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Structure and emission properties of mixed-ligand Cu(I) complexes containing phosphinesulfide ligands

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ABSTRACT

Mixed-ligand Cu(I) complexes containing phosphinesulfide ligands were synthesized, and the structure and emission properties were studied for the Cu(I) complexes. X-ray crystallographic study showed that a chelating phosphinesulfide and diimine are coordinated to Cu(I) center. Coordination geometry around Cu(I) center of each complex is described as a distorted tetrahedron. Some of the complexes show photoluminescence in the solid state.

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1. Introduction

Much attention has been paid to emissive copper(I) complexes containing diimine (polypyridine or phenanthroline) ligands in view of practical applications for chemical sensors, display devices and solar-energy conversion schemes [1]. Among the studies, the photophysical properties of the mixed-ligand Cu(I) complexes involving both diimines and diphosphines are notable, and thus extensive studies of the complexes have been reported [2-4]. [Cu(dmp)(DPEphos)]⁺(dmp = 2,9-dimethyl-1,10-phenanthroline; DPEphos = bis[2-(diphenylphosphino)phenyl]ether) and [Cu(dbp) (DPEphos)]⁺(dbp = 2,9-dibutyl-1,10-phenanthroline) exhibit high quantum yield and a long lifetime in CH₂Cl₂ at room temperature [2,3a]. We have been also studying the emissive properties of mixed-ligand copper(I) complexes with a diimine-type ligand such as dmp and a diphosphine ligand [4]. However, phosphine derivatives have a disadvantage when they are used in emitting devices. Since they have soft and strong σ -donor character of trivalent phosphorus atoms, they are easily oxidized in air or with light in the presence of air, even though triaryl phosphines are relatively stable on coordination with metal complexes (see Scheme 1).

Unlike phosphines, phosphine sulfides have pentavalent phosphorus atoms, so that they are stable for oxidation. In addition, sulfur atoms can function as soft donors, so phosphinesulfide can be used as the alternatives of phosphine ligands (Fig. 1). Several Cu(I) complexes with phosphine sulfides have been reported [5,6]. Reigle et al. reported the luminescence of $[Cu(dmp)-(S=PPh_3)_2]BF_4$ and $[Cu_2(dmp)_2(dppbS_2)_2]BF_4(dppbS_2 = 1,2-bis-(diphenylphosphino)butanedisulfide) only at 77 K [7]. However, most of the studies about Cu(I) complexes with phosphine sulfides are limited to the structural aspects.$

In this study, photophysical properties of the Cu(I) mixedligand complexes containing phosphinesulfides and diimines have been examined. They are the analogies of our previously reported mixed-ligand Cu(I) complexes containing phosphines [4].

2. Experimental

2.1. General procedures

All reactions were carried out under argon atmosphere using Schlenk techniques although the products are air-stable. 2,9-Dimethyl-1,10-phenanthroline (dmp) were purchased from TCI Co. Ltd. [Cu(CH₃CN)]PF₆ [8], 2,9-diphenyl-1,10-phenanthroline (dpp) [9], dppmS₂ and dppaS₂⁻ [10] were synthesized by the literature methods. Elemental analyses of the complexes were performed on the Perkin–Elmer model 2400 CHN Analyser. NMR spectra were obtained using a JEOL Λ -400 spectrometer, in which chemical shifts are referenced to tetramethylsilane (¹H; as internal) or 85% H₃PO₄ (³¹P{¹H}; as external). Absorption and luminescence spectra were measured with an Agilent 8453 spectrometer and a Shimadzu RF-5000 fluorometer, respectively.





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Scheme 1. Synthesis of mixed-ligand complexes.



Fig. 1. Phosphinesulfide ligands.

2.2. X-ray measurements

X-ray crystallographic measurements were made on a Rigaku Saturn 70 CCD area detector with graphite-monochromated Mo Ka radiation. The crystal-to-detector distance was 54.90 mm. The data were collected at a temperature of -150 ± 1 °C to a maximum 2θ value of 57.5°. 1800 oscillation images were collected and the data were processed by using the CrystalClear software [11]. Absorption corrections were made by the numerical method for complex 1 and 2, and by the spherical method for complex 3. The intensity of the diffraction for complex **3** was weaker than that of the others, so complex 3 gave relatively high refinement residuals. The structure was solved by direct methods (sir-92 [12]) and was refined by full matrix least squares procedures (SHELX-97 [13]). The non-hydrogen atoms are refined anisotropically and the positions of all hydrogen atoms were fixed at calculated positions. All calculations were performed by using the CrystalStructure crystallographic software package [14].

