



Two 1D [Cu_xI_x]-based coordination polymers of tetraphosphine ligands

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ABSTRACT

Diffusion reactions of CuI with N,N,N',N'-tetra(diphenylphosphanylmethyl)ethylene diamine (dppeda) and 1,4-N,N,N',N'-tetra(diphenylphosphanylmethyl)benzene diamine (dpppda) in MeCN/toluene in a zig-zag glass tube or solvothermal reactions of CuI and dppeda or dpppda in MeCN produced two [Cu_xI_x]-based coordination polymers, [Cu₂I₂(dppeda)]·2MeCN (**1**·2MeCN) and [Cu₄I₄(dpppda)]·MeCN (**2**·MeCN), respectively. Both compounds were characterized by elemental analysis, IR spectroscopy, ¹H and ³¹P{¹H} NMR, ESI-MS and thermogravimetric analysis, and their structures were determined by single crystal X-ray diffraction. In **1**, [Cu₂I₂] cores are interconnected by dppeda ligands in a μ-η²:η² end-to-end mode to form a 1D chain, while in **2** stair-like [Cu₄I₄] cores are linked by dpppda ligands in a unique Z-shaped μ-η²:η² side-by-side mode to produce the other kind of 1D chain.

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In the past decades, the design and synthesis of multi-phosphine ligands have attracted much attention [1–8] due to their versatile ligating capability as well as rich structural chemistry and unique physical and chemical properties of their metal complexes [9–16]. Among the multiphosphine ligands, several tetraphosphine ligands and their metal complexes that showed interesting luminescent properties, catalytic and anticancer performances, have been extensively investigated [17–20]. In the case of IB metals, the tetraphosphine ligands could react with Au(I) and Ag(I) salts to form luminescent Au(I) complexes [20] and Ag(I) complexes (cage-like) [11–13]. However, synthesis of Cu(I) complexes with tetraphosphine ligands have been virtually not explored, though a great number of Cu(I) complexes with diphosphines [3–5] were reported.

Recently, we have also been interested in the preparation of coinage metal complexes with tetraphosphines. N,N,N',N'-tetra(diphenylphosphanylmethyl)ethylene diamine (dppeda) was used to react with Ag(I) salts to produce a dinuclear Ag(I) complex with a molecular basket structure and a polymeric complex (2D layer) [21]. As an extension of this study and an exploration of the coordination chemistry of tetraphosphines towards Cu(I), we employed CuI to react with dppeda and its analogue 1,4-N,N,N',N'-tetra(diphenylphosphanylmethyl)benzene diamine (dpppda), which resulted in the formation of two different [Cu_xI_x]-based coordination polymers [Cu₂I₂(dppeda)]·2MeCN (**1**·2MeCN) and

[Cu₄I₄(dpppda)]·MeCN (**2**·MeCN). Herein we report their synthesis and structural characterization.

Directly mixing a solution of CuI in MeCN with a solution of dppeda or dpppda in toluene gave rise to an instant white precipitate. Attempts to recrystallize it failed due to its low solubility in common organic solvents. Therefore we decided to employ other strategies to prepare Cu/tetraphosphine complexes. One method is the diffusion reactions in a zigzag tube we developed recently [22]. Stepwisely loading a solution of dppeda or dpppda in toluene, a mixed solution of toluene and MeCN as a buffer band, an acetonitrile solution of CuI and diethyl ether followed by leaving it at ambient temperature for 10 days afforded **1**·2MeCN (61% yield) or **2**·MeCN (56% yield) [23]. The other is the solvothermal reactions in sealed glass tube. Reactions of CuI with dppeda or dpppda in MeCN at 100 °C for one day produced **1**·2MeCN and **2**·MeCN in somewhat higher yields (79% for **1**·2MeCN and 61% for **2**·MeCN). In both cases, changing the molar ratios of tetraphosphines and CuI in 1:1–4 yielded the same compound. Crystals of **1**·2MeCN and **2**·MeCN easily lost solvent when being taking out of their mother liquor. When the solvated MeCN molecules in **1**·2MeCN and **2**·MeCN were removed *in vacuo*, **1** and **2** were stable towards air and moisture, and slightly soluble in DMF and DMSO. The elemental analyses were consistent with their chemical formulae. The IR spectra of **1** and **2** show the stretching vibrations of the phenyl groups at ~1600, 1482 and 1430 cm⁻¹. The ¹H NMR spectra showed signals related to protons of the ethylene group (2.73 ppm for **1**), the methylene groups (3.64 ppm for **1** and 3.34 ppm for **2**), the -NC₆H₄N- group (5.84 ppm for **2**) and the phenyl groups (7.09–7.60 ppm for **1** and 7.18–7.67 ppm for **2**), respectively. In the ³¹P{¹H} NMR spectra (Figs. S1 and S2), there existed the chemical shifts from -25.30 ppm (**1**) to -28.5 ppm (dppeda) or from

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–24.56 ppm (2) to –26.63 ppm (dpppda), which may be due to the coupling between the ^{31}P and ^{63}Cu (^{65}Cu) nuclei. The identities of both compounds were confirmed by X-ray crystallography [24].

