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Synthesis and Structure of $\{[Ag(SalGly)]\cdot 0.33H_2O\}_n$: An Infinite Double Helical Coordination Polymer

Andrea Erxleben

Fachbereich Chemie, Universität Dortmund,
44221 Dortmund, Germany

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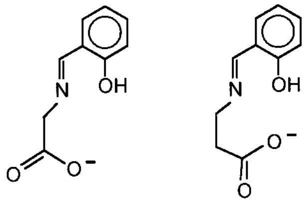
Introduction

Helical structures receive much current attention in coordination chemistry and materials science.[1−13] Reasons for the increasing interest and fascination are the fundamental role of helicity in biology, potential applications in the fields of supramolecular chemistry, asymmetric catalysis, and nonlinear optical materials and the aesthetically appealing topology of helical compounds. Consequently, much effort has been devoted to the preparation of helical metal complexes by careful design of helicating ligands, and since the pioneering work of Lehn[14] a large number of di- and oligonuclear helical complexes have been reported in the literature.[15] In these so-called *helicates* the ligands that are often oligopyridines twist around the helix axis defined by the metal ions. Besides, there are various examples of metallohelices where a 1D coordination polymer spontaneously assumes a helical conformation.[16] However, while numerous di- and oligonuclear double helicates have been structurally characterized to date, infinite double helices are quite rare.[17−19]

Chart 1



SalGly Sal-β-Ala

In the case of silver compounds, only three previous examples have been reported, namely, $[Ag(SCEt_2Me)]_n$,[20] $\{[Ag(1,3\text{-bis-}(4\text{-pyridyl})propane)](CF_3SO_3)\}_n$,[21] and the double helix of $\{[AgL](PF_6)\}_n$, with L being a chiragen derivative.[22,23] Recently, a triple helical Ag coordination polymer was structurally characterized.[24]

In this report, the solid-state structure of a double helical silver coordination polymer derived from the Schiff base ligand α-*N*-(*o*-hydroxybenzyl)glycinate (SalGly, Chart 1) is described along with the crystal structure of polymeric $\{[Ag_2(\beta\text{-ala})_2(NO_3)]NO_3\}_n$ (β-ala = β-alanine) that was obtained in the course of studies on the related Schiff base ligand α-*N*-(*o*-hydroxybenzyl)-*β*-alaninate (Sal-*β*-Ala).

Experimental Section

General. ^{1}H NMR spectra were recorded on a Bruker AC200 spectrometer using sodium 3-(trimethylsilyl)propanesulfonate as internal reference. Infrared spectra were taken on a Bruker IFS 28 FT-spectrometer. All chemicals and solvents were reagent grade and used without further purification.

$\{[Ag(SalGly)]\cdot 0.33H_2O\}_n$ (1·0.33H$_2$O). $AgNO_3$ (812 mg, 4.78 mmol) and glycine (359 mg, 4.78 mmol) were dissolved in water (30 mL) and added to a solution of salicylaldehyde (500 μL, 4.78 mmol) in methanol (30 mL). After overnight stirring at room temperature, a yellow precipitate was filtered off and washed with diethyl ether: yield 250 mg (18%). Recrystallization from methanol at 4 °C afforded X-ray suitable needles of **1·0.33H$_2$O**. Selected IR data (cm^{-1}): 3397 (vbr), 1636 (s), 1591 (s), 1507 (m), 1402 (s), 1297 (m), 1219 (m), 1198 (m), 1146 (m), 1075 (m), 924 (m), 755 (m), 739 (m), 667 (m), 602 (m). ^{1}H NMR (DMSO-d_6): δ = 8.47 (s, 1H, azomethine), 7.45 (d, 1H, aryl), 7.32 (t, 1H, aryl), 6.84 (d, 2H, aryl), 4.19 (s, 2H, =*N*-CH$_2$).

Anal. Calcd for $C_9H_8AgNO_3$ (M_r = 286.04): C, 37.8; H, 2.8; N, 4.9. Found: C, 38.0; H, 3.2; N, 5.0.

