

X-RAY STRUCTURAL INVESTIGATION OF THE CRYSTALS OF THE DOUBLE SALT OF PENTAMMINECOPPER PERCHLORATE AND AMMONIUM PERCHLORATE

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The structure of crystals having the over-all formula $\text{NH}_4\text{Cu}(\text{NH}_3)_5(\text{ClO}_4)_2$ was investigated by the x-ray diffraction method. The crystals belong to the tetragonal system; $a = 10.59$, $c = 7.50$ Å, $N = 2$. Space group $\text{P}\bar{4}2_1\text{m}$. Magnetic moment $\mu_{\text{eff}} = 1.77 \mu_B$. The crystals exhibit a considerable piezo effect. The structure was determined from the (001) and (110) projections of the interatomic function, by geometrical analysis in conjunction with the method of trial and error, and from the (001) and (110) electron density projections with the aid of the Booth correction. The structural elements are $[\text{Cu}(\text{NH}_3)_4]^{2+}$ square complexes, NH_4^+ and ClO_4^- ions, and NH_3 groups. The Cu-NH₃ distances are 2.07 ± 0.02 Å. There are numerous hydrogen bonds between the NH₃ groups and NH₄⁺ and ClO₄⁻ ions.

In the course of studies of the crystal chemistry of complex compounds of bivalent copper (and, in particular, its amines) in the Laboratory of Crystal Chemistry of the Chemical Faculty of Moscow State University crystals of $\text{Cu}(\text{NH}_3)_4\text{PtCl}_4$ [1], $\text{Cu}(\text{NH}_3)_4(\text{NO}_2)_2$ [2], $\text{CuPy}_2(\text{NCS})_2$ [3] were investigated by the x-ray structural method. In this paper we present the results obtained by structure analysis of $\text{NH}_4\text{Cu}(\text{NH}_3)_5(\text{ClO}_4)_2$ crystals.

An account of the preparation of the substance and its crystallographic characteristics are given in an earlier brief communication [4]. The lattice parameters were determined from oscillation x-ray patterns along the [100], [001], and [110] directions and refined from the distant spots of zero layer lines taken with the Wessenberg x-ray goniometer. The cassette radius was determined with the aid of an NaCl standard. The following values were obtained: $a = b = 10.59 \pm 0.02$, $c = 7.50 \pm 0.02$ Å; $N = 2$; $d_{\text{x-ray}} = 1.84$, $d_{\text{pycn.}} = 1.84$ g/cc. The crystals belong to the $D_{4h} = 4/\text{mm}$ diffraction class. In accordance with the extinctions found, the space groups $D_{2d}^7 = \text{P}\bar{4}2_1\text{m}$ and $D_4^2 = \text{P}\bar{4}2_1$ are possible. The absence of an inversion center is confirmed by the strong piezo effect.*

The structure was determined by recording of the zero layer lines during rotation of the crystal about the [001] and [110] axes. The reflection intensities were estimated visually on a blackening point scale. The squares of the structure amplitudes were calculated with the polarization and Lorentz factors taken into account; the absorption factor was disregarded. Crystals of approximately 0.2×0.2 mm square section were chosen. In some instances the crystals were elongated in the [110] direction; this was very convenient for adjustment of the crystal in that direction and diminished the errors due to absorption. After adjustment the crystals were coated with collodion.

Preliminary structure data were obtained by calculation of projections of the interatomic functions in the [110] and [001] directions [because of numerous superpositions of maxima, the projection on the (100) plane did not give any useful results]. Combined analysis of these projections gave the possible vertical (along the Z axis) and horizontal (lying in the XY plane) components of the Cu-Cu, Cu-Cl_I-Cu-Cl_{II}, and Cu-NH₃ vectors (Fig. 1).

Analysis of the occurrence of general and special positions in both possible space groups reveals several structural variants different in principle. However, detailed comparison of the coordinates of the maxima and of the positions which may be occupied by Cu and Cl atoms makes it possible to reject all the variants with the exception of one which belongs to the $\text{P}\bar{4}2_1\text{m}$ group. Thus, analysis of Patterson projections gave the space group and the coordinates of most of the atoms. It was found that Cu atoms occupy the twofold position 2 (a): $000, \frac{1}{2}, \frac{1}{2}, 0$; six Cl

* The piezo effect was measured by V. A. Koptsik (Faculty of Physics, Moscow State University).

