

## Single Strands versus Ladders: Construction of Skeletal Structure via Coordinating Ability of Anions

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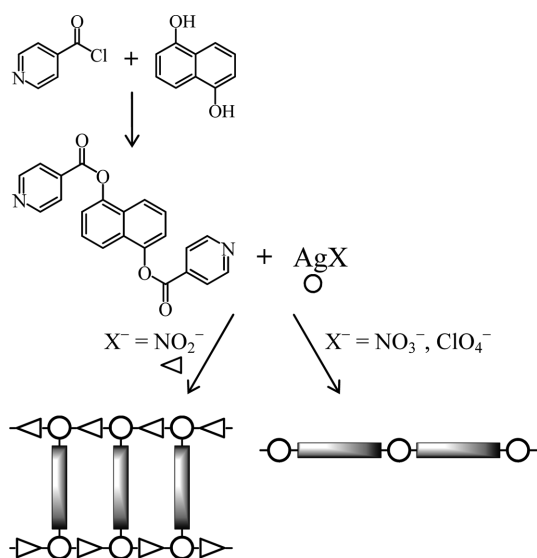
The rational construction of aesthetic coordination polymeric skeletons by means of selection of basic components such as the coordination geometry of metal ions, the bonding mode of donating atoms, and weak interactions, for example van der Waals, MM bonds, electrostatic interaction, hydrogen-bonds, and  $\pi$ - $\pi$  interaction, is a promising issue since the materials have various potential applications such as molecular separation, toxic materials adsorption, gasoline containers, ion exchangers, molecular recognition, and luminescent sensors.<sup>1-6</sup> Various types of intriguing network topologies including chain,<sup>7</sup> ladder,<sup>8</sup> grid,<sup>9</sup> brick-wall,<sup>10</sup> honeycomb,<sup>11</sup> diamondoid,<sup>12</sup> rutile,<sup>13</sup> and  $\alpha$ -polonium<sup>14</sup> by using numerous multidentate ligands with appropriate spacers have been reported. The spacers are basically important factors because they promise greater possibilities for constructing and fine-tuning novel frameworks owing to their flexibility and conformational freedom. In addition, weak interactions via (counter) anions have emerged as another important factor in the formation of molecular skeleton.<sup>15</sup> Concomitantly, research on anion coordinating ability has become a rapidly expanding field owing to a timely interest from environmental pollution, industrial chemical, biological process, ionic liquids, catalysis, lithium battery, and health related perspectives.<sup>16-21</sup> The characteristics of anions such as negative charge, size, a wide range of geometries, significant solvent effects, and pH dependence play key roles in determining the molecular skeleton and behaviors.<sup>22-24</sup> Of these polyatomic anions,  $\text{NO}_2^-$ ,  $\text{NO}_3^-$ , and  $\text{ClO}_4^-$  anion exist in diverse fields and their bonding modes are quite interesting.<sup>25,26</sup>

In this context, research on self-assembly of  $\text{AgX}$  ( $\text{X}^- = \text{NO}_2^-$ ,  $\text{NO}_3^-$ , and  $\text{ClO}_4^-$ ) with a 1,5-bis(isonicotinoyloxy) naphthalene (**L**) was accomplished. We report anion-dependent 1D molecular skeletons, 1D molecular ladders consisting of  $[\text{Ag}_2(\text{NO}_2)_2(\text{L})]_n$  and 1D single strands consisting of  $[\text{Ag}(\text{L})]_n(\text{X})_n$  ( $\text{X}^- = \text{NO}_3^-$  and  $\text{ClO}_4^-$ ). The **L** is a

new ligand that may possess characteristic properties such as a potential bidentate, an  $sp^2$  angle around  $\text{C}=\text{O}$  ( $\sim 120^\circ$ ), a malleable length, conformational nonrigidity, and manageable solubility. Exploitation of naphthalene-based-bipyridyl ligand unit as a linear spacer has remained unexplored.

