

Polymorphism of Copper(I) Halide π -Complexes: Synthesis and Structure of a New Modification of $[\text{Cu}_2\text{Cl}_2(\text{CH}_2=\text{CH}-\text{CH}_2)_2\text{O}]$

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Received February 8th, 2008.

Abstract. A new polymorph of $[\text{Cu}_2\text{Cl}_2(\text{CH}_2=\text{CH}-\text{CH}_2)_2\text{O}]$ has been obtained by an alternating-current electrochemical synthesis starting from CuCl_2 and triallylphosphite. Diallylether was obtained *in statu nascendi* via transesterification of triallylphosphite. The structure is monoclinic, space group $P2_1/n$, $a = 7.6738(7)$, $b = 6.5128(4)$, $c = 18.2689(18)$ Å, $\beta = 91.644(11)^\circ$, $V = 912.67(13)$ Å³ (at 150 K), $Z = 4$; $R_1 = 0.0339$, $wR_2 = 0.0822$. Flexibility of the

organic ligand and weak interactions copper···halogen have been identified as the main reasons enabling polymorphism in molecular π -complexes of copper(I).

Keywords: Polymorphism; Copper; π -Complexes; Diallylether; Crystal structure

1 Introduction

Polymorphism, as the ability of a solid material to exist in more than one form or crystal structure, still remains poorly understood and controlled. Characterization of polymorphs is mainly focused on organic polymorphism, even though in recent years polymorphism of organometallic compounds received some attention [1 – 3]. However, there are difficulties in obtaining new polymorphs. The formation of a new modification is often unpredictable, but on the other hand, it can be a result of a by-product synthesis. Such an example is described in this paper: attempting to obtain a copper(I) chloride π -complex with triallylphosphite, we encountered a new polymorph of copper(I) chloride π -complex with diallylether. In this paper we report on its synthesis and crystal structure. In addition, we have tried to review similar cases of polymorphism in molecular π -complexes of copper(I) halides and to identify the means of stabilization of different polymorphs in these systems.

2 Experimental Section

Synthesis. 1 mmol (0.20 g) of triallylphosphite, $(\text{CH}_2=\text{CH}-\text{CH}_2-\text{O})_3\text{P}$, was added to a solution of 1.5 mmol (0.26 g) $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ in 5 ml of ethanol. The mixture was placed into a glass reservoir and copper electrodes were mounted via an air-tight rubber cork. Under an applied tension of alternating current (50 Hz, 0.4 V, 0.14 mA) good quality prismatic colourless crystals of the complex $[\text{Cu}_2\text{Cl}_2(\text{CH}_2=\text{CH}-\text{CH}_2)_2\text{O}]$ appeared on the electrodes in 48 hours at 0 °C.

Single-crystal X-ray diffraction. The crystal was mounted on a Stoe IPDS diffractometer equipped with an image plate detector and Mo X-ray tube. 180° ω oscillation images with an increment of 1° were collected at 150 K with 10 min exposure, crystal-to-detector distance 60 mm. After integration [4] the data were corrected for Lorentz factor and polarization effects, and numerically for absorption (12 faces for a plate-like crystal, $\mu = 5.2 \text{ mm}^{-1}$, $T_{\text{min,max}} = 0.606, 0.883$). Cell parameters were obtained by a least-squares refinement based on reflection angles in the range $5.8 < 2\theta < 55.9^\circ$. No significant variation of average intensity during the measurement was observed. Systematic absences suggested space group $P2_1/n$ (No. 14). The structure was solved by direct methods [5] with subsequent difference Fourier syntheses. Hydrogen atoms were placed in geometrically calculated positions and refined in a riding model with isotropic temperature factors equal to 1.2U of the neighbouring atom. A full least-squares refinement on F^2 was performed on positional and thermal parameters for all atoms (anisotropically for all non-H atoms) by using SHELXL-97 [5]. Reduced cell calculations and a non-crystallographic symmetry check (PLATON2007) [6] did not indicate higher lattice symmetry or missed symmetry elements. No warnings for twinning or superstructure were observed. Crystal data and details of data collection and structure refinement are given in Table 1. Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication CCDC 675120. Copy of the data can be obtained free of charge on application to CCDC,

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Table 1 Crystal data and details of data collection and structure refinement for the new polymorph of $[\text{Cu}_2\text{Cl}_2(\text{CH}_2=\text{CH}-\text{CH}_2)_2\text{O}]$.

