

Synthesis and crystal structure of three-coordinated silver(I) and copper(I) complexes with N₂P binding set containing a novel tripodal ligand: 1,3,5-tris(benzimidazol-2-ylmethyl)benzene

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Assembly of a novel tripodal ligand, 1,3,5-tris(benzimidazol-2-ylmethyl)benzene (tbib), and triphenylphosphine with silver(I) and copper(I) salts gave the corresponding mononuclear complexes, which were characterized by electrospray mass spectrometry and ³¹P NMR spectroscopy. The structure of the silver(I) complex, [Ag(tbib)(PPh₃)]NO₃ · CH₃OH · H₂O, determined by X-ray crystallography, indicates that the silver(I) atom is coordinated by two nitrogen atoms of one tbib ligand and one phosphorus atom of triphenylphosphine. Another nitrogen atom of the tbib ligand is uncoordinated. The complex provides another example of a structurally characterized silver(I) complex with N₂P binding set.

In the past decade, much progress has been made in metal-directed assembly of supramolecular frameworks with suitable metal ions and rationally designed organic ligands.¹ Especially in recent years, supramolecular complexes with tripodal ligands have attracted much attention from chemists since frameworks with specific topologies and interesting properties have been obtained by the assembly of metal ions with tripodal ligands.^{2–4} For example, 1,3,5-tricyanobenzene and 1,3,5-tris(4-ethylbenzonitrile)benzene react with silver(I) trifluoromethanesulfonate to give honeycomb-like and hinge-like coordination networks, respectively.² In the cases of 2,4,6-tris(4-pyridyl)-1,3,5-triazine and 1,3,5-tris(pyrazol-1-ylmethyl)-2,4,6-triethylbenzene, M₆L₄-type cage-like complexes were obtained by assembly of the ligands with Pd(II).^{3,4} These results indicate that organic bridging ligands have a great impact on the formation of supramolecular complexes. Recently, we developed a new tripodal ligand with imidazole groups, 1,3,5-tris(imidazol-1-ylmethyl)-2,4,6-trimethylbenzene (titmb) and found that the ligand reacts with Cu(II) to form two-dimensional (2D) networks.⁵

In order to further investigate the influence of the linkage mode of bridging ligands on the formation of metal complexes, we designed and synthesized a novel tripodal ligand containing the benzimidazol group, 1,3,5-tris(benzimidazol-2-ylmethyl)benzene (tbib). In this ligand, the pendants are connected by a methylene group at the 2 position of the imidazole

group rather than at the 1 position as in the case of titmb. We are currently engaged in the synthesis and study of these tripodal ligands and their complexes for application in supramolecular chemistry. Such tripodal ligands show a variety of coordination modes to different metal ions.⁵ In this paper, we report the synthesis and characterization of Ag(I) and Cu(I) complexes with tbib and triphenylphosphine ligands.

Experimental

Materials and physical measurements

[Cu(CH₃CN)₄]ClO₄ was prepared according to the reported procedures.⁶ Acetonitrile and methanol solvents were dried and purified by distillation prior to use. Other reagents such as silver nitrate, triphenylphosphine, etc. are commercially available and used as received without further purification. C, H and N analyses were made on a Perkin-Elmer 240C elemental analyzer at the analysis center of Nanjing University. ³¹P and ¹H NMR spectroscopic measurements were performed on a Bruker AM-500 NMR spectrometer [202 (³¹P) and 500 (¹H) MHz]. TMS (SiMe₄) and 85% H₃PO₄ in D₂O were used as internal and external references for ¹H and ³¹P NMR, respectively. Electrospray mass (ES-MS) spectral measurements were carried out on an LCQ System (Finnigan MAT, USA) using methanol as the mobile phase. The spray voltage, tube lens offset, capillary voltage and capillary temperature were set at 4.50 kV, 0 V, 17.00 V and 120 °C, respectively. The quoted *m/z* values are for the major peaks in the isotopic distributions. Cyclic voltammograms were recorded on an EG&G M273 potentiostat/galvanostat system using a platinum working electrode, a platinum wire counter electrode and an Ag–AgCl electrode used as the reference. The measurements were carried out in DMSO solution containing 0.1 M tetrabutylammonium perchlorate as supporting electrolyte at a scan rate of 100 mV s^{–1}. Ferrocene was added at the end of the experiment and used as an internal reference.

