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The Formation of New Complexes from a Decomposition of $[Au^{I}_{6}Ag^{I}_{3}Cu^{II}_{3}]^{3+}$ Parent Framework during Co-crystallization Process

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Abstract

Co-crystallization in supramolecular complexes often encounters significant challenges, particularly when dealing with highly charged ionic species. One major issue is that the parent complex structure may decompose during the process, while the new structure cannot be obtained using the original method. In this study, we reported the formation of new coordination complexes derived from the decomposition of a framework structure composed of $[Au^{I}_{6}Ag^{I}_{3}Cu^{II}_{3}(tdme)_2(D\text{-}pen)_6(H_2O)_3]^{3+}$. Two decomposition products, $[Au^{I}_{6}Ag^{I}_{2}Cu^{II}_{2}(tdme)_2(D\text{-}pen)_6]$ and $[Au^{I}_{6}Ag^{I}_{2}Cu^{II}_{2}(tdme)_2(D\text{-}pen)_6][Au^{I}_{6}Cu^{II}_{3}(tdme)_2(D\text{-}pen)_6]$, were isolated as a result of the interruption of $[Rh^{II}_{4}Zn^{II}_{4}O(L\text{-}cys)_{12}]^{6-}$ through anion-exchange reaction at two different temperature conditions (0 and 25°C) in a co-crystallization process.

Keywords: Au supramolecular complex, single-crystal X-ray diffraction, co-crystallization, anion exchange

1. Introduction

The construction of metal-organic-frameworks (MOFs) has emerged as one of the most outstanding fields in chemistry after three researchers, Prof. Susumu Kitagawa, Prof. Richard Robson, and Prof. Omar M. Yaghi, were awarded the Nobel Prize in chemistry in 2025 [1-6]. MOFs offered a promising alternative to other porous material (e.g., zeolites, clays, MCMs, etc.) for facilitating reactions through host–guest interactions. Currently, strategies for building MOF frameworks vary widely, encompassing one, two-, and three-dimensional architectures with diverse metals and organic ligands [7-10]. Regarding metal nodes, the use of metals is not limited to single or dual systems but extends to triple and multiple-metal combinations to achieve structural uniqueness and expand lattice parameters [11-13]. Similarly, organic ligands in metal–organic systems range from common linkers to polyhedral structures and macromolecules [14-16].

In our group, we have combined organic ligands, such as amino acid group (e.g., penicillamine and cysteine) or phosphine group (e.g., 1,1,1-tris(diphenylphosphinomethyl)ethane) and bis(diphenylphosphino)methane) and various metal ions to create new metalloligand [12-13,17-19] for reconstructing coordination complexes with certain functionalities, such as cation and anion exchanges. Recently, we successfully performed anion-exchange reactions using $K[Co^{III}(EDTA)]$ ($EDTA^{4-}$ = ethylenediaminetetraacetate) and $Na_3[M^{I}_{3}Co^{III}_{2}(pen)]$ (M = Au/Ag; pen^{2-} = penicillaminate) in $[Au^{I}_{6}Ag^{I}_{3}Cu^{II}_{3}(tdme)_2(D\text{-}pen)_6(H_2O)_3](tfa)_3$ (tdme = 1,1,1-tris(diphenylphosphinomethyl)ethane); $D\text{-}pen^{2-}$ = D-penicillaminate; tfa = trifluoroacetate) {[**$Au_6Ag_3Cu_3$**](tfa)$_3$} systems [20-21]. In this study, we evaluated the robustness of the [**$Au_6Ag_3Cu_3$**](tfa)$_3$ framework using a larger anion, $K_6[Rh_4Zn_4(L\text{-}cys)_{12}O]$ ($L\text{-}cys^{2-}$ = L-cysteinate) {K_6[**Rh_4Zn_4**]}. Here, we report the formation of a new single-crystal structure resulting from the decomposition of the parent

structure of [**Au$_6$Ag$_3$Cu$_3$**](tfa)$_3$ during the co-crystallization process. Our investigation focused on single-crystal structure determination, supported by complementary spectroscopic evidence. This work provides deeper insight into the limitations of the [**Au$_6$Ag$_3$Cu$_3$**](tfa)$_3$ framework for subsequent chemical transformations in the term of anion exchange reaction and co-crystallization behavior. Furthermore, the newly obtained single-crystal structures have been deposited in the Cambridge Crystallographic Data Centre (CCDC), offering valuable resources for researchers aiming to design similar coordination complexes containing Au^{I}, Ag^{I}, or Cu^{II} ions.