Empirical formula	$\text{C}_6\text{H}_{10}\text{Br}_2\text{Cu}_2\text{O}$
Formula weight	296.12
Space group	$P2_1/n$
$a/\text{\AA}$	7.6738(7)
$b/\text{\AA}$	6.5128(4)
$c/\text{\AA}$	18.2689(18)
$\beta/^\circ$	91.644(11)
$V/\text{\AA}^3$	912.67(13)
$Z, D_c/\text{g}\cdot\text{cm}^{-3}$	4, 2.155
$F(000)$	584
$2\theta_{\text{max}}/^\circ$	55.8
Refl. collected/unique	7414/2166
Data/restraints/parameters	2166/0/100
R_{int}, R_σ	0.0565, 0.0453
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0339, wR_2 = 0.0761$
R indices (all data)	$R_1 = 0.0533, wR_2 = 0.0822$
Goodness-of-fit on F^2	0.925
$(\Delta/\sigma)_{\text{max}}$	0.000
Largest diff. peak and hole $/e\cdot\text{\AA}^{-3}$	0.80(16) and $-0.48(16)$

12 Union Road, Cambridge CB2 1EZ, UK (Fax, +44-(1223)336-033; E-mail, deposit@ccdc.cam.ac.uk). A list of structure factors can be obtained from the authors.

3 Results and Discussions

An electrochemical synthesis, starting from the $\text{Cu}^{2+}/\text{Cu}^0$ redox pair and triallylphosphite, was originally aimed at obtaining a complex of copper(I) chloride with $(\text{CH}_2=\text{CH}-\text{CH}_2-\text{O})_3\text{P}$. However, we have isolated a complex of copper(I) chloride with diallylether (DAE). Formation of $(\text{CH}_2=\text{CH}-\text{CH}_2)_2\text{O}$ can be explained by a transesterification of triallylphosphite by ethanol, followed by a CuCl-catalyzed etherification [7, 8] of the resulting allyl alcohol into DAE. This rather common transformation of organic molecules has an interesting outcome for coordination chemistry: reaction of CuCl with DAE *in statu nascendi* leads to a new polymorphic modification of $2\text{CuCl}\cdot\text{DAE}$, denoted **B** below. The first known polymorph of $2\text{CuCl}\cdot\text{DAE}$ [9], denoted here as **A**, was obtained by a direct interaction of DAE with CuCl, and also by the electrochemical method. A similar polymorphism has been found in $3\text{CuBr}\cdot\text{DAAC}$ (DAAC = diallylacetylene), where one polymorph was obtained by an alternating-current electrochemical synthesis from CuBr_2/Cu and DAAC [10], and the other by direct interaction of CuBr with DAAC [11]. Therefore, it is likely that for one of the polymorphs the limiting stage of formation is the dissolution of the inorganic salt, while for the other it is a generation of one of the reaction components (copper(I) halide or organic ligand). The kinetic factors are directly linked to the $[\text{CuCl}]/[\text{ligand}]$ concentration ratio, which not only gives rise to the polymorphism of the coordination compounds, but can also determine their stoichiometry. For example, *in statu nascendi* formation of diallylamine (DAA) during its complexation with copper(I) halides results in metal-rich compositions $3\text{CuX}\cdot\text{DAA}$ ($\text{X} = \text{Cl}, \text{Br}$) [12], while the direct use of DAA yields the $2\text{CuX}\cdot\text{DAA}$ compositions [13]. It is remarkable

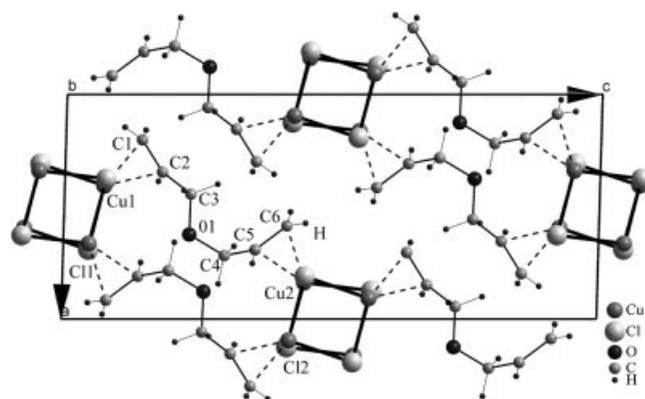


Fig. 1 Crystal structure of the new polymorph of $[\text{Cu}_2\text{Cl}_2(\text{CH}_2=\text{CH}-\text{CH}_2)_2\text{O}]$, as seen in the bc projection. Cu–Cl bonds are shown as thick solid lines, Cu–(C=C) π -interaction is highlighted by dashed lines.