2.3. Synthesis of mixed-ligand Cu(I) complexes

2.3.1. $[Cu(dmp)(dppmS_2)]PF_6(1)$

dmp (0.11 g, 0.50 mmol) and dppmS2 (0.22 g, 0.50 mmol) were dissolved in 10 ml of acetone. $[Cu(CH_3CN)_4]PF_6$ (0.19 g, 0.50 mmol) was then gradually added to the solution. After the solution was stirred for 2 h at room temperature, addition of diethylether (20 ml) to the solution gave orange crystals, which are collected by filtration. Yield 0.33 g (76%). *Anal.* Calc. for $[Cu(C_{14}H_{12}N_2)-$

Table 1							
Crystallographic	data	for	complex	1.	2	and	3

 $(C_{25}H_{22}P_2S_2)]PF6:$ C, 54.13; H, 3.96; N, 3.24. Found: C, 54.26; H, 4.14; N, 2.75%. ³¹P NMR (acetone- d_6 , 25 °C): δ 35.0.

2.3.2. $[Cu(dmp)(dppaS_2^{-})]$ (2)

dmp (0.11 g, 0.5 mmol) and K(dppaS₂⁻) (0.24 g, 0.5 mmol) were dissolved in 10 ml of acetone. [Cu(CH₃CN)₄]PF₆ (0.19 g, 0.5 mmol) was then gradually added to the solution. The solution was stirred for 2 h at room temperature and an orange powder was obtained. Recrystallization of the powder from acetone/diethylether gave orange crystals. Yield 0.31 g (85%). *Anal.* Calc. for [Cu(C₁₄H₁₂N₂)-(C₂₄H₂₀NP₂S₂)]: C, 63.36; H, 4.48; N, 5.83. Found: C, 63.18; H, 4.21; N, 5.69%. ³¹P NMR (acetone-d₆, 25 °C): δ 31.8.

2.3.3. $[Cu(dpp)(dppaS_2^{-})]$ (**3**)

This complex was prepared by a similar method used for the preparation of complex **3**; use of dpp (0.17 g, 0.5 mmol) instead of dmp gave purple crystals. Yield 0.29 g (71%). *Anal.* Calc. for $[Cu(C_{24}H_{16}N_2)(C_{24}H_{20}NP_2S_2)]$: C, 68.27; H, 4.30; N, 4.98. Found: C, 67.84; H, 4.09; N, 5.01%. ³¹P NMR (acetone- d_6 , 25 °C): δ 31.2.

3. Result and discussion

3.1. Synthesis and crystal structures

Treatment of $[Cu(CH_3CN)_4]PF_6$ with equimolar amount of dmp and dppmS₂ in acetone gave the mixed-ligand complex **1**. Attempt to prepare a similar complex to complex **1** by using dppaS₂ instead of dppmS₂ gave complex **2** containing dppaS₂⁻ in which a proton on an amine moiety is eliminated from dppaS₂. Use of KdppaS₂ and dmp also led complex **2** readily. Treatment of KdppaS₂ and $[Cu(CH_3CN)_4]^+$ with dpp instead of dmp gave complex **3**.

The single crystals with X-ray quality of **1–3** were obtained by gradual addition of diethylether into the reaction solution. Structural data are summarized in Table 1. The structure of complex cation **1** is shown in Fig. 2. It was found that the complex has the tetrahedral structure in which each copper center is coordinated by dmp and dppmS₂ chelate ligands. Selected bond lengths, bond angles and torsion angles of **1** are shown in Table 2. The S–P bond lengths of **1** in dppmS₂ are 1.9670(7) and 1.9784(7) Å. Since it has been reported that P–S single-bond distance is 2.14 Å and P=S double-bond distance ranges from 1.916 to 1.959 Å, the P–S bond length in **1** shows the bonds almost have double-bond character [15]. The bond angle of S–Cu–S is 108.10(2)°, which is similar to that in the ideal tetrahedral structure. The chelate ring made of Cu-dppmS₂ moiety is like a twist boat, as deduced from the torsion angles within the chelate ring ranging from 14.6(2)° to 75.38(12)°.

