THE FIRST COPPER(I) HALIDE Π-COMPLEXES WITH ALLYL DERIVATIVES OF UREA AND PARABANIC ACID

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By means of alternating current electrochemical synthesis with wire copper electrodes in methanolic solutions of corresponding ligands and copper(II) halide salts, two novel copper(I) Π-complexes \([\text{Cu}_2(\text{dau})\text{Cl}_{1.87}\text{Br}_{0.42}]\) (I) and \([\text{Cu}_2(\text{dapa})\text{Cl}_{1.74}\text{Br}_{0.26}]\) (II) (where dau and dapa are 1,3-diallylurea and 1,3-diallylparabanate, respectively) were prepared in well-shaped crystalline form and studied with X-ray diffraction method. Compound I crystallizes in orthorhombic crystal system, space group \(P2_12_12_1\): \(a=8.047(3)\ \text Å, b=8.154(3)\ \text Å, c=16.533(5)\ \text Å, V=1085.8(76)\ \text Å^3, Z=4\). Compound II crystallizes in monoclinic crystal system, space group \(I2/a\): \(a=6.420(3)\ \text Å, b=8.088(3)\ \text Å, c=49.020(9)\ \text Å, \beta=90.92(3)^\circ, V=2545.03(16)\ \text Å^3, Z=8\). Both structures contain two crystallographically independent copper(I) ions with one half in trigonal planar coordination environment and the other half in trigonal pyramidal one. Both compounds are built of parallel copper halide polymeric chains of one type in I and two types in II, cross-linked by organic ligand molecules to produce 3D-polymer bulk in I and 2D-layered structure in II. In compound I, 1,3-diallylurea is both Π- and Π-donor ligand for copper(I) halide chains (by means of two allylic \(\text{C}=\text{C}\) bonds and carbonyl O atom), whereas 1,3-diallylparabanate is a solely Π-donor ligand in compound II. The structure of II is noteworthy due to the combination of two different chain types that have been previously observed only separately in copper(I) halide coordination compounds.

Keywords: copper(I) halides, Π-complex, diallylurea, diallylparabanate, crystal structure.

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Introduction

Copper(I) halides are known to form diverse structures in combination with organic ligands [1], including the ones capable of being Π-coordinated, for example described in [2–4].

A large number of olefin–copper(I) Π-complexes is known to date. A common approach toward the design of ligands is combining rigid fragments such as heteroatom-containing molecules (σ-donors) with flexible allyl group substituents (Π-coordination) [5]. Additional hydrogen atoms connected to heteroatoms are involved in hydrogen bonds formation, that further stabilizes the crystal structures.

A good example of such fragment, which possesses both potential Π-donating oxygen atom as well as groups capable of hydrogen bonds formation is urea. Its 1,3-diallyl derivative (dau) has already been investigated as a ligand for Π-complexation of silver [6] due to the structural similarity to 1,3,5-cyanuric acid (Fig. 1) and has demonstrated its ability to be a linker in coordination polymers. Nevertheless, its behavior in presence of compounds that can catenate on their own has never been tested prior to this moment. An example of such partners for dau are copper(I) halides.

Another ligand, structurally related to 1,3-diallylurea, is 1,3-diallylparabanate (dapa) (Fig. 1). This compound has been studied in context of useful polymer materials preparation [7]. Nevertheless, its use as a ligand for copper(I) Π-coordination is novel. This ligand has a similar spatial arrangement of allyl groups, as in 1,3-diallylurea, while not being able to donate hydrogen bonds and being more sterically hindered. 1,3-Diallylparabanate possesses only oxygen atoms as potential Π-donor with their hard basic properties not favorable for bonding with soft acidic Cu+. Consequently, the dapa ligand is a good candidate for solely Π-complexation while possibly...
inspired by respective earlier studied silver complexes of dau [6], two compounds, [Cu(dau)Cl1.58Br0.42] (I) and [Cu(dapa)Cl1.74Br0.26] (II), were prepared and studied by X-ray diffraction in this work.

**Experimental**

**Synthesis of the ligands**

1,3-diallylurea (dau) was prepared starting from the unsubstituted urea and allylamine in a similar approach as described elsewhere [8].

For the synthesis of 1,3-diallylparabanate (dapa), sodium parabanate was prepared starting from urea and diethylxalate according to the known procedure [9]. The salt was then alkylated in methanol with allyl bromide yielding dapa.