Table 2 Selected bond distances \AA and angles $^\circ$ in the **B** polymorph of $[\text{Cu}_2\text{Cl}_2(\text{CH}_2=\text{CH}-\text{CH}_2)_2\text{O}]$.

Cu1–Cl1	2.2890(9)	Cl1–Cu1–Cl1'	95.57(3)
Cu1–Cl1'	2.3159(9)	Cl1–Cu1–Cl2	104.24(3)
Cu1–Cl2	3.1506(9)	Cl1'–Cu1–Cl2	83.86(3)
Cu1–C1	2.037(3)	Cl1–Cu1–m12	133.70(9)
Cu1–C2	2.072(3)	Cl1'–Cu1–m12	129.72(9)
Cu1–m12 ¹	1.939(3)	Cl2–Cu1–m12	91.37(9)
		C1–Cu1–C2	38.6(1)
Cu2–Cl2	2.2925(8)	Cl2–Cu2–Cl2'	98.25(3)
Cu2–Cl2'	2.3091(8)	Cl2–Cu2–Cl1	96.52(3)
Cu2–Cl1	2.9135(9)	Cl2'–Cu2–Cl1	89.61(3)
Cu2–C6	2.048(3)	Cl2–Cu2–m56	126.62(9)
Cu2–C5	2.070(3)	Cl2'–Cu2–m56	132.29(9)
Cu2–m56 ¹	1.942(3)	Cl1–Cu2–m56	99.29(9)
		C5–Cu2–C6	38.8(1)
C1–C2	1.357(5)	C1–C2–C3	123.6(3)
C2–C3	1.506(4)	C2–C3–O1	108.9(2)
C3–O1	1.421(4)	C3–O1–C4	113.4(2)
C4–O1	1.436(4)	O1–C4–C5	110.2(3)
C4–C5	1.505(4)	C4–C5–C6	123.8(3)
C5–C6	1.369(4)		

¹ m12, m56 are the middles of the C1=C2 and C5=C6 bonds.

that in the electrochemical synthesis of copper(I) chloride π -complexes with allylcyanide (AC), the use of different voltages of the alternating current permitted to obtain two different polymorphs of $2\text{CuCl}\cdot\text{AC}$ [14, 15]. The same effect was observed for copper(I) chloride π -complexes with DAE, where the use of different voltages allowed to obtain compounds with different compositions: **A**-polymorph of $2\text{CuCl}\cdot\text{DAA}$ [9] and $5\text{CuCl}\cdot 2\text{DAA}$ [16]. Thus, the electrochemical synthesis using the $\text{Cu}^{2+}/\text{Cu}^0$ redox pair and ligand reveals an impressive flexibility with respect to the kinetical control of the formation of intermediate products and therefore stabilization of different polymorphs of the resulting copper(I) complexes.

As can be seen from Figure 1, the structure of the polymorph **B** contains two crystallographically independent centrosymmetric units Cu_2Cl_2 . These dimers are linked via the π, π -bridging molecule of DAE into an infinite chain running along $[10\bar{1}]$. The inorganic dimers are also weakly

associated along [010] into a zigzag $(\text{Cu}_2\text{Cl}_2)_n$ tape by means of elongated contacts $\text{Cu}2\cdots\text{Cl}1$ 2.9135(9) Å and $\text{Cu}1\cdots\text{Cl}2$ 3.1506(9) Å. Thus, the structure is made of layers situated in the (101) plane, which are associated by Van der Waals interaction. Both copper atoms have an almost flat trigonal environment completed by the apical chlorine ligand to a trigonal pyramid, see Table 2. The π -coordinated double bond is aligned in the plane of equatorial ligands. The $\text{C}1-\text{Cu}1-\text{C}2$ and $\text{C}5-\text{Cu}2-\text{C}6$ angles, $38.5(1)^\circ$ and $38.8(1)^\circ$, respectively, are bigger than in most molecular copper(I) halide π -complexes [17]. This suggests that the $\text{Cu}-(\text{C}=\text{C})$ interaction is rather strong in this complex.