Complex name	Complex 1 acetone	Complex 2 (2 molecules)	Complex 3
Formula	$H_{40}CuF_6N_2P_3S_2C_{42}O$	$C_{76}H_{64}Cu_2N_6P_4S_4$	$C_{48}H_{36}CuN_3P_2S_2$
Crystal size (mm)	$0.25\times0.25\times0.20$	$0.40 \times 0.20 \times 0.10$	$0.20\times0.16\times0.12$
Lattice type	triclinic	monoclinic	monoclinic
space group	P1 (#2)	P2 ₁ /c (#14)	$P2_1/c$ (#14)
a (Å)	10.996(2)	10.649(6)	10.111(4)
b (Å)	11.249(2)	19.599(10)	21.340(8)
c (Å)	17.783(3)	32.222(17)	17.981(7)
α (°)	98.444(3)	90	90
β (°)	92.604(2)	92.990(3)	95.231(2)
γ (°)	107.468(3)	90	90
$V(Å^3)$	2066.1(7)	6716.1(60)	3863.8(25)
Ζ	2	4	4
D_{calcd} (g cm ⁻³)	1.484	1.425	1.452
μ (Mo K α) (cm ⁻¹)	8.09	9.03	7.97
R_1	0.033	0.052	0.079
wR ₂	0.080	0.099	0.219

Table 2



Fig. 2. X-ray structure of complex cation 1.

Selected bond distances (Å), bond angles (°) and torsion angles (°) in complex 1.

Cu(1)–S(1)	2.2771(6)
Cu(1)–S(2)	2.3333(6)
Cu(1)–N(1)	2.1060(16)
Cu(1)–N(2)	2.0578(18)
S(1)-P(1)	1.9784(7)
S(2)–P(2)	1.9670(7)
S(2)-Cu(1)-S(1)	108.10(2)
Cu(1)-S(1)-P(1)	103.91(3)
Cu(1)-S(2)-P(2)	100.09(3)
N(2)-Cu(1)-N(1)	80.91(6)
Cu(1)-S(1)-P(1)-C(39)	-40.47(8)
Cu(1)-S(2)-P(2)-C(39)	-41.55(7)
S(1)-Cu(1)-S(2)-P(2)	63.78(2)
S(2)-Cu(1)-S(1)-P(1)	-23.74(3)
S(1)-P(1)-C(39)-P(2)	75.38(12)
S(2)-P(2)-C(39)-P(1)	-22.75(14

The structures of complex **2** and **3** containing $dppaS_2^-$ are described in Figs. 3 and 4. The selected bond lengths, bond angles and torsion angles of 2 and 3 are summarized in Tables 3 and 4, respectively. Complex 2 showed two independent molecules in the unit cell. The coordination geometry in one of the molecules is almost similar to each other. There are little differences between the structures of two independent molecules; the maximum of deviation for the corresponding bond angles is no more than 3.0°. In the dppaS₂⁻ complexes, the S–P bond lengths range from 2.0043(8) to 2.0145(8) Å, which are slightly longer than those of 1. The elongation should reflect the decrease of the P–S bond order, as shown in other reported transition metal complexes containing dppaS₂⁻ ligand [10]. The bond angle of S-Cu-S in **2** is $117.77(2)^{\circ}$ and $119.01(2)^\circ$, which is larger than that of **1**. It suggests that the chelate ring of dppaS₂⁻ is somewhat more planar than that of $dppmS_2$. In addition, torsion angles within the chelate in 2 $(11.28(4)-44.25(10)^{\circ})$ is smaller than those in **1** and the mean deviations from the least-squares planes which consists of six atoms in Cu-dppaS₂ chelate ring of two independent complexes of **2** are 0.238 and 0.256 Å, whereas that of **1** is 0.439 Å. They also show the planarity of the chelates in 2. It should be caused by the negative charge on the imino nitrogen of the anionic ligand. dppaS₂⁻, which promotes delocalization of π -electron within the S-P-N-P-S chelate. The coordination environment of 3 is similar to that of **2**. However, the torsion angles within the chelate ring of $dppaS_2^-$ are much smaller than those in **2**, and the mean deviation from the least-squares plane in Cu-dppaS₂ chelate ring of **3** is the smallest of the three complexes (0.120 Å). So they suggest that **3** has a more planar chelate ring of dppaS₂⁻. It may be due to the



Fig. 3. X-ray structure of complex of 2. The figure shows one of the two independent molecules in the unit cell.