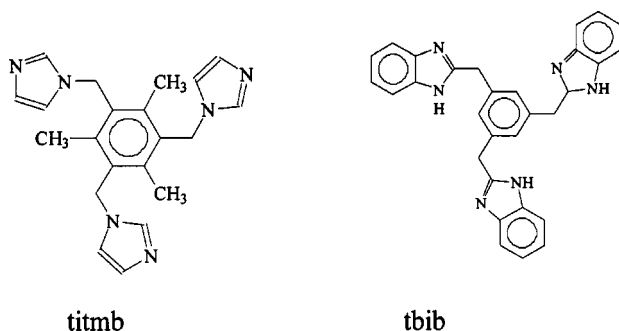


Table 1 Summary of crystal data and refinement results for the complex $[\text{Ag}(\text{tbib})(\text{PPh}_3)]\text{NO}_3 \cdot \text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$, **1**

Empirical formula	$\text{C}_{49}\text{H}_{45}\text{N}_7\text{AgO}_5\text{P}$
Formula weight	950.76
Crystal system	Monoclinic
Space group	$C2/c$
$a/\text{\AA}$	25.504(4)
$b/\text{\AA}$	20.498(3)
$c/\text{\AA}$	19.688(3)
$\beta/^\circ$	118.610(10)
$U/\text{\AA}^3$	9036(2)
Z	8
T/K	296(2)
μ/mm^{-1}	0.537
Measured and independent reflections	8686/7973
R_{int}	0.0136
$R_1 [I > 2\sigma(I)]$	0.0409
$wR_2 [I > 2\sigma(I)]$	0.0898

$$w = 1/[\sigma^2(F_o)^2 + (0.0519 P)^2], P = (F_o^2 + 2 F_c^2)/3$$

Syntheses

1,3,5-Tris(benzimidazol-2-ylmethyl)benzene (tbib). The tbib ligand was synthesized by a similar method reported for the preparation of tris(benzimidazol-2-ylmethyl)amine.⁷ 1,3,5-Benzenetriacetic acid (1.26 g, 5 mmol) and 1,2-phenylenediamine (1.73 g, 16 mmol) were mixed and ground to a fine powder, then heated together at 180–200 °C using an oil bath until the evolution of water vapor ceased (this usually required 2 h). The crude product was recrystallized from methanol to give a colorless powder in 66% yield. ES-MS: m/z 469.4 $[\text{tbib} + \text{H}]^+$. ^1H NMR (CDCl_3 , 298 K): δ 14.99 (s, 3H); 8.41 (s, 3H); 7.71 (m, 6H); 7.36 (m, 6H) and 4.63 (s, 6H).

$[\text{Ag}^{\text{I}}(\text{tbib})(\text{PPh}_3)]\text{NO}_3 \cdot \text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$, **1.** All procedures for the synthesis and recrystallization were carried out in the dark. A solution of PPh_3 (13.1 mg, 0.05 mmol) in methanol (4 ml) was added to 4 ml of a CH_3OH solution of AgNO_3 (8.5 mg, 0.05 mmol). After stirring for 3 h at room temperature, tbib ligand (23.4 mg, 0.05 mmol) in methanol (4 ml) was dropped into the mixture. The reaction mixture was further stirred for 1 h and then filtered. The clear filtrate was left at ambient temperature for several days and almost colorless crystals of the complex $[\text{Ag}(\text{tbib})(\text{PPh}_3)]\text{NO}_3 \cdot \text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$, **1**, were collected in 54% yield. Anal. calcd for $\text{C}_{49}\text{H}_{45}\text{N}_7\text{AgO}_5\text{P}$: C, 61.90; H, 4.77; N, 10.31; found: C, 61.62; H, 4.63; N, 10.26%. ES-MS: m/z 838.5 $[\text{Ag}(\text{tbib})(\text{PPh}_3)]^+$. ^1H NMR ($\text{DMSO}-d_6$, 298 K): δ 7.53 (t, 3H); 7.50 (m, 6H); 7.47 (t, 6H); 7.32 (s, 3H); 7.28 (dd, 6H); 7.17 (m, 6H) and 4.17 (s, 6H).

