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A Remarkable Enantioselectivity of Diastereomers of an L-Cysteinato Co^{III}_3 Complex-anion toward a Racemic 2-Aminoethanethiolato $Co^{III}_2Ag^{I}_3$ Complex-cation

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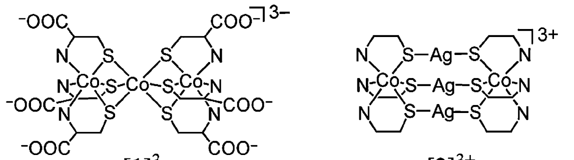
The $(\Lambda_{LLL})_2$ isomer of a Co^{III}_3 trinuclear complex anion, $[Co_3(L\text{-}cys)_6]^{3-}$, selected exclusively the $(\Lambda)_2$ isomer of a $Co^{III}_2Ag^{I}_3$ pentanuclear complex cation, $[Ag_3Co_2(aet)_6]^{3+}$, from its racemic $(\Delta)_2/(\Lambda)_2$ isomer, giving only the $(\Lambda)_2(\Lambda_{LLL})_2$ isomer of a 1:1 complex salt, $[Ag_3Co_2(aet)_6][Co_3(L\text{-}cys)_6]$. Notably, the $(\Lambda)_2$ isomer of $[Ag_3Co_2(aet)_6]^{3+}$ was also selected, but preferentially, by the $(\Delta_{LLL})_2$ isomer of $[Co_3(L\text{-}cys)_6]^{3-}$ in the course of the formation of $[Ag_3Co_2(aet)_6][Co_3(L\text{-}cys)_6]$.

The recent development of asymmetric synthesis and optical resolution of compounds has been driven by the great demand for the isolation of compounds in an optically pure form.[1] In coordination chemistry, optical resolution by use of an appropriate resolving agent has long been a common method to obtain optically pure complexes.[2] As resolving reagents, not only organic/complex ions with asymmetric carbon atom(s) but also those with a helicoidal chirality have widely been used for optical resolution.[3] In many cases, these resolving reagents are available only for a selected metal complex, and thus, it is desirable to explore efficient reagents that can be employed for a wide range of coordination compounds. Chiral multinuclear species would be a potential candidate for this requirement because of the presence of multiple chiral centers.[3,4] In addition, the formation of two or more diastereomers for these species due to the combination of multiple chiral centers provides the chance to improve the resolving ability by the control of their stereoisomerism. Recently, we have reported that the $(\Lambda_{LLL})_2$ isomer of an anionic S-bridged Co^{III}_3 complex with L-cysteinate (L-cys), $[Co_3(L\text{-}cys)_6]^{3-}$ ($[\mathbf{1}]^{3-}$), acts as an efficient resolving reagent toward the racemic $(\Delta)_2/(\Lambda)_2$ isomer of the corresponding cationic Co^{III}_3 complex with 2-aminoethanethiolate (aet), $[Co_3(aet)_6]^{3+}$, producing only the heterochiral $(\Delta)_2(\Lambda_{LLL})_2$ isomer of a 1:1 complex-salt, $[Co_3(aet)_6][Co_3(L\text{-}cys)_6]$, while its $(\Delta_{LLL})_2$ isomer did not show any enantioselectivity toward the racemic $[Co_3(aet)_6]^{3+}$.[5] To investigate the applicability of the diastereomers of $[\mathbf{1}]^{3-}$ as an effective resolving agent toward another complex-cation and to better understand the factors that govern the chiral recognition between complex-cations and anions of this class, we investigated the reactions of $(\Lambda_{LLL})_2$- and $(\Delta_{LLL})_2$-$[\mathbf{1}]^{3-}$ with the racemic $(\Delta)_2/(\Lambda)_2$ isomer of a $Co^{III}_2Ag^{I}_3$ pentanuclear complex, $[Ag_3Co_2(aet)_6]^{3+}$ ($[\mathbf{2}]^{3+}$), which has a tricationic S-bridged structure very similar to that of $[Co_3(aet)_6]^{3+}$, except the presence of a $\{Ag_3\}^{3+}$ moiety between two terminal $[Co(aet)_3]$ units in place of a Co^{3+} ion in $[Co_3(aet)_6]^{3+}$. In this paper, we report that the $(\Lambda_{LLL})_2$ isomer of $[\mathbf{1}]^{3-}$ does not select the $(\Delta)_2$ isomer of $[\mathbf{2}]^{3+}$ but its $(\Lambda)_2$ isomer to give exclusively a homochiral 1:1 complex salt. A remarkable chiral recognition behavior of the $(\Delta_{LLL})_2$ isomer of $[\mathbf{1}]^{3-}$, which selects preferentially the $(\Lambda)_2$ isomer of $[\mathbf{2}]^{3+}$, is also reported (Scheme 1).