**L** was prepared by the reaction of 1,5-dihydroxynaphthalene with isonicotinoyl chloride hydrochloride in the presence of triethylamine. **L** is soluble in chloroform, dichloromethane, tetrahydrofuran, and toluene, but is insoluble in water, methanol, ethanol, acetone, diethyl ether, and *n*-hexane. The slow diffusion of an alcoholic solution of  $\text{AgX}$  ( $\text{X}^- = \text{NO}_2^-$ ,  $\text{NO}_3^-$ , and  $\text{ClO}_4^-$ ) into an organic solution of **L** afforded different skeletal adducts, presumably owing to the different coordinating nature of the respective anions, as will be explained in detail (*Scheme 1*). The reactions were initially conducted in the 1 : 1 molar ratio of the silver(I) ions and **L**, but the products were not significantly affected by either the molar ratio and the concentrations, indicating that the compounds are thermodynamically favorable species. All the compounds are insoluble in water and common organic solvents but dissociated in acetonitrile, dimethyl sulfoxide, and *N,N*-dimethylformamide. In addition, these products are air-stable but slowly turned to gray powder under light. Characteristic IR frequencies of  $\text{NO}_2^-$ ,  $\text{NO}_3^-$ , and  $\text{ClO}_4^-$  appeared at 1270, 1384, and 1087  $\text{cm}^{-1}$ , respectively. Elemental analyses were consistent with the proposed structures.

The crystal structures of  $[\text{Ag}_2(\text{NO}_2)_2(\text{L})]_n$  and  $[\text{Ag}(\text{L})]_n(\text{X})_n$  ( $\text{X}^- = \text{NO}_3^-$  and  $\text{ClO}_4^-$ ) were solved. Crystal parameters and procedural information corresponding to data collection and structure refinements are given in *Table 1*, and selected bond lengths and angles are listed in *Table 2*. They are centrosymmetric molecules as depicted in *Fig. 1*. For  $[\text{Ag}_2(\text{NO}_2)_2(\text{L})]_n$ , combining  $\text{AgNO}_2$  and **L** in a 2 : 1 ratio results in the formation of a unique ladder structure (*Fig. 2*). One spacer **L** bridges two silver(I) atoms to yield



**Scheme 1.** Synthetic procedure.

the rung of the ladder (Ag-N = 2.277(2), 2.375(2) Å; Ag...Ag separation = 20.034(1) Å), where the pyridyl groups are twisted from the naphthyl plane with the dihedral angle of 83.35(5)°. Nitrogen donor of NO<sub>2</sub><sup>-</sup> coordinates to a silver(I) atom (Ag-N(2) = 2.277(2) Å) and two oxygen donors chelate to the other silver(I) atom (Ag-O = 2.457(2), 2.488(2) Å; O(3)-Ag-O(4) = 50.05(6)°). That is,

NO<sub>2</sub><sup>-</sup> anion is an anionic ligand in a tridentate μ<sup>2</sup>-fashion rather than a simple counteranion. The local geometry around the silver(I) atom is an unusual *trigonal pyramidal arrangement* with a nitrogen and two oxygen atoms from two NO<sub>2</sub><sup>-</sup> anions in a basal plane and a nitrogen atom from **L** in an apical position. To our knowledge, such geometrical arrangement on silver(I) complexes is rare.<sup>27</sup> Moreover, the molecular ladder may be stabilized via an intramolecular face-to-face (π-π) stacking (3.574(3) Å).