[Ag(Sal-*β*-Ala)] (2) and $\{[Ag_2(\beta\text{-ala})_2(NO_3)]NO_3\}_n$ (3). $AgNO_3$ (163 mg, 0.96 mmol) and β-alanine (85 mg, 0.95 mmol) were dissolved

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Table 1. Crystallographic Data

	$1\cdot0.33H_2O$	**3**
emp formula	$C_9H_8AgNO_3\cdot0.33H_2O$	$C_6H_{14}Ag_2N_4O_{10}$
fw	292.04	517.95
cryst syst	rhombohedral	monoclinic
space group	*R*3	*Cc*
a, Å	23.001(3)	8.455(1)
b, Å		25.509(1)
c, Å	4.649(1)	6.461(1)
β,°		96.15(1)
V, Å^3	2130.0(6)	1385.5(3)
Z	9	4
d(calcd), g cm^{-3}	2.049	2.483
μ(Mo Kα), mm^{-1}	2.111	2.887
T, K	293	293
R1, wR2 [$I > 2\sigma(I)$][a]	0.050, 0.151	0.025, 0.062
R1, wR2 (all data)[a]	0.059, 0.173	0.041, 0.129

[a] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR2 = [\Sigma w(F_o^2 - F_c^2)^2 / \Sigma w(F_o^2)^2]^{1/2}$; $w^{-1} = \sigma^2(F_o^2) + (aP)^2$; $P = (F_o^2 + 2F_c^2)/3$, $a = 0.121$ for **$1\cdot0.33H_2O$** and 0.0427 for **3**.

Table 2. Selected Bond Lengths (Å) and Angles (deg) for **$1\cdot0.33H_2O$**

Ag−N(1)	2.269(9)	Ag···Ag[b]	3.193(1)
Ag−O(2)[a]	2.226(7)	Ag···Ag[c]	3.193(1)
Ag−O(2)	2.344(7)		
N(1)−Ag−O(2)	73.5(3)	N(1)−Ag−O(2)[a]	147.9(3)
O(2)−Ag−O(2)[a]	136.4(3)	Ag−O(2)−Ag[b]	131.7(3)

[a−c] Symmetry operations: [a] $-y + {}^1/_3 + 1, x - y + {}^2/_3, z + {}^2/_3$; [b] $-y + {}^1/_3 + 1, x - y + {}^2/_3, z - {}^1/_3$; [c] $-x + y + {}^2/_3, -x + {}^1/_3 + 1, z + {}^1/_3$.

Table 3. Selected Bond Lengths (Å) and Angles (deg) for **3**

Ag(1)−O(1)	2.147(5)	Ag(2)−O(2)	2.287(5)
Ag(1)−O(3)	2.139(5)	Ag(2)−O(4)	2.235(5)
Ag(1)−O(5)[a]	2.440(5)	Ag(2)−O(6)	2.384(4)
Ag(1)···Ag(2)	2.8066(7)	Ag(2)···Ag(2)[b]	3.2808(5)
O(1)−Ag(1)−O(3)	164.8(2)	O(2)−Ag(2)−O(4)	161.8(2)
O(1)−Ag(1)−O(5)[a]	97.9(2)	O(2)−Ag(2)−O(6)	81.4(2)
O(3)−Ag(1)−O(5)[a]	95.0(2)	O(4)−Ag(2)−O(6)	116.8(2)
O(1)−Ag(1)···Ag(2)	84.2(1)	O(2)−Ag(2)···Ag(1)	80.6(1)
Ag(2)[b]···Ag(2)···Ag(2)[c]	159.90(4)		

[a−c] Symmetry operations: [a] $x - 1, -y, z - {}^1/_2$; [b] $x, -y, z - {}^1/_2$; [c] $x, -y, z + {}^1/_2$.

