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Synthesis and crystal structure of N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium (*dalltmen*$^{2+}$) compounds: {*dalltmen*}$(ClO_4)_2$ and the copper(I) nitrate π-complex [{*dalltmen*}$_{0.5}$$Cu(NO_3)_2$]

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N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium dichloride ({*dalltmen*}Cl_2) was synthesized from N,N,N',N'-tetramethylethylenediamine (obtained by the Eschweiler-Clarke reaction of ethylenediamine, formaldehyde and formic acid) and allyl chloride. Then an ethanol solution of KOH was added to replace the Cl^- anions by OH^- ones and N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium dihydroxide ({*dalltmen*}$(OH)_2$) was obtained. Single crystals of {*dalltmen*}$(ClO_4)_2$ (I) and the π-complex [{*dalltmen*}$_{0.5}$$Cu(NO_3)_2$] (II) were obtained from alcohol solutions containing the hydroxide of the tetramethylethylenediamine diallyl derivative, previously titrated by $HClO_4$ (or HNO_3), and $Cu(ClO_4)_2{\cdot}6H_2O$ (or $Cu(NO_3)_2{\cdot}3H_2O$) by *ac*-electrochemical synthesis on copper wire electrodes. The structures of compounds I and II were determined by X-ray diffraction. Crystals I are orthorhombic: space group *Pbca*, $Z = 4$, $a = 11.4125(6)$, $b = 10.4604(5)$, $c = 15.2884(8)$ Å, $V = 1825.1(2)$ Å^3. The structure consists of N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium cations and ClO_4^- anions, which are held together by electrostatic interactions and hydrogen bonds. Crystals II are monoclinic: space group $P2_1/c$, $Z = 4$, $a = 7.9620(4)$, $b = 8.2823(4)$, $c = 15.6224(8)$ Å, $\beta = 96.793(2)°$, $V = 1022.97(9)$ Å^3. The structure contains infinite anionic chains $(Cu(NO_3)_2^-)_n$ with only one independent metal atom. The C=C bonds of the centrosymmetric N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium cation are π-coordinated by Cu(I) atoms from parallel inorganic fragments, so that the *dalltmen*$^{2+}$ moities play a bridging role, connecting separate $(Cu(NO_3)_2^-)_n$ polymeric chains into layers (coordination nets). A branched system of hydrogen bonds connects the nets into a framework.

N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium cation / Anionic chains / Copper(I) nitrate π-complex / Hydrogen bonds

Introduction

Organic ligands playing a bridging role are useful in the synthesis of metal compounds with polymeric one-, two- or three-dimensional coordination networks [1]. N-allyl derivatives of ethylenediamine are convenient "materials" for such purposes: variation of the number of allyl groups is the key to obtain a series of polydentatic π-ligands. The similarity of the latter gives an opportunity for structural comparison and observation of crystal engineering regularities. Earlier structural investigations of copper(I) halide π-complexes with the derivatives [2,3], including N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium [4,5], revealed structural diversity on one side and similarities on the other. To continue the research, we undertook the synthesis of new copper(I) ionic compounds; the results of this investigation are presented below.

Experimental section

Synthesis of N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium dihydroxide ({dalltmen}$(OH)_2$, $[C_2H_4N_2(CH_3)_4(C_3H_5)_2](OH)_2$)

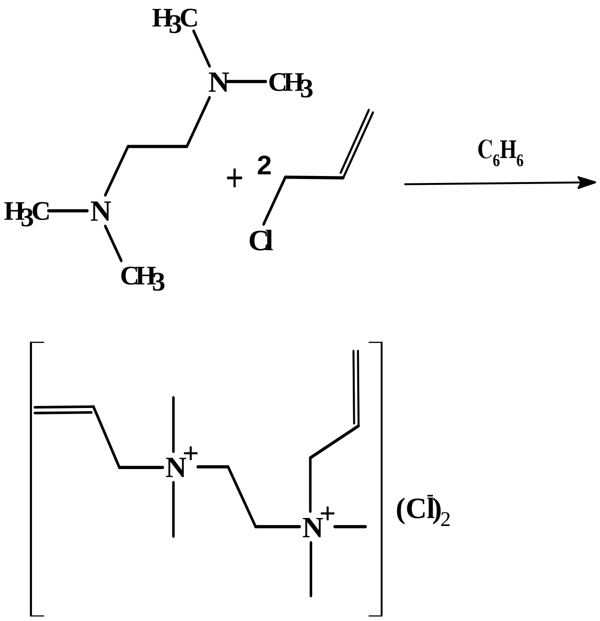
The synthesis of N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium dihydroxide was performed in several steps.

