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The Crystal Structure of Copper Yunainate Trihydrate, $Cu(C_5H_8O_3NS)_2 \cdot 3H_2O$

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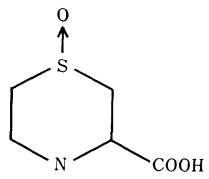
The crystal structure of copper yunainate trihydrate, $Cu(C_5H_8O_3NS)_2 \cdot 3H_2O$, has been studied by means of three-dimensional X-ray analysis. The crystal is monoclinic, with two formula units in a unit cell of the dimensions: $a=15.94$, $b=6.33$, $c=8.60$ Å, and $\beta=95.6°$; the space group is $P2_1$. The intensities were measured visually from equi-inclination integrating Weissenberg photographs taken with Cu$K\alpha$ radiation around the b and c axes. The structure was elucidated by Sim's method, and refined by the block-diagonal-matrix least-squares method, assuming anisotropic thermal motions for all the atoms. The final R factor is 9.2%. The copper atom is coordinated by imino nitrogen and carboxyl oxygen atoms of two yunainate groups, and a water oxygen atom. The first four atoms lie almost on a plane and make a square configuration of the *trans* type. The last one is bonded to the copper atom nearly in the direction of the normal of the plane. Thus, the coordination number of copper is five in the present crystal. Of the three sorts of water of crystallization, one is connected with the copper atom, as has already been mentioned, and the remaining two are lattice water, forming a hydrogen-bonded spiral chain around the screw axis. Further, these water molecules connect complex molecules to one another by their hydrogen bonds, forming a three-dimensional hydrogen-bonded structure.

Yunaine (I) is a kind of natural amino acid isolated from a seaweed, *Chondria crassicaulis* Harvey (Japanese name "Yuna"), by Professor Tsunematsu Takemoto.[1] A projection of the crystal structure of the copper salt of this amino acid, *i.e.*, copper yunainate trihydrate, has already been reported.[2] That work not only confirmed the structural formula (I), but also revealed some interesting facts about the molecular and crystal structure. In order to obtain more detailed information about these aspects, a three-dimensional X-ray crystal analysis of the copper salt has now been carried out.



(I)

Experimental

The sample of yunaine was kindly supplied by Professor Tsunematsu Takemoto, Tohoku University. The copper salt was prepared by dissolving copper carbonate in an aqueous solution of yunaine. Single crystals, suitable for the structure determination, were obtained as blue needles from the aqueous solution by slow evaporation in a desiccator.

The crystal data of copper yunainate trihydrate, obtained from oscillation and Weissenberg photographs in the present experiment, are given in Table 1.

Table 1. Crystal data of copper yunainate trihydrate

Monoclinic	$a=15.94\pm0.04$ Å
	$b=6.33\pm0.03$
	$c=8.60\pm0.03$
	$\beta=95.6\pm0.3°$
Space group	$P2_1$
Z	2
$\rho_{calcd.}$	1.70 g/cm^3

Equi-inclination integrating Weissenberg photographs were taken with Cu$K\alpha$ radiation from the zeroth to the fourth layer around the b axis and from the zeroth to the sixth layer around the c axis. The intensities were estimated visually by the multiple-film technique and were corrected for the Lorentz and polarization factors. Though the cross-sections of the samples used were about 0.2×0.3 mm^2 for the b axis and 0.2×0.4 mm^2 for the c, no correction was made for the absorption effect. The partial resolution of α_1—α_2 doublets was not taken into account, and only the α_1 spot intensities were measured where the doublets were completely resolved.

*1 Deceased 31 May 1967.

1) T. Takemoto, *Yakugaku Kenkyu*, **32**, 645 (1960).

2) A. Furusaki and Y. Tomiie, *Kwansei Gakuin Univ. Ann. Stud.*, **13**, 187 (1964).

The approximate scale and mean temperature factors were estimated by Wilson's method,[3] the latter being 2.83 Å^2. Thus, the absolute values of the structure factors of 1699 independent reflections were obtained out of about 1970 possible reflections.

All the calculations necessary to derive the structure factors in an absolute scale from the intensity data were carried out on a Bendix G-20 at the C. Itoh Electronic Computing Service Co., Ltd., using a program devised in this laboratory.