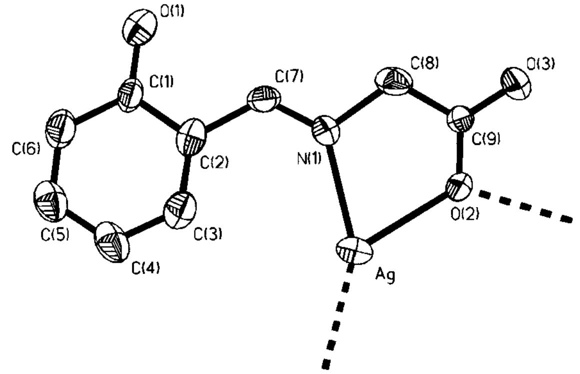

Figure 1. Asymmetric unit of the crystal structure of **$1\cdot0.33H_2O$** with the atom numbering scheme. The water molecule of crystallization is omitted for clarity.

in water (6 mL) and added to a solution of salicylaldehyde (100 μL, 0.96 mmol) in methanol (6 mL). Overnight stirring yielded a yellow precipitate (148 mg) of composition $[Ag(C_{10}H_{10}NO_3)]$ (**2**). Selected IR data (cm^{-1}): 3284 (s, br), 1644 (s), 1587 (s), 1506 (s), 1423 (m), 1306 (m), 1219 (m), 1196 (m), 1049 (m), 856 (m), 742 (m). ^{1}H NMR (DMSO-d_6): $\delta = 13.67$ (s, br, 1H, OH), 8.56 (s, 1H, azomethine), 7.42 (d, 1H, aryl), 7.33 (d, 1H, aryl), 6.86 (t, 2H, aryl), 3.80 (t, 2H, =*N*-CH$_2$), 2.48 (t, 2H, CH$_2$).

Anal. Calcd for $C_{10}H_{10}AgNO_3$ ($M_r = 300.07$): C, 40.0; H, 3.4; N, 4.7. Found: C, 39.6; H, 3.3; N, 4.5.

Slow evaporation of the filtrate at 4 °C afforded colorless plates of $\{[Ag_2(\beta\text{-ala})_2(NO_3)]NO_3\}_n$ (**3**) (109 mg). Selected IR data (cm^{-1}): 2930 (s, vbr), 1634 (s), 1574 (s), 1509 (s), 1387 (s, br), 1157 (m), 1089 (m), 1042 (m), 991 (m), 840 (m), 653 (m), 590 (w), 537 (w), 403 (m), 328 (m). ^{1}H NMR (D$_2$O, pD 6.1): $\delta = 3.17$ (t, 4H, *N*-CH$_2$), 2.56 (t, 4H, CH$_2$).

Anal. Calcd for $C_6H_{14}Ag_2N_4O_{10}$ ($M_r = 517.95$): C, 13.9; H, 2.7; N, 10.8. Found: C, 13.8; H, 2.5; N, 10.9.

Crystal Structure Analyses. Crystal data were collected at room temperature on an Enraf-Nonius-KappaCCD diffractometer[25] using graphite-monochromated Mo Kα radiation ($\lambda = 0.71069$ Å). For data reduction and cell refinement the programs DENZO and SCALEPACK were applied.[26] The structures were solved by conventional Patterson methods and subsequent Fourier syntheses and refined by full-matrix least squares on F^2 using the SHELXTL PLUS and SHELXL-93 programs.[27] The scattering factors were those given in the SHELXTL PLUS program. The Flack parameters[28] converged to $x = 0.08(9)$ for **$1\cdot0.33H_2O$** and to −0.01(4) for **3**. Except for C(9) and O(1w) in **$1\cdot0.33H_2O$** all non-hydrogen atoms were refined anisotropically. Carbon-bound hydrogen atoms were placed at calculated positions and given fixed isotropic temperature factors.

Crystallographic data and details of refinement are reported in Table 1. Selected bond lengths and angles are listed in Tables 2 and 3.