Preparation of N,N,N',N'-tetramethylethylenediamine was carried out by reaction of ethylenediamine with formaldehyde and formic acid according to the Eschweiler-Clarke procedure (by the general method of obtaining tertiary amines from primary or secondary ones) [6].

$$H_2NC_2H_4NH_2 + 4CH_2O + 4HCOOH \xrightarrow{t^\circ} (CH_3)_2NC_2H_4N(CH_3)_2 + 4CO_2 + 4H_2O.$$

Formaldehyde (35% solution, 38 ml) and 90% formic acid (52 ml) were added to ethylenediamine (6.65 ml, 6.0 g). The reaction mixture was refluxed in a water bath. When the vigorous evolution of CO_2 had finished, the heating was continued for six hours (the total time of the synthesis was ~11 h). Then an excess of 36% HCl (22 ml) was added; unreacted formaldehyde and formic acid were removed by distillation. N,N,N',N'-tetramethylethylenediamine was liberated from its hydrochloride by adding an excess of concentrated aqueous KOH to the residue and extracting with benzene. The solvent and other volatile impurities were removed to give a virtually pure product with a yield of ~80%, which then was distilled under reduced pressure (40 mmHg/80°C).

N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium dichloride was synthesized in the following way: allyl chloride (17 g, 18 ml, 0.22 mol, small excess) was added dropwise to 11.6 g (0.1 mol) of N,N,N',N'-tetramethylethylenediamine in benzene.



The mixture was refluxed in a water bath for 20 h. The product, in the form of a white precipitate, was filtered off. The yield was ~40%.

Potassium hydroxide (in C_2H_5OH) was added to an alcohol solution of N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium dichloride:

$$[C_2H_4N_2(CH_3)_4(C_3H_5)_2]Cl_2 + 2KOH \xrightarrow{ethanol} [C_2H_4N_2(CH_3)_4(C_3H_5)_2](OH)_2 + 2KCl\downarrow + 2H_2O.$$

KCl was filtered off and the ethanol solution of the resulting N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium dihydroxide was used in further syntheses.

Preparation of {*dalltmen*}$(ClO_4)_2$ (I)

Colourless crystals of compound **I** were obtained on the copper wires of a reactor containing an ethanol solution of 0.25 g (~0.001 mol) of $[C_2H_4N_2(CH_3)_4(C_3H_5)_2](OH)_2$, previously titrated by 52% $HClO_4$ to pH ~ 4, in presence of 0.40 g (~0.001 mol) of $Cu(ClO_4)_2\cdot 6H_2O$. The crystals were formed after ten days (last three days in a refrigerator at –1°C).

Preparation of [{*dalltmen*}$_{0.5}Cu(NO_3)_2$] (II)

Colourless crystals of complex **II** were obtained by the *ac*-electrochemical technique [7] on copper wire electrodes from an alcohol solution of 0.5 g (~0.002 mol) of $Cu(NO_3)_2\cdot 3H_2O$ and 0.25 g (~0.001 mol) of $[C_2H_4N_2(CH_3)_4(C_3H_5)_2](OH)_2$, previously titrated by 56% HNO_3 to pH = 3.5-4, after five days.

X-ray crystal structure determination

Single crystals of **I** and **II**, previously studied by the photographic method, were investigated using a Rigaku AFC7R single crystal diffractometer (CCD detector, Mo *K*α radiation, graphite monochromator and ω-scanning) equipped with a low-temperature device. The intensities were corrected for Lorentz and polarization factors. The diffraction data were processed with the Rigaku CrystalClear software [8]. The structures were solved by direct methods; the light atoms were found by Fourier difference syntheses. An analytical absorption correction was applied [9]. The structures were solved using SIR-92 [10] and refined with SHELX [11]. All the hydrogen atoms in the compounds were located based on geometrical considerations.