2. Materials and Method

2.1. Materials

All chemicals were used without further purification. The [**Au$_6$Ag$_3$Cu$_3$**](tfa)$_3$ and K$_6$[**Rh$_4$Zn$_4$**] synthesis pathways were prepared under the same conditions as in references [23] and [24], respectively.

2.2. Co-crystallization of [Au$_6$Ag$_3$Cu$_3$](tfa)$_3$ and K$_6$[Rh$_4$Zn$_4$] through anion exchange reaction

Isolated single crystals of [**Au$_6$Ag$_3$Cu$_3$**](tfa)$_3$ (1×10^{-2} M; 1.0 mL) were dissolved with deionized water. In a separated beaker, isolated K$_6$[**Rh$_4$Zn$_4$**] (1×10^{-2} M; 0.5 mL) were also dissolved with deionized water before mixing with the [**Au$_6$Ag$_3$Cu$_3$**](tfa)$_3$ solution. The mixed reaction solutions then stood at 0 (beaker A) and 25°C (beaker B). After one-week, blue needle (**1**) and octahedral single crystals (**2**) appeared in the beakers A and B, respectively. Both single crystals were subsequently analyzed using single-crystal X-ray diffraction (SCXRD) at 100 K under N_2 atmosphere, along with complementary spectroscopic techniques.

Elemental Anal. Calcd. for [Au$_6$Ag$_2$Cu$_2$(tdme)$_2$(D-pen)$_6$][Au$_6$Cu$_3$(tdme)$_2$(D-pen)$_6$]·60 H$_2$O (**1**) = C, 32.64%; H, 4.70%; and N, 2.04%. Found: C, 32.43%; H, 4.48%; and N, 2.32%. Complex **2** has no elemental analysis data due to limited sample amounts.

2.2. Single-crystal X-ray determination

Diffraction data of single crystals of **1** and **2** were recorded on a Rigaku XtaLAB Synergy Custom X-ray diffractometer equipped with a Hypix-6000HE hybrid photon-counting detector and a Rigaku VariMax rotating-anode X-ray source with a Mo target. The intensity data were processed using the CrysAlisPro program (version 1.171.41.122a) and collected using the ω-scan technique. This instrument was available at Department of Chemistry, The University of Osaka, Japan. The structures were solved by direct methods using SHELXS [25], and refined by SHELXL [26] using the Olex2 program [27]. CCDC 2519807 and 2519806 (**1** and **2**, respectively), contain the supplementary crystallographic data for this paper. These data are provided free of charge by The Cambridge Crystallographic Data Centre (https://www.ccdc.cam.ac.uk/).

3. Results and Discussion

3.1. The single-crystal X-ray structure determination and characterization of new complexes

The [**Au$_6$Ag$_3$Cu$_3$**](tfa)$_3$ possesses a three-dimensional architecture that enables chiral discrimination of guest molecules. Previously, this framework successfully separated Λ-[Co^{III}(EDTA)]$^-$ isomer and [M$^{I}_3$Co$^{III}_2$(L-pen)$_6$]$^{3-}$ (M = Au/Ag) complexes through anion-exchange reaction via co-crystallization processes [20-21]. Here, we employed a larger anionic species of [Rh$_4$Zn$_4$]$^{6-}$, compared to [Co^{III}(EDTA)]$^-$, [Au$^{I}_3$Co$^{III}_2$(L-pen)$_6$]$^{3-}$ and [Ag$^{I}_3$Co$^{III}_2$(L-pen)$_6$]$^{3-}$, to train the limitation of parent framework.

Two temperature conditions were applied (0 and 25°C) as previous reports suggested that structural variations could occur under such circumstances. After one-week, blue needle (**1**, 0 °C) and octahedral single crystals (**2**, 25 °C) appeared at the bottom of each reaction solution. Based on their appearance and subsequent X-ray fluorescence (XRF) analysis, it was confirmed that the parent crystals recrystallized without incorporating the [**Rh$_4$Zn$_4$**]$^{6-}$ anion. Nevertheless, both crystals were suitable for SCXRD measurements. Elemental analysis suggested the incorporation of 60 hydrated water molecules in the crystal structure of **1**. The single crystal X-ray analysis suggested that **1** has a monoclinic crystal system with C_2 space group (Table 1, Figures 1-2). The single crystal color looked blue, indicating that the exchange from tfa$^-$ to [**Rh$_4$Zn$_4$**]$^{6-}$ was not successful.