1·2MeCN and **2**·MeCN crystallize in the triclinic space group $P\bar{1}$, and the asymmetric unit of **1** contains half of one $[\text{Cu}_2\text{I}_2(\text{dppeda})]$ molecule and one MeCN solvent molecule while that of **2** has half of one $[\text{Cu}_4\text{I}_4(\text{dpppda})]$ molecule and half of one MeCN solvent molecule [24]. In **1**, two Cu(I) and two I atoms form a $[\text{Cu}_2\text{I}_2]$ core with a crystallographic center of symmetry being at the midpoint of Cu(1)··Cu(1A) contact (**1**) (Fig. 1). Each Cu(I) atom is tetrahedrally coordinated to two μ -I atoms and two P atoms. The Cu(1)··Cu(1A) contact (3.175(1) Å) is too long to include any metal–metal interactions. The mean Cu– μ -I and Cu–P bond lengths (2.6582(9) Å vs. 2.2776(13) Å) are close to those of the corresponding ones in $[\text{Cu}(\mu\text{-I})(\text{L})_2]$ (2.662(2) Å vs. 2.272(1) Å for L = bis(diphenylphosphino)propane) [3], 2.635(3) Å vs. 2.281(2) Å for L = bis(diphenylphosphino)butane [26].

In **2**, four copper(I) centers are held together via two doubly-bridging and two triply-bridging iodides to form a stair-like Cu_4I_4 core [27,28], which has a crystallographic center of symmetry at

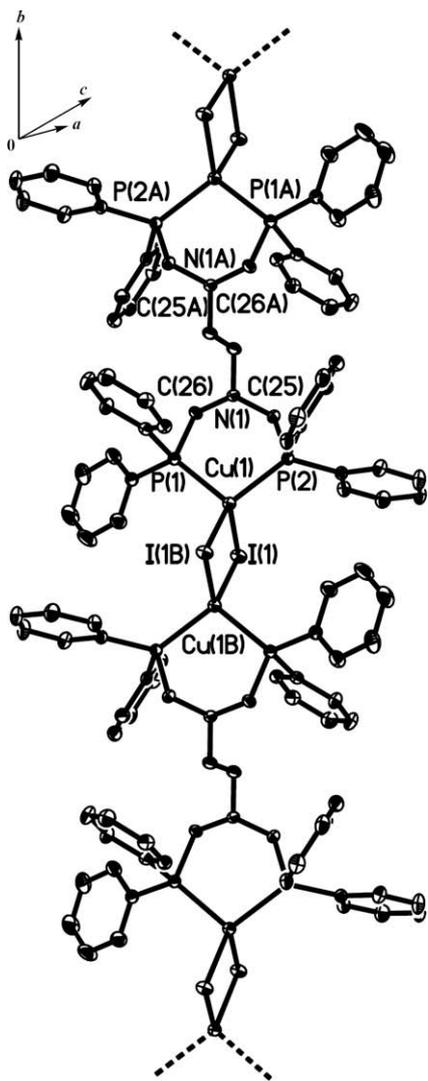


Fig. 1. View of a section of the 1D chain of **1** extending along the *b*-axis. All hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (°): Cu(1)–P(1) 2.2764(13); Cu(1)–P(2) 2.2788(10); Cu(1)–I(1) 2.6664(9); Cu(1)–I(1B) 2.6500(9); P(1)–Cu(1)–P(2) 103.10(4); P(1)–Cu(1)–I(1) 105.42(4); P(1)–Cu(1)–I(1B) 109.20(4); P(2)–Cu(1)–I(1) 108.21(4); P(2)–Cu(1)–I(1B) 123.02(4); I(1)–Cu(1)–I(1B) 106.66(2). Symmetry codes: (A) $-1-x, 1-y, -z$; (B) $-1-x, -y, -z$.