Selected crystallographic parameters and a summary of the data collections for compounds **I** and **II** are given in Table 1. Atomic positional and displacement parameters for **I** and **II** are given in Tables 2 and 3.

The figures were prepared using DIAMOND v3.1 software [12].

Table 1 Crystallographic characteristics and summary of the structure determinations for **I** and **II**.

Parameter	**I**	**II**
Empirical formula	$C_{12}H_{26}N_2\cdot 2(ClO_4)$	$C_6H_{13}N_3O_6Cu$
Formula weight	397.25	286.73
Temperature, K	200	200
Wavelength, Å	0.71069 (Mo *K*α)	0.71069 (Mo *K*α)
Crystal system, space group	Orthorhombic, *Pbca*	Monoclinic, $P2_1/c$
Unit cell dimensions		
a, Å	11.4125(6)	7.9620(4)
b, Å	10.4604(5)	8.2823(4)
c, Å	15.2884(8)	15.6224(8)
β, °	–	96.793(2)
Volume, $Å^3$	1825.12(16)	1022.97(9)
Z	4	4
Calculated density	1.446	1.862
Absorption coefficient, mm^{-1}	0.40	2.16
F(000)	840	588
Crystal size, mm	0.12 × 0.1 × 0.1	0.28 × 0.2 × 0.08
Shape, colour	chunk, colorless	plate, colorless
θ range for data collection, deg	from 3.0 to 29.0	from 2.6 to 29.1
Limiting indices	$-14 < h < 15$ $-8 < k < 14$ $-12 < l < 20$	$-10 < h < 6$ $-10 < k < 11$ $-12 < l < 20$
Refinement method	full-matrix least-squares on F^2	full-matrix least-squares on F^2
Measured reflections	7225	4240
Independent reflections	2241	2303
Reflections with $F > 4\sigma(F)$	1836	1876
Free parameters	110	146
Weighting scheme*	$1/[\sigma^2(F_o^2) + (0.1702P)^2 + 1.1071P]$	$1/[\sigma^2(F_o^2) + (0.088P)^2 + 2.2316P]$
Goodness-of-fit on F^2	1.12	1.11
Final R indices [$I > 2\sigma(I)$]	R = 0.088, wR = 0.26	R = 0.059, wR = 0.16
R indices (all data)	R = 0.099, wR = 0.28	R = 0.070, wR = 0.17
Largest peaks and holes, e $Å^{-3}$	1.29, −0.83	1.02, −0.90

* $P = (F_o^2 + 2F_c^2)/3$

Results and discussion

The structure of the organic salt N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium perchlorate (**I**) consists of centrosymmetric *dalltmen*$^{2+}$ cations and ClO_4^- anions (Fig. 1). The length of the C=C bond is equal to 1.302(4) Å, the angle C(2)–C(3)=C(4) is equal to 122.9(2)° (Table 4). The electrostatic interaction between the ions is supplemented by H-bonds. Hydrogen atoms of the methyl and allyl groups form (C)H…O contacts (2.48-2.70 Å), which fix the organic moieties in the crystal and prevent disorder.

The structure of **II** contains one independent copper(I) atom with a trigonal-pyramidal coordination environment formed by the C=C-bond of an allyl group of centrosymmetric *dalltmen*$^{2+}$, two oxygen atoms of bridging NO_3^- anions and one oxygen atom from a monodentate nitrate anion (Fig. 2). The apical position is occupied by an O atom. The π-interaction Cu–(C=C) is characterized by notable strength. The displacement of the copper(I) atom from the equatorial plane is only ~0.08 Å. The length of the coordinated C=C bond is 1.386(5) Å; the distance from the metal atom to the middle of the double bond is 1.893(2) Å; the contact Cu–O(3) is 2.549(3) Å, *i.e.* relatively long compared to the other two Cu–O distances (1.975(2) and 2.029(2) Å) (Table 4). A comparison of the interatomic distances shows an elongation for the N–O bonds with O atoms that are coordinated by copper(I) (1.280(3) Å for N(2)–O(1) and 1.249(4) Å for N(2)–O(3), in contrast with 1.229(3) Å for N(2)–O(2)). Weakening of the bond N(3)–O(5) is caused by the formation of the hydrogen contact C(2)H(21)...O(5).