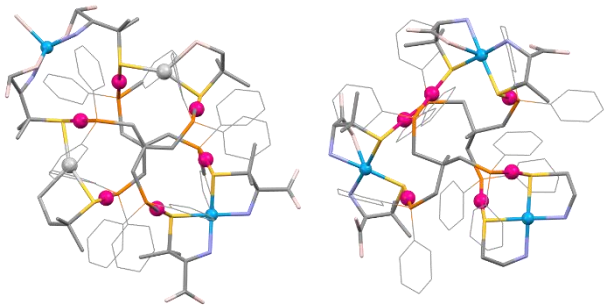


Figure 1. A perspective view of the asymmetric unit of **1** (0 °C) containing [**Au$_6$Ag$_2$Cu$_2$**] and [**Au$_6$Cu$_3$**] moieties. Color codes: red, Au; blue, Cu; pale grey, Ag; orange, P; yellow, S; pink, O; pale blue, N; grey, C. The H atoms are omitted for clarity.

According to the crystal refinement, the suggested chemical structure was [Au$_6$Ag$_2$Cu$_2$(tdme)$_2$(D-pen)$_6$][Au$_6$Cu$_3$(tdme)$_2$(D-pen)$_6$]. The presence of two independent molecules, [Au$_6$Ag$_2$Cu$_2$(tdme)$_2$(D-pen)$_6$] {[**Au$_6$Ag$_2$Cu$_2$**]} and [Au$_6$Cu$_3$(tdme)$_2$(D-pen)$_6$] {[**Au$_6$Cu$_3$**]} in

the asymmetric unit reduces the overall symmetry compared to the original structure (from $F4_132$ to C_2; see also Table 1, Figures 1-2, Ref. 22).

Table 1. Crystallographic data of complexes **1** and **2**.

Complex	**1** (0 °C)	**2** (25 °C)
CCDC No.	2519807	2519806
Color, form	Blue, octahedral	Blue, needle
λ=Wavelength/ Å	0.71073	0.71073
Crystal system	Monoclinic	Orthorhombic
Space group	C_2	$P2_12_12_1$
a/Å	25.7911(8)	22.7984(16)
b/Å	22.0072(5)	24.1337(14)
c/Å	24.5590(5)	26.4647(12)
α/°	90	90
β/°	91.478(2)	90
γ/°	90	90
V/Å^3	13,934.8(6)	14,561.2(15)
Z	4	2
T/K	293	293
F(000)	9188	7802
ρ_{calcd}/g·cm^{-3}	2.277	1.817
$\mu(\lambda)$/mm^{-1}	7.681	3.853
Flack parameter	0.023(6)	0.038(13)
Limiting indices	-33 ≤ h ≤ 34	-16 ≤ h ≤ 31
	-28 ≤ k ≤ 29	-24 ≤ k ≤ 33
	-30 ≤ l ≤ 34	-35 ≤ l ≤ 34
$R1$ (I>2σ(I))[a]	0.1487	0.1232
w$R2$ (all data)[b]	0.3463	0.3058
GOF	1.945	1.109

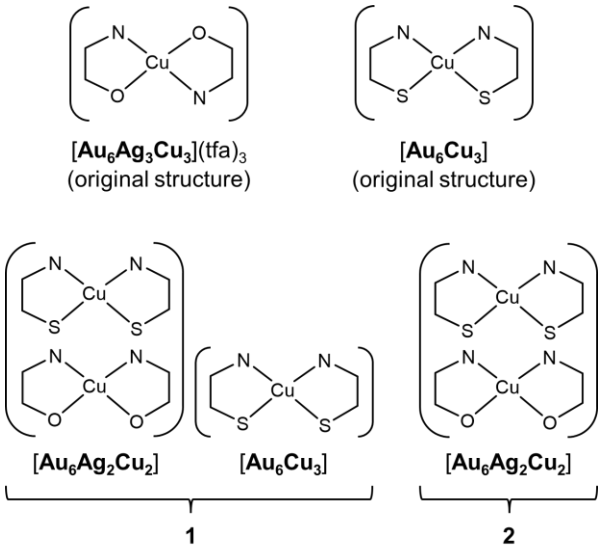
[a] $R1 = \Sigma||F_o|-|F_c|| / \Sigma|F_o|$.

