The Crystal and Molecular Structure of *N*-Salicylidene-*α*-aminoisobutyratoaquocopper(II)

Hiroto Fujimaki,[1] Isao Oonishi, Fumio Muto, Akitsugu Nakahara,* and Yoshimichi Komiyama[2]

Department of Applied Chemistry, Faculty of Engineering, Yamanashi University, Kofu

* *Institute of Chemistry, College of General Education, Osaka University, Machikaneyama, Toyonaka*

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The crystal structure of *N*-salicylidene-α-aminoisobutyratoaquocopper(II), $[Cu\{OOCC(CH_3)_2N{=}CHC_6H_4O\}(H_2O)]$, has been determined from the three-dimensional X-ray diffraction data. The crystals are monoclinic. The cell dimensions are: $a=12.30$, $b=8.45$, $c=23.84$ Å, and $\beta=91.0°$. The space group is $P2_1/c$, with eight formula units in a unit cell. The structure has been refined by the least-squares method, with anisotropic temperature factors, to an R-value of 0.10. The molecular structures of the complex are essentially the same as those of *N*-salicylideneglycinatoaquocopper(II) hemihydrate and tetrahydrate. The environments of the copper(II) ions are square pyramidal, with one long and four short coordination bonds. The average distances of the two copper environments are: Cu–O, 1.912 and 1.945; Cu–N, 1.961; Cu–O(H_2O), 1.989, and Cu–O′ (the bond to the carboxyl oxygen of the adjacent molecule), 2.344 Å. The average bond distances of N=C in the salicylaldimine residue and of N–C in the α-aminoisobutyrate group are 1.271 and 1.504 Å respectively. Neighboring molecules related by a center of symmetry are linked together alternately by hydrogen bonds and coordination bonds (Cu–O′), an infinite chain being formed along the *b*-axis. However, there are no interactions other than van der Waals forces along the *a*- and *c*-axes.

An extensive study has recently been made of the copper(II) complexes with tridentate Schiff bases derived from salicylaldehyde and various amino acids by Nakahara and his co-workers.[3]

Kakudo and his co-workers have determined the structure of *N*-salicylideneglycinatoaquocopper(II) hemihydrate[4] (SGCH) and tetrahydrate (SGCT).[5] Concerning the transamination reaction, Kakudo *et al.* concluded, from their consideration of the bond distances around the nitrogen atom, that the N–C single bond of the salicylaldimine moiety and the N=C double bond of the glycine moiety are electron-rich.

It seemed that it would be of interest to investigate whether bulky groups such as the *t*-methyl group affect the bonds around the nitrogen atom of the Schiff-base linkage. Another point of interest is the coordination configuration about the copper atoms. In SGCH and SGCT, the coordination configurations about the copper atoms are both square pyramidal; however, in SGCH the fifth ligand atom is a 'free' carboxyl oxygen of the adjacent complex, while in SGCT it is a water oxygen atom.

In order to obtain more information about these aspects, we attempted to determine the crystal structure for *N*-salicylidene-α-aminoisobutyratoaquocopper(II), $[Cu\{OOCC(CH_3)_2N{=}CHC_6H_4O\}(H_2O)]$.

Experimental

N-Salicylidene-α-aminoisobutyratoaquocopper(II) was prepared and purified according to the procedure described in a previous paper.[3] The final pure product appears as green plates. The unit-cell dimensions were determined from the higher-order reflections of Weissenberg photographs (Cu*Kα*, $\lambda=1.5412$ Å). The systematic absences were: *h*0*l* for *l* odd and 0*k*0 for *k* odd. Hence, the space group was unequivocally determined. The crystal data are listed in Table 1. Sets of multiple-film equi-inclination Weissenberg photographs were taken about the *b*-axis (0 to 6th layers), the *a*-axis, and the *c*-axis (0th layer). Cu*Kα* radiation was employed throughout. The crystal used was a rod with dimensions of 0.1×0.3×1.0 mm. The intensities were estimated visually with a standard film strip and were converted to $|F_o(hkl)|$ by applying the usual Lorentz, polarization, and spot-shape corrections. No correction was made for absorption and extinction. The range of relative intensities was from 1 to 7200. 2750 independent reflections fell within this range, whereas 1389 others were too weak to be observed.