**Preparation of [Cu(dau)Cl1.58Br0.42] (I)**

To 4 mL of ethanol solution of CuCl2.H2O (2.4 mmol, 0.409 g), 0.322 g (2.3 mmol) of dau and 0.247 g (2.4 mmol) of NaBr were added. The mixture was carefully stirred and the resulting solution was placed into a 5 mL test tube and then copper-wire electrodes in cork were inserted. With the application of an alternating current (voltage of 0.56 V, frequency of 50 Hz) for 3 days, good quality colorless crystals of I appeared on the copper electrodes in a very small amount.

**Preparation of [Cu(dapa)Cl1.74Br0.26] (II)**

To methanol solution (4 mL) of CuCl2.2H2O (2.4 mmol, 0.409 g), 0.322 g (2.3 mmol) of dau and 0.247 g (2.4 mmol) of NaBr were added. The mixture, after careful stirring, was subjected to an alternating current (voltage of 0.70 V, frequency of 50 Hz) for 4 days. Crystals of II appeared on the copper electrodes.

**Results and discussion**

1,3-Diallyl urea (dau) in the structure of π-complex [Cu(dau)Cl1.58Br0.42] (I) serves as a ligand possessing both σ- and π-coordination abilities. Atom Cu1 in I adopts trigonal pyramidal coordination surrounding (τ1=0.78, where τ1 is the geometry index calculated elsewhere [10]) that consists of C7=C8 bond of the allyl group and O1 atom of the same dau molecule and two halogen ions (Hal1 & Hal2) of the endless copper(I) halide polymer chain. Cu1 deviates from the base plane of coordination polyhedron by 0.28(1) Å, whereas the angle between C7=C8 line and the base plane is 9.6(1)°. Ion Cu2 in I possesses planar trigonal coordination environment formed by Cl0=Cl1 bond of the other allyl group of dau and two halogen ions of the neighboring infinite chain (Fig. 2, Table 2). π,σ-Coordination of the one half of copper ions and π-coordinated to the other half of copper shows similarity to dau’s behavior in silver(I) complexes [6].

Copper halide 1D-polymer chains (Fig. 3) stretch along [010] in the crystal of the complex I. The chains are linked by the bridging molecules of dau into 3D-polymer.

1,3-Diallyl parabanate is exclusively π-coordinated in the structure of the compound [Cu3(C10H10N2O3)Cl1.74Br0.26] (II). Ion Cu1 forms trigonal pyramidal surrounding (τ1=0.76) that consists of Hal1, Hal1i and Hal1ii ions of the ladder-like copper halide polymer chain and C7=C8–bond of...
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Note: CCDC 1890065 and 1870895 contain the supplementary crystallographic data for this paper. Copies of the data can be obtained free of charge on applications to the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: int.code +(1223)336–033; e-mail for inquiry: fileserv@ccdc.cam.ac.uk).

Table 1

| Characteristics          | I                | II               |
|--------------------------|------------------|------------------|
| CCDC number              | 1890065          | 1870895          |
| Empirical formula        | C₇H₁₂Br₀.₄₂Cl₁₅₈Cu₂N₂O | C₉H₁₀Br₂₀₂Cl₁₇₆Cu₂N₂O₂ |
| Formula weight           | 356.95           | 403.78           |
| Temperature, K           | 100              | 100              |
| Wavelength               | MoKα             | MoKα             |
| Crystal system, space group | Orthorhombic, P2₁₂₁₂₁ | Monoclinic, I2/a |
| Unit cell dimensions: a, Å | 8.047(3)         | 6.420(3)         |
|                          | b, Å             | 8.154(3)         |
|                          | c, Å             | 16.533(5)        |
| β, °                      | 90               | 90.92(3)         |
| V, Å³                    | 1084.8(7)        | 2545.0(16)       |
| Z                        | 4                | 8                |
| Calculated density, g cm⁻³ | 2.186            | 2.108            |
| Absorption coeff., mm⁻¹  | 5.831            | 4.526            |
| F(000)                   | 702              | 1590             |
| Crystal size, mm³         | 0.32×0.13×0.12   | 0.15×0.09×0.04   |
| Color                    | colorless        | colorless        |
| Theta range for data collection, ° | 2.5–29.6       | 4.1–28.0         |
| Measured reflections     | 57513            | 1739             |
| Used in refinement       | 2945             | 1248             |
| Refined parameters       | 130              | 105              |
| R[F²>2σ(F²)], wR(F²), S  | 0.0192, 0.0407, 1.097 | 0.0805, 0.2176, 1.005 |

