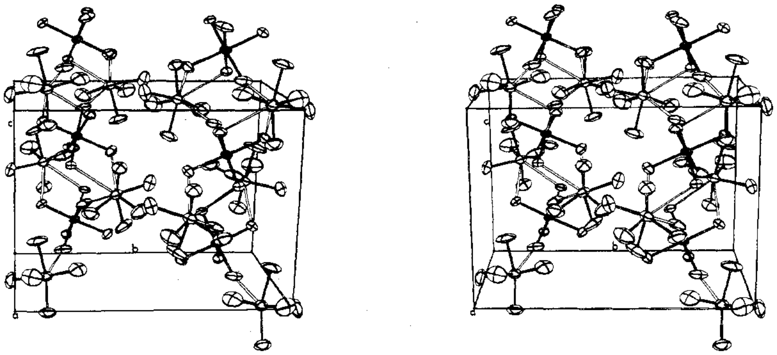

Figure 5. Stereogram of the $[XeF_5^+]_2[PdF_6^{2-}]$ unit cell, showing the packing of linked $[XeF_5^+][AsF_6^-]$-like rings.

$[RuF_6^-]$[5] the cation coordination in anion F ligands is four—not three.

It is not yet clear why certain anions (like PtF_6^- and RuF_6^-) provide four F ligands to coordinate to XeF_5^+, whereas PdF_6^{2-} and AsF_6^- [6] provide three. The similarity of the XeF_5^+ coordination in the PdF_6^{2-} and AsF_6^- salts is striking. As a consequence, essentially the same ring (shown for $[XeF_5]_2$-$[PdF_6]$ in Figure 3) of two XeF_5 groups linked to two MF_6 groups occurs in each structure. It is of relevance to the hypothesis of steric activity of the nonbonding Xe(VI) valence-electron "pairs" that the XeF_5 groups do not share a common axis in the ring but avoid one another (see the stereogram, Figure 4).

It appears probable that the $2XeF_6 \cdot MF_4$ (M = Ge, Sn) complexes reported[3] by Pullen and Cady will prove to be structurally related to $[XeF_5^+]_2[PdF_6^{2-}]$. The ability of XeF_6 to donate F^- to a tetrafluoride to form $[XeF_5^+]_2$-$[PdF_6^{2-}]$ indicates that it is a moderately good base. It remains, however, to be seen if $[XeF_5^+]_3[MF_6^{3-}]$ salts can be derived from the metal trifluorides.

Registry No. $[XeF_5^+]_2[PdF_6^{2-}]$, 39797-61-0.

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Crystal and Molecular Structure of a Pentacoordinate Copper(II) Hippurate Dimer

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The crystal and molecular structure of tetraaquobis(μ-hippurato-*O*)-bis(hippurato-*O*)dicopper has been determined from a single-crystal X-ray diffraction study. The compound $Cu[(C_6H_5)CONHCH_2COO]_2 \cdot 4H_2O$ crystallizes in the monoclinic space group $P2_1/c$ with unit cell constants $a = 7.253$ (1), $b = 40.169$ (3), $c = 7.466$ (1) Å, and $\beta = 102.81$ (1)°. The structure was solved by the heavy-atom method and refined by block-diagonal least-squares methods to a final $R = 0.074$ for the 1693 reflections considered as observed. The compound exists as a dimer with each copper having distorted [4 + 1] tetragonal-pyramidal geometry and bridging is through two carboxyl oxygens with distances 1.93 and 2.37 Å. The hippurate ligands are unexpectedly monodentate.

Introduction

Authentic five-coordinate complexes of the first-row transition metals are known.[1,2] In general, they are formed with simple monatomic or diatomic ligands, such as halide or carbonyl, or they are formed by polydentate ligands which tend to force a particular geometry. Both types of coordination compounds are known for copper(II). The simple five-coordinate type is represented by $CuCl_5^{2-}$ which has been shown to have a trigonal-bipyramidal structure.[3] The more complex species are represented by $\{Cu[N,N'\text{-di-(3-aminopropyl)piperazine}]Cl\}\{ClO_4\}$ which has been found to have a square-pyramidal geometry with N atoms in the base and the chloride in the apical position.[4] In addition, copper(II) forms five-coordinate species with a variety of amino acids and peptide model compounds.[5] In these complexes, the geometry about the copper atom is approximately square pyramidal with amino or peptide nitrogens and carboxyl or peptide oxygens in the base and either a water molecule or a carboxyl oxygen (from a neighboring ligand) in the apical position. Only recently have examples of oxygen-bridged dimers been reported.[6]

In a recent communication,[7] we reported the preparation and characterization of a five-coordinate Cu(II) dimer of the hippurate anion. This report details the complete crystallographic study of this complex.