TABLE 1. CRYSTAL DATA

$[Cu\{OOCC(CH_3)_2N{=}CHC_6H_4O\}(H_2O)]$
Monoclinic
$a=12.30\pm0.01$ Å
$b=\ 8.45\pm0.01$ Å
$c=23.84\pm0.02$ Å
$\beta=91.0\ \pm0.5°$
$Dx=1.54\ g\cdot cm^{-3}$
$Dm=1.54\ g\cdot cm^{-3}$
$Z=8$
Space group $C^5_{2h}-P2_1/c$
Linear absorption coefficient for Cu*Kα*,
$\mu=43.9\ cm^{-1}$

Structure Analysis

The presence of eight formula units in a unit cell of the space group $P2_1/c$ requires that the complex molecules occupy two sets of general positions. Three-dimensional Patterson syntheses were performed. From these Patterson maps, the positions of the Cu atoms

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[1] Present address: Tokyo Laboratory, Kureha Chemistry Industry Co., Ltd., Hyakunin-cho, Shinjuku, Tokyo.

[2] Deceased Aug. 1, 1968.

[3] Y. Nakao, K. Sakurai, and A. Nakahara, This Bulletin **40**, 1536 (1967).

[4] T. Ueki, T. Ashida, Y. Sasada, and M. Kakudo, *Acta Crystallogr.* **22**, 870 (1967).

[5] T. Ueki, T. Ashida, Y. Sasada, and M. Kakudo, *ibid.*, **B25**, 328 (1969).

TABLE 2. ATOMIC PARAMETERS AND THEIR E.S.D.'S[a] ($\times 10^4$)

The expression of the temperature factor is given by
$\exp[-(h^2B_{11}+k^2B_{22}+l^2B_{33}+hkB_{12}+hlB_{13}+klB_{23})]$.

Molecule I

Atom	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cu(1)	1679(1)	240(3)	601(1)	42	210	15	35	−7	14
O(1)	2090(7)	1857(14)	1125(4)	34	197	18	50	−10	−20
O(2)	1273(7)	−1473(14)	91(4)	37	202	14	21	−12	−34
O(3)	1969(8)	−3388(13)	−443(4)	57	137	21	73	−22	−55
W(1)	122(6)	742(13)	742(4)	22	178	16	35	8	−1
N(1)	3099(7)	−829(13)	629(4)	24	83	11	55	−6	−8
C(1)	2053(10)	−2363(20)	−76(5)	24	175	13	−11	6	−10
C(2)	3177(9)	−2199(20)	224(5)	17	181	9	5	−6	−16
C(3)	4018(12)	−1801(22)	−224(6)	54	213	16	−41	16	−8
C(4)	3437(14)	−3758(22)	552(6)	103	117	13	88	−4	12
C(5)	3901(9)	−436(20)	935(5)	30	169	10	11	−7	−2
C(6)	3901(9)	943(18)	1310(5)	24	153	7	−21	−6	−8
C(7)	4880(12)	1177(25)	1628(6)	50	283	12	−19	−6	5
C(8)	4983(14)	2444(26)	2001(7)	84	285	23	10	−15	−33
C(9)	4122(15)	3525(24)	2063(7)	102	193	20	−9	−27	−53
C(10)	3147(13)	3258(22)	1777(6)	84	189	14	−36	−15	−31
C(11)	3017(11)	2017(20)	1386(6)	43	144	13	−9	−1	−11