$[\mathbf{1}]^{3-}$ $[\mathbf{2}]^{3+}$

$(\Lambda_{LLL})_2$-$[\mathbf{1}]^{3-}$ + $(\Delta)_2/(\Lambda)_2$-$[\mathbf{2}]^{3+}$ ⟶ **3** : $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ only

$(\Delta_{LLL})_2$-$[\mathbf{1}]^{3-}$ + $(\Delta)_2/(\Lambda)_2$-$[\mathbf{2}]^{3+}$ ⟶ **4** : $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ rich

Scheme 1. Chiral selectivity between the isomers of $[Co_3(L\text{-}cys)_6]^{3-}$ ($[\mathbf{1}]^{3-}$) and $[Ag_3Co_2(aet)_6]^{3+}$ ($[\mathbf{2}]^{3+}$).

Treatment of the racemic $(\Delta)_2/(\Lambda)_2$ isomer of $[Ag_3Co_2(aet)_6](BF_4)_3$ ($[\mathbf{2}](BF_4)_3$)[6] with the $(\Lambda_{LLL})_2$ isomer of $K_3[Co_3(L\text{-}cys)_6]$ ($K_3[\mathbf{1}]$)[7] in a 1:1 ratio in water afforded black plate crystals **3** in a reasonable yield.[8] X-ray fluorescence spectrometry indicated the presence of Co and Ag atoms in **3**, and its elemental analytical data were consistent with the formula for a 1:1 adduct of $[Ag_3Co_2(aet)_6]^{3+}$ and $[Co_3(L\text{-}cys)_6]^{3-}$.[9] In the IR spectrum, **3** shows a strong C=O stretching band at $1616\,cm^{-1}$, indicating that L-cys carboxylate groups exist in a deprotonated form.[8,10] From these data, together with the absorption spectrum of **3** that is identical with the 1:1 mixture of $[\mathbf{2}]^{3+}$ and $[\mathbf{1}]^{3-}$, it is confidently concluded that **3** is a 1:1 complex-salt of $[\mathbf{2}]^{3+}$ and $[\mathbf{1}]^{3-}$, $[Ag_3Co_2(aet)_6][Co_3(L\text{-}cys)_6]$. To check the enantioselectivity of $[\mathbf{2}]^{3+}$ incorporated in **3**, an aqueous solution of the bulk sample of **3** was chromatographed on a cation-exchange column (SP-Sephadex C-25). When the column was treated with water, a brownish green band containing $(\Lambda_{LLL})_2$-$[\mathbf{1}]^{3-}$ was eluted, while a purple band containing $[\mathbf{2}]^{3+}$ was adsorbed on the top of the column, which was eluted with a 0.3 M aqueous solution of $NaClO_4$. It was confirmed from the absorption and CD spectral measurements that the purple eluate contains only the $(\Lambda)_2$ isomer of $[\mathbf{2}]^{3+}$,[8] indicating the enantioselective uptake of $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ into **3** in combination with $(\Lambda_{LLL})_2$-$[\mathbf{1}]^{3-}$.