For [Ag(**L**)]<sub>n</sub>(X)<sub>n</sub> (X<sup>-</sup> = NO<sub>3</sub><sup>-</sup> and ClO<sub>4</sub><sup>-</sup>), each **L** connects two silver(I) atoms in a μ<sup>2</sup>-fashion to give single strands (Ag-N = 2.157(4), 2.163(4) Å; Ag...Ag separation = 19.582(1) Å for [Ag(**L**)]<sub>n</sub>(NO<sub>3</sub>)<sub>n</sub>; Ag-N = 2.16(3), 2.17(3) Å; Ag...Ag separation = 19.757(5) Å for [Ag(**L**)]<sub>n</sub>(ClO<sub>4</sub>)<sub>n</sub>) as shown in Fig. 2. Interestingly, the NO<sub>3</sub><sup>-</sup> anions closely interact with the silver(I) ions compared to ClO<sub>4</sub><sup>-</sup> anions, which is consistent with the coordinating ability of the anions.<sup>28</sup> The distance of Ag...ONO<sub>2</sub><sup>-</sup> is 2.776(5) Å whereas the distances of Ag...O<sub>2</sub>ClO<sub>2</sub><sup>-</sup> are 2.89(6) and 3.08(4) Å. The weak Ag...X interactions are less than the sum of the van der Waals radii (3.20 Å) of Ag and O.<sup>29</sup> For [Ag(**L**)]<sub>n</sub>(ClO<sub>4</sub>)<sub>n</sub>, the local geometry around the silver(I) atom approximates a distorted linear arrangement (N-Ag-N = 169.7(2)°), presumably owing to the presence of weak Ag...O<sub>2</sub>ClO<sub>2</sub><sup>-</sup> interaction in abidentate-like mode. For [Ag(**L**)]<sub>n</sub>(NO<sub>3</sub>)<sub>n</sub>, the geometry around the silver(I) atom

**Table 1.** Crystal data and structure refinement for [Ag<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>(**L**)<sub>n</sub>], [Ag(**L**)]<sub>n</sub>(NO<sub>3</sub>)<sub>n</sub>, and [Ag(**L**)]<sub>n</sub>(ClO<sub>4</sub>)<sub>n</sub>·nH<sub>2</sub>O

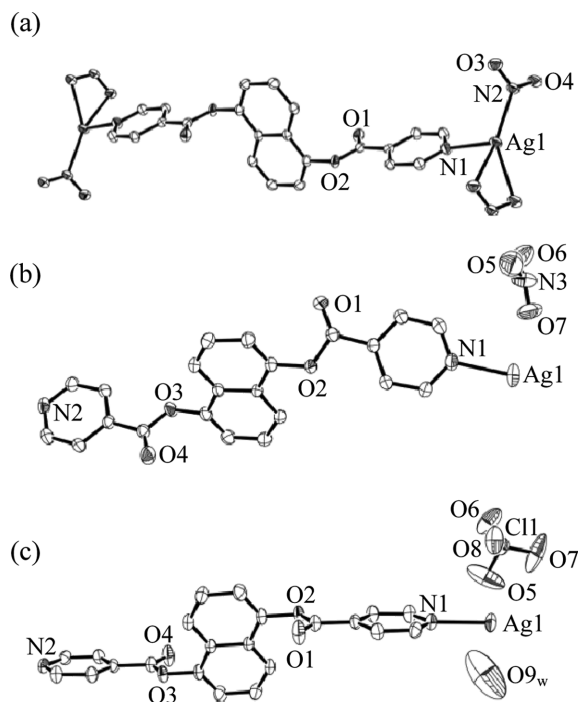
	[Ag <sub>2</sub> (NO <sub>2</sub> ) <sub>2</sub> ( <b>L</b> ) <sub>n</sub> ]	[Ag( <b>L</b> )] <sub>n</sub> (NO <sub>3</sub> ) <sub>n</sub>	[Ag( <b>L</b> )] <sub>n</sub> (ClO <sub>4</sub> ) <sub>n</sub> ·nH <sub>2</sub> O
Formula	C <sub>11</sub> H <sub>7</sub> N <sub>2</sub> O <sub>4</sub> Ag	C <sub>22</sub> H <sub>14</sub> N <sub>3</sub> O <sub>7</sub> Ag	C <sub>22</sub> H <sub>16</sub> N <sub>2</sub> O <sub>9</sub> ClAg
M <sub>w</sub> (g mol <sup>-1</sup> )	339.06	540.23	595.69
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>n</i>	<i>P</i> 2 <sub>1</sub> / <i>c</i>	<i>P</i> 1
<i>a</i> (Å)	5.0787(1)	11.2075(2)	8.3926(2)
<i>b</i> (Å)	14.7949(2)	12.9542(3)	10.6444(3)
<i>c</i> (Å)	14.5423(1)	14.0872(3)	13.5560(3)
α (°)	90	90	94.248(1)
β (°)	96.32(1)	100.84(2)	94.112(1)
γ (°)	90	90	102.422(1)
<i>V</i> (Å <sup>3</sup> )	1086.05(3)	2008.74(7)	1174.63(5)
<i>Z</i>	4	4	2
density (Mg/m <sup>3</sup> )	2.074	1.786	1.684
μ (mm <sup>-1</sup> )	1.864	1.057	1.027
<i>F</i> (000)	664	1080	596
<i>R</i> <sub>int</sub>	0.0275	0.0361	0.0777
GOF on <i>F</i> <sup>2</sup>	1.043	1.054	0.901
<i>R</i> <sub>1</sub> [ <i>I</i> > 2σ( <i>I</i> )] <sup>a</sup>	0.0249	0.0522	0.0564
<i>wR</i> <sub>2</sub> (all data) <sup>b</sup>	0.0588	0.1765	0.1727