[b] $wR2 = [\Sigma(w(F_o^2-F_c^2)^2) / \Sigma w(F_o^2)^2]^{1/2}$.

On the other hand, the X-ray analysis indicated that **2** crystallized in orthorhombic crystal system and $P2_12_12_1$ space group, with the predicted chemical formula $[Au_6Ag_2Cu_2(tdme)_2(D\text{-}pen)_6]$ (Table 1, Figure 3). Unfortunately, the number of hydrated water molecules in **2** could not be determined due to the insufficient amount of crystal available for elemental analysis. For the asymmetric units in compounds **1** and **2**, it was found that one Ag^+ and Cu^{2+} ions released from the $[\mathbf{Au_6Ag_3Cu_3}]^{3+}$ framework structure to the reaction solution. Consequently, we did not observe regions of high electron density surrounding the Au and penicillamine ligands, which could be assigned as Ag and Cu atoms, respectively. This observation provides evidence supporting the release of both metal ions during the co-crystallization process. In the crystal structures, the observed average S-Ag bond lengths (2.454 pm in **1**; and 2.394 pm in **2**) were comparable to those in $[\mathbf{Au_6Ag_3Cu_3}](tfa)_3$ (2.440 pm) [see Ref. 22]. In **1**, the packing arrangement forms a three-dimensional structure through interactions between two independent molecules, resulting in a total porosity smaller than that of the parent $[\mathbf{Au_6Ag_3Cu_3}](tfa)_3$ framework (porosity = 66%) [Figure 1, see Ref. 22]. Furthermore, the geometry transformation of Cu metals centers in **1** and **2** were summarized in Scheme 1 and discussed in the following section.

3.2. The geometry transformation of Cu metals center

Based on the single-crystal refinement, the structure of **1** resembles a combination of pristine $[\mathbf{Au_6Ag_3Cu_3}](tfa)_3$ and discrete $[\mathbf{Au_6Cu_3}]$ molecules (see refs. 9,22), whereas **2** is close to the parent framework of $[\mathbf{Au_6Ag_3Cu_3}](tfa)_3$ (see ref. 22). Previous studies reported that the present of Ag can induce new Ag-S interaction, which promotes the inclusion of water molecules and results in the formation of $[\mathbf{Au_6Ag_3Cu_3}](tfa)_3$ [21]. As shown in Scheme 1, the initial geometries around Cu metal center were *trans*-N_2,O_2 (pristine $[\mathbf{Au_6Ag_3Cu_3}](tfa)_3$) and *cis*-$N_2,S_2$ (pristine $[\mathbf{Au_6Cu_3}]$) [see Refs. 9,22].



Scheme 1. Geometry of Cu^{II} ions comparison in $[\mathbf{Au_6Ag_3Cu_3}](tfa)_3$ and $[\mathbf{Au_6Cu_3}]$ pristine molecules, **1**, and **2**.

In this investigation, the Cu^{II} environment of **1** changed to partial *cis*-N_2,O_2 and *cis*-N_2,S_2 in $[\mathbf{Au_6Ag_2Cu_2}]$, with an unchanged geometry of the $[\mathbf{Au_6Cu_3}]$ moiety compared with that reported in the literature. In complex **2**, the geometry transformation in the $[\mathbf{Au_6Ag_2Cu_2}]$ moiety was comparable to that in **1**. The metal coordination environment transformation strongly associated with interference from the $[\mathbf{Rh_4Zn_4}]^{6-}$ species during the anion-exchange reaction, even though it was not co-crystallized with the cationic framework. The present of $[\mathbf{Rh_4Zn_4}]^{6-}$ initiated two important phenomena: the release of Ag^{I}-Cu^{II} metal ions and the scrambling of bond formation around the Cu center in **1** and **2**. Consequently, the distinct final crystal structures of **1** (0 °C) and **2** (25 °C) can also be attributed to differences in the co-crystallization temperature. Overall, we conclude that temperature variations significantly influence the coordination environment around the Cu^{II} center. The combined effects of co-crystallization temperature and the interruption

caused by the [**Rh$_4$Zn$_4$**]$^{6-}$ anion contributed to the release of Ag^{I} and Cu^{II} metal ions, both of which are major factors in the decomposition of the [**Au$_6$Ag$_3$Cu$_3$**](tfa)$_3$ parent molecule.