Results and Discussion

$\{[Ag(SalGly)]\cdot0.33H_2O\}_n$ ($1\cdot0.33H_2O$). The title compound was obtained by reacting AgNO$_3$ with glycine and salicylaldehyde in methanol−water followed by recrystallization from methanol. The elemental C, H, and N analysis and NMR spectroscopic characterization are consistent with the formation of a 1:1 silver complex of the Schiff base ligand SalGly. X-ray analysis revealed a one-dimensional, double helical coordination polymer of composition $\{[Ag(SalGly)]\cdot0.33H_2O\}_n$ (**$1\cdot0.33H_2O$**). Figure 1 gives a view of the asymmetric unit; relevant bond lengths and angles are listed in Table 2. Silver coordinates to the imine nitrogen and carboxylate oxygen of the side arm of the tridentate ligand with the Ag−O and Ag−N bond lengths falling in the usual range [Ag−N(1) 2.269(9) Å and Ag−O(2) 2.344(7) Å], while the phenolic oxygen does not participate in the coordination. The carboxylate oxygen forms a monatomic bridge to the next silver so that each metal ion has an NO$_2$ coordination sphere. Owing to the narrow bite of the chelating ligand side arm, silver assumes a distorted Y-shaped coordination geometry with the metal center being displaced by 0.170(5) Å out of the least-squares plane defined by the donor atoms. The carboxylate-bridged Ag centers are 3.193(1) Å apart, and the Ag−O(2)−Ag and O(2)−Ag−O(2a) angles are 131.7(3)° and 136.4(3)°, respectively. The Ag−O bonds are slightly asymmetric [Ag−O(2) 2.344(7) Å and Ag−O(2) 2.226(7) Å, $-y + {}^1/_3 + 1, x - y + {}^2/_3, z + {}^2/_3$]. The formation of monatomic carboxylate bridges, which is relatively rare in metal carboxylate complexes,[29] results in an infinite chain of alternating silver and oxygen atoms extending along a 3-fold screw axis. Two

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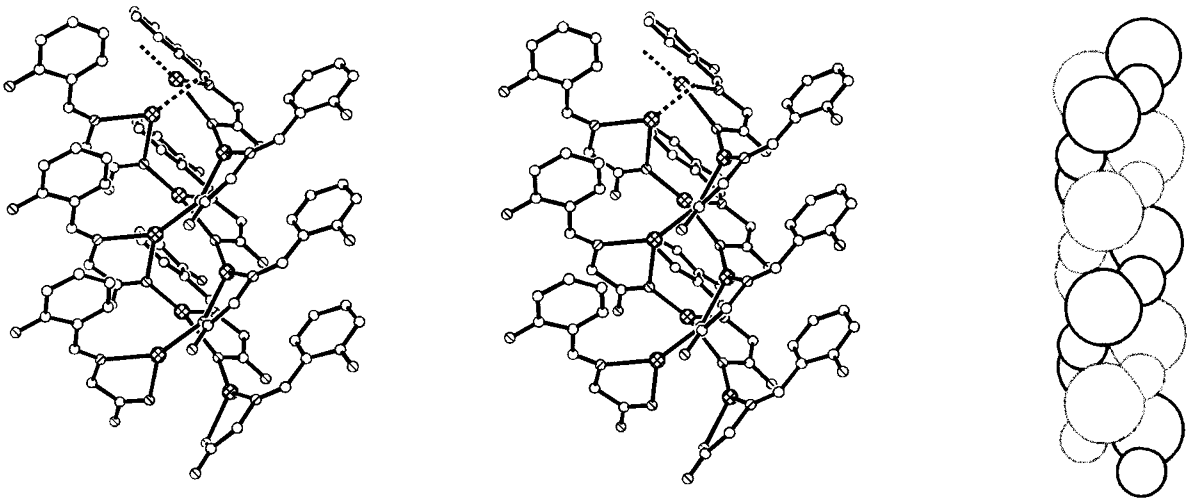

Figure 2. Stereoview of a section of the infinite double helix of **1·0.33H₂O**: (left) Ag, crosshatched circles; N,O, hatched circles; C, empty circles; (right) space-filling presentation of the intertwined Ag−O chains.

