Z = 3$. $\rho_{calcd} = 1.568$ g cm^{-3}, $\mu = 8.24$ cm^{-1}; $R = 0.0655$, $R_w = 0.1822$, and GOF = 0.999 for 116 parameters, 894 reflections with $F_o > 4\sigma(F_o)$.

2: The synthesis of **2** was similar to that of **1**, except silver(I) perchlorate (1.0 mmol) was used in place of silver(I) nitrate. Yellow-orange block-shaped crystals of **2** (ca. 65% yield based on the ligand) were obtained. Elemental analysis calcd for $C_{54}H_{60}Ag_2Cl_2N_6O_{14}$: C 49.75, H 4.64, N 6.45; found: C 49.58, H 4.62, N 6.36; IR (KBr): $\tilde{\nu} = 3050$w, 3015w, 2938m, 2868m, 1637vs, 1609s, 1581m, 1532s, 1482s, 1440m, 1349m, 1285m, 1236w, 1187s, 1145s, 1089vs, 927w, 885m, 765m, 625m, 568w, 547w, 456w cm^{-1}. Crystal data for **2**:[23] $C_{54}H_{60}Cl_2N_6O_{14}Ag_2$, $M_r = 1303.72$, trigonal, space group $R\bar{3}$ (No. 148), $a = 15.725(2)$, $b = 15.725(2)$, $c = 19.628(4)$ Å, $V = 4203.3(11)$ Å^3, $Z = 3$. $\rho_{calcd} = 1.545$ g cm^{-3}, $\mu = 8.64$ cm^{-1}. $R = 0.0642$, $R_w = 0.1851$, and GOF = 1.066 for 124 parameters, 1149 reflections with $F_o > 4\sigma(F_o)$.

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supplementary publication nos. CCDC-112414 and -112415. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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The Li$^+$-Initiated Twofold Dehydrogenation and C–C Bond Formation of Hexaphenylbenzene to the Dilithium Salt of the 9,10-Diphenyltetrabenz[*a*,*c*,*h*,*j*]anthracene Dianion[**]

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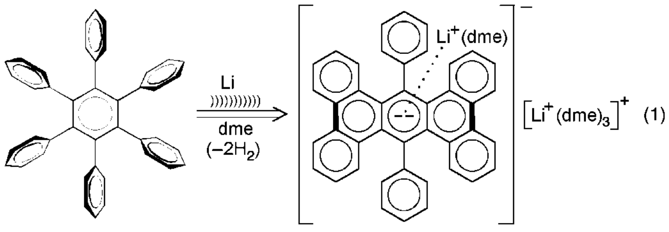
Katayoun Gharagozloo-Hubmann, and Mark Sievert

Structures of molecular crystals are often a well-suited starting point for hypersurface calculations[1] aiming to reveal essential sections of microscopic reaction pathways. Examples that take into account the energetically important cation solvation[2] are the reductive ring opening of dibenzothiophene to the dimeric sodium sulfide salt[1c] or the reductive C6/C6′ azulene dimerization to the solvent-wrapped contact ion triple $[(\text{diglyme}_2\text{Na}^+)\cdots{}^-\text{azulene}-\text{azulene}^-\cdots(\text{Na}^+\text{diglyme}_2)]$.[1d] Here we report on the ultrasonically activated reaction of hexaphenylbenzene in 1,2-dimethoxyethane (DME) with lithium metal powder,[3] which enforces twofold dehydrogenation and formation of two additional C–C bonds [Eq. (1)].



The crystal structure determination (Figure 1)[3] proves the formation of a partly solvent separated and partly solvent shared contact ion triple of the 9,10-diphenyltetrabenz[*a*,*c*,*h*,*j*]anthracene dianion, the threefold DME solvated Li$^+$ countercation and the singly DME solvated contact ion pair anion, which stack alternatingly along the crystallographic *x* axis (Figure 1 A).

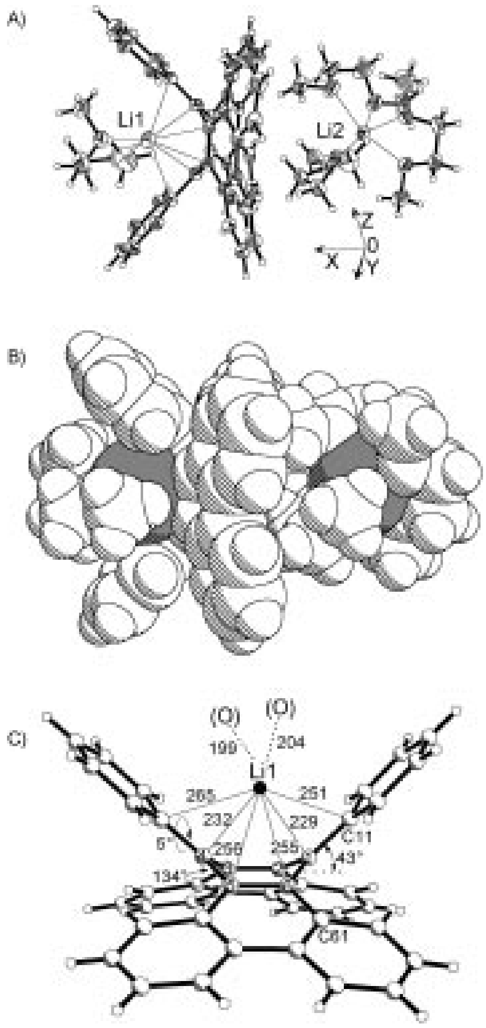


Figure 1. Crystal structure of the contact ion triple $[(\text{dme})\text{Li}^+\cdots\text{C}_{42}\text{H}_{26}{}^{2-}]^-[\text{Li}^+(\text{DME})_3]^+$ at 180 K: A) Packing (monoclinic $P2_1/n$, $Z=4$) along the *x* axis (50% thermal ellipsoids). B) Space-filling representation (Li$^+$ grey). C) Boat conformation of the 9,10-diphenyltetrabenzanthracene dianion with Li$^+$ contact distances and bending angles. Selected bond lengths [pm] and angles [°]: C1–C6 148, C5–C6 138, C1–C11 143, C1–C61 145; C2-C1-C6 111, C1-C6-C5 117, C6-C5-C4 116, Li2-O 202 (Li1-O 216). See text for details.

The molecular skeleton of the 9,10-diphenyltetrabenz[*a*,*c*,*h*,*j*]anthracene dianion in its {(DME)Li$^+$} contact ion pair shows a drastic distortion comparable to those of the tetraphenylethene dianion,[4a] the 1,2,4,5-tetrakis(trimethylsilyl)benzene dianion,[4b] or the rubrene tetraanion[4c] in their alkali metal cation aggregates. In analogy to the π-hydrocarbon salt presented here (Figure 1 B) they are impressively solvent-wrapped and therefore kinetically stabilized. In the central planar hexacarbon ring of the precursor hexaphenylbenzene [Eq. (1); see Figure 2 A][5] the *para*-positioned centers C1 and C4 are each bent upward by 43° (!), whereas the two phenanthracene subunits at C2/C3 as well as C5/C6 are bent downward with an interplanar angle of only 143° (!) (Figure 1 C). The *para*-phenyl substituents, twisted out of the

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