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Experimental Section

A reasonable single crystal ($0.27 \times 0.21 \times 0.10$ mm^3) of the title compound was grown from a methanol-water mixture as previously described.[7] The crystals grow in layers perpendicular to b^* (40.169-Å axis) and exhibit large intensity differences in ϕ scans at $\chi = 90°$. The crystal ultimately used for this study showed a 1.4:1.0 intensity difference. It was mounted with the cylindrical (111) axis coincident with the ϕ axis of a General Electric XRD-5 diffractometer. The reciprocal lattice showed $2/m$ symmetry with extinctions $k = 2n + 1$ for ($0k0$) and $l = 2n + 1$ for ($h0l$), uniquely characterizing the space group as $P2_1/c$. Lattice constants were determined by a least-squares fit of 19 carefully measured values of the copper Kα_1 and Kα_2 doublet for reflections with $2\theta > 68°$ under fine conditions (1° takeoff angle and 0.05° slit). The resultant lattice constants and their estimated standard deviations are $a = 7.253 \pm 0.001$ Å, $b = 40.169 \pm 0.003$ Å, $c = 7.466 \pm 0.001$ Å, and $\beta = 102.81 \pm 0.01°$. The calculated density of 1.54 g/cm^3, assuming four monomers per unit cell, agrees with the experimental density of 1.50 ± 0.05 g/cm^3 (by flotation methods).

An initial, limited set of intensity data was collected and used to solve the structure, as previously reported. A complete set of intensity data was collected on a General Electric XRD-490 fully automated diffractometer by the stationary-crystal, stationary-counter method using balanced nickel and cobalt filters and copper Kα radiation. A total of 4419 reflections were measured to a 2θ maximum of 140° ($d = 0.82$ Å), and of this total 1693 were considered statistically significant by the criterion

$$[I_{\mathrm{Ni}} - 2\sigma(I_{\mathrm{Ni}})] - [I_{\mathrm{Co}} + 2\sigma(I_{\mathrm{Co}})] > 100$$

where $\sigma(I)$ was based on counting statistics with all reflections counted for 10 sec. The intensities were corrected for Lorentz-polarization effects and Kα_1-Kα_2 splitting[8] in the usual manner, for absorption as a function of ϕ, with a transmission factor of 1.4:1.0 (linear $\mu = 18.0$ cm^{-1} for Cu Kα radiation), and for crystal decay as a linear function of time (uniform 8% overall decay in three standard reflections monitored throughout data collection).

Structure Determination

The coordinates previously determined[7] were used as a starting point for initial least-squares refinement using the new larger set of data. The coordinates and isotropic temperature factors of all 31 nonhydrogen atoms[9] were refined by block-diagonal least squares, using $1/\sigma^2$ weights, to an R value 0.09. A difference electron density map was calculated to locate the hydrogen atoms. Positive regions of electron density were found at the expected hydrogen positions but were rather diffuse. Therefore, the coordinates of the 16 hydrogen atoms (excluding those for water) were calculated, based on expected geometry, and, initially, were included only in the structure factor calculation (with $B = 4.0$ Å^2) but were not varied in the least-squares refinement. Subsequently, the isotropic temperature factors of the nonhydrogen atoms were converted to anisotropic temperature factors. At this point, refinement was resumed with all coordinates (including those for hydrogen atoms) and the anisotropic temperature factors of the nonhydrogens being allowed to vary while the isotropic temperature factors of the hydrogen atoms remained fixed at their initial value of 4.0 Å^2. Refinement was continued until all of the shifts were less than one-tenth the estimated standard deviations of the respective parameters. The final value of the reliability index was $R = 0.074$.

Results and Discussion

The final coordinates and anisotropic temperature factors of the nonhydrogen atoms with estimated standard deviations of each parameter are summarized in Table I. The hydrogen atom coordinates are listed in Table II. The estimated standard deviations (esd's) are less than 0.008 Å for Cu-O distances and 0.02 Å for light-atom bond distances. Likewise, angles involving Cu atoms have esd's less than 0.5° and angles among light atoms have esd's less than 1.5°. Figure 1 shows the coordination distances and angles about the copper atom and Figure 2 is a schematic drawing of the hippurate ions with distances and angles indicated.