X-ray analysis was performed for a single crystal picked from the bulk sample of **3**, which revealed the presence of $[Ag_3Co_2(aet)_6]^{3+}$ ($[\mathbf{2}]^{3+}$) and $[Co_3(L\text{-}cys)_6]^{3-}$ ($[\mathbf{1}]^{3-}$) in a 1:1 ratio, besides water molecules of crystallization.[11] As shown in Figure 1, $[\mathbf{2}]^{3+}$ and $[\mathbf{1}]^{3-}$ adopt the $(\Lambda)_2$ and the $(\Lambda_{LLL})_2$ configurations, respectively, consistent with the assignment described above. In **3**, each of six carboxylate groups of $(\Lambda_{LLL})_2$-$[\mathbf{1}]^{3-}$ has an axial orientation with a *lel* (δ for Λ) conformational N,S-chelate ring,[12] which forms an intramolecular hydrogen bond with an amine group (av. $N\cdots O = 2.977(6)$ Å). In addition, one of six carboxylate groups of $(\Lambda_{LLL})_2$-$[\mathbf{1}]^{3-}$ coordinates weakly to a Ag atom of an adjacent $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cation ($Ag\text{–}O = 2.578(3)$ Å), and the other two

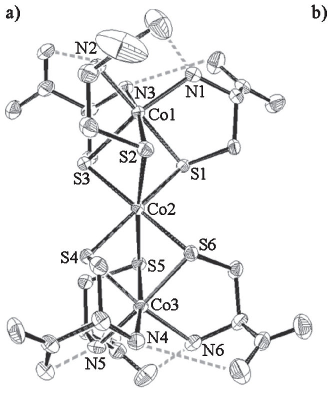


Figure 1. ORTEP drawings of a) $(\Lambda_{\mathrm{LLL}})_2$-$[\mathrm{Co}_3(\text{L-cys})_6]^{3-}$ and b) $(\Lambda)_2$-$[\mathrm{Ag}_3\mathrm{Co}_2(\mathrm{aet})_6]^{3+}$ in **3**. H atoms are omitted for clarity.

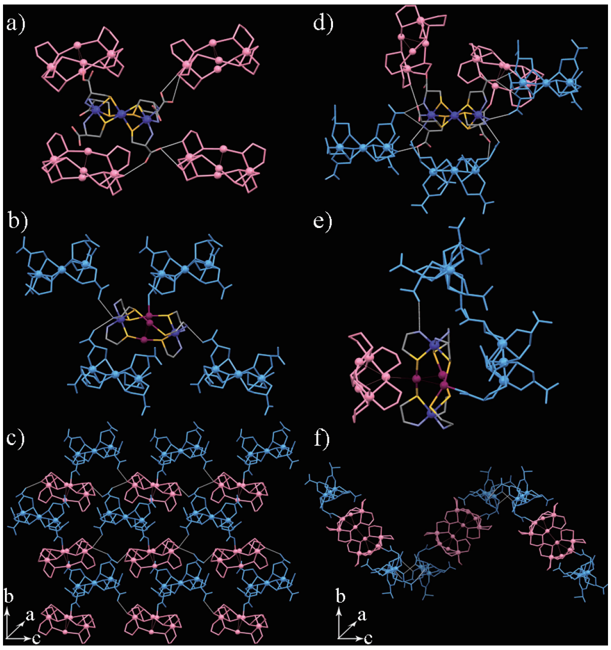


Figure 2. Intermolecular interactions around a) each complex-anion and b) each complex-cation and c) a dimensional structure in **3**. Intermolecular interactions around d) each complex-anion and e) each complex-cation and f) a dimensional structure in **4′**. Pink and blue molecules represent complex-cations and complex-anions, respectively. Expanded figures are given in Supporting Information.[8]