Helical infinite $(Cu(NO_3)_2)_n^{n-}$ chains are formed in the structure of **II**. The nitrate anion in this compound plays a bridging role, similarly to the halide anions in the structures of the previously studied complexes of copper(I) with N-allyl derivatives of ethylenediamine [2,4]. A polymeric $(Cu(NO_3)_2)_n^{n-}$ chain is formed by assembling Cu-centered trigonal pyramids through

O atoms of NO_3^- anions. The copper(I) atoms are separated from each other by 4.50 Å. Formation of polymeric cuprous nitrate fragments in Cu(I) π-complexes is rare. In the majority of known Cu(I) π-compounds, the NO_3^- anion is located in an outer coordination sphere [13], or the structures contain islet copper(I) nitrate units [14,15]. The crystal structures of only a few copper(I) π-complexes with infinite cuprous nitrate fragments are described in the literature. One of them, the compound $[Cu(C_3H_5CN)(NO_3)]$, has a zigzag-shaped chain $(Cu(NO_3)_2)_n$ [16]. In the structure of another π-complex, $[\{(C_3H_5)NHC(NH_2)_2\}Cu(NO_3)_2]$, a similar helical anion $(Cu(NO_3)_2)_n^{n-}$ is formed, but it has more twisted shape [17]. The *dalltmen*$^{2+}$ cations in **II** display a bridging role, connecting separate anionic chains into two-dimensional layers (Fig. 3). Such coordination behavior is typical for this organic ligand [4,5] and is similar to the behavior of other N-allyl derivatives of ethylenediamine in the structures of cuprous halide π-complexes.

As it is usually for coordination compounds, weak interactions are significant for the structure organization [18]. As mentioned above, in **I** they play a role in the structure ordering, making the interaction between *dalltmen*$^{2+}$ cations and ClO_4^- anions more directional. In the structure of **II** hydrogen bonds connect the coordination nets $\{Cu(NO_3)_2$–(*dalltmen*)–$Cu(NO_3)_2\}_m$ into a framework (Table 5). In the case of branched ligands, such as N,N'-diallyl-N,N,N',N'-tetramethylethylenediammonium, the role of the hydrogen bonds, which fix the distinct parts of the structure, increases significantly.

