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SYNTHESIS AND CRYSTAL STRUCTURE OF COPPER(I) CHLORIDE π-COMPLEX BASED ON 3-ALLYLTHIO-4-ALLYL-5-(2-PYRIDYL)-4*H*-1,2,4-TRIAZOLE

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Abstract

This work is devoted to the synthesis and structure characterization of a new copper(I) η^2-π-complex [$Cu_4(\mathit{Apt})_2Cl_4$] (1) based on 3-allylthio-4-allyl-5-(2-pyridyl)-4*H*-1,2,4-triazole (*Apt*). Crystals of the compound were obtained by means of alternating current-electrochemical technique and studied by X-ray single crystal diffraction. The π-complex crystallizes in the monoclinic centrosymmetric space group $P2_1/n$ (a = 10.884(3), b = 10.944(3), c = 13.860(4) Å, β = 93.96(3) °, V = 1647.0(8) Å^3, Z = 2) and is built of polymeric chains. In its crystal structure, two crystallographically independent copper(I) atoms have different coordination arrangements: Cu1 forms a close to trigonal-pyramidal coordination environment composed of two triazole N atoms, an allylic C=C bond, and one halogen atom, while distorted tetrahedral surrounding of Cu2 is formed by pyridyl N atom and tree bridging Cl atoms. The pyridyl-substituted 1,2,4-triazole defines the formation of an infinite coordination polymer in 1.

Keywords: copper(I); 1,2,4-triazole; allyl derivative; crystal structure.

СИНТЕЗ ТА КРИСТАЛІЧНА СТРУКТУРА π-КОМПЛЕКСУ КУПРУМ(I) ХЛОРИДУ НА ОСНОВІ 3-АЛІЛТІО-4-АЛІЛ-5-(2-ПІРИДИЛ)-4*Н*-1,2,4-ТРИАЗОЛУ

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Анотація

Робота присвячена синтезу та характеристиці структури нового купрум(I) η^2-π-комплексу [$Cu_4(\mathit{Apt})_2Cl_4$] (1) на основі 3-алілтіо-4-аліл-5-(2-піридил)-4*Н*-1,2,4-триазолу (*Apt*). Кристали сполуки були отримані методом змінно-струмного електрохімічного синтезу та досліджені за допомогою рентгеноструктурного аналізу. π-Комплекс кристалізується в моноклінній центросиметричній просторовій групі $P2_1/n$ (a = 10,884(3), b = 10,944(3), c = 13,860(4) Å, β = 93,96(3) °, V = 1647.0(8) Å^3, Z = 2) і побудований з полімерних ланцюгів. У кристалічній структурі два кристалографічно незалежні атоми купруму(I) мають різне координаційне оточення: Cu1 утворює близьке до тригонально-пірамідального координаційне оточення, що складається з двох атомів N триазолу, алільного зв’язку C=C та одного місткового атома хлору, тоді як деформоване тетраедричне тетраедричне оточення Cu2 утворене атомом N піридильного замісника та трьома містковими атомами Cl. Піридил-заміщений 1,2,4-триазол визначає формування нескінченного координаційного полімеру в 1.

Ключові слова: купрум(I); 1,2,4-триазол; алільне похідне; кристалічна структура.

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Introduction

1,2,4-Triazoles are fascinating nitrogen-rich heterocyclic scaffolds for the synthesis and design of versatile medicinal compounds and a variety of transition-metal organometallic compounds, that form stable mononuclear or polynuclear complexes and coordination polymers possessing luminescent, nonlinear, catalytic, biochemical, and spin-crossover activities [1–5]. Among them, allyl derivatives of 5-phenyl-1,2,4-triazole-3-thiols were used in the preparation of mixed-valence and heterometallic copper chloride π,σ-coordination compounds with magnetic and nonlinear optical properties [6; 7]. Copper(I) π-complexes with allyl derivatives of heterocycles are appealing objects for modern interdisciplinary research and may also have a number of potential applications in advanced laser-operated materials [8–17]. The allylic substituent attached to the triazole ring serves as the key for the specific competitive co-coordination to the soft Cu^+ acid of the unsaturated C=C bond, heterocyclic N atoms, and the anion of hard basicity [18; 19]. That's why similar organic compounds have proven to be constructive ligands for the preparation of unique π-coordination compounds of unusual stereochemistry [20–22].