<sup>a</sup>*R*<sub>1</sub> = Σ||*F*<sub>o</sub>|-|*F*<sub>c</sub>||/Σ|*F*<sub>o</sub>|. <sup>b</sup>*wR*<sub>2</sub> = (Σ*w*(*F*<sub>o</sub><sup>2</sup>-*F*<sub>c</sub><sup>2</sup>)/Σ*wF*<sub>o</sub><sup>2</sup>)<sup>1/2</sup>

**Table 2.** Selected bond lengths (Å) and angles (°) of  $[\text{Ag}_2(\text{NO}_2)_2(\text{L})]_n$ ,  $[\text{Ag}(\text{L})]_n(\text{NO}_3)_n$ , and  $[\text{Ag}(\text{L})]_n(\text{ClO}_4)_n$ 

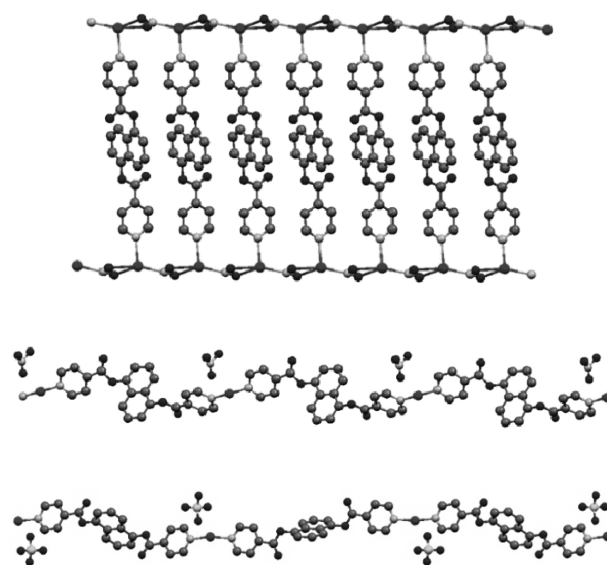
$[\text{Ag}_2(\text{NO}_2)_2(\text{L})]_n$		$[\text{Ag}(\text{L})]_n(\text{NO}_3)_n$		$[\text{Ag}(\text{L})]_n(\text{ClO}_4)_n$	
Ag–N(1)	2.375(2)	Ag–N(1)	2.157(4)	Ag–N(1)	2.17(3)
Ag–N(2)	2.277(2)	Ag–N(2) <sup>#2</sup>	2.163(4)	Ag–N(2)	2.16(3)
Ag–O(3) <sup>#1</sup>	2.488(2)	Ag⋯ONO <sub>2</sub>	2.776(5)	Ag⋯OCLO <sub>3</sub>	2.89(6)
Ag–O(4) <sup>#1</sup>	2.457(2)				3.08(4)
N(1)–Ag–N(2)	106.56(7)	N(1)–Ag–N(2)	173.4(1)	N(1)–Ag–N(2)	169.7(2)
N(1)–Ag–O(3) <sup>#1</sup>	145.84(7)	N(1)–Ag⋯O	89.4(2)		
N(2)–Ag–O(3) <sup>#1</sup>	145.84(7)	N(2)–Ag⋯O	93.9(2)		
O(3) <sup>#1</sup> –Ag–O(4) <sup>#1</sup>	50.05(6)				