Structure Determination

A three-dimensional Patterson function, $P(u,v,w)$, was calculated on the computer mentioned above. From the Patterson map, the y parameters of the copper and two sulfur atoms were obtained easily, since the x and z parameters of these atoms had already been determined by two-dimensional analysis.[2] For the elucidation of the positions of the light atoms, the Fourier synthesis using Sim's method[4] was carried out on a HITAC 5020 computer at the University of Tokyo using a program written by the present authors. In case of non-centrosymmetry, the statistical weight for each structure factor, W, is given by the following equation:

$$W = I_1(X)/I_0(X)$$
$$X = 2|F||F_H|/\sum f_L^2$$

where F is the observed structure factor in an absolute scale, F_H is that calculated with heavy atoms alone, $\sum f_L^2$ is the sum of the squares of the scattering factors of the light atoms in a unit cell, and I_0 and I_1 are, respectively, the zero-order and first-order modified Bessel functions of the first kind.

From the Fourier map thus obtained, the peaks corresponding to all twenty-one light atoms could be easily picked out because of the distinct difference in height between these peaks and the ghost peaks. These atomic parameters were refined at first by the diagonal-matrix least-squares method, with individual isotropic temperature factors, on the HITAC 5020 computer. After three cycles of the refinement, the R factor dropped from 0.159 to 0.125. In these calculations, the standard atomic scattering factors listed in "International Tables for X-Ray Crystallography, Vol. III" were used. For the carbon, nitrogen, oxygen, and sulfur atoms, the scattering factors of neutral atoms were used, while for the copper atom, that of the Cu^{2+} ion was used without any correction for the anomalous dispersion. Figure 1 shows a composite diagram of the (010) sections, taken as through the centers of the atoms, of the three-dimensional electron density distribution at this stage. The positional parameters were further refined by the block-diagonal-matrix least-squares method, with anisotropic temperature factors of the form:

$$\exp(-B_{11}h^2-B_{22}k^2-B_{33}l^2-B_{12}hk-B_{23}kl-B_{31}lh)$$

After six cycles of the refinement, including a real dispersion correction of the copper atom for Cu$K\alpha$ radiation, $\Delta f' = -2.1$, the R factor dropped to 0.092, excluding non-observed reflections. The final atomic coordinates and anisotropic temperature factors are given in Table 2.

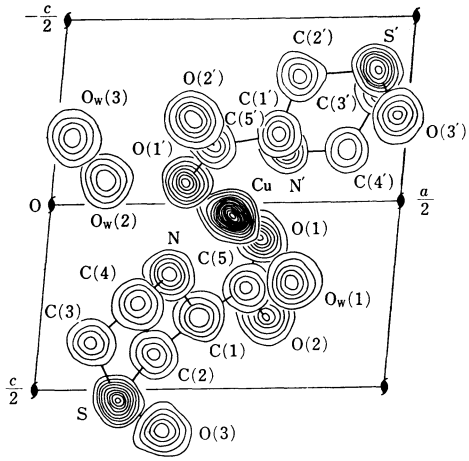


Fig. 1. The electron density distribution of copper yunainate trihydrate. A composite diagram of the (010) sections taken as through the atomic centers. Contours are at intervals of 5 e/Å^3 for a copper atom, 4 e/Å^3 for sulfur atoms and 2 e/Å^3 for the others, beginning at 2 e/Å^3.

Discussion

The molecular framework of the yunainate ions thus obtained is given in Fig. 2. Since the yunaine molecule has been proved to be of the L-type,[1] the yunainate ions should have the absolute configuration shown in Fig. 2, as long as the inversion into the D-type does not occur in the process of the mild reaction of yunaine with copper carbonate.

The two yunainate ions surrounding a copper atom are crystallographically independent. The bond lengths and angles of these two kinds of yunainate ions are given in Fig. 3. The average standard deviations of the atomic coordinates are 0.018 Å for carbon atoms, 0.013 Å for nitrogen and oxygen atoms, 0.005 Å for sulfur atoms, and 0.003 Å for the copper atom. Therefore, the mean standard deviations of various interatomic distances are estimated to be as is shown in Table 3. Some of the corresponding bond lengths and angles of the two kinds of yunainate groups are somewhat different from each other, but these differences do not seem to be significant in view of the accuracy of the present experiment. These bond lengths and

3) A. J. C. Wilson, *Nature*, **150**, 152 (1942).
4) G. A. Sim, *Acta Crystallogr.*, **12**, 813 (1959).