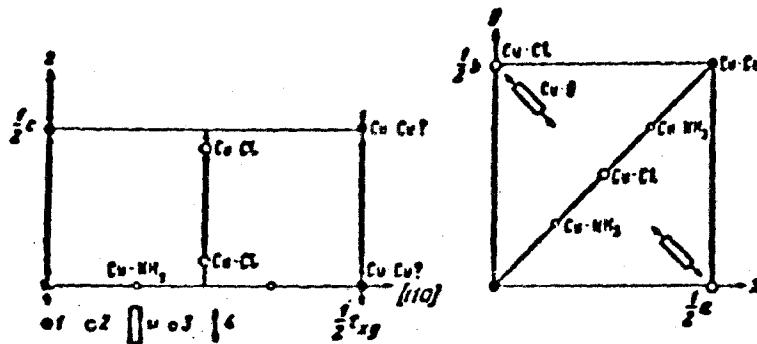


Fig. 1. Distribution of the maxima in Patterson projections (the Cu-Cu, Cu-Cl, Cu-O, and Cu-NH₃ vectors): 1) Highest maxima; 2) maxima of medium height; 3) low maxima; 4) direction of "diffusion" of the maximum and the possible vector displacement associated with it.

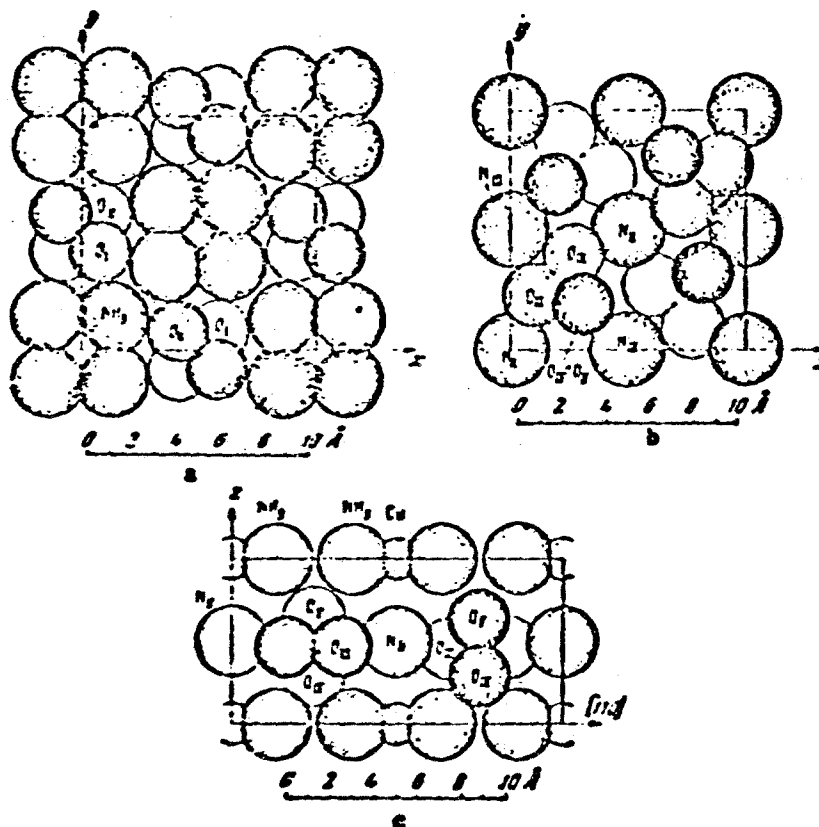


Fig. 2. Atomic packing in the structure: a) Layer parallel to (001), with $z=0$; b) layer parallel to (001), with $z=1/2$; c) layer parallel to (110), with $z=0$.