#1 x+1,y,z #2 x+1,y,z-1

**Fig. 1.** ORTEP drawings of  $[\text{Ag}_2(\text{NO}_2)_2(\text{L})]_n$  (a),  $[\text{Ag}(\text{L})]_n(\text{NO}_3)_n$  (b), and  $[\text{Ag}(\text{L})]_n(\text{ClO}_4)_n \cdot n \text{H}_2\text{O}$  (c) with thermal ellipsoids at the 30% level. Hydrogen atoms were omitted for clarity.

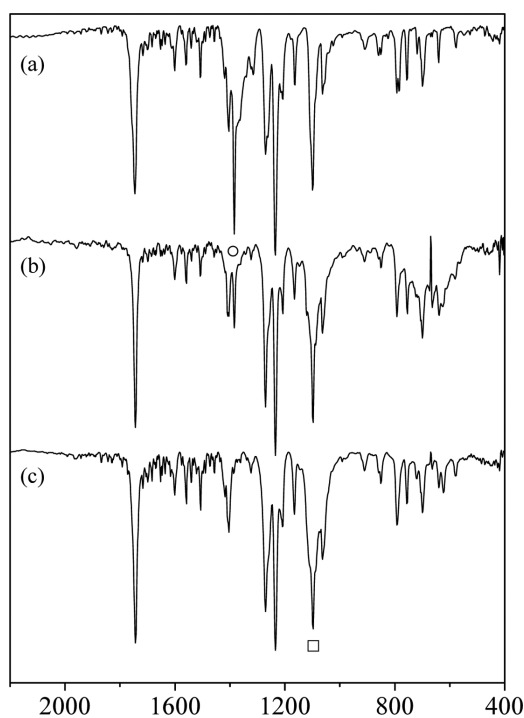
may be best described as T-shape ( $\text{N–Ag–N} = 173.4(1)^\circ$ ;  $\text{N–Ag}\cdots\text{ONO}_2 = 89.4(2), 93.9(2)^\circ$ ).

Our intentional combinations of stable **L** and a variety of Ag(I) ions have formed a series of anion-dependent motifs. Each motif was efficiently constructed irrespective of the stoichiometry, the solvent type, and the concentration. Less coordinating anions,  $\text{NO}_3^-$  and  $\text{ClO}_4^-$ , afford the spacer-abundant species, 1 : 1 adducts while the coordinating  $\text{NO}_2^-$  anions afford the 2 : 1 adducts. From the local geometry and the bond length of  $\text{Ag}\cdots\text{X}$ , the coordinating nature of the anions may be ordered as  $\text{NO}_2^- \gg \text{NO}_3^- > \text{ClO}_4^-$ . This order is consistent with the Hoffmeister

**Fig. 2.** Infinite structures of  $[\text{Ag}_2(\text{NO}_2)_2(\text{L})]_n$  (top),  $[\text{Ag}(\text{L})]_n(\text{NO}_3)_n$  (middle), and  $[\text{Ag}(\text{L})]_n(\text{ClO}_4)_n$  (bottom). Hydrogen atoms are omitted for clarity.

series that is usually given in terms of the ability of anions to stabilize the structure of macromolecules.<sup>28</sup> The overall motifs including the local geometry and mole ratio of the products appear to be delicately associated with the nature of anions. Of course,  $\pi$ - $\pi$  interactions are another important factor for the formation of the present skeletons.