Molecule II

Atom	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cu(2)	−669(1)	4724(3)	963(1)	46	202	11	40	4	1
O(4)	−430(7)	3046(14)	1488(4)	57	209	7	71	10	4
O(5)	−935(7)	6452(14)	442(4)	47	208	12	108	8	4
O(6)	−2143(8)	8324(15)	223(4)	72	233	13	135	7	38
W(2)	788(6)	4307(13)	652(4)	27	195	10	48	13	−2
N(2)	−1775(7)	5768(14)	1412(4)	30	99	8	38	8	−3
C(12)	−1762(10)	7336(19)	547(6)	39	122	14	6	2	15
C(13)	−2267(11)	7189(21)	1131(6)	40	179	12	32	13	11
C(14)	−3512(12)	6859(25)	1060(7)	38	275	25	84	−6	62
C(15)	−1973(16)	8713(25)	1466(7)	140	188	18	2	4	−48
C(16)	−2080(9)	5330(21)	1898(5)	35	183	10	31	7	−6
C(17)	−1658(10)	3947(20)	2195(5)	30	184	6	11	9	2
C(18)	−2032(13)	3711(24)	2740(6)	76	230	13	−40	4	8
C(19)	−1656(14)	2453(26)	3077(7)	86	274	17	−19	23	27
C(20)	−925(13)	1366(22)	2867(6)	79	187	14	15	8	−9
C(21)	−511(12)	1605(23)	2317(6)	68	228	10	12	−19	20
C(22)	−850(10)	2878(19)	1986(5)	36	141	9	12	−3	−3

[a] e.s.d.'s in parentheses

were easily deduced, but those of lighter atoms could not be fixed. The structure factors, $h0l$, were calculated with the parameters of the Cu atoms. The discrepancy factor, R, was 0.50. The parameters of the eight lighter atoms coordinated to the Cu atoms were deduced from the two-dimensional electron density diagrams, $\rho(x,z)$ and $\rho(y,z)$, the signs of which were calculated from the parameter values of the copper atoms. After two cycles of calculations of the structure factors and the electron densities, $\rho(x,z)$ and $\rho(y,z)$, the parameters of all the atoms were fixed. At this stage, the discrepancy factors, R_{h0l} and R_{0kl}, dropped 0.20 and 0.25 respectively.

The structure thus obtained was refined by a block-diagonal, least-squares method with a HBLS-4 program written by T. Ashida. After three cycles of the refinements, the R-value was 0.14. Six more cycles of the refinements were carried out on the introduction of anisotropic temperature factors. The following weighting scheme was employed:

$$w = 0.2, \quad \text{if } F_0 \leqq F_{min}\ (=15.0);$$

$$w = 1.0, \quad \text{if } F_{min} < F_0 \leqq F_{max}\ (=200.0);$$

and

$$w = F_{max}/F_0, \text{ if } F_0 > F_{max}$$

The final discrepancy factor, $R=\sum||F_o|-|F_c||/\sum|F_o|$, was 0.10 for all the observed reflections. The atomic scattering factors were taken from the International Tables for X-ray Crystallography.[6] The final atomic parameters and their estimated standard deviations are summarized in Table 2. The agreement between the

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[6] International Tables for X-Ray Crystallography, Vol. III, Kynoch Press, Birmingham (1962), p. 202.

observed and calculated structure amplitudes is reasonable.[7]

Description of the Structure and Discussion

The intramolecular bond distances and angles are listed, with their estimated standard deviations, in Tables 3 and 4, and are shown in Figs. 1 and 2. These distances and angles were calculated by using the DAPH program written by T. Ashida.

The *N*-salicylidene-α-aminoisobutyrate groups is coordinated to the copper atom as a tridentate ligand; it is linked to the metal atom through the two oxygen atoms—carboxylic and phenolic—, and the one nitrogen atom of the Schiff-base linkage.

The coordination configurations about the copper atoms in the molecules I and II are both square pyramidal (Figs. 3 and 4). This type of 5-coordinated configuration has been already found in various copper(II) complexes, for instance, di-μ-hydroxobis[dimethylaminecopper(II)]sulfate monohydrate,[8] sodium glycylglycylglycinocuprate monohydrate,[9] and other copper(II) glycine-peptide complexes, pyruvidene-β-alaninatoaquocopper(II) dihydrate,[10] *N*-salicylidene-

TABLE 3. BOND DISTANCES AND THEIR ESTIMATED STANDARD DEVIATIONS[a]