$[\text{Cu}^{\text{I}}(\text{tbib})(\text{PPh}_3)]\text{ClO}_4$, **2.** The synthesis was performed under an argon atmosphere. PPh_3 (13.1 mg, 0.05 mmol) and $[\text{Cu}^{\text{I}}(\text{CH}_3\text{CN})_4]\text{ClO}_4$ (16.4 mg, 0.05 mmol) were dissolved in dioxxygen-free methanol (15 ml). After stirring for half an hour at room temperature, tbib ligand (23.4 mg, 0.05 mmol) was added as a solid into the mixture. The reaction mixture was further stirred for half an hour and then filtered. The filtrate was left at room temperature for several days, after which

Table 2 Selected bond distances (\AA) and angles ($^\circ$) for the complex $[\text{Ag}(\text{tbib})(\text{PPh}_3)]\text{NO}_3 \cdot \text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$, **1**

$\text{Ag}-\text{N}(1)$	2.249(3)	$\text{C}(31)-\text{P}-\text{Ag}$	105.97(12)
$\text{Ag}-\text{N}(3)$	2.341(3)	$\text{C}(43)-\text{P}-\text{Ag}$	117.11(12)
$\text{Ag}-\text{P}$	2.4205(10)	$\text{C}(7)-\text{N}(1)-\text{Ag}$	127.3(2)
$\text{N}(1)-\text{Ag}-\text{N}(3)$	118.57(10)	$\text{C}(1)-\text{N}(1)-\text{Ag}$	127.9(2)
$\text{N}(1)-\text{Ag}-\text{P}$	127.87(8)	$\text{C}(16)-\text{N}(3)-\text{Ag}$	132.5(3)
$\text{N}(3)-\text{Ag}-\text{P}$	109.07(8)	$\text{C}(17)-\text{N}(3)-\text{Ag}$	122.4(3)
$\text{C}(37)-\text{P}-\text{Ag}$	117.76(12)		

colorless microcrystals of the complex were isolated in 61% yield (after drying in vacuum). Anal. calcd for $\text{C}_{48}\text{H}_{39}\text{N}_6\text{ClCuO}_4\text{P}$: C, 64.50; H, 4.40; N, 9.40; found: C, 64.57; H, 4.63; N, 9.56%. ES-MS: m/z 792.5 $[\text{Cu}(\text{tbib})(\text{PPh}_3)]^+$. ^1H NMR ($\text{DMSO}-d_6$, 298 K): δ 7.52 (br, 6H); 7.46 (t, 3H); 7.38 (t, 9H); 7.17–7.20 (m, 12H) and 4.20 (s, 6H). **Caution!** Perchlorate salts of copper(I) complexes with organic ligands are potentially explosive and should be handled with care.