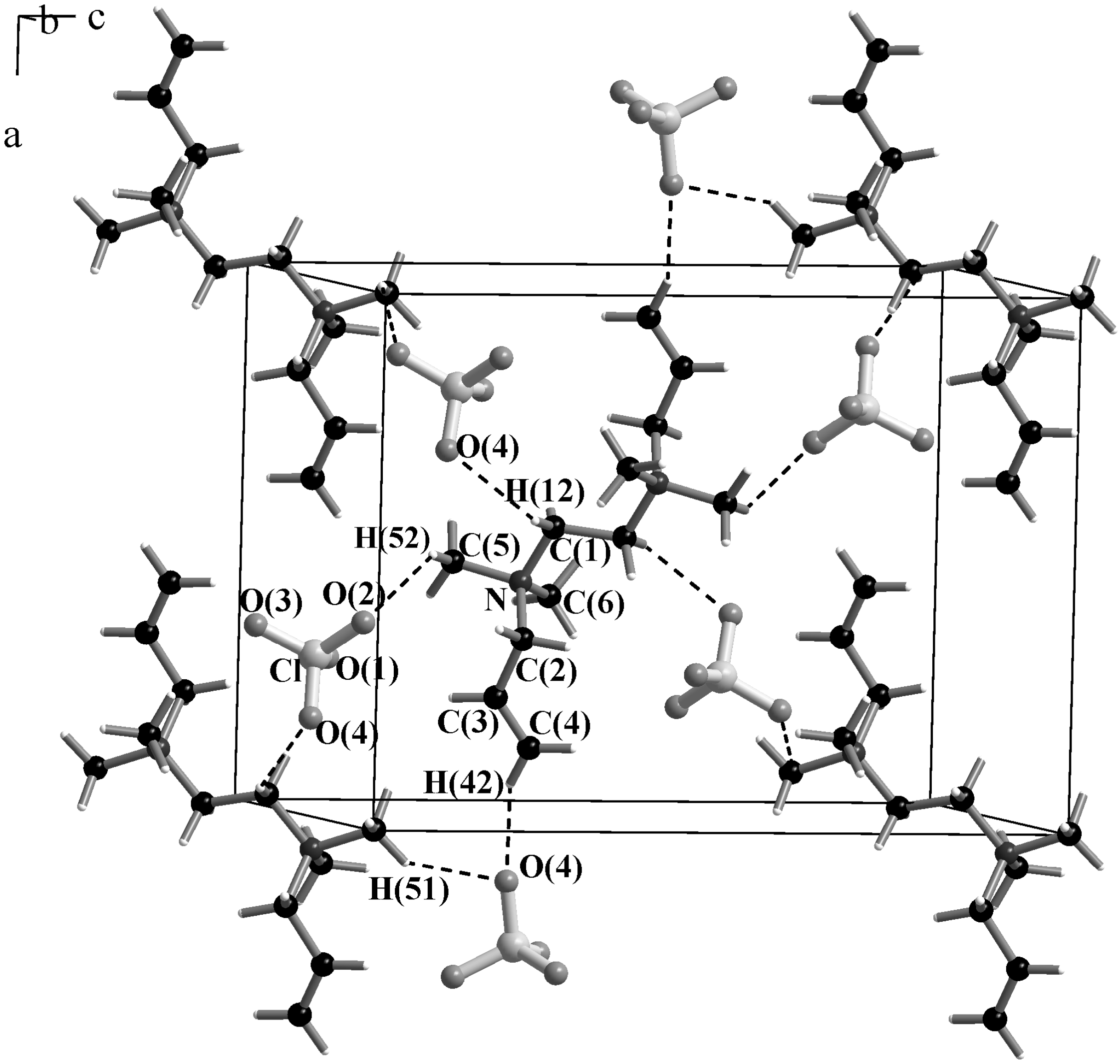


Fig. 1 Fragment of the crystal structure of **I**.

Table 2 Fractional atomic coordinates and isotropic/equivalent isotropic displacement parameters (Å^2) for **I**.

Atom	*x*	*y*	*z*	U_{iso}/U_{eq}[a], Å^2
Cl(1)	0.75531(4)	0.21339(5)	0.65202(3)	0.04086(16)
O(1)	0.76023(17)	0.0777(2)	0.64324(13)	0.0588(5)
O(2)	0.8154(2)	0.2544(2)	0.72945(13)	0.0748(6)
O(3)	0.8086(2)	0.2705(2)	0.57815(14)	0.0782(7)
O(4)	0.63712(18)	0.2516(2)	0.65621(18)	0.0898(8)
N(1)	0.40957(14)	−0.03727(15)	0.60420(10)	0.0352(4)
C(1)	0.50848(17)	0.0133(2)	0.54831(13)	0.0402(5)
H(11)	0.581	−0.025	0.567	0.048
H(12)	0.515	0.104	0.557	0.048
C(2)	0.29862(19)	0.0371(2)	0.58438(16)	0.0488(6)
H(21)	0.311	0.125	0.600	0.059
H(22)	0.284	0.033	0.521	0.059
C(3)	0.19224(19)	−0.0098(3)	0.63053(17)	0.0516(6)
H(31)	0.190	−0.007	0.691	0.062
C(4)	0.1012(2)	−0.0551(3)	0.58961(19)	0.0690(8)
H(41)	0.100	−0.059	0.528	0.083
H(42)	0.036	−0.083	0.621	0.083
C(5)	0.4453(2)	−0.0162(2)	0.69791(13)	0.0471(5)
H(51)	0.387	−0.052	0.736	0.071
H(52)	0.452	0.073	0.709	0.071
H(53)	0.519	−0.056	0.708	0.071
C(6)	0.3915(2)	−0.1775(2)	0.59060(15)	0.0449(5)
H(61)	0.384	−0.219	0.646	0.067
H(62)	0.457	−0.212	0.559	0.067
H(63)	0.321	−0.191	0.557	0.067