Molecule I		Molecule II	
Cu(1)-O(1)	1.914(10) Å	Cu(2)-O(4)	1.910(10) Å
O(2)	1.948(19)	O(5)	1.941(10)
W(1)	1.996(9)	W(2)	1.982(9)
O(6″)	2.385(11)	O(3′)	2.302(10)
N(1)	1.966(10)	N(2)	1.956(10)
C(1)-O(2)	1.288(17)	C(12)-O(5)	1.289(17)
O(3)	1.235(17)	O(6)	1.226(18)
C(2)	1.551(19)	C(13)	1.539(21)
C(2)-C(3)	1.537(21)	C(13)-C(14)	1.562(23)
C(4)	1.562(21)	C(15)	1.556(24)
N(1)	1.511(16)	N(2)	1.497(18)
C(5)-N(1)	1.262(17)	C(16)-N(2)	1.279(17)
C(6)	1.469(18)	C(17)	1.457(19)
C(7)-C(6)	1.426(21)	C(18)-C(17)	1.399(22)
C(8)	1.395(25)	C(19)	1.406(25)
C(9)-C(8)	1.409(26)	C(20)-C(19)	1.385(25)
C(10)	1.387(25)	C(21)	1.429(23)
C(11)-C(10)	1.410(22)	C(22)-C(21)	1.393(21)
C(6)	1.429(19)	C(17)	1.439(19)
O(1)	1.297(18)	O(4)	1.312(17)

O(3′) and O(6″) are related to O(3) and O(6) through the operations ($-x$, $-y$, $-z$) and ($-x$, $1-y$, $-z$) respectively.

[a] e.s.d.'s × 10^3 in parentheses

TABLE 4. BOND ANGLES AND THEIR ESTIMATED STANDARD DEVIATIONS[a]

Molecule I	
N(1)-Cu(1)-O(1)	94.7(4)°
N(1)-Cu(1)-O(2)	84.2(4)
W(1)-Cu(1)-O(1)	88.9(4)
W(1)-Cu(1)-O(2)	91.5(4)
O(6″)-Cu(1)-O(1)	96.3(4)
O(6″)-Cu(1)-O(2)	85.8(4)
O(6″)-Cu(1)-N(1)	92.1(4)
O(6″)-Cu(1)-W(1)	105.9(4)
Cu(1)-O(2)-C(1)	116.2(9)
O(2)-C(1)-O(3)	125.1(13)
O(3)-C(1)-C(2)	116.9(12)
O(2)-C(1)-C(2)	117.9(12)
C(1)-C(2)-C(3)	107.7(11)
C(1)-C(2)-C(4)	109.2(11)
C(3)-C(2)-C(4)	113.4(12)
C(3)-C(2)-N(1)	109.0(11)
C(4)-C(2)-N(1)	110.0(11)
C(1)-C(2)-N(1)	107.3(10)
C(2)-N(1)-Cu(1)	113.3(8)
C(5)-N(1)-Cu(1)	125.7(9)
N(1)-C(5)-C(6)	123.6(12)
C(5)-C(6)-C(11)	126.1(12)
C(7)-C(6)-C(11)	118.8(12)
C(6)-C(7)-C(8)	120.7(15)
C(7)-C(8)-C(9)	120.3(17)
C(8)-C(9)-C(10)	119.2(17)
C(9)-C(10)-C(11)	122.2(15)
C(10)-C(11)-C(6)	118.6(13)
C(6)-C(11)-O(1)	122.5(13)
C(11)-O(1)-Cu(1)	127.4(9)