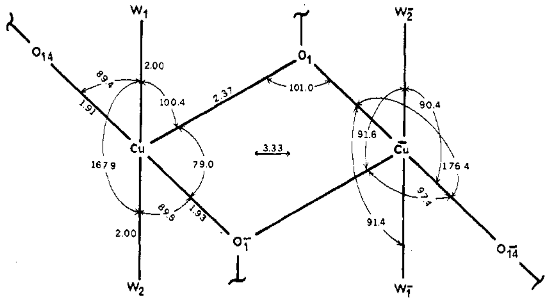

Figure 1. Schematic drawing of the copper coordination sphere with distances and angles indicated.

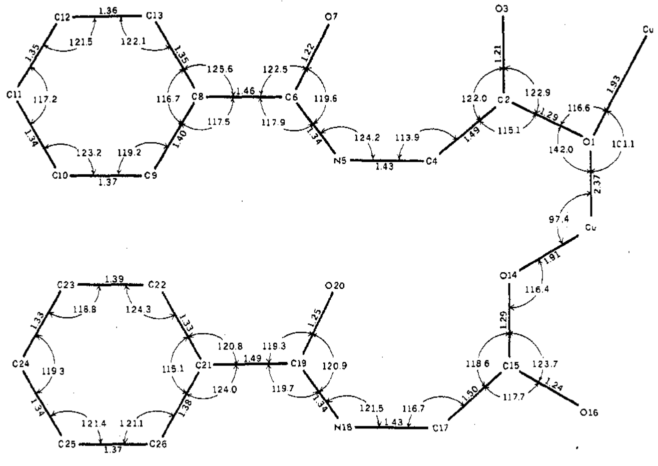

Figure 2. Schematic drawing of the hippurate ions with distances and angles indicated.

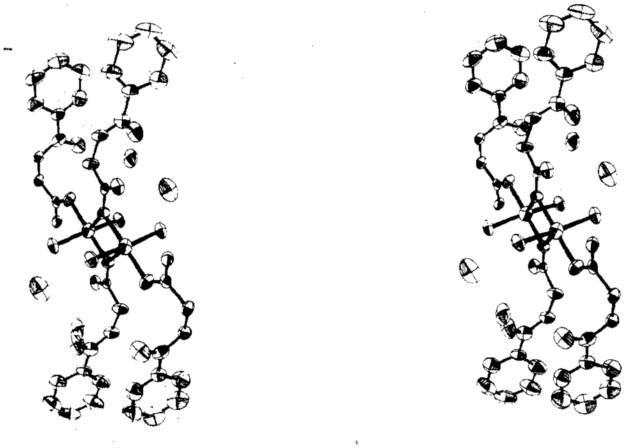

Figure 3. ORTEP stereo drawing of the dimer.

The molecule exists as a dimer, utilizing the crystallographic center of symmetry, as shown in the ORTEP[10] stereo drawing (Figure 3), with each Cu coordinated to five ligands forming a distorted [4 + 1] square-based pyramid. Each copper is uniquely coordinated to one hippurate ion and two water molecules and shares coordination to two other hip-

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[8] A. Tulinsky, C. Worthington, and E. Pignataro, *Acta Crystallogr.*, 12, 623 (1959)

[9] The scattering factors used were from D. Cromer and J. Waber, *Acta Crystallogr.*, 18, 104 (1965), for the nonhydrogens; R. Stewart, E. Davidson, and W. Simpson, *J. Chem. Phys.*, 42, 3175 (1965), for the hydrogens. The reliability index cited throughout, is the usual crystallographic R value, namely, $R = \Sigma \left| k|F_{\mathrm{obsd}}| - |F_{\mathrm{calcd}}| \right| / \Sigma k |F_{\mathrm{obsd}}|$. Among the programs used in this study were the following. LSLAT, K. N. Trueblood, modified by R. L. R. Towns, 1970: a program of least-squares 2θ values of a given set of reflections to determine the best fit of lattice constants. INCON, R. E. Davis, modified by R. L. R. Towns, 1970: program for data reduction. BDLS, R. Sparks and K. N. Trueblood, modified by J. N. Brown, 1969: a block-diagonal least-squares program revised and modified for the PDP-10. FOUR, J. N. Brown, 1970: a three-dimensional Fourier program for the PDP-10.

[10] C. K. Johnson, ORTEP, Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tenn., 1965.