atoms occupy two positions: Cl_I a twofold position 2(c): $0 \frac{1}{2} z, \frac{1}{2} 0 z$ with z close either to zero or to $\frac{1}{2}$; Cl_{II} occupies the fourfold position 4(e): $x, \frac{1}{2} + x, z$ with $x = \frac{1}{4}$, and z close to $\frac{1}{2}$ or to zero; of the ten NH₃ groups, eight occupy the general position 8(f): xyz with $x=y=0.13$ and $z=0$. The remaining two NH₃ groups and the two NH₄⁺ ions must occupy twofold positions 2(b): $\frac{1}{2} 00, \frac{1}{2} \frac{1}{2} \frac{1}{2}$ and 2(c): $0 \frac{1}{2} z, \frac{1}{2} 0 \bar{z}$. As these groups are indistinguishable by the x-ray method, we use the symbol N_{II} to denote the group in position 2(b), and N_{III} for the group in position 2(c). In analysis of the positions of the oxygen atoms we took into account the obvious fact that they are in tetrahedral coordination around the chlorine atoms. This gave two positions 4(e) for the eight oxygen atoms O_I and O_{II} surrounding the Cl atoms, and three positions: 8(f), 4(e), and 4(c) for the 16 oxygen atoms O_{III}, O_{IV}, and O_V surrounding the Cl_{II} atoms. The coordinates of the O_I and O_{II} atoms are found from Patterson Projections.

Three problems remained to be solved for a complete determination of the atomic arrangement.

1. Selection of one of the two possible variants for distribution of Cl_I and Cl_{II} atoms along the height z of the cell (Cl_I with $z \approx 0$ and Cl_{II} with $z \approx 1/2$, or vice versa).
2. Determination of the coordinate z of the N_{III} atom in position 2(c).
3. Determination of the orientation of the $[\text{Cl}_{II}\text{O}_4]^-$ perchlorate group: i.e., determination of the coordinates of the atoms O_{III} , O_{IV} , and O_V .

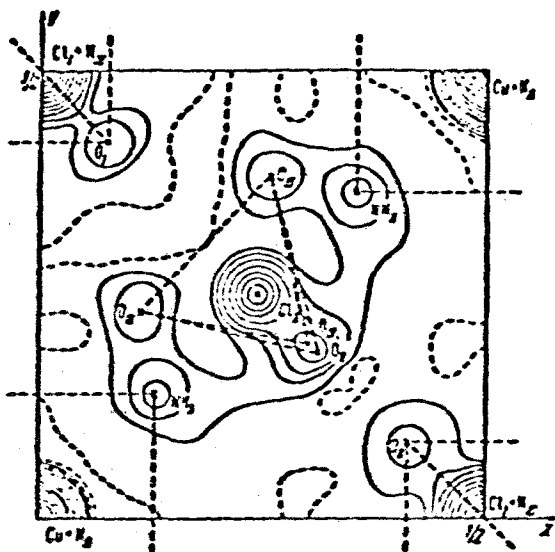


Fig. 3. Electron-density projection on the (001) plane. The curves are drawn at intervals starting at the $2 \text{ e}/\text{Å}^2$ level.

NH_3 groups surrounding the copper atoms in a square arrangement. The characteristic feature of the noncentrosymmetric projection (110) is that for many atoms (linked by the plane of symmetry parallel to the projection plane) there are maxima of double height, so that the precision in determination of their coordinates is increased considerably; moreover, the two independent chlorate groups both appear twice in different regions of the projection. The theoretical (001) and (110) projections were calculated, with the Booth correction, for determination of the displacements of the maxima due to termination of the series and superposition of maxima of atoms in close proximity in the projections. The final values were the x and y coordinates averaged over two projections and the z coordinates from the (110) projection averaged for independent maxima. In the averaging differences between the errors of the coordinates found from the centrosymmetric and noncentrosymmetric coordinates [5] and from maxima of single or double height were taken into account. The final atomic coordinates are given in Table 1.

These structural details were elucidated by geometrical analysis in conjunction with the method of trial and error. In analysis of the possible spatial distribution of the structural elements, the ClO_4^- tetrahedrons were assumed to be regular, and the $\text{Cl}-\text{O}$ distances were taken as 1.50 Å; the values adopted for the intermolecular radii were 1.40 Å for O, and 1.70 Å for NH_3 groups and NH_4^+ ions. Values of $F_{\text{calc.}}$ and $F_{\text{meas.}}$ were compared for reflections of the $h0k$ and $00l$ type. As the result of somewhat laborious work on rejection of geometrically improbable positions, and variations of the positional parameters for improving the agreement between $F_{\text{calc.}}$ and $F_{\text{meas.}}$, we obtained the configuration shown in Fig. 2a, b, and c, which is a schematic diagram of the atomic packing in individual layers of the unit cell.