Previously studied allyl derivatives of 5-phenyl-1,2,4-triazole-3-thiols reveal a strong tendency to form with copper(I) dimeric fragments, which act as the main building blocks in crystal structure organization. In order to diversify the coordination abilities of triazole allyl derivatives, we suggest the functionalization of the heterocyclic core with additional rigid donor group, such as pyridyl to create a multidentate ligand with an additional chelating or bridging sites. In this report, we designed a new allyl-containing ligand, namely 3-allylthio-4-allyl-5-(2-pyridyl)-4*H*-1,2,4-triazole (*Apt*) and based on this, we focused on the synthesis and structural characterization of a novel copper(I) π-compound $[Cu_4(Apt)_2Cl_4]$ (**1**).

Results and their discussion

π-Complex **1** crystallizes in the centrosymmetric space group $P2_1/n$, with one organic molecule and two copper(I) atoms in the asymmetric unit (Fig. 1). Cu(1) adopts a typical for π-bonded copper(I), close to trigonal-pyramidal coordination environment (four-coordinate geometry index τ_4 [23] is 0.87) including in the basal plane C=C bond of the η^2-allylthio group, N1 and N2 atoms of two neighbouring triazole rings (Table 1). The apical position of the Cu(I) polyhedron is occupied by a μ_2-Cl1 atom. The main organometallic part in the complex is represented by the $\{Cu_2(Apt)_2\}^{2+}$ dimers, which contain a six-membered $\{Cu_2N_4\}$ ring comprised of two pairs of triazole fragments and two six-membered rings formed by chelating mode of the *Apt*. Similar organometallic moieties have been previously found as the main building blocks in the construction of copper(I) halide π-complexes with 3-allylthio-4-allyl-5-phenyl- and 3-allylthio-4,5-diphenyl-4*H*-1,2,4-triazoles (Fig. 2a) [6; 7].

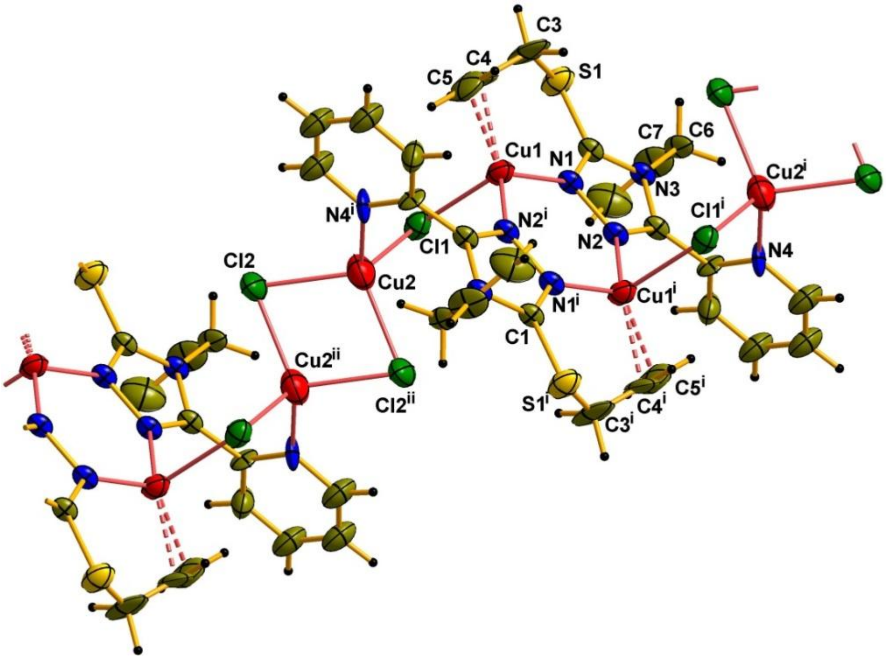


Fig. 1. Fragment of 1 crystal structure. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (*i*) –*x*+1, –*y*, –*z*+1; (*ii*) –*x*+1, –*y*–1, –*z*+1