[a] For non-hydrogen atoms U_{eq} is defined as one-third of the trace of the orthogonalized U_{ij} tensor $U_{eq} = {}^{1}\!/_{3}\sum_i\sum_j U_{ij} a_i^* a_j^* (\vec{a}_i \vec{a}_j)$, for hydrogen – $U_{iso.}$

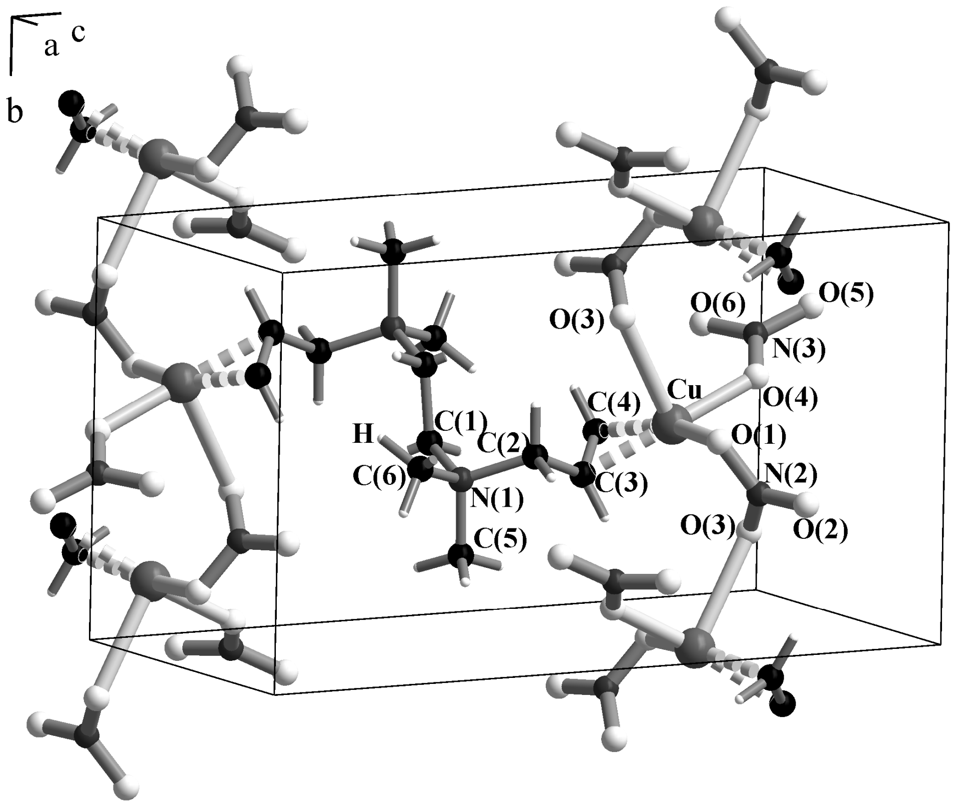


Fig. 2 Coordination behavior of the N,N′-diallyl-N,N,N′,N′-tetramethylethylenediammonium cation in **II.**

Table 3 Fractional atomic coordinates and isotropic/equivalent isotropic displacement parameters (Å^2) for **II**.