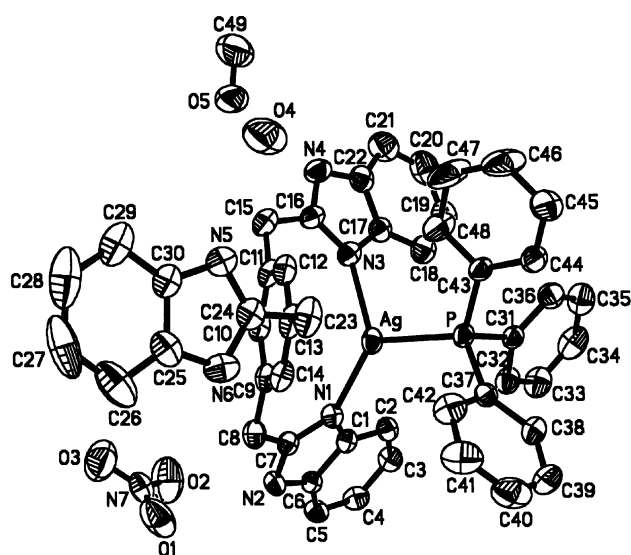
X-Ray crystallography

Single crystals of complex **1** suitable for X-ray diffraction were obtained by slow evaporation of the filtrate. A crystal with $0.48 \times 0.46 \times 0.34$ mm dimensions was mounted on a Siemens P4 automatic four-circle diffractometer and the intensity data were collected in the variable ω -scan mode using graphite-monochromated Mo $\text{K}\alpha$ radiation ($\lambda = 0.71073$ \AA) at 296(2) K. The structure was solved by Patterson methods and refined by full-matrix least-square calculations on F^2 with SHELXL-93.^{8,9} All non-hydrogen atoms were refined anisotropically, whereas the hydrogen atoms were generated geometrically. Calculations were performed on a PC-586 computer using Siemens SHELXTL program package.^{10,11} Details of the crystal parameters†, data collection and refinements are listed in Table 1, and selected bond distances and angles are given in Table 2.

Results and discussion

Crystal structure of $[\text{Ag}^{\text{I}}(\text{tbib})(\text{PPh}_3)]\text{NO}_3 \cdot \text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$, **1**

The molecular structure of the complex **1** determined by X-ray crystallography is shown in Fig. 1 with the atom numbering scheme. The silver(I) atom is three-coordinated by two nitrogen atoms of the tbib ligand and one phosphorus atom of PPh_3 . The coordination angles around the $\text{Ag}(\text{I})$ atom are in the range of $109.07(8)^\circ$ to $127.87(8)^\circ$ and the $\text{Ag}-\text{P}$ bond distance is $2.4205(10)$ \AA (Table 2), which is similar to those reported for three-coordinated silver(I) phosphine complexes.^{12–15} The silver atom is nonsymmetrically coordinated to the tbib ligand with $\text{Ag}-\text{N}$ distances of $2.249(3)$ and $2.341(3)$ \AA .

**Fig. 1** Molecular structure of $[\text{Ag}(\text{tbib})(\text{PPh}_3)]\text{NO}_3 \cdot \text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$ (**1**) with atom numbering scheme; thermal ellipsoids are drawn at the 50% probability level and hydrogen atoms are omitted for clarity.

† CCDC reference number 440/183. See <http://www.rsc.org/suppdata/nj/b0/b002450n/> for crystallographic files in .cif format.

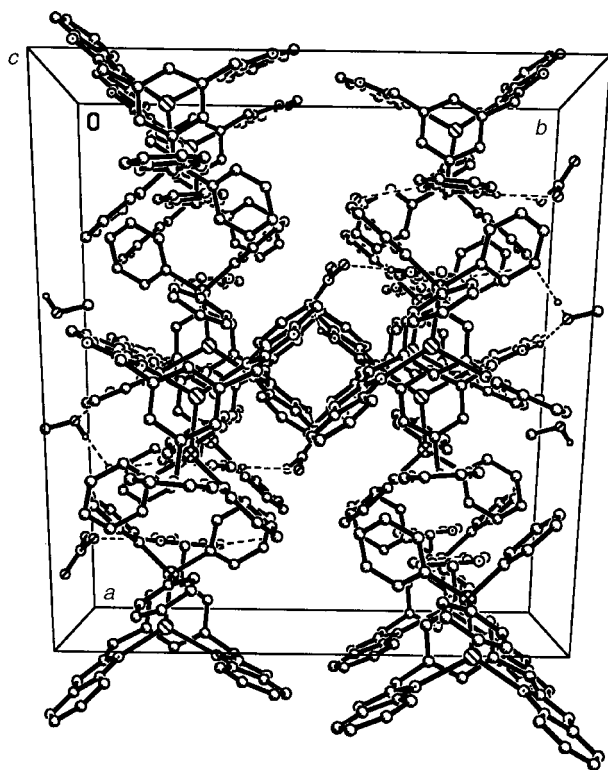


Fig. 2 Crystal packing diagram of $[\text{Ag}(\text{tbib})(\text{PPh}_3)]\text{NO}_3 \cdot \text{CH}_3\text{OH} \cdot \text{H}_2\text{O}$ (**1**) with hydrogen bonds indicated by dashed lines.