the midpoint of the N(1)··N(1A) contact (**2**) (Fig. 2). Cu(1) is coordinated by one P, μ -I and two μ_3 -I atoms, adopting a distorted tetrahedron geometry, while Cu(2) by one P, μ -I and one μ_3 -I atoms, adopting a planar trigonal geometry. The Cu(1)··Cu(1A) and the Cu(1)··Cu(2) separations are 3.523(1) Å and 3.302(1) Å, which exclude any metal–metal interactions. For tetrahedrally-coordinated Cu, the mean Cu(1)– μ_3 -I bond length (2.7460(14) Å) is longer than that of Cu(1)– μ -I(2) bond and that of the corresponding ones in $[\text{Cu}_4\text{I}_4(\text{PPh}_3)_4]$ (2.685(3) Å) [29]. The trigonally-coordinated Cu(2)– μ_3 -I(1) and Cu(2)– μ -I(2) bond lengths are almost the same, but are in-between those of the corresponding ones of $[\text{Cu}_4\text{I}_4(\text{PPh}_3)_4]$ (2.559(3) Å) and **1**. The mean Cu–P bond length (2.240(3) Å) is in between that of **1** and that of $[\text{Cu}_4\text{I}_4(\text{PPh}_3)_4]$ (2.235(2) Å).

From a topological view, each $[\text{Cu}_2\text{I}_2]$ core in **1** or $[\text{Cu}_4\text{I}_4]$ core in **2** connects its equivalent ones via dppeda or dpppda bridges to form a 1D chain extended along the *b* (**1**) or *a* (**2**) axis. The adjacent chains are 12.37 Å apart with MeCN solvent molecules lying between the chains. The different coordination modes of dppeda and dpppda towards Cu(I) deserve comments. It seems like that the spacer of the tetraphosphine ligand influences its coordination modes. Relative to the $-\text{CH}_2\text{CH}_2-$ spacer in dppeda, the phenyl spacer in dpppda is longer and more rigid. Therefore, dppeda in **1** has a smaller side-space and takes a μ - η^2 : η^2 end-to-end mode

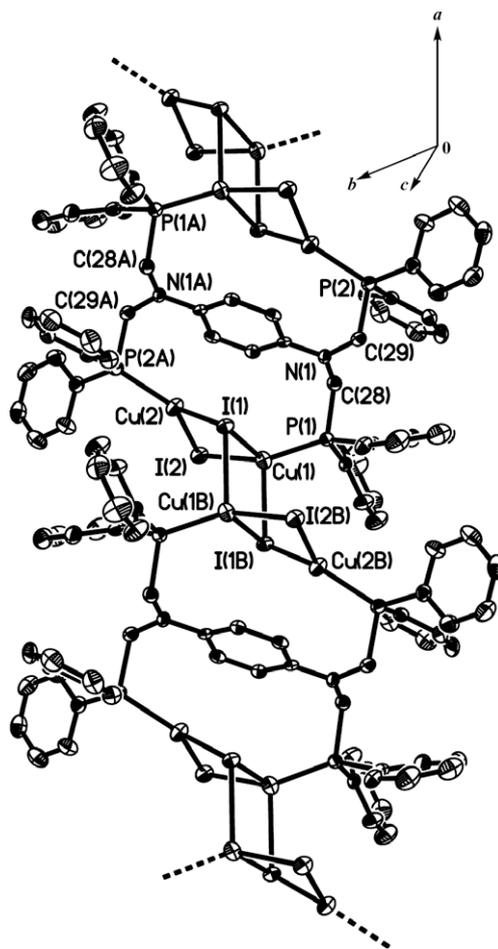
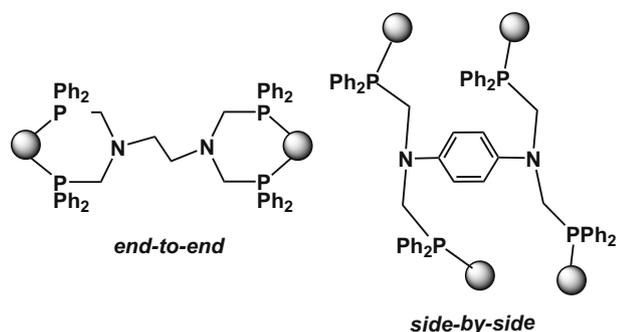


Fig. 2. View of a section of the 1D chain of **2** extending along the *a*-axis. All hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (°): Cu(1)–P(1) 2.247(3); Cu(2)–P(2A) 2.238(3); Cu(1)–I(1) 2.7493(16); Cu(1)–I(2) 2.6795(14); Cu(1)–I(1B) 2.7427(14); Cu(2)–I(1) 2.6135(14); Cu(2)–I(2) 2.6137(15); P(1)–Cu(1)–I(1) 121.07(8); P(1)–Cu(1)–I(2) 119.97(7); P(1)–Cu(1)–I(1B) 110.58(8); I(1)–Cu(1)–I(2) 98.85(5); I(1)–Cu(1)–I(1B) 100.19(5); I(2)–Cu(1)–I(1B) 103.11(5); P(2A)–Cu(2)–I(1) 129.87(7); P(2A)–Cu(2)–I(2) 124.96(8); I(1)–Cu(2)–I(2) 104.17(5). Symmetry codes: (A) $1-x, 1-y, 1-z$; (B) $-x, 1-y, 1-z$.