The final electron-density projections are given in Figs. 3 and 4. The maxima on the projections were found by interpolation from parabolic curves (electron-density sections passing through the maximum) and determinations of the points where the first derivatives are zero. The (001) centrosymmetric projection clearly reveals the maxima corresponding to all five independent oxygen atoms and the

TABLE 1. Atomic Coordinates in the Crystal Structure of $\text{NH}_4[\text{Cu}(\text{NH}_3)_2](\text{ClO}_4)_2 \cdot \text{NH}_3$

Atoms	Position	x	y	z	Atoms	Position	x	y	z
Cu	2 (a)	0	0	0	O_{IV}	4 (e)	0,309	0,191	0,698
Cl_I	2 (c)	0	$1/2$	0,067	O_V	4 (e)	0,307	0,193	0,368
Cl_{II}	4 (c)	$1/2$	$1/4$	0,530	NH_3	8 (f)	0,138	0,138	-0,002
O_I	4 (e)	0,082	0,418	0,187	N_{II}	2 (b)	0	0	$1/2$
O_{II}	4 (e)	0,685	0,585	-0,053	N_{III}	2 (c)	0	$1/2$	0,567
O_{III}	8 (f)	0,109	0,230	0,532					

Neither the experimental nor the theoretical (110) projection shows clearly the maxima corresponding to N_{III} atoms. In both projections they are hidden to an equal extent in the slopes of high maxima corresponding to two Cl_{II} atoms and two O_{IV} atoms. However, the small difference between the electron densities in the experimental

and theoretical projections in regions where N_{III} atoms may be present confirms that it is correct to locate them half way between the perchlorate groups, i.e., with $z_{N_{III}} = 0.566$. The N_{II} maximum on the (110) projection is also in the region of the stronger Cl_{II} maximum. Here, however, the coordinate $z = 1/2$ is determined by the crystal symmetry.

Nevertheless two other variants were tested involving displacement of the N_{II} atom along the z axis to a position with $z \approx 0.17$, i.e., into the region of a diffuse "spur" of one of the maxima which cannot be accounted for by the other atoms. If the twofold position of the N_{II} atom was to be retained, it was necessary to assume either a lowering of symmetry in the structure to $P2_12_12$, or a statistical distribution of N_{II} atoms over one half of the points of the fourfold position $4(d): 00z, 00z, 1/2, 1/2 z, 1/2, 1/2 z$, with $z \approx 0.17$. However, both variants increased the divergence factor and worsened the general contours of the (110) projection. Moreover, a similar "spur" was found on the theoretical electron-density projection. Its existence is therefore a consequence of series termination.

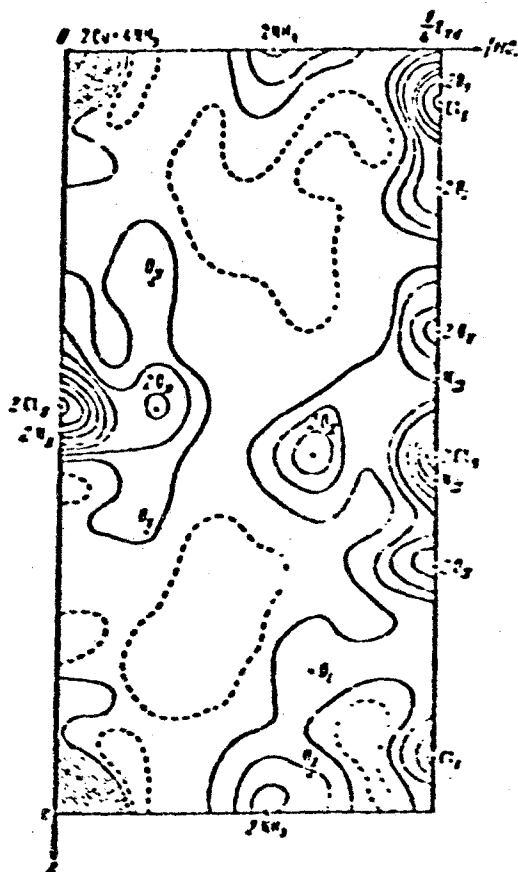


Fig. 4. Electron-density projection on the (110) plane. The curves are drawn at intervals of $4 \text{ e}/\text{Å}^2$.