Fig. 4. X-ray structure of complex of 3.

repulsion from bulky phenyl rings of dpp, which forces the chelate ring of the $dppaS_2^-$ to be more planar.

3.2. Spectroscopic properties

NMR spectra have been measured to study the structures of the mixed-ligand complexes in solution. In the ³¹P NMR spectra of **1**, a signal assigned as dppmS₂ was observed as a distinct singlet at δ 35.0, which is slightly shifted compared with the free ligand (δ 35.3), and no signals assigned to free ligand were observed. In addition, the phosphorus signal of **1** was shifted to higher field than that of [Cu(dppmS₂)₂]PF₆, which shows a singlet signal at δ 35.3. It suggests that the complexes keep mixed-ligand coordination even in solution. The spectra of **2** and **3** also show only a singlet signal of dppaS₂⁻ moiety.

The absorption spectra of **1–3** in acetone are shown in Fig. 5. The absorption maximum of complex **1** is 457 nm with moderate intensity. ($\varepsilon = 3.1 \times 10^3 \text{ mol}^{-1} \text{ dm}^3 \text{ cm}^{-1}$) It has been reported that mixed-ligand copper(I) complexes containing phenanthroline derivatives and diphosphines show absorption maxima ranging from 400 to 360 nm which have been assigned to MLCT transitions [2–4]. The band maximum of **1** are shifted to lower energy compared with the [Cu(dimine)(diphosphine)] complexes, but the

Table	
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Selected bond distances (Å), bond angles (°) and torsion angles (°) in complex 2.

Cu(1)-S(1)	2.2761(7)
Cu(1)-S(2)	2.2957(7)
Cu(1)-N(1)	2.126(2)
Cu(1)-N(2)	2.124(2)
S(1)-P(1)	2.0130(8)
S(2)-P(2)	2.0043(8)
S(1)-Cu(1)-S(2)	117.77(2)
Cu(1)-S(1)-P(1)	100.92(3)
Cu(1)-S(2)-P(2)	102.06(3)
N(1)-Cu(1)-N(2)	79.18(8)
$\begin{array}{l} Cu(1)-S(1)-P(1)-N(3)\\ Cu(1)-S(2)-P(2)-N(3)\\ S(1)-Cu(1)-S(2)-P(2)\\ S(2)-Cu(1)-S(1)-P(1)\\ S(1)-P(1)-N(3)-P(2)\\ S(2)-P(2)-N(3)-P(1) \end{array}$	44.25(10) 40.71(10) -24.10(4) -11.28(4) -40.4(2) -13.9(2)
$\begin{array}{l} Cu(2)-S(3)-P(3)-N(6)\\ Cu(2)-S(4)-P(4)-N(6)\\ S(3)-Cu(2)-S(4)-P(4)\\ S(4)-Cu(2)-S(3)-P(3)\\ S(3)-P(3)-N(6)-P(3)\\ S(4)-P(4)-N(6)-P(4)\\ S(4)-P(4)-N(6)-P(3)\\ Cu(2)-S(3)\\ Cu(2)-S(4)\\ Cu(2)-N(4)\\ Cu(2)-N(4)\\ Cu(2)-N(5)\\ S(3)-P(3)\\ S(4)-P(4) \end{array}$	$\begin{array}{r} -39.21(10\\ -39.51(10\\ 23.85(4)\\ 9.10(4)\\ 35.2(2)\\ 15.5(2)\\ 2.2768(7)\\ 2.2909(7)\\ 2.128(2)\\ 2.118(2)\\ 2.0145(8)\\ 2.0068(8)\end{array}$
S(3)-Cu(2)-S(4)	119.01(2)
Cu(2)-S(3)-P(3)	101.62(3)
Cu(2)-S(4)-P(4)	101.26(3)
N(4)-Cu(2)-N(5)	79.60(8)

Table 4									
Selected	bond distances	(Å), bond a	angles (°)	and	torsion	angles (°) in	com	olex 3.