Scheme 1. The *end-to-end* coordination mode of dppda in **1** (left) and the *side-by-side* coordination mode of dpppa in **2** (right).

(Scheme 1) [17–21] to chelate at each of the two Cu atoms of the $[\text{Cu}_2\text{I}_2]$ core via two Cu–P bonds. On the contrary, dpppa in **2** adopts a rare Z-shaped $\mu\text{-}\eta^2\text{:}\eta^2$ *side-by-side* mode (Scheme 1) to have more side-space to coordinate at the four Cu(I) centers of the $[\text{Cu}_4\text{I}_4]$ core via four Cu–P bonds. Such a coordination mode was uncommon in the chemistry of tetraphosphines [20] because one tetraphosphine ligand usually employs a $\mu\text{-}\eta^2\text{:}\eta^2$ *side-by-side* mode to bind to only two metal centers [15,30,31].

In order to gain more insight into the stability of **1**·2MeCN and **2**·MeCN in solution and in solid state, we measured their ESI-MS and made their thermogravimetric analysis. The positive ESI-MS spectra of **1**·2MeCN and **2**·MeCN in DMSO (ionized by 0.1 M HCl) revealed a set of peaks assignable to $[\text{Cu}(\text{DMSO})_2]^+$ ($m/z = 219.1$), $[\text{Cu}(\text{PPh}_2)]^+$ ($m/z = 247.1$), $[\text{Cu}_2\text{ICl}_3]^+$ ($m/z = 359.2$), $[\text{Cu}_2(\text{PPh}_2)]^+$ ($m/z = 437.1$), $[\text{Cu}_2(\text{PPh}_2)]^+$ ($m/z = 501.2$), $[\text{Cu}_3\text{I}_4 + \text{H}]^+$ ($m/z = 699.2$), and $[\text{Cu}_4\text{I}_3(\text{DMSO})]^+$ ($m/z = 713.1$) (Figs. S3 and S4). The existence of these cationic fragments implied that the polymeric chains of **1** and **2** may not be stable in solution under the acidic and mass conditions, and that the resulting cations may be formed by decomposition of the $[\text{Cu}_2\text{I}_2]$ or $[\text{Cu}_4\text{I}_4]$ core and the tetraphosphine ligands followed by recombination of the decomposed species.

The thermogravimetric analysis showed that solids **1** and **2** were stable at ambient temperature (Fig. S5). Their TGA curves displayed three stages of decomposition. The first weight loss of 6.2% (**1**·2MeCN) and 2.3% (**2**·MeCN) in the region of 60–110 °C or 110–250 °C corresponds to the removal of the MeCN solvent molecules in **1**·2MeCN or **2**·MeCN. The second weight loss (33.7% for **1**·2MeCN and 52.0% for **2**·MeCN) in the region of 270–430 °C (**1**·2MeCN) or 280–510 °C (**2**·MeCN) amounts roughly to the loss of the tetraphosphine ligands in **1**·2MeCN or **2**·MeCN. Upon heating up to 660 °C (**1**·2MeCN) or 780 °C (**2**·MeCN), all iodine and phosphorous were removed and the final residue was assumed to be copper (9.7% for **1**·2MeCN and 14.9% for **2**·MeCN) according to X-ray fluorescence analysis.

Acknowledgements

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Appendix A. Supplementary material

CCDC 708477 and 708478 contain the supplementary crystallographic data for **1**·2MeCN and **2**·MeCN. 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. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.inoche.2009.08.014.

