The Crystal Structure of *N*-Salicylidene-1-phenylalaninatoaquacopper(II) Dimer

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The Schiff base-type complex *N*-salicylidene-1-phenylalaninatoaquacopper(II) dimer crystallizes in the monoclinic system with \( Z = 12 \). The space group is \( C2 \) and the lattice parameters are \( a = 22.053(6) \) Å, \( b = 12.715(3) \) Å, \( c = 16.476(3) \) Å and \( \beta = 89.98(2) \degree \). The compound was studied by single-crystal X-ray diffraction using the counter method and the structure was solved by direct methods combined with Fourier syntheses. Refinement by block-diagonal least-squares technique reduced the \( R \) value to 0.090 for 2091 reflections. The compound consists of dimeric units with copper–copper distances of 3.30(1) Å. The asymmetric unit is made up of one and a half dimeric units. The copper atoms are five-coordinated (4 + 1), a tridentate *N*-salicylidene-1-phenylalanine group and a water molecule forming the coordination plane. The Cu–O distances in these planes vary between 1.89 and 1.99 Å and the Cu–N distances between 1.91 and 1.92 Å. The fifth coordination atom, an oxygen atom from an adjacent salicylidene group, lies at the apex of the square pyramid at 2.41 – 2.45 Å from the copper ion. This contact also serves to join the monomeric complexes into dimeric units. The sixth site is taken up by the phenylalanine ring of the adjacent complex, the closest carbon atom lying at a distance of 3.31 – 3.40 Å from the copper atom. The hydrogen bond distances between the dimeric units vary between 2.66 and 3.10 Å.

The syntheses of several copper complexes formed with salicylaldehyde and amino acids have been reported in the literature.\(^1\)\(^-\)\(^2\) As part of a series of investigations on metal chelate structures where at least one component is an amino acid,\(^3\)\(^-\)\(^5\) we have synthesized and investigated the structure of the Schiff base copper(II) complex formed with salicylaldehyde and 1-phenylalanine.

**Experimental**

Preparation and analyses. The title compound was synthesized according to Laurie\(^1\) by dissolving equimolar quantities of salicylaldehyde (2.12 cm\(^3\)) and copper(II) acetate monohydrate (4.0 g) in a mixture of 1 M hydrochloric acid (30 cm\(^3\)) and ethanol (30 cm\(^3\)). The solution was heated to 50 °C and a stoichiometric amount of 1-phenylalanine (Merck AG) dissolved in a minimum volume of the same mixture was added. The pH of the solution was adjusted to 5 by dropwise addition of 0.2 M sodium hydroxide. Microscopic examination of the fine green powder which separated as product, revealed two different crystalline forms. Thus, in addition to the Schiff base complex, one or other of the possible bis complexes seemed to be formed. However, further concentration of the mother liquid gave green prismatic crystals of one kind only. These were of expected composition and suitable for X-ray data collection. To check the composition, copper was analyzed by electrodeposition. The decomposition temperature and density were measured, and the IR-spectrum and thermogravimetric curve were recorded. All results were in agreement with values given in the literature.\(^1\)

Measurements. Preliminary rotation and equi-inclination Weissenberg photographs taken with Ni-filtered CuK\(_\alpha\) radiation showed that the crystals belong to the monoclinic system. The cell parameters were calculated by least-squares treatment of the 2\( \theta \) values of fourteen high order reflections measured on a SYNTEX-P2\(_1\) diffractometer using graphite monochromated MoK\(_\alpha\) radiation (\( \lambda = 0.71069 \) Å). Crystal data:

\[
\begin{align*}
a & = 22.053(6) \text{ Å} \\
b & = 12.715(3) \\
c & = 16.476(3) \\
\beta & = 89.98(2) \degree \\
Z & = 12
\end{align*}
\]

\( V = 4620.1 \) Å\(^3\) 

\( F.W. = 348.85 \) 

\( D_{\text{obs}} = 1.52 \) g cm\(^{-3}\) 

\( D_{\text{calc}} = 1.52 \) g cm\(^{-3}\) 

Space group \( C2 \)

The intensities of 4447 reflections were collected by the $o$-scan technique with $2o_{\text{min}} = 5.0^\circ$ and $2o_{\text{max}} = 50.0^\circ$ and variable scan rate (2.0 - 15.0 min$^{-1}$). The crystal used in data collection had the approximate dimensions 0.15 x 0.20 x 0.40 mm and it was mounted with the $c$ axis nearly parallel to the spindle axis of the goniometer head. Systematic absences for $hkl$ when $h + k$ odd, $h0l$ when $h$ odd and $0k0$ when $k$ odd indicated $C2$, $Cm$ or $C2/m$ as the possible space groups; and since the compound has

Table 1. Fractional atomic coordinates ($\times 10^4$) and thermal parameters ($\times 10^3$) with standard deviations in parentheses.

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* The anisotropic thermal parameters are of the form exp$[-2\pi^2(la^2b^2)U_{11} + \cdots + 2hka^*b^*U_{12} + \cdots]$.

only one enantiomer the structure determination was based on C2. During the data collection, the intensity of one standard reflection was measured every 60 reflections. The intensities were corrected for Lorentz and polarization effects but not for absorption \( \mu(\text{MoK}\alpha) = 15.1 \text{ cm}^{-1} \).