Table 2

(a) The final atomic coordinates

Atom	x/a	y/b	z/c	Atom	x/a	y/b	z/c
Cu	0.2600	0.0002	0.0329	N	0.1774	−0.0703	0.1891
S	0.1202	−0.1322	0.5306	N′	0.3249	0.0552	−0.1505
S′	0.4496	0.3003	−0.3577	C(1)	0.2288	−0.2005	0.3105
O(1)	0.3038	−0.2908	0.0896	C(2)	0.1684	−0.3199	0.4061
O(2)	0.3239	−0.4886	0.3088	C(3)	0.0716	0.0333	0.3745
O(3)	0.1916	−0.0187	0.6126	C(4)	0.1379	0.1138	0.2695
O(1′)	0.1903	0.2365	−0.0531	C(5)	0.2898	−0.3458	0.2281
O(2′)	0.1928	0.4984	−0.2276	C(1′)	0.3157	0.2869	−0.1830
O(3′)	0.4948	0.4208	−0.2266	C(2′)	0.3370	0.3429	−0.3472
$O_w(1)$	0.3578	0.1298	0.2121	C(3′)	0.4551	0.0304	−0.2917
$O_w(2)$	0.0740	−0.3061	−0.0636	C(4′)	0.4178	−0.0166	−0.1359
$O_w(3)$	0.0244	0.0881	−0.1750	C(5′)	0.2267	0.3452	−0.1557

(b) The anisotropic temperature factors in form of exp $(-B_{11}h^2-B_{22}k^2-B_{33}l^2-B_{12}hk-B_{23}kl-B_{31}lh)$

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{23}	B_{31}
Cu	0.00213	0.01554	0.00727	0.00131	0.00279	0.00226
S	0.00276	0.02077	0.00823	−0.00128	0.00081	0.00333
S′	0.00236	0.03792	0.01245	−0.00198	0.01085	0.00428
O(1)	0.00373	0.01237	0.00997	0.00032	−0.00415	0.00417
O(2)	0.00453	0.02175	0.01176	0.01129	0.00589	0.00263
O(3)	0.00390	0.02503	0.01050	−0.00043	−0.00773	−0.00003
O(1′)	0.00219	0.01819	0.00904	0.00136	0.00651	0.00410
O(2′)	0.00354	0.03827	0.01965	−0.01097	0.00796	−0.00041
O(3′)	0.00500	0.02104	0.01412	0.00945	0.01582	0.00699
$O_w(1)$	0.00221	0.01611	0.01889	0.00163	−0.00731	−0.00004
$O_w(2)$	0.00406	0.02702	0.01245	−0.00066	−0.00450	0.00407
$O_w(3)$	0.00403	0.02763	0.01687	0.00381	−0.00005	0.00189
N	0.00185	0.01813	0.00651	0.00425	0.00261	0.00146
N′	0.00233	0.01379	0.00831	−0.00042	0.00136	0.00312
C(1)	0.00252	0.00564	0.00778	0.00472	0.00324	0.00287
C(2)	0.00319	0.01393	0.00958	0.00196	0.00537	0.00393
C(3)	0.00328	0.02077	0.00948	0.00505	0.00766	0.00626
C(4)	0.00330	0.01341	0.00923	0.00496	−0.00365	0.00513
C(5)	0.00307	0.02187	0.00884	0.00281	−0.00364	0.00028
C(1′)	0.00185	0.01810	0.00902	−0.00119	0.00175	−0.00127
C(2′)	0.00188	0.03132	0.01066	0.00237	0.01454	0.00143
C(3′)	0.00485	0.02447	0.01788	0.00806	0.00956	0.01138
C(4′)	0.00242	0.03288	0.01492	0.00769	0.01452	0.00720
C(5′)	0.00349	0.02083	0.01011	0.00158	0.00331	0.00357

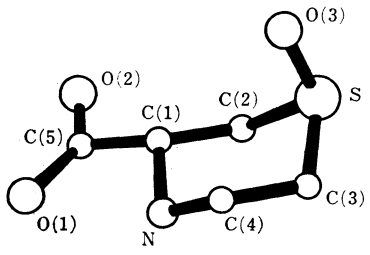


Fig. 2. The framework of a yunainate ion.

angles agree, within the limits of experimental error, with those found in the hydrochloride of cycloalliin, a methyl derivative of yunaine.[5]

In either of the two kinds of yunainate ions, the heterocyclic ring takes a chair form similar to that of cyclohexane. The sulfinyl oxygen atom is attached to the sulfur in the axial configuration, while the carboxyl group, a bulky substituent of the ring, is equatorial. The copper atom occupies the equatorial position of the nitrogen; hence, the hydrogen atom of the imino group should be axial.