Cu(1)–S(1)	2.2470(11
Cu(1)-S(2)	2.3048(11
Cu(1)–N(1)	2.106(3)
Cu(1)–N(2)	2.154(3)
S(1)-P(1)	2.0065(15
S(2)-P(2)	2.0021(14
S(1)-Cu(1)-S(2)	121.14(4)
Cu(1)-S(1)-P(1)	104.00(5)
Cu(1)-S(2)-P(2)	104.88(5)
N(1)-Cu(1)-N(2)	79.57(13)
Cu(1)-S(1)-P(1)-N(3)	11.25(16)
Cu(1)-S(2)-P(2)-N(3)	-0.14(17)
S(2)-Cu(1)-S(1)-P(1)	9.91(7)
S(1)-Cu(1)-S(2)-P(2)	-14.88(7)
S(1)-P(1)-N(3)-P(2)	-39.8(4)
S(2)-P(2)-N(3)-P(1)	32.1(4)

band is reasonably assigned to MLCT involving the π^* orbitals of the dmp ligand, because the coordination of the sulfide having weaker σ -donor character leads to a decrease of energy levels of $d\sigma^*$ orbitals interactions in the complex and a decrease of the CT excitation energy [16]. However, there is another shoulder band in the lower energy region (500–550 nm) for complex **1**. The two bands may arise from two MLCT transitions to different π^* orbitals of the phen ligand as shown in [Ru(bpy)(CN)₄]^{2–} which contains two quite distinct MLCT absorptions associated with the bpy ligand [17]. A second possibility is that the second absorption band might be assigned to a LLCT (ligand-to-ligand charge transfer) from the electron-rich S-donor ligand to the electron-poor phen ligands.

The absorption maximum of complex **2** is 427 nm. The absorption band is also assigned to MLCT transition involving the π^* orbital of dmp, whereas the maximum is shifted to higher energy



Fig. 5. Absorption spectra of complex cation 1 (solid line), 2 (dotted line) and 3 (dashed line) in acetone at room temperature.

than that of complex **1** which should reflect the stronger ligand field of dppaS₂⁻. The shoulder band in lower energy region may be assigned to the second MLCT or LLCT as discussed for complex **1**. Complex **3** has an absorption maximum at 578 nm, which is shifted to lower energy due to extended π conjugated system of dpp compared with dmp. In order to confirm this hypothesis, the redox processes have been studied by cyclic voltammetry. Unfortunately, the redox potential in the cathodic region is too negative to estimate the potential for a diimine/diimine⁻ redox couple. However, shoulder cathodic current in complex **3** was observed at -1.85 V versus SCE, which are clearly less negative than that of complex **1** and **2**. It may suggest that the red shift observed for dpp complex is reflected in a less negative ligand-based reduction process.

Both **1** and **2** show photoluminescence in the solid state as shown in Fig. 6. The emission maxima for **1** and **2** are 592 and 661 nm, respectively. The emissive excited states are assigned to MLCT transitions involving of the π^* orbital of dmp, as deduced from the luminescent behavior of [Cu(dmp)2]⁺ and other mixed-ligand complexes. The Stokes shift observed for **2** (240 nm) is considerably large, whereas that of **1** is ca. 140 nm. It has been shown that a relatively large Stokes shift observed in Cu(I) com-



Fig. 6. Solid-state emission spectra of ${\bf 1}$ (solid line) and ${\bf 2}$ (dotted line) at room temperature.

plexes results from flattening distortion which occurs in the MLCT excited state [1a]. It suggests that the extent of distortion on excitation may become larger in **2** than **1**, which has relatively higher planarity of phosphinesulfide chelate than **1** as shown by X-ray analysis.

For both **1** and **2**, no emission is observed in solution. It contrasts with the fact that $[Cu(dmp)(dppe)]^+$ and $[Cu(dmp)(dppp)]^+$ emit in solution [2,4b]. It may be caused by the fact that coordination sphere around the copper atom of the diphosphine sulfide complexes is less crowded than the diphosphine complexes. The less crowded structure allows (1) the metal center to facilitate tetragonal flattening distortion of the excited luminophore and (2) the solvent molecules to attack the metal center in the excited states. These quenching mechanism [1a] should effectively affect the photophysical properties in solution.

4. Conclusions

Mixed-ligand Cu(I) complexes containing diimine and chelating phosphinesulfide ligands were synthesized. The isolated complexes, which have pseudo tetrahedral structure, were relatively stable in air. Two complexes of dmp show emission in the solid state, and their bands are assigned to MLCT.

5. Supplementary data

CCDC 715718, 715719 and 715720 contain the supplementary crystallographic data for **1**, **2** and **3**, respectively. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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