The experimental and calculated structure amplitudes are compared in Table 2. Reduction to the absolute scale and determination of the average temperature correction for all the atoms were effected by comparison of F_{calc} with F_{meas} [6]. For $hk0$ reflections $B = 2.8 \text{ Å}^2$, and for reflections of the hhl type $B = 2.3 \text{ Å}^2$. The divergence factor, calculated for all the reflections up to $(\sin \theta / \lambda)_{\text{max}} = 0.63$ was found to be 0.226 for reflections of the $hk0$ type (0.197 without zero reflections). For reflections of the hhl type it was 0.194. The probable errors in determination of the atomic coordinates, calculated from Vainshtein's formula [7-8] for a two-dimensional electron density distribution, were:

$$\begin{aligned} \Delta(x_{Cl}) &= \Delta(z_{Cl}) = 0.006, & \Delta(z_{N_{III}}) &= 0.013, \\ \Delta(x_{N}) &= \Delta(z_{N}) = 0.014, & \Delta(x_{N_{II}}) &= \Delta(z_{N_{II}}) = 0.019 \text{ Å}. \end{aligned}$$

Description of the Structure

The most important interatomic distances in Å and bond angles have the following values:

Distances and angles in ClO_4 groups

$Cl_I - O_I$	$= 1.52 \pm 0.02$	$\angle O_I - Cl_I - O_I$	$= 108^\circ$
$Cl_I - O_{II}$	$= 1.55 \pm 0.02$	$\angle O_I - Cl_I - O_{II}$	$= 110^\circ$
$O_I - O_I$	$= 2.46 \pm 0.03$	$\angle O_{II} - Cl_I - O_{II}$	$= 110^\circ$
$O_{II} - O_{II}$	$= 2.54 \pm 0.03$		
$O_{II} - O_I$	$= 2.52 \pm 0.02$		
$Cl_{II} - O_{III}$	$= 1.51 \pm 0.02$	$\angle O_{III} - Cl_{II} - O_{III}$	$= 107^\circ$
$Cl_{II} - O_{IV}$	$= 1.52 \pm 0.02$	$\angle O_{IV} - Cl_{II} - O_{IV}$	$= 109^\circ$
$Cl_{II} - O_V$	$= 1.48 \pm 0.02$	$\angle O_{IV} - Cl_{II} - O_{III}$	$= 111^\circ$
$O_{III} - O_{III}$	$= 2.42 \pm 0.03$	$\angle O_V - Cl_{II} - O_{III}$	$= 111^\circ$
$O_{IV} - O_V$	$= 2.46 \pm 0.02$		
$O_{III} - O_{IV}$	$= 2.49 \pm 0.02$		
$O_{III} - O_V$	$= 2.46 \pm 0.02$		

Environment of the NH_2 group

$NH_2 - NH_2$	$= 2.92 \pm 0.01$ (in complex)		
$NH_2 - NH_2$	$= 3.36 \pm 0.05$	$\angle Cu - N_I - O_{II}$	$= 124^\circ$
$NH_2 - O_{IV}$	$= 2.97 \pm 0.02$	$\angle Cu - N_I - O_{IV}$	$= 127^\circ$
$NH_2 - O_{II}$	$= 3.01 \pm 0.02$	$\angle O_{II} - N_I - O_{IV}$	$= 64^\circ$

TABLE 2. Comparison of $F_{meas.}$ and $F_{calc.}$ for Reflections of the $hk0$ and hkl Types

$hk0$	$F_{meas.}$	$F_{calc.}$	$hk0$	$F_{meas.}$	$F_{calc.}$	$hk0$	$F_{meas.}$	$F_{calc.}$	$hk0$	$F_{meas.}$	$F_{calc.}$
200	17	+7	520	7	-18	640	40	+26	770	7	+6
400	69	+73	620	66	+61	740	10	-8	870	0	0
600	9	+6	720	10	+14	840	49	+46	970	0	+8
800	75	+58	820	18	+18	940	8	+8	10.70	0	+1
10.00	17	+20	920	16	-11	10.40	17	+13	11.70	4	+3
12.00	23	+27	10.20	33	+34	11.40	0	-4	850	49	+47
210	20	-14	11.20	0	+4	12.40	28	+26	980	5	+4
310	24	-10	12.20	13	+7	550	21	-29	10.80	9	-9
410	46	+49	13.20	0	0	650	0	0	990	9	+8
510	24	+29	330	29	+14	750	9	+6			
610	17	-35	430	15	-18	850	9	-4			
710	31	+34	530	40	+47	950	10	+6			
810	0	0	630	8	+8	10.50	0	+1			
910	12	+15	730	10	-6	11.50	0	+5			
10.10	7	+6	830	10	+10	12.50	0	-2			
11.10	5	-11	930	9	+1	660	71	+69			
12.10	0	-1	10.30	0	-7	760	0	+1			
13.10	0	+4	11.30	10	+16	860	16	+9			
220	172	+181	12.30	0	-2	960	0	-5			
320	11	+11	440	85	+89	10.60	26	+28			
420	52	-54	540	0	+7	11.60	6	+3			