Table I. Fractional Coordinates and Anisotropic Thermal Parameters[a]

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cu	0.3839 (2)	0.4904 (0)	0.2879 (2)	173 (4)	6 (0)	146 (4)	−3 (1)	24 (3)	4 (1)
O1	0.4870 (10)	0.4708 (2)	0.5934 (9)	153 (20)	3 (1)	121 (19)	0 (3)	30 (16)	2 (3)
C2	0.4982 (13)	0.4441 (2)	0.6917 (14)	66 (26)	3 (1)	140 (29)	0 (4)	47 (22)	3 (4)
O3	0.5723 (9)	0.4434 (2)	0.8530 (9)	141 (20)	7 (1)	60 (18)	4 (3)	−9 (15)	2 (3)
C4	0.4129 (16)	0.4139 (3)	0.5914 (15)	272 (39)	3 (1)	159 (33)	2 (5)	124 (29)	6 (4)
N5	0.4762 (13)	0.3833 (2)	0.6833 (12)	253 (29)	3 (1)	133 (26)	0 (4)	106 (22)	6 (4)
C6	0.6250 (16)	0.3659 (3)	0.6571 (15)	233 (37)	5 (1)	136 (33)	1 (5)	54 (28)	0 (5)
O7	0.7150 (11)	0.3759 (2)	0.5477 (11)	235 (26)	12 (1)	228 (26)	12 (4)	134 (21)	17 (4)
C8	0.6764 (16)	0.3359 (3)	0.7657 (15)	254 (40)	4 (1)	139 (33)	3 (5)	62 (28)	−5 (4)
C9	0.8598 (19)	0.3234 (3)	0.7804 (17)	301 (43)	7 (1)	187 (36)	3 (6)	89 (32)	−3 (6)
C10	0.9092 (20)	0.2939 (3)	0.8701 (19)	364 (51)	9 (1)	275 (46)	28 (7)	5 (38)	−3 (6)
C11	0.7905 (21)	0.2761 (3)	0.9462 (18)	570 (61)	5 (1)	222 (43)	13 (7)	90 (40)	6 (6)
C12	0.6149 (21)	0.2886 (3)	0.9341 (20)	516 (60)	7 (1)	363 (51)	18 (7)	254 (44)	13 (6)
C13	0.5585 (19)	0.3174 (3)	0.8426 (19)	373 (50)	5 (1)	316 (46)	9 (6)	146 (38)	14 (6)
O14	0.2597 (10)	0.4506 (2)	0.1851 (10)	171 (21)	5 (1)	135 (20)	−9 (3)	50 (17)	3 (3)
C15	0.1570 (13)	0.4528 (2)	0.0201 (14)	84 (28)	4 (1)	135 (30)	2 (4)	71 (22)	1 (4)
O16	0.1557 (10)	0.4775 (2)	−0.0796 (10)	114 (20)	5 (1)	164 (21)	−2 (3)	11 (16)	12 (3)
C17	0.0353 (15)	0.4239 (3)	−0.0571 (14)	119 (30)	5 (1)	96 (30)	−4 (4)	4 (23)	−1 (4)
N18	0.0441 (12)	0.3952 (2)	0.0584 (12)	138 (25)	4 (1)	177 (28)	−2 (3)	75 (20)	2 (4)
C19	0.1931 (16)	0.3745 (3)	0.0878 (15)	230 (36)	5 (1)	138 (32)	−9 (5)	69 (27)	−4 (5)
O20	0.3279 (11)	0.3793 (2)	0.0114 (11)	295 (27)	9 (1)	170 (23)	10 (4)	155 (20)	12 (4)
C21	0.1993 (15)	0.3456 (3)	0.2140 (15)	209 (35)	4 (1)	150 (32)	−2 (5)	93 (27)	2 (4)
C22	0.3542 (18)	0.3271 (3)	0.2621 (18)	251 (42)	8 (1)	243 (41)	0 (6)	103 (33)	2 (6)
C23	0.3675 (19)	0.2986 (3)	0.3697 (20)	332 (49)	7 (1)	368 (52)	7 (6)	62 (40)	14 (7)
C24	0.2180 (21)	0.2891 (3)	0.4328 (18)	467 (56)	9 (1)	217 (42)	−6 (7)	113 (38)	15 (6)
C25	0.0584 (20)	0.3070 (3)	0.3887 (19)	411 (53)	8 (1)	318 (47)	−15 (7)	225 (40)	10 (7)
C26	0.0477 (18)	0.3352 (3)	0.2842 (19)	305 (47)	6 (1)	308 (45)	−1 (6)	157 (36)	7 (6)
W1	0.6067 (10)	0.4782 (2)	0.1848 (10)	150 (20)	6 (1)	143 (21)	8 (3)	61 (17)	1 (3)
W2	0.1424 (9)	0.5073 (2)	0.3401 (9)	114 (19)	7 (1)	134 (19)	6 (3)	39 (15)	6 (3)
W3	0.7019 (11)	0.4005 (2)	0.1995 (12)	192 (25)	11 (1)	310 (29)	−1 (4)	140 (22)	8 (4)
W4	0.9421 (13)	0.4440 (2)	0.4536 (12)	362 (34)	17 (1)	216 (29)	25 (5)	24 (24)	−6 (5)

[a] Anisotropic thermal parameters $\times 10^4$; estimated standard deviations in parentheses refer to the last decimal place.