Molecule II	
N(2)-Cu(2)-O(3)	94.4(4)°
N(2)-Cu(2)-O(5)	84.2(4)
W(2)-Cu(2)-O(4)	89.1(4)
W(2)-Cu(2)-O(5)	92.2(4)
O(3′)-Cu(2)-O(4)	94.9(4)
O(3′)-Cu(2)-O(5)	85.2(4)
O(3′)-Cu(2)-N(2)	91.8(4)
O(3′)-Cu(2)-W(2)	109.6(4)
Cu(2)-O(5)-C(12)	115.9(9)
O(5)-C(12)-O(6)	124.5(13)
O(6)-C(12)-C(13)	118.0(13)
O(5)-C(12)-C(13)	117.5(12)
C(12)-C(13)-C(14)	109.1(12)
C(12)-C(13)-C(15)	107.6(13)
C(14)-C(13)-C(15)	115.0(13)
C(14)-C(13)-N(2)	107.0(12)
C(15)-C(13)-N(2)	110.2(12)
C(12)-C(13)-N(2)	107.6(11)
C(13)-N(2)-Cu(2)	113.4(8)
C(16)-N(2)-Cu(2)	125.6(9)
N(2)-C(16)-C(17)	124.4(12)
C(16)-C(17)-C(22)	125.3(12)
C(18)-C(17)-C(22)	118.1(13)
C(17)-C(18)-C(19)	121.9(15)
C(18)-C(19)-C(20)	120.3(16)
C(19)-C(20)-C(21)	118.8(15)
C(20)-C(21)-C(22)	121.3(14)
C(21)-C(22)-C(17)	119.4(13)
C(17)-C(22)-O(4)	122.0(12)
C(22)-O(4)-Cu(2)	128.0(9)

a) e.s.d.'s × 10 in parentheses

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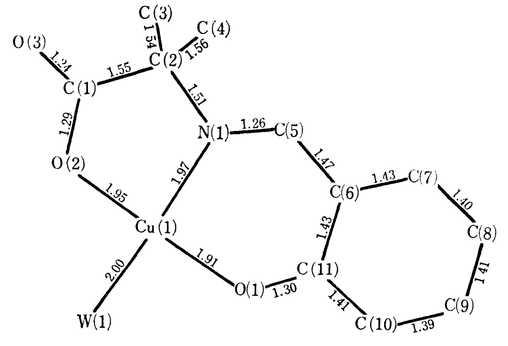
7) A complete list of the observed and calculated structure factors has been submitted to, and is kept as Document No. 7101 at the office of the Bulletin of the Chemical Society of Japan, 1-5 Kanda-Surugadai, Chiyoda-ku, Tokyo. A copy may be secured by citing the Document number and by remitting, in advance, ¥200 for photoprints. Pay by check or money order payble to: The Chemical Society of Japan.

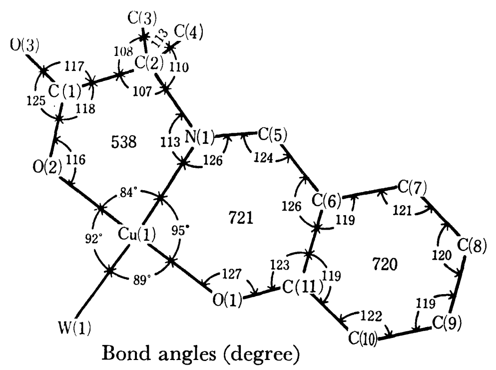
8) Y. Iitaka, K. Simizu, and T. Kwan, *Acta Crystallogr.* **20**, 803 (1966).

9) H. C. Freeman, J. C. Schoone, and J. G. Sime, *ibid.*, **18**, 381 (1965).

10) T. Ueki, T. Ashida, Y. Sasada, and M. Kakudo, *ibid.*, **B24**, 1361 (1968).

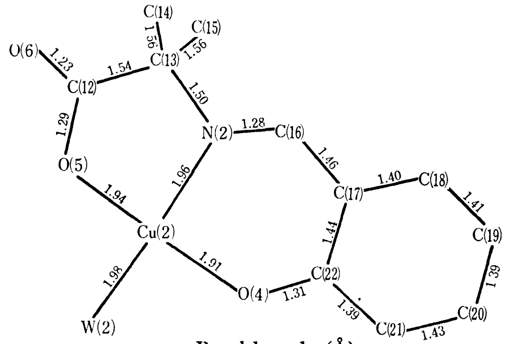


Bond lengths (Å)

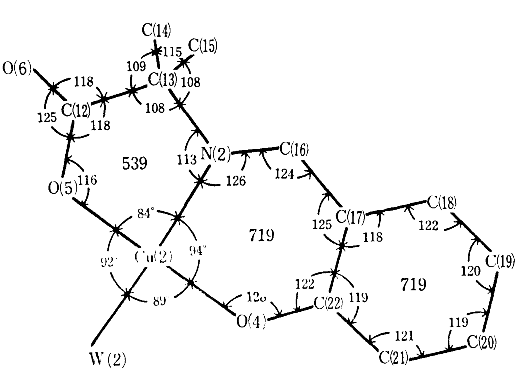


Bond angles (degree)

Fig. 1. Bond lengths and angles in the molecule I.