hkl	$F_{meas.}$	$F_{calc.}$	Phase	hkl	$F_{meas.}$	$F_{calc.}$	Phase	hkl	$F_{meas.}$	$F_{calc.}$	Phase
002	169	189	0°	228	7	5	45°	556	28	22	35.9°
003	27	30	0°	330	34	14	0°	557	16	21	331.2°
004	66	90	0°	331	54	49	25.1°	558	20	24	19.6°
005	9	9	0°	332	59	63	21.6°	650	84	75	0°
006	49	51	0°	333	18	20	19°	661	21	7	16.8°
007	0	2	0°	334	58	53	6°	662	54	54	0.7°
008	0	1	0°	335	38	33	355.1°	663	11	4	0°
009	5	8	0°	336	40	36	24.4°	664	22	33	358°
111	41	21	315°	337	24	23	0°	665	7	6	70.2°
112	72	64	26.5°	338	24	30	17.3°	666	11	19	337.7°
113	31	39	346.6°	440	102	94	0°	770	11	6	0°
114	47	55	25.8°	441	64	51	348.7°	771	17	14	344.8°
115	27	45	303.4°	442	70	70	356°	772	22	21	24.4°
116	44	53	46.5°	443	38	36	340.3°	773	24	19	339°
117	58	41	318.5°	444	18	47	6.1°	774	22	24	24.4°
118	37	37	26.2°	445	14	20	16.7°	775	14	21	317.7°
119	14	19	325.6°	446	26	29	324.5°	880	54	54	0°
220	169	183	0°	447	0	10	0°	881	16	5	288.2°
221	21	23	272.0°	448	0	10	320.0°	882	31	33	358.3°
222	92	91	351.1°	550	31	32	180.0°	883	14	10	335.6°
223	32	28	315°	551	31	26	356.6°	990	7	8	0°
224	28	47	10.1°	552	33	20	33.1°	991	5	8	262.6°
225	19	5	249°	553	31	20	334.1°				
226	31	29	340.5°	554	31	25	29.9°				
227	8	7	296.4°	555	21	22	326.3°				

Environment of the $(\text{NH}_2)_{\text{II}}$ group

$$N_{\text{II}} - O_{\text{III}} = 2.71 \pm 0.02 \quad \angle O_{\text{III}} - N_{\text{II}} - O_{\text{III}} = 90.4^\circ$$

Environment of the $(\text{NH}_m)_{\text{III}}$ group

$$\begin{array}{ll} N_{\text{III}} - O_{\text{I}} = 3.10 & \angle O_{\text{I}} - N_{\text{III}} - O_{\text{I}} = 51^\circ \\ N_{\text{III}} - O_{\text{II}} = 3.14 & \angle O_{\text{II}} - N_{\text{III}} - O_{\text{II}} = 51^\circ \\ N_{\text{III}} - O_{\text{III}} = 3.11 & \angle O_{\text{III}} - N_{\text{III}} - O_{\text{III}} = 51^\circ \\ N_{\text{III}} - O_{\text{V}} = 2.92 & \angle O_{\text{V}} - N_{\text{III}} - O_{\text{V}} = 160^\circ \end{array}$$

The structure is composed of $[\text{Cu}(\text{NH}_2)_4]^{2+}$, NH_4^+ , ClO_4^- ions and NH_3 groups. The complex $[\text{Cu}(\text{NH}_2)_4]^{2+}$ ions have plane square structure with $\text{Cu}-\text{NH}_2$ distances of 2.07 ± 0.02 Å. The ClO_4^- tetrahedrons are only slightly distorted; nearly all the differences between the $\text{Cl}-\text{O}$ and $\text{O}-\text{O}$ distances lie within the possible error limits. The average distances are 1.52 Å for $\text{Cl}-\text{O}$ and 2.48 Å for $\text{O}-\text{O}$.