The inorganic fragment in the polymorph **A** is quite different. It is made of the corrugated $(-\text{Cu}-\text{Cl}-)_n$ chains, associated into two-dimensional layers via elongated $\text{Cu}\cdots\text{Cl}$ contacts [9]. The ability of copper(I) chloride to form different inorganic fragments and the flexibility of the DAE molecule, which allows to link them into the dense structures, are the evident reasons of the polymorphism of $2\text{CuCl}\cdot\text{DAE}$. Similar structures are formed by CuCl with other flexible organic molecules. For example, a similar combination of centrosymmetric dimers Cu_2Cl_2 as in the polymorph **B** was observed in the copper(I) chloride π -complex with 1,5-hexadiene (HD), $2\text{CuCl}\cdot\text{HD}$ [9]. In this structure the dimers are associated more closely ($\text{Cu}\cdots\text{Cl}$ 2.765(3) Å), and therefore the $(\text{Cu}_2\text{Cl}_2)_n$ zigzag tape is slightly less corrugated than in **B** $2\text{CuCl}\cdot\text{DAE}$. The conformation of the allyl groups in HD molecules ($\text{C}=\text{C}-\text{C}-\text{C}$ torsion angles $144(1)^\circ$ and $97(1)^\circ$) is close to the one observed for DAE ($\text{C}=\text{C}-\text{C}-\text{O}$ $146.6(3)^\circ$ and $104.7(3)^\circ$). Despite a strong similarity between the two structures, they can not be considered homologous. While in the **B** polymorph of $2\text{CuCl}\cdot\text{DAE}$ each $(\text{Cu}_2\text{Cl}_2)_n$ zigzag tape is connected via the organic molecules with two other tapes into layers, in $2\text{CuCl}\cdot\text{HD}$ they are linked with four other tapes into a three-dimensional framework.

A big spread of torsion angles observed for DAE molecule in five structurally characterized π -complexes (Figure 2) shows that it adjusts to the symmetry and composition of an inorganic fragment. Moreover, it can act both as a π,π -bridging and a π,π -chelating ligand. In all four complexes with Cu^I this molecule is bridging, and in the complex with Pd^0 [18] it forms a six-membered chelate ring. While in the two $2\text{CuCl}\cdot\text{DAE}$ complexes the organic molecule is totally asymmetric (see Figures 2a and 2b), in $5\text{CuCl}\cdot 2\text{DAE}$ [16] it takes the symmetry of the two-fold axis (Figure 2c) and in $2\text{Cu}(\text{O}_3\text{SCH}_3)\cdot\text{DAE}$ [19] and $[\text{Me}_3\text{P}\cdot\text{Pd}\cdot\text{DAE}]$ [18] it takes a pseudo-symmetry of the mirror plane (see Figures 2d and 2e). The conformational flexibility of DAE in the complexes listed above suggests that it is the main reason which favours polymorphism in $2\text{CuCl}\cdot\text{DAE}$.

Polymorphic modifications of ionic copper(I) complexes can be stabilized via different mechanisms, such as an order-disorder of weakly coordinated anions and redistribution of strong hydrogen bonds $(\text{N})\text{H}\cdots\text{O}$ [20]. Such polymorphism, involving only small atomic displacements, re-

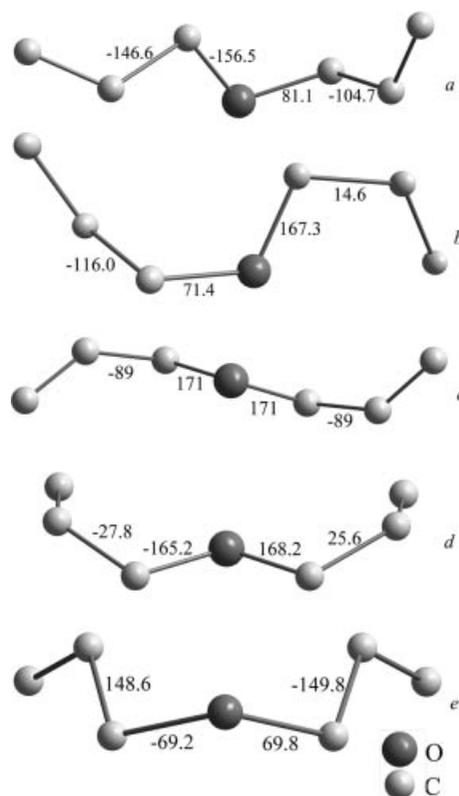


Fig. 2 Torsion angles in DAE molecule in crystal structures of the **B** polymorph of $2\text{CuCl}\cdot\text{DAE}$ (a), the **A** polymorph of $2\text{CuCl}\cdot\text{DAE}$ [9] (b), $5\text{CuCl}\cdot 2\text{DAE}$ [16] (c), $2\text{Cu}(\text{O}_3\text{SCH}_3)\cdot\text{DAE}$ [19] (d) and $[\text{Me}_3\text{P}\cdot\text{Pd}\cdot\text{DAE}]$ [18] (e).