The configuration of the atoms bonded to the sulfur is geometrically similar to that in dimethyl

5) K. J. Palmer and Kay Sue Lee, *Acta Crystallogr.* **20**, 790 (1966).

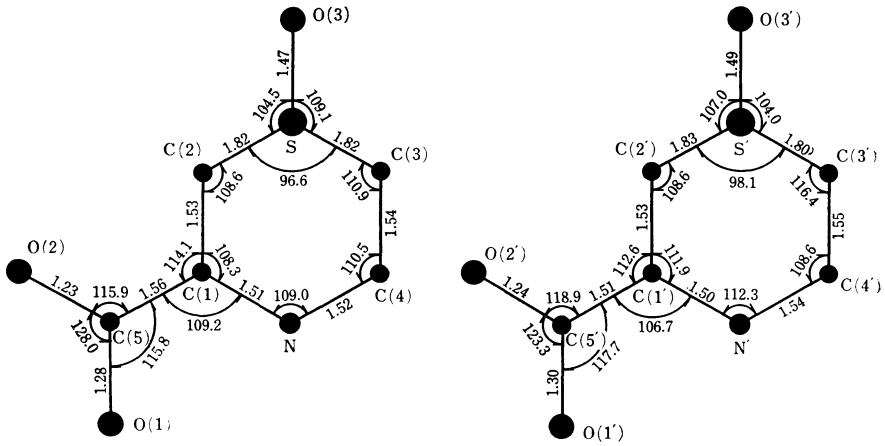


Fig. 3. The bond lengths (Å) and angles (°) of the two yunainate ions.

TABLE 3. THE MEAN STANDARD DEVIATIONS OF VARIOUS INTERATOMIC DISTANCES (in Å)

Cu···Cu	0.005	S···C	0.019
Cu···S	0.006	O···O	0.018
Cu···O	0.013	O···N	0.018
Cu···N	0.013	O···C	0.022
Cu···C	0.018	N···N	0.018
S···S	0.007	N···C	0.022
S···O	0.014	C···C	0.025
S···N	0.014		

sulfoxide.[6] The average length of the four independent sulfur-carbon bonds and that of the two independent sulfur-oxygen bonds found in the present molecules are 1.82 and 1.48 Å respectively, while the corresponding values in dimethyl sulfoxide are 1.82 and 1.47 Å respectively. The mean value of the four C–S–O angles and that of the two C–S–C angles are 106 and 97° respectively, while the corresponding angles in dimethyl sulfoxide are 107 and 100° respectively.

The mean of the two C–N–C angles, 111°, is very close to the tetrahedral angle, 109.5°, while the average value of the four nitrogen-carbon bond distances, 1.52 Å, is nearly in agreement with that in copper D,L-proline dihydrate, 1.525 Å.[7]

The conformation of the carboxyl group around the C(1)–C(5) or C(1′)–C(5′) bond is somewhat different in the two kinds of yunainate ions; that is, the azimuthal angle of the C(1)–N and C(5)–O(1) bonds around the C(1)–C(5) bond is about 19°, while the corresponding angle of the C(1′)–N′ and C(5′)–O(1′) bonds is about 30°.

As is shown in Fig. 4, the copper atom is coordinated by two imino nitrogen atoms, two carboxyl oxygen atoms, and a water oxygen atom.

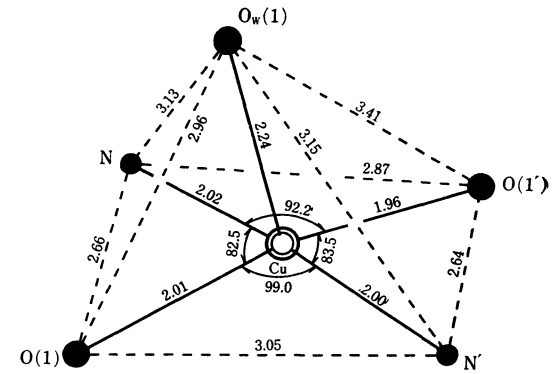


Fig. 4. The tetragonal pyramid of the five coordinating atoms.