The idealized packing pattern of the structural elements may be described as follows. The $[\text{Cu}(\text{NH}_2)_4]^{2+}$ complexes and $(\text{ClO}_4^-)_{\text{I}}$ ions lie in the (001) plane at height $z = 0$ and alternate in checkerwork order (Cu at the points 000 and $1/2 \ 1/2 \ 0$, Cl at the points $0 \ 1/2 \ 0$ and $1/2 \ 00$). The remaining ("fifth") NH_3 groups and NH_4^+ ions are arranged in the same manner in another layer at height $z = 1/2$. Between them (in the same layer) the four $(\text{ClO}_4^-)_{\text{II}}$ perchlorate groups are situated at the points $1/4 \ 1/4$, $1/4 \ 3/4$, $3/4 \ 1/4$, $3/4 \ 3/4$. As the NH_3 groups and NH_4^+ ions are indistinguishable by the x-ray method, it is not possible to determine which is in position N_{II} (at the point $001/2$) and which at N_{III} (at the point $1/2 \ 0 \ 1/2$); statistical distribution between these positions is not excluded. The actual coordination of $(\text{NH}_2)_{\text{II}}$ and $(\text{NH}_m)_{\text{III}}$ groups is determined by displacements of ClO_4^- ions from their "ideal" positions and by differences in the orientation of $(\text{ClO}_4^-)_{\text{I}}$ and $(\text{ClO}_4^-)_{\text{II}}$ groups. The $(\text{NH}_2)_{\text{II}}$ group is surrounded by four oxygen atoms which form a very flattened tetrahedron around it (two Cu atoms at a distance of 3.75 Å are more distant neighbors). The distances $N_{\text{II}} - O_{\text{III}} = 2.71$ Å indicate the existence of stable hydrogen bonds. The $(\text{NH}_m)_{\text{III}}$ group is surrounded by ten oxygen atoms. Here the existence of less stable hydrogen bonds may be postulated: $N_{\text{III}} - O_{\text{V}} = 2.92$ Å. However, the average $(\text{NH}_m)_{\text{III}} - \text{O}$ distance is considerably greater than $(\text{NH}_2)_{\text{II}} - \text{O}$ (3.08 Å as against 2.71 Å). The $(\text{NH}_m)_{\text{III}}$ group is in a larger cavity and has a greater coordination number than $(\text{NH}_2)_{\text{II}}$. As was pointed out by G. B. Bokii, this fact suggests that the ammonia molecule and not the ammonium ion plays the role of this "clathrate" group.

The perchlorate groups form hydrogen bonds with the $(\text{NH}_2)_{\text{I}}$ groups contained in the $[\text{Cu}(\text{NH}_2)_4]^{2+}$ complex. The $N_{\text{I}} - O_{\text{II}}$ and $N_{\text{I}} - O_{\text{IV}}$ distances are 3.00 and 2.97 Å respectively. Apparently the tendency to form hydrogen bonds determines to a considerable extent the distribution of perchlorate groups along z . The considerable piezo effect observed in the crystals is probably associated with the presence of numerous hydrogen bonds.

The formally square environment of the Cu atoms is supplemented in the crystal to a bipyramidal one by $(\text{NH}_2)_{\text{II}}$ groups situated on the z axis half way between the Cu atoms. However, the distance $\text{Cu} - (\text{NH}_2)_{\text{II}} = 3.75$ Å is so large that it is difficult to suppose the existence of a chemical bond (electron exchange) between $(\text{NH}_2)_{\text{II}}$ groups and Cu atoms. The position of the $(\text{NH}_2)_{\text{II}}$ groups at points $001/2$ is evidently determined by considerations of packing only: a fairly large cavity (in the form of a flattened tetrahedron), formed by the four O atoms of the perchlorate groups, is present here. Therefore, it may be assumed that in this compound the coordination of the copper atom is of the square type.

The question of purely "square" coordination around bivalent copper in organic compounds has been controversial until recently and has been solved positively only by the very latest investigations. Square coordination around a Cu^{II} atom was discovered in the recently revised structure of azurite [9], in certain copper silicates [10], and finally, in the complex tetramine compound described in the present communication.

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