Bond lengths (Å)



Bond angles (degree)

Fig. 2. Bond lengths and angles in the molecule II.

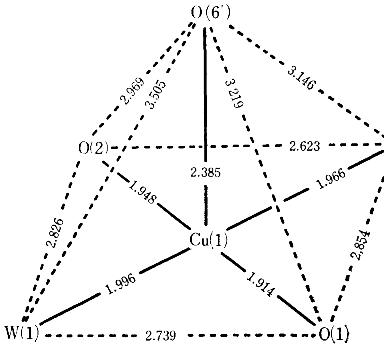


Fig. 3. Five ligand atoms disposed around the copper atom in the molecule I; bond distances are given in Å.

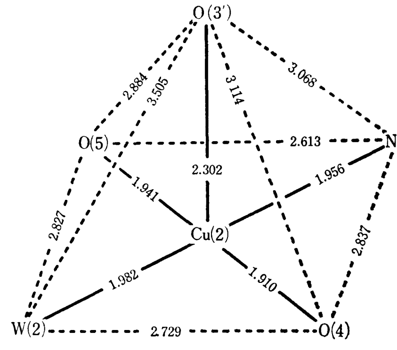


Fig. 4. Five ligand atoms disposed around the copper atom in the molecule II; bond distances are give in Å.

glycinatoaquocopper(II) hemihydrate,$^{4)}$ and tetrahydrate,$^{5)}$ and copper(II) yunainate trihydrate.$^{11)}$

The fifth coordination bond in one molecule is formed by the 'free' carboxyl oxygen atom of the adjacent, crystallographically-independent molecule, as in the case of SGCH, while in SGCT and pyruvidene-β-alaninatoaquocopper(II) dihydrate it is formed by a water oxygen atom.

The Cu–O distances are 1.914 and 1.948 Å in the molecule I, and 1.910 and 1.941 Å in the molecule II, all shorter than those found in SGCH and SGCT. Cu–O distance is 1.966 Å in the molecule I and 1.956 Å in the molecule II, both values being longer than those found in SGCH and SGCT. The Cu–W (W represents the water oxygen atom) distance is 1.966 Å in the molecule I and 1.982 Å in the molecule II. These values are longer than that of 1.965 Å in SGCT, but shorter than that of 2.016 Å in SGCH. The fifth coordinating atoms, O(6″) and O(3′), lie at the apex of the square pyramid at distances of 2.385 and 2.302 Å respectively from the copper atoms in the molecules I and II.

The Cu–O′ bond (O′ means the apical oxygen atom mentioned above) makes an angle of 80.5° in the molecule I, and one of 79.1° in the molecule II, with the plane of the square base. In the present work, no significant displacement of the Cu atom toward the apical oxygen atom was observed such as is found in the cases of SGCH and SGCT. The angles of N(1)–Cu(1)–O(1) and N(1)–Cu(1)–O(2) in the molecule I are 94.7 and 84.2° respectively. The corresponding angles of N(2)–Cu(2)–O(4) and N(2)–Cu(2)–O(5) in the molecule II are 94.4 and 84.2°. These values are in good agreement with those observed in SGCH and SGCT. The other bond lengths and angles generally agree well with those found in SGCH and SGCT, except for the bond distances around the nitrogen

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11) A. Furusaki and Y. Tomiie, This Bulletin, **43**, 736 (1970).