A similar situation has been found in $[\text{Ag}\{\text{P}(\text{C}_6\text{H}_4\text{-}p\text{-Me})_3\}\{\text{BPh}_2(\text{pz})_2\}]$ (pz = pyrazolyl) with different Ag–N lengths of 2.411(4) and 2.194(4) Å.¹² Therefore, the geometry around the silver atom in **1** is distorted tripodal planar and the Ag(I) atom lies 0.28 Å out of the plane formed by the P,

Table 3 ES mass spectral data for complexes **1** and **2**

<i>m/z</i>	1 Assignment	<i>m/z</i>	2 Assignment
400.8	$[\text{Ag}^+(\text{PPh}_3)(\text{CH}_3\text{OH})]^+$	531.2	$[\text{Cu}^+(\text{tbib})]^+$
469.4	$[\text{tbib} + \text{H}]^+$	589.9	$[\text{Cu}^+(\text{PPh}_3)_2]^+$
575.4	$[\text{Ag}^+(\text{tbib})]^+$	630.9	$[\text{Cu}^+(\text{tbib})\text{ClO}_4]^+$
633.0	$[\text{Ag}^+(\text{PPh}_3)_2]^+$	792.5	$[\text{Cu}^+(\text{tbib})(\text{PPh}_3)]^+$
838.5	$[\text{Ag}^+(\text{tbib})(\text{PPh}_3)]^+$	848.6	$[\text{Cu}^+(\text{PPh}_3)_3]^+$
892.6	$[\text{Ag}^+(\text{PPh}_3)_3]^+$	894.4	$[\text{Cu}^+(\text{tbib})(\text{PPh}_3)\text{ClO}_4]^+$

N(1) and N(2) atoms. Complex **1** provides another example of three-coordinated silver(I) with an N_2P binding set. A search in the Cambridge Structural Database revealed that up to now only five complexes with the same N_2P coordination mode, $[\text{Ag}\{\text{P}(\text{C}_6\text{H}_4\text{-}p\text{-Me})_3\}\{\text{BPh}_2(\text{pz})_2\}]$, $[(\eta^5\text{-C}_5\text{Me}_5)(\text{pz})\text{-Ir}(\mu\text{-pz})_2\text{Ag}(\text{PPh}_3)]$, $[\text{Ag}_2(\text{pz})_2(\text{PPh}_3)_2]$, $[\text{Ag}_2(\text{pz})_2(\text{PPh}_3)_3]$ and $[\text{Ag}\{1,2\text{-(C}_5\text{H}_4\text{NS)}_2\text{-1,2-C}_2\text{B}_{10}\text{H}_{10}\}(\text{PPh}_3)]^+$, have been reported.^{12–15}

The crystal structure of complex **1** shows that, in the solid state, only two of the three nitrogen atoms, N(1) and N(3), of the tbib ligand participate in the coordination and another nitrogen atom, N(5), is uncoordinated (Fig. 1). This may be interpreted by two considerations. First, it is geometrically difficult for the three nitrogen atoms of one tbib ligand to coordinate with a single silver atom since the central benzene group is too large to allow such coordination. Secondly, there would be steric hindrance between the benzimidazole group of the tbib ligand and the phenyl group of the PPh_3 when three nitrogen atoms of a tbib ligand and PPh_3 attempt to coordinate to one silver atom to form a four-coordinated complex.

It is interesting that tbib acts as a bidentate ligand in complex **1**, while in the reported Cu(II) complexes with titmb ligand, each titmb binds to three metal ions and acts as a bridging ligand to form 2D networks.⁵ No multinuclear species were observed in the tbib–Ag(I) system, even in the presence of excess metal ions. These results indicate that