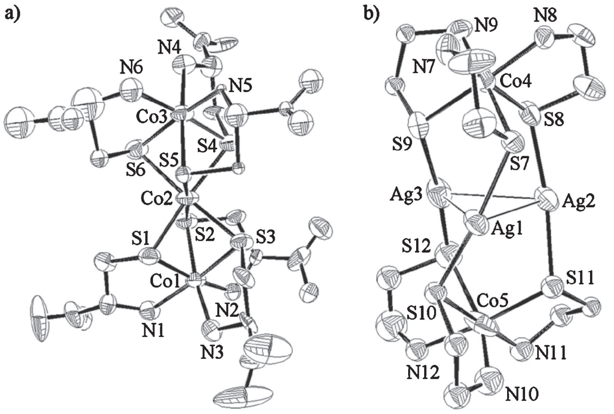


Figure 3. ORTEP drawings of a) $(\Delta_{\mathrm{LLL}})_2$-$[\mathrm{Co}_3(\text{L-cys})_6]^{3-}$ and b) $(\Lambda)_2$-$[\mathrm{Ag}_3\mathrm{Co}_2(\mathrm{aet})_6]^{3+}$ in **4′**. H atoms are omitted for clarity.

carboxylate groups are hydrogen-bonded to amine groups of three adjacent $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cations (av. N···O = 2.927(6) Å). Thus, each $(\Lambda_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anion in **3** is connected with four $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cations through a Ag–OOC coordination bond and NH···OOC hydrogen bonds (Figure 2a). Similarly, each $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cation in **3** is connected with four $(\Lambda_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anions through a Ag–OOC coordination bond and NH···OOC hydrogen bonds (Figure 2b). As a result, the $(\Lambda_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anions and the $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cations are alternately arranged to construct a 2D sheet-like structure in **3** (Figure 2c).

When the racemic $(\Delta)_2/(\Lambda)_2$ isomer of $[\mathbf{2}](\mathrm{BF}_4)_3$ was treated with the $(\Delta_{\mathrm{LLL}})_2$ isomer of $\mathrm{K}_3[\mathbf{1}]$ in a 1:1 ratio in water, a dark-brownish-green crystalline powder **4** was obtained.[8] Like **3**, **4** was characterized to be a 1:1 complex-salt of $[\mathbf{2}]^{3+}$ and $[\mathbf{1}]^{3-}$, $[\mathrm{Ag}_3\mathrm{Co}_2(\mathrm{aet})_6][\mathrm{Co}_3(\text{L-cys})_6]$, based on the absorption and IR spectroscopies, together with X-ray fluorescence and elemental analyses.[8,9] The cation-exchange column chromatography and the subsequent absorption and CD spectral measurements of the eluates indicated that the bulk sample of **4** contains both the $(\Delta)_2$ and $(\Lambda)_2$ isomers of $[\mathbf{2}]^{3+}$ in a ratio of ca. 2:3 (20% ee for the $(\Lambda)_2$ isomer),[8] besides the $(\Delta_{\mathrm{LLL}})_2$ isomer of $[\mathbf{1}]^{3-}$. Thus, the $(\Lambda)_2$ isomer of $[\mathbf{2}]^{3+}$ was preferentially incorporated into **4** in combination with $(\Delta_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$. It is reasonable to consider that the bulk sample of **4** is not a single isomer that contains $(\Lambda)_2$- and $(\Delta)_2$-$[\mathbf{2}]^{3+}$ in a 3:2, but a mixture of some isomers of $[\mathrm{Ag}_3\mathrm{Co}_2(\mathrm{aet})_6][\mathrm{Co}_3(\text{L-cys})_6]$, including $(\Lambda)_2(\Delta_{\mathrm{LLL}})_2$ and $(\Delta)_2$-$(\Delta_{\mathrm{LLL}})_2$. Indeed, a single crystal of the $(\Lambda)_2(\Delta_{\mathrm{LLL}})_2$ isomer of $[\mathrm{Ag}_3\mathrm{Co}_2(\mathrm{aet})_6][\mathrm{Co}_3(\text{L-cys})_6]$ (**4′**) was obtained from the reaction solution after the removal of the crystalline powder of **4**, and its structure was established by X-ray analysis.[13]