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- Method 1: To a zigzag glass tube was carefully loaded in order a solution of dppda/dpppa (0.005 mmol) in toluene (1 mL), 5 mL of toluene and MeCN (v/v = 1:1), a solution of CuI (0.01 mmol) in MeCN (1.5 mL), and 1 mL of diethyl ether. The glass tube was left at ambient temperature for 10 days. Colorless blocks of **1**·2MeCN or **2**·MeCN were collected by filtration and washed with toluene and MeCN (v/v = 2:1). Yield: 61% for **1**·2MeCN or 56% for **2**·MeCN. Method 2: To a Pyrex glass tube was added dppda/dpppa (0.01 mmol), CuI (0.02 mmol) and 1.0 mL of MeCN. The tube was then sealed and heated up to 100 °C for 24 h. After being cooled to ambient temperature at a rate of 5 °C/h, the resulting colorless blocks of **1**·2MeCN or **2**·MeCN were formed. Yield: 79% for **1**·2MeCN or 61% for **2**·MeCN. For **1**: Anal. Calcd for $\text{C}_{54}\text{H}_{52}\text{N}_2\text{P}_4\text{Cu}_2\text{I}_2$: C, 52.57; H, 4.25; N, 2.27. Found: C, 52.51; H, 4.31; N, 2.20%. IR (KBr, cm^{-1}): 3441 (m), 3046 (m), 2924 (w), 1966 (w), 1895 (w), 1885 (w), 1570 (m), 1482 (s), 1433 (s), 1370 (w), 1257 (w), 1184 (w), 1097 (s), 1026 (m), 859 (s), 739 (s), 692 (s), 509 (s), 482 (m). ^1H NMR (400 MHz, DMSO-d_6 , ppm): δ 7.09–7.60 (m, 40H, -Ph), 3.64 (s, 8H, -PCH₂N-), 2.73 (s, 4H, -CH₂CH₂-). ^{31}P NMR (400 MHz, DMSO-d_6 , ppm): δ -25.30 (s, -PPh₂). For **2**: Anal. Calcd for $\text{C}_{58}\text{H}_{52}\text{N}_2\text{P}_4\text{Cu}_4\text{I}_4$: C, 41.90; H, 3.15; N, 1.68. Found: C, 41.81; H, 3.13; N, 1.66%. IR (KBr, cm^{-1}): 3441 (m), 3049 (m), 2920 (w), 1960 (w), 1888 (w), 1608 (w), 1517 (s) 1482 (s), 1435 (s), 1348 (w), 1246 (m), 1217 (m), 1137 (m), 1098 (s), 1026 (m), 998 (m), 863 (m), 795 (m), 743 (s), 693 (s), 508 (s), 480 (m), 440 (w). ^1H NMR (400 MHz, DMSO-d_6 , ppm): δ 7.18–7.67 (m, 40H, -Ph), 5.84 (m, 4H, -Ph-), 3.34 (s, 8H, -PCH₂N-). ^{31}P NMR (400 MHz, DMSO-d_6 , ppm): δ -24.56 (s, -PPh₂).
- [24] Crystal data for **1**·2MeCN: $\text{C}_{58}\text{H}_{58}\text{N}_4\text{P}_4\text{Cu}_2\text{I}_2$, $M_r = 1315.84$, colorless crystal (0.10 × 0.40 × 0.28 mm), triclinic, space group $P1$, $a = 8.4994(17)$ Å, $b = 13.175(3)$ Å, $c = 13.383(3)$ Å, $\alpha = 67.52(3)^\circ$, $\beta = 82.70(3)^\circ$, $\gamma = 84.15(3)^\circ$, $V = 1371.1(5)$ Å³, $Z = 1$, $D_c = 1.594$ g cm^{-3} , $F(000) = 658$ and $\mu = 2.060$ mm⁻¹, $T = 193$ K, 8693 reflections collected, 4729 unique ($R_{\text{int}} = 0.0307$), $R_1 = 0.0303$, $wR_2 = 0.0621$ and $S = 0.983$ based on 4083 observed reflections with $I > 2.00\sigma(I)$. Crystal data for **2**·MeCN: $\text{C}_{60}\text{H}_{55}\text{N}_3\text{P}_4\text{Cu}_4\text{I}_4$, $M_r = 1703.71$, orange crystal (0.40 × 0.16 × 0.10 mm), triclinic, space group $P1$, $a = 10.363(2)$ Å, $b = 12.367(3)$ Å, $c = 13.540(3)$ Å, $\alpha = 108.27(3)^\circ$, $\beta = 106.76(3)^\circ$, $\gamma = 91.03(3)^\circ$, $V = 1566.6(7)$ Å³, $Z = 1$, $D_c = 1.806$ g cm^{-3} , $F(000) = 924$ and $\mu = 3.451$ mm⁻¹, $T = 193$ K, 15,313 reflections collected, 5710 unique ($R_{\text{int}} = 0.0480$), $R_1 = 0.0580$, $wR_2 = 0.1311$ and $S = 1.107$ based on 4234 observed reflections with $I > 2.00\sigma(I)$. Data collections were performed on a Rigaku Mercury CCD

diffractometer with graphite-monochromated Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$). The crystal structures were solved by direct methods and refined with full-matrix least-squares technique using SHELXL-97 program [25]. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms.

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