TABLE 5. LEAST-SQUARES PLANES IN THE MOLECULE I

Coefficients of least-squares plane equation, $AX+BY+CZ+D=0$

	A	B	C	$D(\times 10^4)$
P(1) (all the atoms except methyl groups)	2311	6371	−7354	6444
P(2) (square coordination atoms)	1399	6085	−7812	9074
P(3) (Cu-α-aminoisobutyrato)	2761	6099	−7428	4449
P(4) (benzene ring)	3528	5719	−7406	2006
P(5) (Cu-salicylaldimine)	3468	5761	−7402	2186

$X(\text{Å})=ax+cz\cos\beta \quad Y=by \quad Z=cz\sin\beta$

Normal distances from the planes ($\times 10^3$ Å)

	P(1)	P(2)	P(3)	P(4)	P(5)
Cu(1)	192	(198)	69		−17
O(1)	255	120	(107)	(2)	12
O(2)	53	199	− 44		
O(3)	161		(157)		
W(1)	−229	− 75	(−453)		−(687)
N(1)	− 28	−159	− 49		19
C(1)	90		60		
C(2)	− 32		− 9		(105)
C(5)	−130		(−122)	(18)	6
C(6)	− 49			17	11
C(7)	−205			−11	−24
C(8)	−150			−17	−25
C(9)	78			22	23
C(10)	160			−22	−14
C(11)	144			17	22

( ) not included in the least-squares calculations

TABLE 6. LEAST-SQUARES PLANES IN THE MOLECULE II

Coefficients of least-squares plane equation, $AX+BY+CZ+D=0$

	A	B	C	$D(\times 10^4)$
P(1) (all the atoms except methyl groups)	−6167	−6323	−4689	32335
P(2) (square coordination atoms)	−5442	−6122	−5737	34892
P(3) (Cu-α-aminoisobutyrato)	−6765	−5918	−4383	28296
P(4) (benzene ring)	−7338	−5682	−3725	22987
P(5) (Cu-salicylaldimine)	−7228	−5637	−3997	24728

$X(\text{Å})=ax+cz\cos\beta \quad Y=by \quad Z=cz\sin\beta$

Normal distances from the planes ($\times 10^3$ Å)

	P(1)	P(2)	P(3)	P(4)	P(5)
Cu(2)	165	(198)	44		−72
O(4)	308	201	(152)	(−51)	32
O(5)	12	183	− 69		
O(6)	168		(223)		
W(2)	−378	−143	(−643)		(−881)
N(2)	− 44	−205	− 13		1
C(12)	52		70		
C(13)	−123		− 29		(212)
C(16)	−109		(− 34)	(−10)	33
C(17)	− 15			18	42
C(18)	−200			2	−16
C(19)	−181			−23	63
C(20)	74			20	1
C(21)	233			3	25
C(22)	171			−19	25

( ) not included in the least-squares calculations

atoms.

Kakudo and his co-workers[5)] concluded, from their consideration of the bond distances, that the N–C single bond and the N=C double bond in SGCT are electron-rich.

In the present work, the N–C single bonds have normal values, and the N=C double bonds are rather shorter than the normal double bond distance (1.29—1.30 Å), while in SGCT and SGCH the N–C bonds are rather shorter than the normal values (1.47—1.49 Å) and the N=C bonds are normal. Thus, such bulky groups as *t*-methyl will certainly affect the bonds around the nitrogen atom of the Schiff-base linkage.