distribution of weak interactions and a symmetry change, is demonstrated by Cu^I nitrate π -complex with diallylamine, $\text{CuNO}_3\cdot\text{DAA}$ [21]. One or the other polymorph in this system can be stabilized by merely using different solvents under otherwise identical conditions of electrochemical synthesis. While it is known that the weak interactions, such as $\text{Cu}\cdots$ apical ligand and hydrogen bonds, play the major role in polymorphism of ionic copper(I) π -complexes (see the review [20]), in molecular complexes the conformational flexibility of organic ligand is instrumental in matching different copper(I) halide fragments, and therefore favouring polymorphism.

Unlike strong $(\text{N})\text{H}\cdots\text{O}$ bonds in ionic complexes, much weaker hydrogen bonds $(\text{C})\text{H}\cdots\text{O}$ can not promote polymorphism in molecular complexes. The other type of weak interactions, the elongated contacts from copper atoms to the apical ligands, may still play an important role in their polymorphism. In order to verify that, we have analyzed structural parameters of coordination polyhedra for copper atoms in four known polymorphic pairs of molecular π -complexes of copper(I) halides. Selected characteristics for these complexes are listed in Table 3. Those polymorphs which according to our experimental observations are the more stable ones are denoted as **A**, and the less stable ones – as **B**. In all eight structures, copper atoms have a trigonal pyramidal coordination with double $\text{C}=\text{C}$ bond and two

Table 3 Selected characteristics for polymorphic modification of molecular π -complexes of copper(I) halides. DAE stands for diallylether, HDOL – 1,5-hexadiene-3-ol, AC – allylcyanide, DAAC – diallylacetylene.

	2CuCl·DAE		2CuCl·HDOL		2CuCl·AC		3CuBr·DAAC	
Polymorph	A [9]	B (this work)	A [24]	B [24]	A [14]	B [15]	A [10]	B [11]
Space group, Z	$Bb, 4$	$P2_1/n, 4$	$P2_1/n, 4$	$P2_1/b, 4$	$P2_1/b, 4$	$Pcab, 8$	$P2_1/a, 4$	$Pca2_1, 4$
Cell parameters /Å, °	$a=19.795(7)$ $b=6.358(3)$ $c=7.635(4)$ $\gamma=108.73(9)$	$a=7.6738(7)$ $b=6.5128(4)$ $c=18.2689(18)$ $\beta=91.644(11)$	$a=8.951(3)$ $b=7.288(2)$ $c=14.423(4)$ $\gamma=97.77(2)$	$a=7.657(2)$ $b=7.362(2)$ $c=16.771(5)$ $\gamma=93.96(3)$	$a=8.358(3)$ $b=7.457(2)$ $c=11.851(3)$ $\gamma=98.77(3)$	$a=15.365(3)$ $b=13.283(3)$ $c=7.056(1)$	$a=15.197(6)$ $b=11.784(5)$ $c=6.893(5)$ $\gamma=108.32(2)$	$a=20.269(6)$ $b=8.582(3)$ $c=6.823(3)$
Density /g·cm ⁻³	2.161	2.155	2.110	2.086	2.412	2.445	3.041	2.950
Cu–X/Å	2.243–2.294(4)	2.2890–2.3159(9)	2.274–2.426(1)	2.261–2.315(3)	2.260(4), 2.411(4)	2.252(1), 2.273(1)	2.349–2.462(6)	2.370–2.430(6)
Cu···X(apical)/Å	2.890(3), 3.021(3)	2.913(1), 3.151(1)	2.888(1)	3.325(3)	2.791(4)	3.174(2)	2.895(6), 3.028(7)	3.050(7), 3.363(7)

halide atoms in the equatorial plane and one halide atom in the apical position. The comparison reveals that the more stable polymorphs have shorter Cu···X(apical) distances. Moreover, in three of the four pairs, the shortening of Cu···X(apical) is accompanied by a decrease of the unit cell volume, and consequently by an increase of the calculated density. The latter correlates with higher thermodynamic stability of these phases. As it concerns small energy changes, the structural differences can not be seen for the strong covalent bonds, but only for weak contacts. It resembles the polymorphism in hydrogen bonded frameworks which, along with conformational polymorphism, covers most cases of this phenomenon for majority of organic and for some coordination compounds [22, 23]. Thus, the diversity of copper(I) halide frameworks along with the flexibility of metal atom's coordination sphere and of the organic ligands are the main factors that enable polymorphism in molecular π -complexes.

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