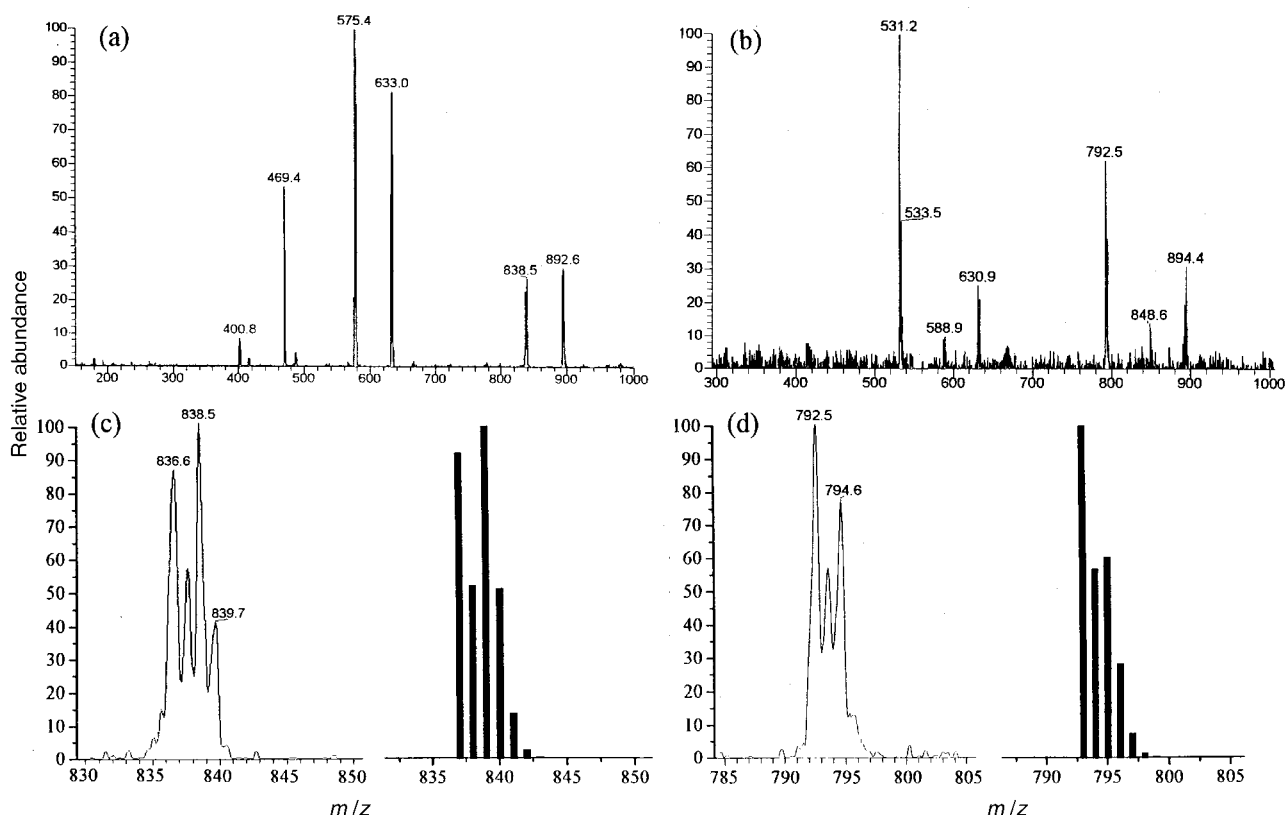


Fig. 3 ES-MS spectrum of (a) complex **1** and (b) complex **2**. Comparison of the observed (traces) and calculated (bars) isotopic distributions for the molecular cations at (c) m/z 838.5 for **1** and (d) m/z 792.5 for **2**. The quoted m/z values represent the major peaks in the isotopic distribution.

change of the appending group of tripodal ligands has a great influence on the formation of frameworks.

In addition to the $[\text{Ag}(\text{tbib})(\text{PPh}_3)]^+$ cation and NO_3^- anion, there are methanol and water molecules in the crystal packing of **1** as illustrated in Fig. 2. In all, six inter-molecular hydrogen bonds were found in **1**: $\text{N}(2)-\text{H}\cdots\text{O}(1)$, 3.00; $\text{O}(4)-\text{H}\cdots\text{N}(5)^{\#1}$, 2.78; $\text{N}(4)-\text{H}\cdots\text{O}(5)$, 2.76; $\text{N}(6)-\text{H}\cdots\text{O}(2)^{\#1}$, 3.20; $\text{N}(6)-\text{H}\cdots\text{O}(3)^{\#1}$, 3.24 and $\text{O}(5)-\text{H}\cdots\text{O}(4)^{\#1}$, 2.67 Å (symmetry transformations used to generate equivalent atoms: $\#1\ 1-x, y, 1.5-z$).