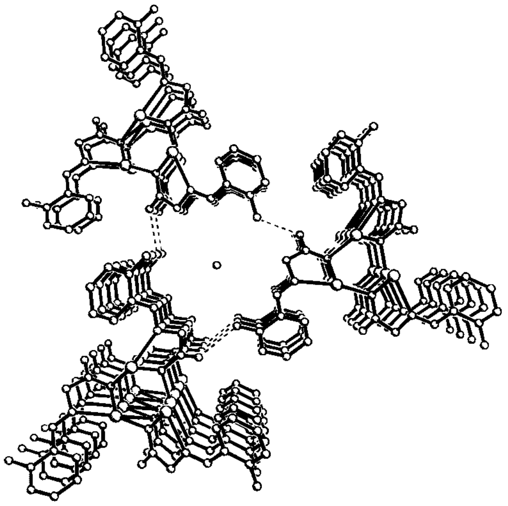

Figure 3. Crystal packing of **1·0.33H₂O** viewed down the *z*-axis.

unconnected helical chains then wind around each other to form a double helix as shown in Figure 2. The helix has a pitch height of 9.298(1) Å and a diameter of about 13 Å. In the crystal lattice, three double helices are connected through hydrogen-bonding interactions involving the phenolic OH groups and the uncoordinated carboxylate oxygens O(3) [O(1)···O(3) 2.67(2) Å, $-x + y + 1$, $-x + 1$, $z + 1$] so that long channels along the crystallographic *z*-direction are produced (Figure 3). These channels having diameters of about 5 Å are filled with water molecules of crystallization.

An interesting point is the driving force for the intertwining of the two separate chains to the double helix. Neighboring phenolic rings of the two strands are essentially coplanar—the dihedral angle between the ring planes is 15.2(9)°—but the distance of about 4.6 Å clearly rules out stabilization by π-stacking interactions. Stabilization by interchain hydrogen-bonding interactions can likewise be excluded. The shortest interstrand Ag···Ag contact is 3.193(1) Å which is less than twice the van der Waals radii for silver (3.44 Å[30a]) and may indicate weak (ligand-unsupported) metallophilic ($d^{10}-d^{10}$) interactions. However, the assignment of bonding or nonbonding interactions is not unambiguous and the presence of metal−metal bonding is difficult to assess on the basis of the metal−metal distance.[31] For example, the stabilization of the double helix in $\{[Ag(1,3\text{-bis(4-pyridyl)propane})](CF_3SO_3)\}_n$[21] where adjacent Ag ions are 3.089(1) Å apart is attributed to weak interstrand Ag···Ag interactions, while the metal−metal distance of 2.886(4) Å found in double helical $[Ag(SCEt_2Me)]_n$[20] is interpreted as nonbonding. Short $M^I\cdots M^I$ contacts in the absence of supportive bridging ligands are of particular interest, and several examples of unsupported Ag···Ag attractive interactions are documented in the literature, for example, $\{[Ag(2,4'\text{-bipyridine})]ClO_4\}_n$ [3.1526(6) Å],[16c] $\{[Ag_2(\text{trimethylammoniopropionate})_2(H_2O)](ClO_4)_2\}_n$ [3.269(2) and 3.346(2) Å],[32a] $[Ag_3(NO_3)_3(4,4'\text{-dipyridyl sulfide})_2(H_2O)]_n$ [3.436(2) Å],[32b] $[Ag_3(2\text{-}(3(5)\text{-pz})py)_3]_2$ [3.227(2) Å] (2-(3(5)-pzH)py = 2-[3(5)-pyrazolyl]pyridine),[32c] $\{[Ag_2(H_2L)_3](NO_3)_2\}_n$ [2.934(2) Å] and $\{[Ag_2(H_2L)_3](ClO_4)_2\}_n$ [2.946(2) Å] (H_2L = *N*,*N*′-bis(salicylidene)-1,4-diaminobutane).[32d]