The first four atoms lie almost on a plane and make a square configuration of the *trans* type. The last one is bonded to the copper atom nearly in the direction of the normal of the plane.

In the present crystal, there is no atom at a distance smaller than 3.5 Å from the copper atom except for the coordinated five atoms and except for some carbon atoms which are forced to approach the copper in consequence of the formation of the square coordination. On the side of the coordination square opposite to the coordinated water molecule described above, two oxygen atoms are present at distances of 3.57 and 3.67 Å respectively from the copper; one of them is the oxygen atom, $O_w(2)$, of another water molecule, and the other, the sulfinyl oxygen, O(3), of the adjacent complex molecule. However, both of these approaches to the copper seem to be due to the formation of hydrogen bonds with the N and N′ nitrogen atoms respectively rather than to the formation of the coordination bond with the copper. Thus, it is reasonable to conclude that the coordination number of the copper atom is five in the present crystal.

In the present case, the absence of the sixth atom

6) O. Bastiansen and H. Viervoll, *Acta Chem. Scand.*, **2**, 702 (1948).

7) A. M. Mathieson and H. K. Welsh, *Acta Crystallogr.*, **5**, 599 (1952).

to be coordinated to the copper may be closely connected with the fact that, as has already been mentioned, the two oxygen atoms, $O_W(2)$ and O(3), form hydrogen bonds with the N and N′ nitrogen atoms respectively in the axial direction of the six-membered ring and lie just in the neighborhood of the sixth coordination position. If, on the contrary, one of the water oxygen atoms should, for example, occupy the sixth position, neither of the two imino nitrogen could form a normal hydrogen bond because of the steric hindrance of the sixth coordinating atom. It is an interesting fact that, as is actually found, the formation of one of coordination bonds of a copper atom is given up, and instead that of two hydrogen bonds of the nitrogen atoms is achieved.

The five atoms, N, O(1), N′, O(1′), and $O_W(1)$, connected with the copper form a tetragonal pyramid, as is shown in Fig. 4. The lengths of two of the edges, N–O(1) and N′–O(1′), are nearly equal, while those of the other two edges, N–O(1′) and N′–O(1), are somewhat different from each other, the latter being 0.18 Å larger. This distortion of the tetragonal configuration may come from the deformation of the whole complex molecule of copper yunainate, a deformation which is due to the formation of hydrogen bonding at several parts of the complex. It is further found that the orientation of the Cu–$O_W(1)$ bond is inclined to some extent toward the side of the O(1) atom. This inclination may also be due to the strong hydrogen bond formed at a distance of 2.63 Å with the O(1) oxygen atom of the adjoining molecule. The two copper-nitrogen distances, Cu–N and Cu–N′, are 2.02 and 2.00 Å respectively, while the two copper-oxygen distances, Cu–O(1) and Cu–O(1′), are 2.01 and 1.96 Å respectively. These correspond approximately to the ordinary values found in many other copper complexes, such as copper D,L-proline dihydrate (1.99 Å for Cu–N and 2.03 Å for Cu–O).[7] The remaining copper-oxygen bond, Cu–$O_W(1)$, is somewhat weaker than the other two Cu–O bonds, its length being 2.24 Å. This value also nearly corresponds to that in cupric acetate dihydrate, 2.20 Å.

The best plane determined by the least-squares method throughout the four atoms, N, O(1), N′, and O(1′), is described by the equation:

$$0.5577X + 0.5182Y + 0.6484Z = 2.253$$

where X, Y, and Z are rectangular coordinates in the Å unit; $X=x+z\cos\beta$, $Y=y$, and $Z=z\sin\beta$. The deviations of the atoms from this plane are shown in Fig. 5. The copper atom is about 0.23 Å displaced from the plane toward the coordinated water molecule, and the direction of the Cu–$O_W(1)$ bond makes an angle of about 10° with the normal of the plane.

It may be said that the whole complex molecule, composed of a copper atom, a water molecule, and two yunainate groups, has approximately a symmetry of C_2. The centers of all the atoms in the puckered complex molecule lie between two parallel planes, a distance of about 1 Å apart, except in some hydrogen atoms and three oxygen atoms; the latter three oxygen atoms, O(3), O(3′), and $O_W(1)$, stand out to the same side of the plane.