The molecules may be described in terms of a set

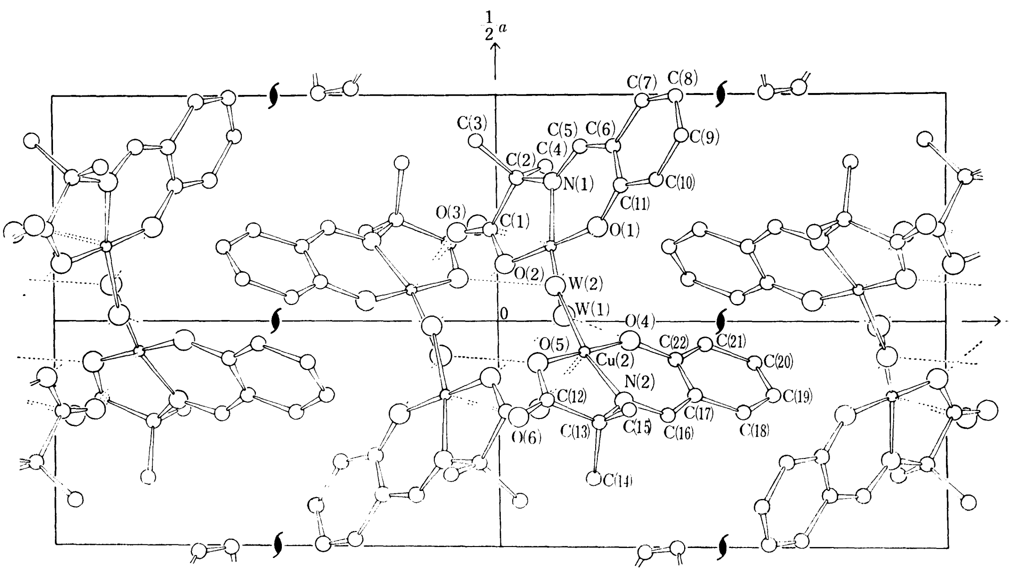

Fig. 5. Projection of the unit cell contents on the (010) plane.

of various plane groups. Tables 5 (molecule I) and 6 (molecule II) list the equations for various least-square planes and the deviations of the atoms therefrom. The largest deviation of any atom from the plane P(1), determined in any of the atoms of the molecule I (except for the two methyl groups) is 0.26 Å for the O(1) atom, whereas it is —0.38 Å for the W(2) atom in the molecule II. Thus, the planarity of the molecule I is slightly better than that of the molecule II, in which several atoms deviate by more than 0.3 Å from the plane. The plane through the α-aminoisobutyrate residue, P(3), and the one containing the salicylaldimine residue, P(5), make dihedral angles of 4.5° in the molecule I and 3.8° in the molecule II; these angles are considerably smaller than the corresponding values of 9.5° in SGCH and 16.5° in SGCT.

The structure projected upon a plane normal to the *b*-axis is shown in Fig. 5. The important intermolecular contacts are listed in Table 7.

The molecules are alternately linked together by the fifth coordination bonds described above to form zigzag chains along the *b*-axis. Two of these zigzag chains are related by the centres of symmetry and are bound laterally by the hydrogen bonds, indicated by dotted lines in Fig. 5. However, there is no interaction other than the van der Waals forces along the *a*- and *c*-axes.

TABLE 7. INTERMOLECULAR CONTACTS NOT EXCEEDING 3.6 Å

O(1)-O(4) (1)	3.385(14) Å	O(3)-C(12)(1)	3.355(17) Å
O(16)(2′)	3.219(15)	O(4)-W(1) (1)	2.731(13)
W(2) (1)	2.839(13)	C(20) (3)	3.596(19)
O(2)-O(5)(1″)	3.352(14)	O(5)-W(2) (2′)	2.697(13)
O(6) (2′)	2.969(14)	O(6)-W(1) (2′)	3.505(14)
W(1) (2)	2.674(13)	W(2) (2′)	3.492(14)
O(3)-O(4) (2)	3.114(14)	N(1) (2′)	3.146(15)
O(5) (2)	2.884(14)	C(1) (2′)	3.433(18)
W(1) (2)	3.472(14)	W(1)-W(2) (1)	3.130(13)
W(2) (1″)	3.586(14)	C(1) (2)	3.377(17)
W(2) (2)	3.505(14)	C(15)(1″)	3.566(21)
N(2) (2)	3.068(14)	W(2)-C(12)(1″)	3.415(17)

1) $x, \quad y, \quad z$ 1′) $x, 1+y, \quad z$ 1″) $x, -1+y, z$
2) $-x, \quad -y, -z$ 2′) $-x, 1-y, -z$
3) $-x, \frac{1}{2}+y, \frac{1}{2}-z$
e.s.d.'s × 10^3 in parentheses

The numerical calculations were carried out on the FACOM231 computer at this University and the HITAC5020E computer at the Computer Center, the University of Tokyo.

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