[Ag(Sal-*β*-Ala)] (2) and {[Ag₂(*β*-ala)₂(NO₃)]NO₃}*ₙ* (3). Treating $AgNO_3$ and salicylaldehyde with β-alanine in methanol−water resulted in precipitation of a yellow powder of composition $[Ag(C_{10}H_{10}NO_3)]$ (**2**). Schiff base formation can be deduced from the ^1H NMR spectrum (DMSO-d_6) displaying a signal of an azomethine proton at 8.56 ppm. As is evident from a broadened resonance at 13.67 ppm, the phenolic oxygen is protonated as found for **1** and does not bind to silver. No bands corresponding to nitrate as a potential counterion are observed in the infrared spectrum, which is consistent with the proposed formula [Ag(Sal-β-Ala)] for **2**.

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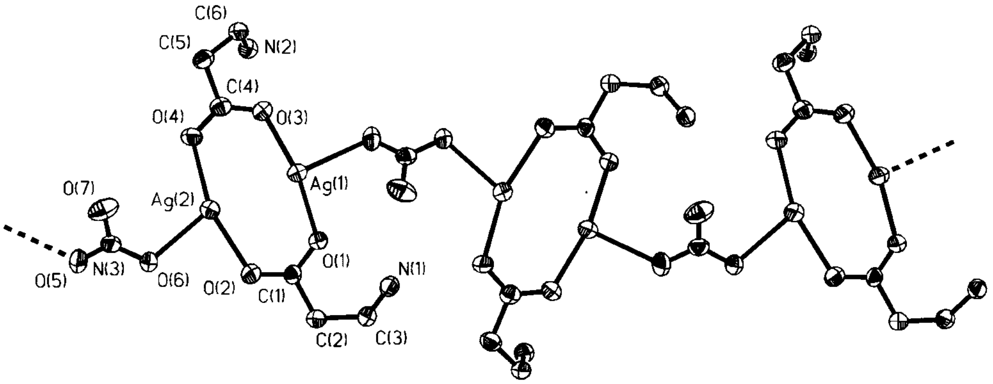

Figure 4. Section of the polymeric cation of $\{[Ag_2(\beta\text{-ala})_2(NO_3)]NO_3\}_n$.

Concentration of the filtrate of the reaction mixture by slow evaporation resulted in crystallization of the β-alanine complex $\{[Ag_2(\beta\text{-ala})_2(NO_3)]NO_3\}_n$ (**3**). The crystal structure of $[Ag_2(\beta\text{-ala})_2](NO_3)_2$ has been reported by Kamwaya et al.,[33] and the basic coordination pattern in both complexes is identical: Two silver ions are bridged by two β-alanine carboxylate groups at a distance of 2.855(4) Å in $[Ag_2(\beta\text{-ala})_2](NO_3)_2$ and 2.8066(7) Å in **3**. However, while $[Ag_2(\beta\text{-ala})_2](NO_3)_2$ contains discrete, centrosymmetric $Ag_2(\beta\text{-ala})_2$ units, in **3** dinuclear $Ag_2(\beta\text{-ala})_2$ entities are linked through bridging, bidentate nitrates [Ag−O 2.384(4) and 2.440(5) Å] to an infinite chain (Figure 4).[34] Thus, the silver ions have T-shaped O_3 coordination spheres in **3**. In the crystal packing, short contacts between Ag(2) ions of neighboring chains are observed [Ag(2)···Ag(2) 3.2808(5) Å, $x, -y, z - {}^1/_2$] with an Ag(2)−Ag(2)−Ag(2) angle of 159.90(4)°.

In summary, two 1D Ag coordination polymers have been characterized, one of which features a double helical structure. Future work will involve other Schiff base ligands derived from related aldehydes and amines to further explore the structural versatility of Ag coordination compounds.

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Supporting Information Available: Packing diagram of compound **3**. Two crystallographic files, in CIF format. This material is available free of charge via the Internet at http://pubs.acs.org.

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