Atom	*x*	*y*	*z*	$U_{iso}/U_{eq}{}^{a}$, Å^2
Cu	0.39261(5)	−0.02858(5)	1.25955(2)	0.03092(11)
O(1)	0.6474(3)	−0.0370(3)	1.25994(16)	0.0324(5)
O(2)	0.8498(3)	−0.1684(3)	1.33559(17)	0.0421(6)
O(3)	0.5946(3)	−0.2628(3)	1.32169(16)	0.0365(6)
O(4)	0.4301(3)	0.0766(3)	1.37368(15)	0.0404(6)
O(5)	0.3482(3)	0.2200(3)	1.47675(15)	0.0408(6)
O(6)	0.1743(3)	0.1704(4)	1.36275(19)	0.0565(8)
N(1)	0.1808(3)	−0.1568(3)	1.00286(16)	0.0255(5)
N(2)	0.7000(3)	−0.1584(3)	1.30626(16)	0.0292(6)
N(3)	0.3138(3)	0.1576(3)	1.40433(17)	0.0296(6)
C(1)	0.0044(3)	−0.0920(3)	1.00382(18)	0.0242(6)
H(11)	−0.037	−0.124	1.057	0.029
H(12)	−0.068	−0.139	0.956	0.029
C(2)	0.3028(4)	−0.0945(4)	1.07933(19)	0.0267(6)
H(21)	0.416	−0.129	1.072	0.032
H(22)	0.301	0.022	1.079	0.032
C(3)	0.2577(4)	−0.1539(4)	1.1641(2)	0.0298(7)
H(31)	0.304	−0.251	1.185	0.036
C(4)	0.1505(4)	−0.0718(4)	1.2126(2)	0.0343(7)
H(41)	0.102	0.025	1.192	0.041
H(42)	0.126	−0.114	1.265	0.041
C(5)	0.1698(4)	−0.3380(4)	1.0079(2)	0.0346(8)
H(51)	0.164	−0.369	1.066	0.052
H(52)	0.268	−0.385	0.987	0.052
H(53)	0.070	−0.374	0.972	0.052
C(6)	0.2518(4)	−0.1128(4)	0.9217(2)	0.0349(7)
H(61)	0.354	−0.052	0.935	0.052
H(62)	0.171	−0.048	0.886	0.052
H(63)	0.275	−0.209	0.891	0.052

[a] For non-hydrogen atoms U_{eq} is defined as one-third of the trace of the orthogonalized U_{ij} tensor $U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* (\vec{a}_i \vec{a}_j)$, for hydrogen – U_{iso}.

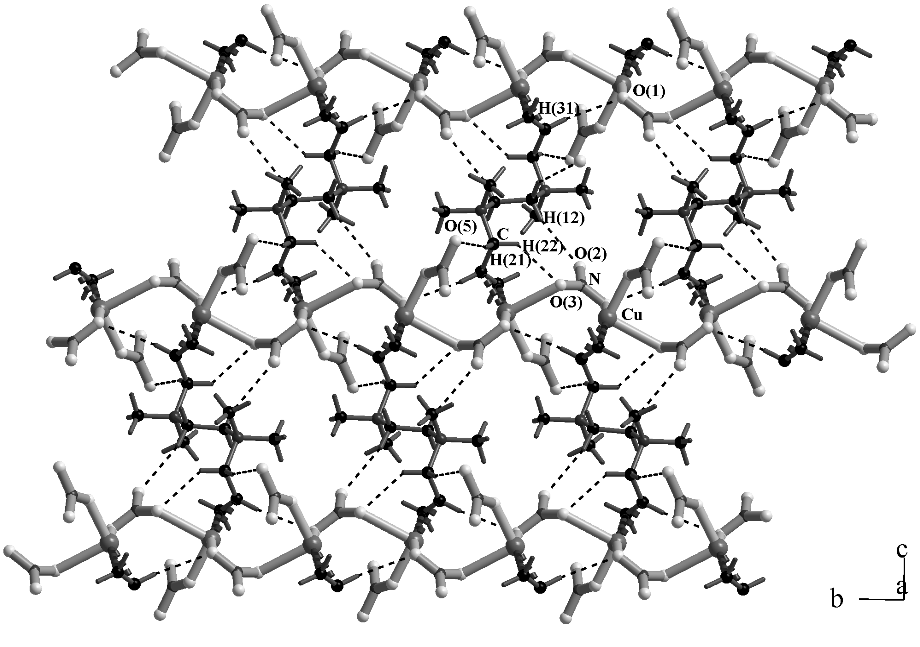


Fig. 3 Fragment of the coordination net in the crystal structure of **II**.