Table 1

Selected geometric parameters (Å, °) of complex 1

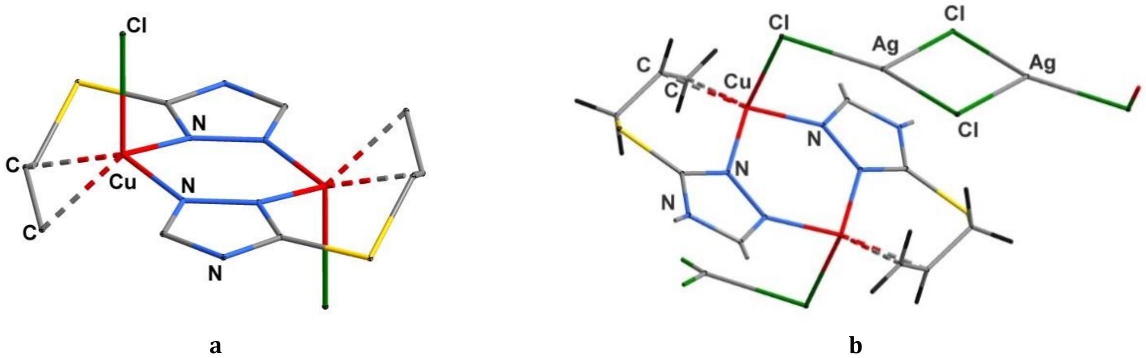
Cu1—Cl1	2.461(3)	Cl1—Cu1—N1	102.2(2)
Cu1—N1	1.939(7)	Cl1—Cu1—*m*	105.3(3)
Cu1—N2^{i} [a]	2.008(6)	N1—Cu1—*m*	110.4(3)
Cu1—*m* [b]	2.020(9)	N2^{ii}—Cu1—*m*	126.4(3)
C4—C5	1.40(1)	N1—Cu1—N2^{i}	110.6(2)
Cu2—Cl1	2.332(3)	Cl1—Cu2—N4^{i}	103.1(2)
Cu2—Cl2	2.363(3)	Cl2—Cu2—N4^{i}	102.9(2)
Cu2—Cl2^{i}	2.421(2)	Cl1—Cu2—Cl2	117.4(1)
Cu2—N4^{i}	2.089(7)	Cu2—Cl2—Cu2^{ii}	73.83(9)

[a] Symmetry codes: (*i*) $-x+1, -y, -z+1$; (*ii*) $-x+1, -y-1, -z+1$;

[b] *m* is the mid-point of C4—C5 bond

Due to back-donation from an occupied 3*d* metal orbital to a low-lying empty π*-orbital of the olefin, the π-connected to the metal center C4=C5 bond in **1** is elongated to 1.40(1) Å in comparison with the uncoordinated allylic C═C bond with a typically observed 1.31÷1.33 Å bond length [7;21]. Allylthio has a synclinal conformation relative to the S1—C3 bond and anticlinal conformation relative to the C3—C4 bond (corresponding torsion C1—S1—C3—C4 and S1—C3—C4—C5 angles are 70.5(10)° and 146.3(8)°). In contrast to the thioallyl substituent, N-allyl group, which is located in **1** almost orthogonally to the triazole ring, remains inactive regarding to copper(I). This group has a synperiplanar conformation relative to its S1—C3 bond (N3—C6—C7—C8 7.4(15)°).

Cu2 environment (τ_4 = 0.91) includes the pyridyl N atom of the *Apt* molecule and three chlorine atoms. These two Cu2 atoms in **1** form centrosymmetric inorganic subunits {ClCuCl$_2$CuCl} through two μ_2-Cl2 atoms. A similar inorganic subunit was previously observed in the structure of heterometallic (Cu^{I} – Ag^{I}) π-complexes with the 3-allylthio-4-allyl-5-phenyl-triazole, in which the second metal position (occupied by Cu2 in **1**) is occupied by Ag(I) with exclusively trigonal coordination (Fig. 2b) [7]. Previously, the analogue coordination polymer based on a homometallic complex could not be obtained, and realization of this in **1** became possible only through additional coordination of the pyridyl substituent to the σ-bonded copper(I) of the inorganic subunit (Fig 3). Comparing theheterometallic (Cu^{I} – Ag^{I}) π-complex and **1** one mey note, that in contrast to the almost flat {ClAgCl$_2$AgCl} subunit due to the trigonal planar environment of Ag(I), homometallic {ClCuCl$_2$CuCl} subunit undergoes distortion, which is stipulated by an increase of the Cu(I) coordination number through pyridyl ring connection (Table 1).



a

b

Fig. 2. (*a*) The main constructive unit in the copper(I) halides π-complexes with the 3-allylthio-4-allyl-5-phenyl-triazole; (*b*) Fragment of heterometallic (Cu^{I} – Ag^{I}) coordination polymer based on the same organic ligand