The anions at the surface of the molecular strands can be exchanged in a typical aqueous media.<sup>6</sup> To investigate the exchange procedure, the anion exchange of  $[\text{Ag}(\text{L})]_n(\text{NO}_3)_n$  with  $\text{ClO}_4^-$  was monitored by the characteristic IR bands of the anions. The addition of  $\text{NaClO}_4$  to a suspension of microcrystalline  $[\text{Ag}(\text{L})]_n(\text{NO}_3)_n$  in water with a 5 : 1 mole ratio showed that, after 40 min, the  $\text{NO}_3^-$  anions are successively exchanged with  $\text{ClO}_4^-$  anions. In detail, intense  $\text{ClO}_4^-$  band at  $1087 \text{ cm}^{-1}$  appears while the  $\text{NO}_3^-$  band at  $1384 \text{ cm}^{-1}$  disappears (Fig. 3). The other peaks of the spec-



**Fig. 3.** Partial IR spectra during the anion exchange of  $[\text{Ag}(\text{L})]_n(\text{NO}_3)_n$  with  $\text{NaClO}_4$  after 0 min (a), 20 min (b), and 40 min (c). The circle and square indicate the  $\text{NO}_3^-$  and  $\text{ClO}_4^-$  bands, respectively.

trum remain virtually unchanged, suggesting the preservation of the skeletal structure during the anion exchange process. The reverse exchange, however, was not easily carried out under the same conditions. The reverse exchange reaction is unfavorable both in entropy and water-solubility.  $[\text{Ag}(\text{L})]_n(\text{ClO}_4)_n$  is hardly soluble in water, but  $[\text{Ag}(\text{L})]_n(\text{NO}_3)_n$  is marginally soluble in water. That is, the anion exchangeability is strongly dependent on both water-solubility and entropy rather than the anion coordinating ability.

In conclusion, the formation of skeletal structure is strongly dependent upon the coordinating ability of anions. An ideal ladder coordination polymer was constructed via self-assembly of Ag(I) ions with a new bipyridyl spacer. The results may contribute to the design and development of desirable materials such as anion sensors, molecular control,<sup>30</sup> ion exchangers, chemical delivery, and intercalators.

## EXPERIMENTAL

All chemicals including 1,5-dihydroxynaphthalene and  $\text{AgX}$  ( $\text{X} = \text{NO}_2^-$ ,  $\text{NO}_3^-$ , and  $\text{ClO}_4^-$ ) were purchased from Aldrich, and used without further purification. Infrared spectra were obtained on a Nicolet 380 FTIR spectrom-

eter with samples prepared as KBr pellets.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Varian Mercury Plus 300 operating at 300.00 MHz and 75.00 MHz, respectively. Elemental microanalyses (C, H, N) were performed on solid samples by the Pusan center, KBSI, using a Vario-EL III.

### 1,5-Bis(isonicotinoyloxy)naphthalene (L)

Triethylamine (10.56 mmol, 1.47 mL) was added to a stirred mixture of 1,5-dihydroxynaphthalene (2 mmol, 0.320 g) and isonicotinoyl chloride hydrochloride (4.8 mmol, 0.854 g) in chloroform (120 mL) at room temperature. The reaction mixture was refluxed for 24 h. The solution was filtered, and then the filtrate was washed with 0.5 N NaOH and water several times. The chloroform layer was dried using magnesium sulfate and filtered. Evaporation of the chloroform gave a deep red product. The product was finally recrystallized in a solvent pair of chloroform and *n*-hexane to give **L** as yellow crystals in a 79% yield. IR (KBr): 1743(s), 1600(m), 1407(s), 1234(s), 1097(s), 792(m)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$ 8.95 (d,  $J = 4.2$  Hz, 4H), 8.14 (d,  $J = 4.2$  Hz, 4H), 7.88 (d,  $J = 8.7$  Hz, 2H), 7.59 (d,  $J = 7.5$  Hz, 2H), 7.45 (d,  $J = 7.2$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta$ 179.86, 151.19, 146.70, 128.19, 126.55, 123.39, 119.75, 119.17, 100.09; *Anal.* Calcd. for  $\text{C}_{22}\text{H}_{14}\text{N}_2\text{O}_4$ : C, 71.35; H, 3.81; N, 7.56. Found: C, 71.34; H, 3.81; N, 7.56.