Table 2

| Bond                  | d, Å   | Angle             | α, °   |
|-----------------------|--------|-------------------|--------|
| Cu₁–C7                | 2.098(3) | Hal1–Cu1–m₁       | 124.14(9) |
| Cu₁–C8                | 2.071(3) | O1–Cu1–m₁         | 97.17(10)  |
| Cu₁–m₁                | 1.970(3) | Hal₂–Cu1–m₁       | 123.64(9)  |
| C7–C8                 | 1.362 (5) | Hal1–Cu1–Hal₂     | 107.42(2)  |
| Cu₁–Hal₁              | 2.301(10) | O1–Cu1–Hal₂       | 92.23(5)   |
| Cu₁–Hal₂              | 2.375(10) | O1–Cu1–Hal₁       | 102.28(5)  |
| Cu₁–O1                | 2.279(2) | Hal₂–Cu2–m₂       | 126.56(9)  |
| Cu₂–C₁₀               | 2.092(3) | Hal₁–Cu2–m₂       | 128.76(9)  |
| Cu₂–C₁₁               | 2.060(3) | Hal₂–Cu₂–Hal₁     | 104.04(2)  |
| Cu₂–m₂                | 1.962(3) | Hal₁–Cu1–Hal₂     | 107.42(2)  |
| C₁₀–C₁₁               | 1.359 (4) | Cu₁–Hal₁–Cu₂      | 90.57(3)   |
| Cu₂–Hal₁              | 2.308(6) | Cu₂–Hal₂–Cu₃      | 117.06(3)  |
| Cu₂–Hal₂              | 2.295(0) | Cu₂–Hal₂–Cu₃      | 105.37(3)  |
| Cu₂–Hal₃              | 2.295(0) | Cu₂–Hal₂–Cu₃      | 105.37(3)  |

Note: ‘m₁’ is the midpoint of C7=C8 bond; ‘m₂’ is the midpoint of C₁₀=C₁₁ bond. Symmetry codes: (i) x+1/2, −y+1/2, −z+1; (ii) −x+1/2, −y+1, z+1/2; (iii) −x+1/2, −y+1, z−1/2; (iv) x−1/2, −y+1/2, −z+1. Hal stands for statistically present halogen ions: chloride and bromide.
-coordination is not observed for any oxygen atom in dapa in comparison to oxygen atom in dau, what is in consistence with harder Lewis basicity.

The two types of infinite copper(I) halide polymer chains propagating in parallel direction are cross-linked by the dapa molecules into 2D-sheets and for every a-type double strand chain (Cu1&Hal1) two b-type chains (Cu2&Hal2) are present (Fig. 5).

The sheets are stacked on each other with protrusions of the sheet entering the depressions of the adjacent sheets and vice versa. Cu2 and Hal2 ions in the adjacent sheets approach each other close to the sum of van der Waals radii threshold providing electrostatic attraction that holds the sheets together.

The O3 and C5 atoms in the neighboring dapa moieties approach each other to a distance shorter than the sum of respective van der Waals radii, which could be interpreted as a consequence of dipole-dipole interaction or packing effects.

Compounds I and II demonstrate the joint action of copper(I) halide catenation and cross-linkage by organic molecules resulting into formation of polymers of higher dimension than copper(I) halide chains on their own. Their case illustrates how increase in hardness of potential σ-donating atoms causes a transition from one coordination mode to the other in ligands with similar geometry of copper(I) binding site.

Conclusions

Two copper(I) π-complexes, [Cu2(dau)Cl0.63Br0.37] (I) and [Cu2(dapa)Cl1.70Br0.30] (II), (where dau and dapa are 1,3-diallylurea and 1,3-diallylparabanate, respectively) were prepared and characterized for the first time in this work. Both structures contain two crystallographically independent copper(I) ions with one half in trigonal planar coordination environment and the other half in trigonal pyramidal one. Both compounds are built of parallel copper halide polymeric chains of one type in I and two types in II, cross-linked by organic ligand molecules to produce 3D-polymer bulk in I and 2D-layered structure in II. The structure of II is noteworthy due to the combination of two different chain types that have been previously observed only separately in copper(I) halide coordination compounds.