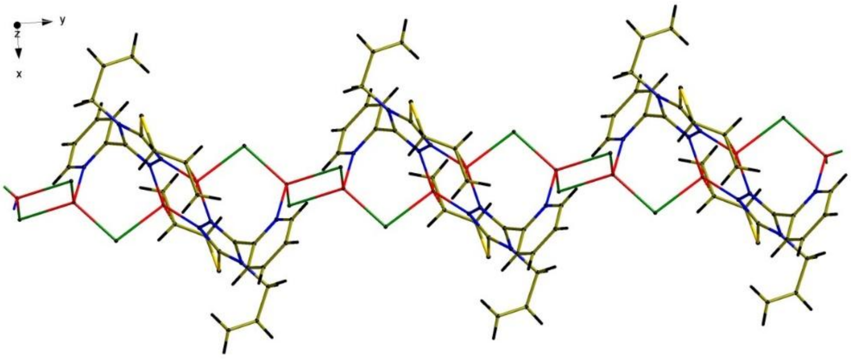


Fig. 2. An infinite chain in crystal structure of 1

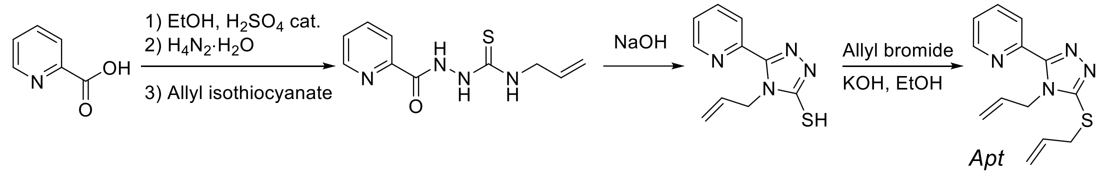
Experimental part

General consideration

All chemicals were obtained from a commercial source (Sigma Aldrich) and used without purification. ^{1}H and ^{13}C NMR spectra were recorded on Varian Unity Plus 400 (400 MHz for ^{1}H) and Bruker 170 Avance 500 (126 MHz for^{13}C) spectrometers in $CDCl_3$ solutions, using TMS as internal references. Mass spectral analyses were performed using an Agilent 1100 series LC/MSD with API-ES/APCI mode (200 eV). IR spectrum for **1** was recorded with Shimadzu FTIR spectrometer IRAffinity. Diffraction data for **1** were collected on an Agilent Xcalibur four-circle diffractometer using Mo K_a radiation (λ = 0.71073 Å).

Preparation of Apt

Ligand *Apt* (2-(4-allyl-5-(allylthio)-4H-1,2,4-triazol-3-yl)pyridine, $C_{13}H_{14}N_4S$) was obtained from 2-pyridinecarboxylic acid, in several steps, similarly to the reported method for 3-phenyl-4-allyl-5-allylsulfanyl-4*H*-1,2,4-triazole [6]:



The overall yield is 67%. IR (cm^{-1}): 3083,47 (w), 2982,21 (w), 2360,38 (w), 1718,59 (w), 1588,80 (w), 1464,72 (m), 1446,18 (w), 1416,23 (m), 1208,00 (w), 1045,41 (w), 982,66 (m), 922,76 (m), 791,55 (m), 774,43 (w), 743,06 (m), 730,22 (w), 701,70 (m), 668,89 (w), 626,11 (w), 616,12 (w), 590,45 (m), 577,62 (m), 549,09 (m), 543,39 (m), 524,85 (w), 510,58 (m); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (br.s, 1H, H_{Py}-6), 8.27 (d, *J* = 7.7 Hz, 1H, H_{Py}-3), 7.80 (t, *J* = 7.2 Hz, 1H, H_{Py}-4), 7.38 – 7.24 (m, 1H, H_{Py}-5), 6.06 – 5.87 (m, 2H, H_{allyl}), 5.33 – 5.21 (m, 3H, H_{allyl}), 5.19 – 5.10 (m, 2H, H_{allyl}), 5.03 (d, *J* = 17.2 Hz, 1H, H_{allyl}), 3.91 (d, *J* = 7.0 Hz, 2H, H_{allyl}); ^{13}C NMR (126 MHz, $CDCl_3$) δ 152.83, 152.61, 148.68, 147.81, 136.91, 132.65, 132.27, 123.95, 123.26, 119.01, 117.92, 47.74, 36.27; LCMS (m/z, ES-API) 259 (M^++1); Calculated for $C_{13}H_{14}N_4S$ C, 60.44; H, 5.46; N, 21.69; Found: C, 60.71; H, 5.34; N, 21.53%.