Electrospray mass spectra of complexes **1** and **2**

The synthesized complexes were further characterized by mass spectroscopy and the spectral data and assignments are summarized in Table 3. The ES mass spectra of complexes **1** and **2** are shown in Fig. 3. Six main peaks were observed for each complex. All the assignments were confirmed by good agreement between the observed and calculated isotopic distributions. Figs. 3(c) and 3(d) show typical examples for the comparison between the experimental and calculated isotopic distributions for the peaks at m/z 838.5 and 792.5. The observation of peaks at m/z 838.5 and 792.5 confirms the existence of molecular cations, $[\text{Ag}(\text{tbib})(\text{PPh}_3)]^+$ for **1** and $[\text{Cu}(\text{tbib})(\text{PPh}_3)]^+$ for **2**, respectively. It is noted that in the ES-MS spectrum of **2** the majority of peaks are Cu(I) species but there are some Cu(II) species as well [Fig. 3(b) and Table 3]. The appearance of $[\text{Cu}^{\text{II}}(\text{tbib})\text{ClO}_4]^+$ and $[\text{Cu}^{\text{II}}(\text{tbib})(\text{PPh}_3)\text{ClO}_4]^+$ indicates that some of the Cu(I) was oxidized to Cu(II) under the electrospray conditions. When the capillary temperature was set at 200 °C the relative intensity of the peak at m/z 792.5 decreased and the cation at m/z 630.9 became the major species. Complex **2** is stable in the solid state at room temperature.

^{31}P NMR spectra and electrochemical properties

In the $^{31}\text{P}\{^1\text{H}\}$ NMR spectra of complexes **1** and **2** in DMSO- d_6 at 298 K, a single broad peak was observed at 13.8 ppm for **1** and at -0.5 ppm for **2**, respectively. The reported complexes $[(\eta^5\text{-C}_5\text{Me}_5)\text{Ir}(\text{pz})_3\text{M}(\text{PPh}_3)]$ [$\text{M} = \text{Ag}(\text{I}), \text{Cu}(\text{I})$], in which the Ag(I) or Cu(I) atom is also three-coordinated with N_2P binding set, exhibited a signal at 15.7 ppm for Ag(I) and at 2.6 ppm for the Cu(I) complex at room temperature.¹³ The NMR results suggest that **1** and **2** are bound with the phosphorus atom of triphenylphosphine in solution.

The electrochemical properties of complexes **1** and **2** were investigated by cyclic voltammetry. Complex **1** showed the absence of any metal-based redox response up to 1.5 V with DMSO as solvent. This might be explained by a very slow redox process at the electrode surface.¹⁶ Complex **2** exhibited oxidation and reduction potentials at 0.27 and -0.35 V *vs.* ferrocene ($i_{\text{pc}}/i_{\text{pa}} = 0.85$), respectively. A large separation between the oxidation and reduction potentials is considered to be caused by the different geometric requirements of Cu(I) and Cu(II).¹⁷ Cu(I) complexes coordinated by nitrogen atoms of oligopyridine ligands with tetrahedral environments have been reported to have Cu(I)/Cu(II) redox potentials in a range of 0.5 to 1.0 V *vs.* SCE in various solvents.^{17,18}

Conclusion

The ^1H NMR spectral data and elemental analysis indicate that the ratio of the tbib and PPh_3 ligands in complex **2** is

1 : 1, the same as in **1**. In addition, similar species were observed in the ES-MS spectra of **1** and **2** as shown in Fig. 3 and Table 3. These spectral data suggest that the structure of **2** is the same as **1**, meaning that **2** is probably a mononuclear complex with the same N_2P binding set and is thus an analog of the Ag(I) complex.¹³

In conclusion we report a rationally designed novel tripodal ligand, 1,3,5-tris(benzimidazol-2-ylmethyl)benzene (tbib), and its silver(I) and copper(I) complexes **1** and **2** obtained by reaction of the tbib and PPh_3 ligands with AgNO_3 and $[\text{Cu}(\text{CH}_3\text{CN})_4]\text{ClO}_4$, respectively. The absence of multinuclear networks indicates that the change in appending group of the tripodal ligand has a great influence on the formation of supramolecular complexes. These new complexes provide examples of three-coordinated Ag(I) and Cu(I) with an N_2P binding set. As far as we know only five structurally characterized Ag(I) complexes with N_2P coordination mode have been reported up to now.

Acknowledgements

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