### Synthesis of $[\text{Ag}_2(\text{NO}_2)_2(\text{L})]_n$

An ethanol solution (5 mL) of  $\text{AgNO}_2$  (0.01 mmol, 0.0015 g) was layered onto a tetrahydrofuran solution (5 mL) of **L** (0.005 mmol, 0.0019 g). After 7 days, yellow crystals suitable for X-ray crystallography were obtained in a 57% yield (0.0020 g) based on Ag(I) salt. IR (KBr): 1743(s), 1558(m), 1407(s), 1270(s,  $\mu(\text{NO}_2^-)$ ), 1234(s), 1097(m), 792(m)  $\text{cm}^{-1}$ ; *Anal.* Calcd. for  $\text{C}_{23}\text{H}_{17}\text{N}_4\text{O}_8\text{Ag}_2$ : C, 39.85; H, 2.47; N, 8.08. Found: C, 39.82; H, 2.50; N, 8.09.

### Synthesis of $[\text{Ag}(\text{L})]_n(\text{NO}_3)_n$

A methanol solution (5 mL) of  $\text{AgNO}_3$  (0.01 mmol, 0.0017 g) was layered onto a dichloromethane solution (5 mL) of **L** (0.01 mmol, 0.0037 g). After 4 days, yellow crystals suitable for X-ray crystallography were obtained in a 77% yield (0.0042 g) based on Ag(I) salt. IR (KBr): 1745(s), 1600(m), 1403(m), 1384(s,  $\mu(\text{NO}_3^-)$ ), 1234(s), 1099(s), 792(m)  $\text{cm}^{-1}$ ; *Anal.* Calcd. for  $\text{C}_{22}\text{H}_{14}\text{N}_3\text{O}_7\text{Ag}$ : C, 48.91; H, 2.61; N, 7.78. Found: C, 48.91; H, 2.63; N, 7.79.

### Synthesis of $[\text{Ag}(\text{L})]_n(\text{ClO}_4)_n \cdot n\text{H}_2\text{O}$

A methanol solution (5 mL) of  $\text{AgClO}_4$  (0.01 mmol, 0.002 g) was layered onto a tetrahydrofuran solution (5

mL) of **L** (0.01 mmol, 0.0037 g). After 4 days, yellow crystals suitable for X-ray crystallography were obtained in a 78% yield (0.0045 g) based on Ag(I) salt. IR (KBr): 1743(s), 1600(m), 1407(m), 1234(s), 1097(s), 1087(s,  $\mu(\text{ClO}_4^-)$ ), 792(m)  $\text{cm}^{-1}$ ; *Anal.* Calcd. for  $\text{C}_{22}\text{H}_{16}\text{N}_2\text{O}_9\text{ClAg}$ : C, 44.36; H, 2.71; N, 4.70. Found: C, 44.33; H, 7.76; N, 4.68.

### Crystal Structure Determinations

X-ray data were collected on a Bruker SMART automatic diffractometer with a graphite-monochromated  $\text{Mo K}\alpha$  ( $\lambda=0.71073 \text{ \AA}$ ) and a CCD detector at ambient temperature. Thirty six frames of two dimensional diffraction images were collected and processed to obtain the cell parameters and orientation matrix. The data were corrected for Lorentz and polarization effects. Absorption effects were corrected by the multi-scan method. The structures were solved by the direct method (SHELXS 97) and refined by full-matrix least squares techniques (SHELXL 97).<sup>31</sup> The non-hydrogen atoms were refined anisotropically, and hydrogen atoms were placed in calculated positions and refined only for the isotropic thermal factors. Crystallographic data for the structures reported here have been deposited with the Cambridge Crystallographic Data Centre (CCDC-831384 for  $[\text{Ag}_2(\text{NO}_2)_2(\text{L})]_n$ , 831385 for  $[\text{Ag}(\text{L})]_n(\text{NO}_3)_n$ , and 831386 for  $[\text{Ag}(\text{L})]_n(\text{ClO}_4)_n \cdot n\text{H}_2\text{O}$ ). The data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/perl/catreq/catreq.cgi> (or from the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1233 336033; e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk)).

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