**Structure determination.** The structure was solved with the MULTAN -74 system\(^7\) and Fourier methods of the X-Ray-76 program system.\(^8\) Atomic coordinates were refined by the block-diagonal least-squares method with isotropic thermal parameters to an \( R \) value of 0.116 (2091 reflections with \( |F_o| > 6.25\sigma|F_o| \) were included) and with anisotropic parameters for copper, oxygen and nitrogen atoms to the final value 0.090 (\( R = \Sigma |F_o| - |F_c|/\Sigma |F_o| \)). The weighting scheme used was \( w = 1/(65.0 + |F_o| + 0.01|F_o|^{1.5}) \) and the average shift/error ratio in the last cycle was 0.24. The positional and thermal parameters are listed in Table 1. The atomic scattering factors were those given by Cromer and Mann.\(^9\) Anomalous dispersion corrections were applied for the copper atom, using the values from International Tables for X-Ray Crystallography given by Cromer and Liberman.\(^10\) Calculations were carried out on a UNIVAC 1108 computer.

A list of the observed and calculated structure factors is obtainable on request from the authors.

**STRUCTURE OF THE COMPOUND**

The geometry and numbering scheme of one asymmetric unit is shown in Fig. 1. The structure

<table>
<thead>
<tr>
<th>Table 2. Interatomic distances (Å) and angles (°).</th>
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* Refers to equivalent position \( -x+1, y, -z \). * The distance CuB—O1C. * The distance CuC—O1B.

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**Table 2. Continued.**

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<td>O4—Cu—N</td>
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<td>C13—C14—C15</td>
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<tr>
<td>C14—C15—C16</td>
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is composed of dimeric units with CuB—CuC and CuA—CuA\(_1\) (I = \( -x+1, y, -z \)) distances of 3.30 Å; the asymmetric unit consists of one and a half dimeric units.

Fig. 1. Stereoscopic view of the asymmetric unit.

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Angles between planes (°)

| 1 and 2 | 10.9 | 10.8 | 12.5 |
| 1 and 3 | 23.8 | 23.4 | 24.2 |
| 1 and 4 | 38.6 | 21.9 | 24.2 |

Table 3. Deviations (Å) of selected atoms from least-squares planes. Atoms indicated with an asterisk were omitted from the calculations.

Table 4. Intermolecular distances below 3.10 Å. 
I = x, y, z II = −x + 1, y, −z and III = −x + 1, y, −z + 1.

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<td>O1B − O1C</td>
<td>2.81(3)</td>
<td>O4B − O3C</td>
<td>2.75(2)</td>
</tr>
<tr>
<td>O4B − O4C</td>
<td>2.96(2)</td>
<td>O2C − O4C</td>
<td>2.74(3)</td>
</tr>
<tr>
<td>O4A − O4B</td>
<td>3.10(2)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The monomeric complexes in the dimeric units are joined together through the coordinated salicylidene oxygen atoms which each in turn occupies the apical position of the square pyramid of the adjacent monomer (Cu−O distance 2.41–2.45 Å). The shortest copper–copper distance between the dimeric units is 5.13 Å. There are hydrogen bonds between the coordinated carboxyl oxygen atom O2A and the water oxygen O4B (2.66 Å) and between the uncoordinated carboxyl oxygen atom O3A and the coordinated water oxygen O4C (2.73 Å). Further contacts are found between O4A and O2B (2.71 Å), between O4A and O4B (3.10 Å), and between O4B and O4C (2.96 Å). Contact distances (<3.10 Å) between asymmetric units, are listed in Table 4.

Each monomeric complex may be described in terms of four approximately planar groups, as was found also in catena-μ-(N-salicylidene-η-tyrosinato-O,O')copper(II):5 the coordination plane, the salicylidene and phenylalanine rings, and the carboxylate group. The angles between the above-mentioned planes in the same complex molecule vary in the range 10.9–38.6, 10.8–23.4 and 12.5–24.2° for complexes A, B and C, respectively. Deviations of selected atoms from least-squares planes and angles between the planes are given in Table 3.

The coordination geometry about the copper atoms is square pyramidal (4+1). The tridentate $N$-salicylidene-$\tau$-phenylalanine group occupies three of the square planar coordination sites, with the oxygen atom of the water molecule completing the plane. The fifth coordinated atom, the salicylidene oxygen of the monomeric partner, lies at the apex of the pyramid. The sixth site is taken up by the phenylalanine ring of the adjacent molecule, with the closest carbon atom lying 3.31 – 3.40 Å from the copper ion. The bond lengths and angles around the copper atoms (Table 2) are in fairly good agreement with the values found in $N$-salicylidene-glycinatoaquacopper(II) tetrahydrate,\cite{11} $N$-salicylidenedeglycinatoaquacopper(II) hemihydrate\cite{12} and catena-$\mu$-($N$-salicylidene-$\tau$-tyrosinato-$O,O'$)-copper(II).\cite{5}

All the copper ions are displaced from the square plane towards the apical salicylidene oxygen atom (0.18 – 0.29 Å), which is a common feature of the square pyramid configuration of the copper(II) ion in the crystalline state. The shift of the nitrogen atom from the carboxyl plane, 0.27 – 0.33 Å, agrees with the values found in $N$-salicylidene-glycinatoaquacopper(II) hemihydrate\cite{12} and catena-$\mu$-($N$-salicylidene-$\tau$-tyrosinato-$O,O'$)-copper(II).\cite{5}

There are no significant differences in molecular dimensions as found in the three monomers, and the bond lengths and angles are, if standard deviations are taken into account, compatible with values given in the literature for analogous compounds.\cite{5,11}

REFERENCES