The overall $\mathrm{Co^{III}}_2\mathrm{Ag^{I}}_3$ pentanuclear structure of the $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cation in **4′** is very similar to that of the $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cation in **3** (Figures 1b and 3b). The $\mathrm{Co^{III}}_3$ trinuclear structure of the $(\Delta_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anion in **4′** is also similar to that of the $(\Lambda_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anion in **3** (Figures 1a and 3a). However, a significant difference is found in the orientation of carboxylate groups between the $(\Delta_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anion in **4′** and the $(\Lambda_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anion in **3**. That is, one of six carboxylate groups of the $(\Delta_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anion in **4′** has an axial orientation with *ob* (δ for Δ) conformation and another one is disordered with axial and equatorial orientations, while the remaining four carboxylate groups have an equatorial orientation with *lel* (λ for Δ) conformation.[12] In addition, each $(\Delta_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anion and each $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cation are surrounded by both of the cations and anions, which is in sharp contrast to the case for **3**. As shown in Figure 2d, each $(\Delta_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anion is connected with three adjacent $(\Delta_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anions through NH···OOC hydrogen bonds (av. N···O = 2.91(2) Å), besides with two adjacent $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cations through a Ag–OOC coordination bond (Ag–O = 2.534(11) Å) and NH···OOC hydrogen bonds (av. N···O = 3.03(5) Å). On the other hand, each $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cation is connected with two adjacent $(\Delta_{\mathrm{LLL}})_2$-$[\mathbf{1}]^{3-}$ anions through a Ag–OOC coordination bond and NH···OOC hydrogen bonds, besides with one $(\Lambda)_2$-$[\mathbf{2}]^{3+}$ cation through a Ag···Ag interaction with a distance of 2.965(3) Å (Figure 2e). As a result, in **4′** the cationic dimers of $(\Lambda)_2$-[**2**]$^{3+}$ and the anionic dimers of $(\Delta_{\mathrm{LLL}})_2$-[**1**]$^{3-}$ are alternately arranged so as to construct a 1D helix structure (Figure 2f).[14]