The arrangements of the complex molecules, viewed down along the b and c axes, are shown in Figs. 6 and 7 respectively, together with some intermolecular distances smaller than 4.0 Å. The copper atom, the approximate center of gravity of a complex molecule, is situated very close to the particular position of $x/a=1/4$ and $z/c=0$. Consequently, the arrangement of the copper atoms alone forms nearly a (001) face-centered lattice; the smallest distance among the copper atoms is 6.33 Å along the b axis.

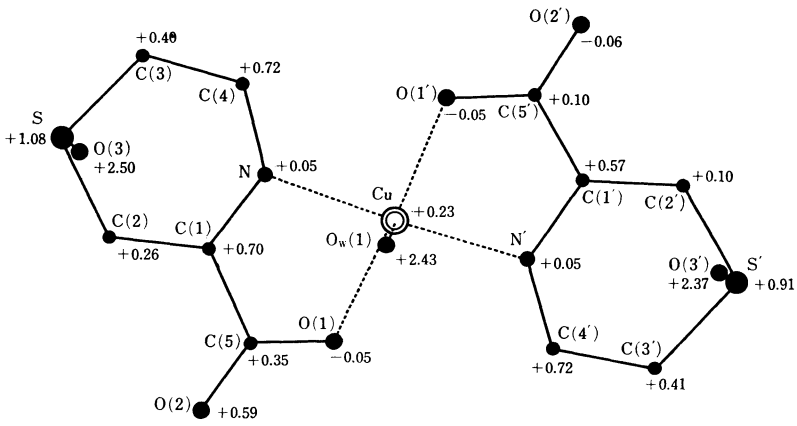


Fig. 5. The deviations (Å) from the square coordination plane, $0.5577X+0.5182Y+0.6484Z-2.253=0$. The complex molecule is viewed down along the normal of this plane.

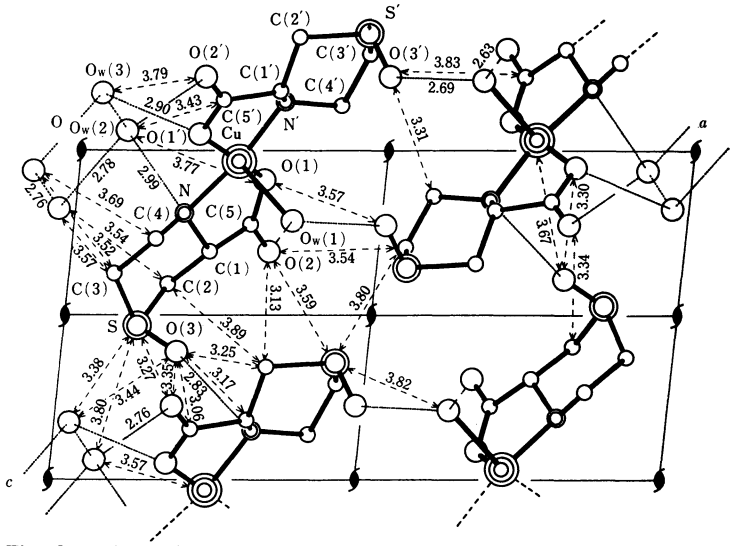


Fig. 6. The molecular arrangement viewed down along the *b* axis in the copper yunainate trihydrate crystal.

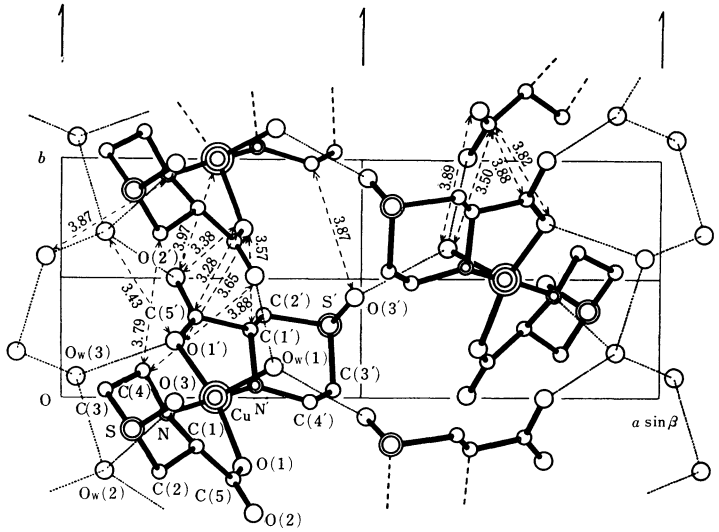


Fig. 7. The molecular arrangement viewed down along the *c* axis in the copper yunainate trihydrate crystal.