Table 4 Selected bond lengths and angles in the structures of **I** and **II.**

	Bond	*d*, Å	Angle	*ω*, deg
I	N–C(6)	1.496(3)	C(6)NC(2)	111.1(2)
	N–C(5)	1.506(3)	C(5)NC(2)	110.0(2)
	N–C(1)	1.511(2)	C(1)NC(2)	109.3(2)
	N–C(2)	1.517(3)	NC(1)C(1)^{i}	113.0(2)
	C(1)–C(1)^{i} [a]	1.516(4)	C(3)C(2)N	114.7(2)
	C(2)–C(3)	1.488(3)	C(2)C(3)C(4)	122.9(2)
	C(3)=C(4)	1.302(4)		
II	Cu–O(1)	2.029(2)	C(4)CuC(3)	40.2(1)
	Cu–O(3)	2.549(3)	O(1)CuO(4)	88.2(1)
	Cu–O(4)	1.975(2)	O(1)CuO(3)	86.1(4)
	Cu–*m* [b]	1.893(2)	O(3)CuO(4)	93.3(4)
	C(3)=C(4)	1.386(5)	C(4)CuO(4)	116.3(2)
	N(2)–O(1)	1.280(3)	C(3)CuO(1)	115.4(2)
	N(2)–O(2)	1.229(3)	C(2)C(3)C(4)	123.5(3)
	N(2)–O(3)	1.249(4)	O(2)N(2)O(3)	121.7(3)
	N(3)–O(4)	1.281(4)	O(3)N(2)O(1)	118.1(2)
	N(3)–O(5)	1.244(3)	O(6)N(3)O(5)	122.4(3)
	N(3)–O(6)	1.223(4)	O(5)N(3)O(4)	117.8(3)

[a] Symmetry code: (i) −*x*+1, −*y*, −*z*+1.

[b] *m* is the midpoint of the C(3)=C(4) bond.

Table 5 Geometry of selected H-contacts in the structures of **I** and **II**.

№	Contact *D*–H…*A*	Bond length, Å			Angle *D*–H…*A*, deg	Atom coordinates of *A*
		D–H	H…*A*	*D*…*A*		
I	C(1)-H(11)…O(3)	0.97	2.48	3.320(3)	144	1.5-*x*, −0.5+*y*, *z*
	C(1)-H(12)…O(4)	0.97	2.57	3.330(3)	135	*x*, *y*, *z*
	C(4)-H(42)…O(4)	0.93	2.68	3.539(4)	154	0.5−*x*, −0.5+*y*, *z*
	C(5)-H(51)…O(4)	0.96	2.65	3.429(4)	139	1−*x*, −0.5+*y*, 1.5−z
	C(5)-H(52)…O(2)	0.96	2.63	3.383(3)	136	−0.5+*x*, *y*, 1.5−*z*
	C(6)-H(63)…O(3)	0.96	2.68	3.580(3)	156	1−*x*, −*y*, 1−*z*
	C(6)-H(62)…O(3)	0.96	2.70	3.471(4)	138	1.5−*x*, −0.5+*y*, *z*
II	C(1)–H(11)...O(6)	0.97	2.44	3.308(4)	149	−*x*, −0.5+*y*, 2.5−*z*
	C(1)–H(12)...O(2)	0.97	2.50	3.405(4)	156	−1+*x*, −0.5−*y*, −0.5+z
	C(2)–H(21)...O(5)	0.97	2.45	3.380(4)	161	1−*x*, −0.5+*y*, 2.5−*z*
	C(2)-H(22)...O(5)	0.97	2.72	3.530(5)	142	*x*, 0.5−*y*, −0.5+*z*
	C(2)-H(22)...O(3)	0.97	2.44	3.211(4)	136	1−*x*, 0.5+*y*, 2.5−*z*
	C(3)–H(31)...O(1)	0.93	2.53	3.440(4)	165	1−*x*, −0.5+*y*, 2.5−*z*
	C(4)-H(41)…O(2)	0.93	2.61	3.425(5)	147	1−*x*, 0.5+*y*, 2.5−*z*
	C(4)-H(42)…O(2)	0.93	2.62	3.340(5)	135	−1+*x*, *y*, *z*
	C(5)–H(53)...O(2)	0.96	2.63	3.481(4)	149	−1+*x*, −0.5−*y*, −0.5+z

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