It was found in this study that the $(\Lambda_{\mathrm{LLL}})_2$ isomer of [**1**]$^{3-}$ ($[Co_3(\text{L-cys})_6]^{3-}$) selects exclusively the $(\Lambda)_2$ isomer of [**2**]$^{3+}$ ($[Ag_3Co_2(aet)_6]^{3+}$) from its racemic $(\Delta)_2/(\Lambda)_2$ isomer, producing only a homochiral 1:1 complex-salt, $(\Lambda)_2(\Lambda_{\mathrm{LLL}})_2$-$[Ag_3Co_2(aet)_6][Co_3(\text{L-cys})_6]$ (**3**), upon crystallization. This is in line with the enantioselectivity of this isomer toward $[Co_3(aet)_6]^{3+}$.[5] In this case, however, the $(\Delta)_2$ isomer of $[Co_3(aet)_6]^{3+}$ was selected from its racemic isomer to afford a heterochiral 1:1 complex-salt, $(\Delta)_2(\Lambda_{\mathrm{LLL}})_2$-$[Co_3(aet)_6][Co_3(\text{L-cys})_6]$. Thus, the introduction of a $\{Ag_3\}^{3+}$ moiety, in place of a central Co^{3+} ion in $[Co_3(aet)_6]^{3+}$, leads to the selectivity with an opposite chiral configuration to afford the homochiral aggregate. The presence of an intermolecular Ag–OOC coordination bond, besides NH···OOC hydrogen bonds, is most likely responsible for this result. On the other hand, the $(\Delta_{\mathrm{LLL}})_2$ isomer of [**1**]$^{3-}$ was found to show poorer enantioselectivity toward [**2**]$^{3+}$. A similar trend has been observed for the system of [**1**]$^{3-}$ and $[Co_3(aet)_6]^{3+}$, in which no crystalline product was obtained from the reaction of $(\Delta_{\mathrm{LLL}})_2$-[**1**]$^{3-}$ and $(\Delta)_2/(\Lambda)_2$-$[Co_3(aet)_6]^{3+}$ in water.[5] X-ray analytical study of **4′** revealed that the carboxylate groups in the $(\Delta_{\mathrm{LLL}})_2$ isomer of [**1**]$^{3-}$ prefer to take an equatorial orientation with a *lel* conformational N,S-chelate ring. This orientation appears to be favorable to form multiple NH···OOC hydrogen bonds with adjacent [**1**]$^{3-}$ anions, which in turn prevents effective interaction with the [**2**]$^{3+}$ cations. On the other hand, all carboxylate groups in the $(\Lambda_{\mathrm{LLL}})_2$ isomer of [**1**]$^{3-}$ have an axial orientation such that each [**1**]$^{3-}$ anion is surround by the [**2**]$^{3+}$ cations, as shown by the crystal structure of **3**. Thus, the orientation of carboxylate groups in [**1**]$^{3-}$, which significantly affects the degree of interactions with the cationic [**2**]$^{3+}$, is an important factor that governs its chiral recognition ability. Finally, it should be noted that the $(\Delta_{\mathrm{LLL}})_2$ isomer of [**1**]$^{3-}$ prefers the $(\Lambda)_2$ isomer of [**2**]$^{3+}$, as does the $(\Lambda_{\mathrm{LLL}})_2$ isomer of [**1**]$^{3-}$, although its enantioselectivity is not excellent. As far as we know, such a coordination system that shows the selectivity toward the same configurational enantiomer by diastereomers having an opposite helicoidal chirality is unprecedented, and this implies for the first time that the local chirality (D or L) due to asymmetric carbon centers, rather than a helicoidal chirality (Δ or Λ) about metal centers, could predominate the chiral recognition behavior of diastereomers. A further study on the enantioselectivity of the diastereomers of [**1**]$^{3-}$ toward other racemic complex-cations is currentl underway.

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9 Calcd for $\mathbf{3}\cdot 14H_2O = C_{30}H_{94}N_{12}O_{26}S_{12}Co_5Ag_3$: C, 17.64; H, 4.64; N, 8.23%. Found: C, 17.68; H, 4.36; N, 8.26%. Yield for $\mathbf{3}\cdot 14H_2O$: 69% (based on $(\Lambda)_2$-[**2**]$^{3+}$). Calcd for $\mathbf{4}\cdot 14H_2O = C_{30}H_{94}N_{12}O_{26}S_{12}Co_5Ag_3$: C, 17.64; H, 4.64; N, 8.23%. Found: C, 17.73; H, 4.63; N, 8.05%. Yield for $\mathbf{4}\cdot 14H_2O$: 75% (based on $(\Delta)_2/(\Lambda)_2$-[**2**]$^{3+}$).

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11 Crystal data for $(\Lambda)_2(\Lambda_{\mathrm{LLL}})_2$-$\mathbf{3}\cdot 24H_2O$: fw 2222.31, Monoclinic, $P2_1$, $a = 11.7872(5)$ Å, $b = 22.5815(8)$ Å, $c = 14.8742(6)$ Å, $V = 3926.0(3)$ Å^3, $Z = 2$, $D_{\mathrm{calcd}} = 1.880$ Mg m^{-3}, 38809 reflections measured, 17228 independent. $R_1 = 0.036$ ($I > 2\sigma(I)$), $wR_2 = 0.099$ (all data). CCDC: 805731.

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14 The 1D helices are further connected to each other through hydrogen bonds to give a 3D network structure.[8]