The three kinds of water of crystallization contained within the crystal seem to play an important part in connecting the complex molecules with one another. The W(1) water molecule, coordinated to the copper atom, binds not only two complex molecules adjoining along the *b* axis by the Cu-O_W(1) coordination bond and the O_W(1)–H···O(2) hydrogen bond, but also those related by the screw axis with each other, by the O_W(1)–H···O(3′) hydrogen bond. By these bonds and another hydrogen bond, N′–H···O(3), a molecular layer is formed parallel to the (100) plane. Such molecular layers are further connected by several hydrogen bonds formed by water molecules of the two remaining kinds, W(2) and W(3), which form a hydrogen-bonded spiral chain around the two-fold screw axis.

As has been described above, the W(1) water molecule forms two hydrogen bonds, O_W(1)–H··· O(2) and O_W(1)–H···O(3′), their lengths being 2.63 and 2.69 Å respectively. The two hydrogen atoms of the water molecule seem to be considerably off the O···O line, since the O(2)–O_W(1)–O(3′) angle, 130°, is markedly larger than the H–O–H angle of free water molecules in the gas phase, 104.5°.[8] Both of the two hydrogen bonds make

8) R. Mecke and W. Baumann, *Phys. Z.*, **33**, 833 (1932); B. T. Darling and D. M. Dennison, *Phys. Rev.*, **57**, 128 (1940); D. W. Posener and M. W. P. Standberg, *ibid.*, **95**, 374 (1954).

an angle of about 114° with the $O_W(1)$–Cu coordination bond. The total sum of the three angles, O(2)-O_W(1)-O(3′), Cu-O_W(1)-O(2), and Cu-O_W(1)-O(3′), 358°, is very close to the angle (360°) which would be found if the three bonds were coplanar.

The N′–H···O(3) hydrogen bond is the only one formed directly between two complex molecules, its length being 2.83 Å. As is shown in Fig. 8, the direction of this hydrogen bond is fairly much inclined to the side of the carboxyl group (C(1′), O(1′) and O(2′)) from the plane which bisects the C(1′)–N′–C(4′) angle perpendicularly. This may be due to some strong attractive force between the sulfinyl oxygen atom, O(3), and the carboxyl group, since they approach each other quite closely, as will be described later in more detail.

The hydrogen-bonded spiral structure of water molecules alone is one of the remarkable features found in the present crystal. The two kinds of hydrogen bonds, $O_W(2)\cdots O_W(3)$ and $O_W(2)\cdots O_W$-(3′), making the spiral are, respectively, 2.76 and 2.78 Å in length; these lengths are nearly comparable to that found in the hexagonal structure of ice, 2.76 Å, while the angles between the hydrogen bonds, $O_W(2)\cdots O_W(3)\cdots O_W(2')$ and $O_W(3)\cdots O_W$-(2)$\cdots O_W(3')$, are 97 and 108° respectively. The spiral is about 3 Å in diameter if only the nuclei of the water oxygen atoms are taken into account, and it is so right-handed a one that it takes, on the average, a rotation of about 1 radian to move it by 1 Å forward in the direction of the *b* axis.

Besides the hydrogen bonds mentioned above, the W(2) and W(3) water molecules form several hydrogen bonds with the complex molecules around them. The former makes two hydrogen bonds, one with the carboxyl oxygen atom, O(2′), and the other with the nitrogen atom, N; their lengths are 2.76 and 2.99 Å respectively. On the other hand, the latter water molecule forms a hydrogen bond with the carboxyl oxygen atom, O(1′), its length being 2.90 Å. It seems certain that the W(2) and W(3) water molecules direct one of their two hydrogen atoms toward the O(2′) and O(1′) atoms respectively, since neither of these carboxyl oxygen has any hydrogen atom. On the other hand, it is difficult to determine distinctly whether the other hydrogen of W(2) is donated to W(3), and that of W(3), to W(2′), or *vice versa*. Judging only from the angles among the hydrogen bonds shown in Fig. 9, the former case seems to be more reasonable. If, in this case, the hydrogen atoms lie exactly on the O···O lines, the H–O–H angles of the W(2) and W(3) water molecules should be about 115 and 102° respectively.