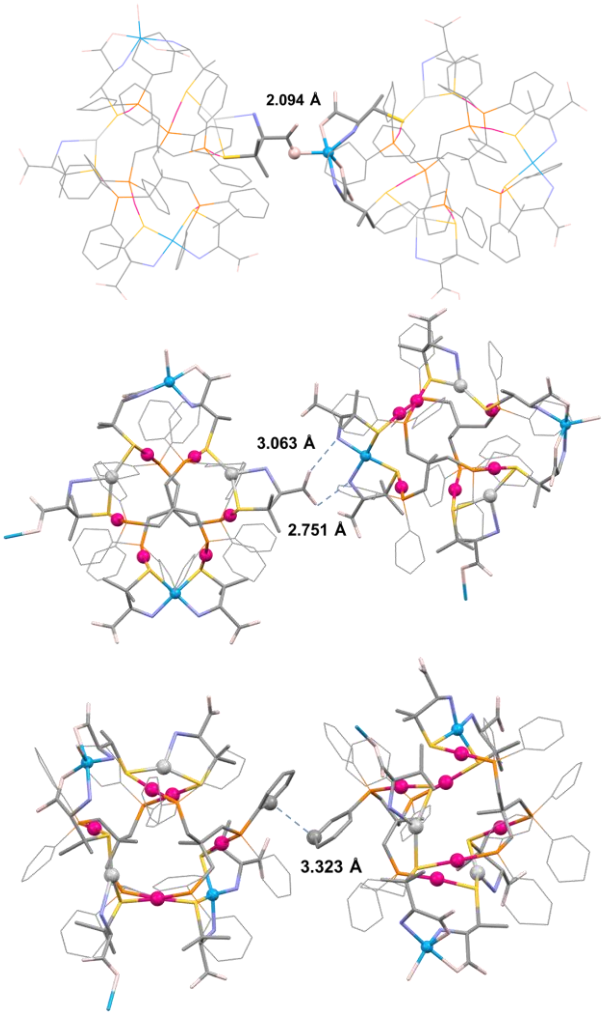


Figure 2. Crystal structure of **1**. Interaction between [Au$_6$Ag$_2$Cu$_2$(tdme)$_2$(D-pen)$_6$] and [Au$_6$Cu$_3$(tdme)$_2$(D-pen)$_6$] molecules through Cu^{II}-oxygen (top), hydrogen bonding (middle), and benzene rings (bottom). Color codes: red, Au; blue, Cu; pale grey, Ag; orange, P; yellow, S; pink, O; pale blue, N; grey, C. The H atoms are omitted for clarity.

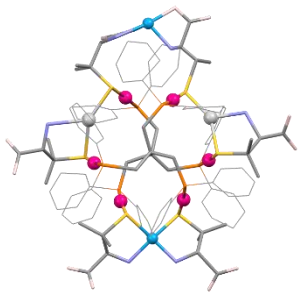


Figure 3. A perspective view of the asymmetric unit of **2** (25 °C) containing [Au$_6$Ag$_2$Cu$_2$(tdme)$_2$(D-pen)$_6$]. Color codes: red, Au; blue, Cu; pale grey, Ag; orange, P; yellow, S; pink, O; pale blue, N; grey, C. The H atoms are omitted for clarity.

4. Conclusion

The decomposition of the [**Au$_6$Ag$_3$Cu$_3$**](tfa)$_3$ framework was investigated using single-crystal X-ray diffraction technique, as the formation of decomposition complexes could not be achieved through conventional methods. The combined effects of anionic molecular size and temperature facilitated the release of Ag^{I} and Cu^{II} metal ions and subsequently altered the coordination environment around the remaining Cu^{II} centers.

Author contributions

Methodology: Benny Wahyudianto and Tatsuhiro Kojima. **Validation:** Tatsuhiro Kojima and Nobuto Yoshinari. **Formal analysis and writing—original draft preparation:** Benny Wahyudianto. Single-crystal X-ray measurement: Benny Wahyudianto, Tatsuhiro Kojima. **Writing—review and editing:** Benny Wahyudianto **Supervision:** Benny Wahyudianto, Nobuto Yoshinari, and Takumi Konno. All authors have read and agreed to the published version of the manuscript.

Conflicts of interest

There are no conflicts to declare.

Acknowledgement

The corresponding author (B.W.) is currently working at the National Institute of Advance Industrial Science and Technology (AIST) in Tsukuba, Japan.

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