Preparation of $Cu_4(apt)_2Cl_4]$ (1)

Crystals of the π-complex **1** were obtained under conditions of the alternating-current electrochemical technique [6; 22] starting from an acetonitrile solution of the triazole compound and copper(II) chloride. For this, a solution of *Apt* (1.0 mmol, 0.258 g) and $CuCl_2·2H_2O$ (1.5 mmol, 0.256 g) in 5.0 mL of acetonitrile was prepared. The prepared green solution was placed into a small 5.5 mL test tube, and then copper-wire electrodes in a cork were inserted. By the application of an alternating current (frequency 50 Hz) of 0.80 V for three days, dark yellow crystals of **1** appeared on copper electrodes. The yield is about 25%. M.p. 157-158°C. IR (cm^{-1}): 3054,95 (w), 2983,63 (w), 2893,78 (w), 2360,38 (w), 1718,59 (w), 1404,82 (m), 1380,57 (m), 1362,03 (w), 1347,77 (w), 1242,23 (m), 1209,43 (m), 1193,74 (w), 1149,53 (w), 1091,05 (w), 1049,69 (w), 1024,02 (w), 1002,63 (w), 994,07 (m), 921,33 (m), 909,92 (m), 901,37 (m), 882,83 (m), 790,12 (s), 751,61 (m), 728,79 (m), 714,53 (m), 687,43 (m), 668,89 (w), 661,76 (w), 626,11 (m), 604,71 (m), 569,06 (m), 549,09 (m), 514,86 (m), 492,04 (w), 472,08 (w), 466,37 (w).

Single crystal X-ray diffraction studies

Diffraction data for **1** were collected on a Agilent Xcalibur diffractometer with MoK_a radiation (λ = 0.71073 Å). The structure was solved by ShelXT and refined by the least squares method on F^2 using ShelXL software with the graphical user interface OLEX^2 [24–26]. Atomic displacements for non-hydrogen atoms were refined using an anisotropic model. Hydrogen atoms were placed on geometrically calculated positions and refined as riding atoms with relative isotropic displacement parameters. The crystal parameters, data collection, and refinement are summarized in Table 2.

Table 2

Selected crystal data and structure refinement parameters of 1

Crystal data	
Empirical formula	$C_{26}H_{28}Cl_4Cu_4N_8S_2$
CCDC number	2542912
Formula weight (g·mol^{-1})	912.64
Crystal system	monoclinic
Space group	$P2_1/n$
a (Å)	10.884(3)
b (Å)	10.944(3)
c (Å)	13.860(4)
α (°)	90
β (°)	93.96(3)
γ (°)	90
V (Å^3)	1647.0(8)
Z	2
μ (mm^{-1})	3.032
F(000)	912
Crystal size (mm)	0.06×0.08×0.2
Crystal color and shape	Colourless, block
Calculated density, g/cm^3	1.840
Temperature, K	293
θ range for data collection (º)	2.3–28.7
Measured reflections	15296
Independent reflections	3629
Refined parameters	163
$R[F^2 > 2\sigma(F^2)]$	0.0659
$wR(F^2)$	0.0792
$\Delta\rho_{max}/\Delta\rho_{min}$ (e Å^{-3})	0.736/–0.554

Conclusions

To sum up, we presented the synthesis and structure characterization of a new copper(I) η^2-π-complex $[Cu_4(\mathit{Apt})_2Cl_4]$ (**1**) based on 3-allylthio-4-allyl-5-(2-pyridyl)-4*H*-1,2,4-triazole (*Apt*). *Apt* molecule in **1** acts as chelate-bridging ligand and is coordinated to the copper(I) atoms by means of its triazole N atoms, N atom of pyridyl substituent and allylthio group. The *Apt* molecule does not fully utilize its coordination possibilities, leaving the 4-allyl group inactive toward copper. Instead, the participation of pyridyl N atom in metal binding stabilizes inorganic $\{ClCuCl_2CuCl\}$ subunit isotypic to which was detected only in the heterometallic (Cu^I – Ag^I) π-complexes with the 3-allylthio-4-allyl-5-phenyl-triazole. The possibility of realizing coordination polymers with a ligand provides grounds to expect the realization of stable complexes of ionic copper(I) salts possessing advanced non-linear optical properties.

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