As is shown in Figs. 6 and 7, in the present crystal some close intermolecular contacts are found aside from the hydrogen-bonded ones already discussed. In particular, the approach between the sulfinyl group (S and O(3)) and the carboxyl

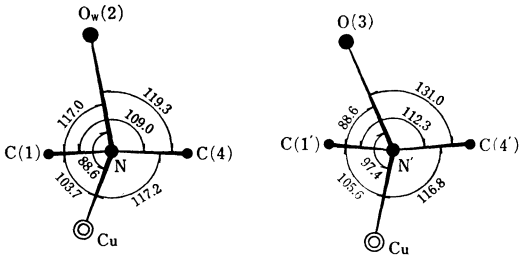


Fig. 8. The bond angles (°) of the imino nitrogen atoms.

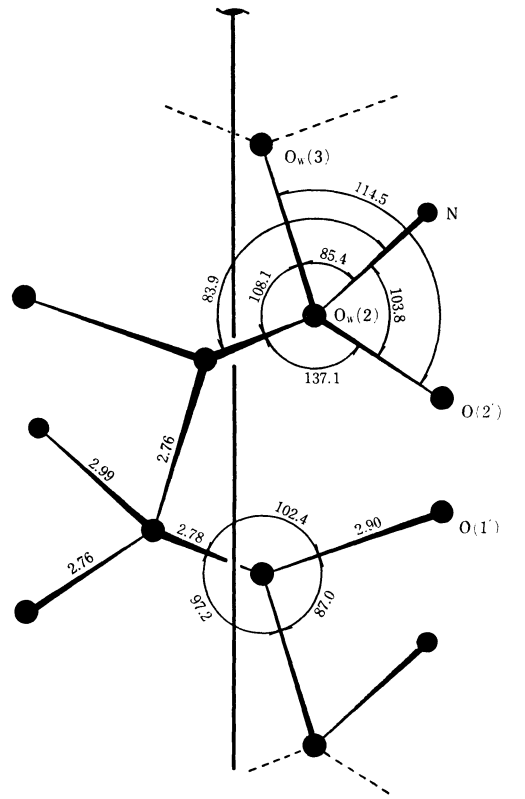


Fig. 9. The hydrogen-bonded spiral chain of the water molecules.

group (O(1′), O(2′), and C(5′)) is of interest in connection with the strong interaction between the two polar groups. The sulfinyl group of a complex molecule is close to the carboxyl groups of the two complex molecules adjoining each other along the *b* axis. The O(3) oxygen atom is in quite close contact with one of the two molecules at four points in all, *i.e.*, O(1′), C(1′), C(2′), and C(5′); the distances of these four atoms from O(3) are, respectively, 3.30, 3.17, 3.25, and 3.06 Å. On the other hand, the sulfinyl group is in contact with the carboxyl oxygen atom, O(2′), of the other complex molecule, the S···O(2′) and O(3)···O(2′) distances being 3.27 and 3.35 Å respectively. The former corresponds nearly to the sum of the van

der Waals radius of sulfur atoms, 1.85 Å, and that of oxygen atoms, 1.40 Å. These close contacts, particularly that of the O(3)···C(5′), seem to suggest that the O(3) sulfinyl oxygen atom and the carboxyl group exert some strong attractive force on each other.

Few atoms are in close contact with the other sulfinyl group (S′ and O(3′)) except for the hydrogen-bonded one which is in close contact with the W(1) water molecule. The sulfinyl groups of this kind are arranged, sufficiently apart from one another, around the two-fold screw axis.

It is interesting to notice that the C(1) carbon atom has no intermolecular contact with a distance smaller than 4.0 Å. This may be partly due to the environment of the other atoms in the same yunainate group, but it does not seem to be attributable to only that cause, since the C(1′) carbon atom, in a similar condition, has contacts with the O(1) and O(3) oxygen atoms of the adjacent molecule. This vacancy around the C(1) atom may be caused by the formation of many hydrogen bonds, which disturb the very close packing of the complex molecules.

Tables of the observed and calculated structure factors are preserved by the Chemical Society of Japan.

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