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Fig. 4. Numbering scheme in structure of compound II. Thermal ellipsoids are shown at 50% probability level. Symmetry codes:
(i) $-x+1/2, y, -z+1$; (ii) $-x+1, -y, -z+1$; (iii) $x+1/2, -y+1, z$

Fig. 5. Two types of copper(I) halide chains in structure of compound II. Symmetry codes:
(i) $-x+1/2, y, -z+1$; (ii) $-x+1, -y, -z+1$; (iii) $x+1/2, -y+1, z$; (iv) $x-1/2, -y+1, z$
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ПЕРШІ ★-КОМПЛЕКСИ ГАЛОГЕНІДІВ КУПРУМУ(I) З АЛІЛЬНИМИ ПОХІДНИМИ СЕЧОВИННИ І ПАРАБАНОВОЇ КИСЛОТИ

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Шляхом електрохімічного синтезу зі змінним строєм у метанолному розчині реагентів на мідних електродах було одержано два нових π-комплекси міді(I): [Cu\textsubscript{2}(dau)Cl\textsubscript{2}Br\textsubscript{2}] (I) і [Cu\textsubscript{2}(dau)Cl\textsubscript{2}Br\textsubscript{2}] (II) (dau – 1,3-діалілпірамідин). Сполучення I кристалізується в отро- робичній сингонії, просторова група P2\textsubscript{2}12\textsubscript{2}; a=8.047(3) Å, b=8.154(3) Å, c=16.533(5) Å; V=2545.03(16) Å\textsuperscript{3}, Z=8. Сполучення II кристалізується в монохіліній сингонії, просторова група I2/a: a=6.420(3) Å, b=8.088(3) Å, c=49.020(9) Å; V=2545.03(16) Å\textsuperscript{3}, Z=8. Обидва структури містять по два кра- сталографічно незалежних атоми Купрума, з яких одна поло- вина знаходиться в прямокутному координаційному оточенні, тоді як інша знаходиться у прямокутно-пірамідальному ото-ченні. Обидві сполуки побудовані із паралелельних ланцюгів галогенідів купруму(I) одного типу в І і двох типів у II, сполучених між собою молекулами органічних лігандів, що призводить до тримірного каркаса в I і двовимірних шарів у II. В I 1,3-діалілпірамідина однорядно π- і π-еїз як із ланцюками галогенідів купрума(I) за рахунок двох аляльних грун і карбоній- ного атома Оксигену, тоді як в II 1,3-діалілпірамідина – виключно π-донорний ліганд. Структура сполуки II варта ува- ги через сумісну присутність двох різних типів полімерних лан- цюгів галогенідів купруму(I), які до цього спостерігалися в кра- сталічних структурах лише окремо.

Ключові слова: галогениди купруму(I), π-комплекс, діалілпірамідина, діалілпірамідина, кристалічна структура.
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By means of alternating current electrochemical synthesis with wire copper electrodes in methanolic solutions of corresponding ligands and copper(II) halide salts, two novel copper(I) π-complexes [Cu(dau)Cl]_{x,y}Br_{z,w} (I) and [Cu(dapa)Cl]_{x,y}Br_{z,w} (II) (where dau and dapa are 1,3-diallylurea and 1,3-diallylparabanate, respectively) were prepared in well-shaped crystalline form and studied with X-ray diffraction method. Compound I crystallizes in orthorhombic crystal system, space group P2_12_2_1: a=8.047(3) Å, b=8.154(3) Å, c=16.533(5) Å, V=1085.8(76) Å³, Z=4. Compound II crystallizes in monoclinic crystal system, space group I2/a: a=6.420(3) Å, b=8.088(3) Å, c=49.020(9) Å, β=90.92(3)°, V=2545.03(16) Å³, Z=8. Both structures contain two crystallographically independent copper(I) ions with one half in trigonal planar coordination environment and the other half in trigonal pyramidal one. Both compounds are built of parallel copper halide polymeric chains of one type in I and two types in II, cross-linked by organic ligand molecules to produce 3D-polymer bulk in I and 2D-layer structure in II. In compound I, 1,3-diallylurea is both π- and α-honded to copper(I) halide complexes (by means of two allylic C=C bonds and carbonyl O atom), whereas 1,3-diallylparabanate is a solely π-donor ligand in compound II. The structure of II is noteworthy due to the combination of two different chain types that have been previously observed only separately in copper(I) halide coordination compounds.

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