Table II. Hydrogen Fractional Coordinates[a]

Atom[b]	x	y	z
H1	0.5677 (32)	0.4962 (11)	0.8186 (29)
H2	0.6358 (34)	0.5857 (11)	0.7310 (30)
H4	0.0602 (34)	0.5626 (11)	0.1882 (29)
H6	−0.0595 (33)	0.4314 (11)	0.1500 (29)
H6′	−0.1816 (33)	0.4878 (11)	0.1393 (29)
H7	−0.2133 (34)	0.3749 (11)	0.2059 (30)
H7′	−0.1657 (33)	0.4299 (11)	0.3382 (29)
H9	0.1598 (33)	0.4416 (11)	0.6440 (29)
H11	0.5085 (33)	0.4171 (11)	0.8918 (29)
H11′	0.5007 (34)	0.3647 (11)	0.7710 (31)
H12	0.2976 (32)	0.3515 (11)	0.8839 (29)
H12′	0.4861 (35)	0.3051 (11)	0.9570 (30)
H14	−0.0180 (33)	0.3473 (11)	0.5749 (29)
H15	−0.1348 (34)	0.2507 (11)	0.3079 (29)
H15′	−0.2763 (34)	0.2905 (10)	0.3077 (30)
H16	−0.0809 (33)	0.1690 (11)	0.4835 (30)
H16′	−0.2072 (34)	0.2107 (11)	0.5036 (30)
H17	0.0730 (34)	0.2642 (11)	0.7724 (31)
HM13	0.3497 (34)	0.2629 (11)	0.6155 (29)
HM13′	0.1933 (34)	0.2010 (11)	0.5448 (30)
HM13″	0.4195 (34)	0.2107 (11)	0.7647 (30)
HO3	0.5164 (35)	0.6571 (11)	0.4974 (31)
HO17	0.1816 (33)	0.1638 (11)	0.8780 (29)

[a] Isotropic thermal parameter fixed at 4.0 Å². [b] Numbers following H refer to bonding atom.

Table III. Intermolecular Contact Distances Less Than 3.5 Å

Atom 1	Atom 2	Eq pos 2	Distance, Å
O1	O14	x, y, z	3.23
O3	O16	$x, y, 1 + z$	3.45
O3	C17	$1 + x, y, 0.5 + z$	3.37
O3	O20	$x, y, 1 + z$	3.48
O3	W1	$x, y, 1 + z$	2.81
O3	W3	$x, y, 1 + z$	3.08
C4	O14	x, y, z	3.33
N5	O20	$x, y, 1 + z$	2.89
O7	W3	x, y, z	2.76
O7	W4	x, y, z	3.35
C10	C26	$1 + x, y, 1 + z$	3.45
C13	O20	$x, y, 1 + z$	3.39
O14	W4	$x - 1, y, z$	3.38
C15	W2	$-x, 1 - y, -z$	3.45
C15	W2	x, y, z	3.26
O16	O16	$-x, 1 - y, -z$	3.31
O16	W1	$1 - x, 1 - y, -z$	2.71
O16	W2	$-x, 1 - y, -z$	2.64
O16	W2	x, y, z	3.37
N18	W3	$x - 1, y, z$	2.91
W1	W1	$1 - x, 1 - y, -z$	3.34
W1	W3	x, y, z	3.19
W1	W4	x, y, z	3.11
W2	W4	$x - 1, y, z$	3.14
W2	W4	$1 - x, 1 - y, -z$	2.64
W3	W4	x, y, z	2.89

purate ions which act as bridges in the dimer. Although the hippurate anion possesses four potential coordination sites, coordination occurs only through the terminal carboxyl oxygen in a monodentate arrangement. This was not anticipated since bidentate coordination through the nitrogen and terminal carboxyl oxygen would have led to a five-membered chelate ring as has been found in similar situations.[11,12]

The coordination distances are similar to those previously reported for copper(II) compounds. The two copper-water bonds are 2.00 Å and only slightly longer than the 1.95 Å reported by Ueki[12] for a water ligand in the basal plane (defined by W1, W2, O14, $\overline{\text{O1}}$). The copper to unique hippurate oxygen bond distance of 1.91 Å is shorter than the 1.95 Å reported by Norton[11] for a carboxyl oxygen and the 1.97 Å observed in pyridine *N*-oxide coordination.[6] The bond angles within the basal plane, varying ±2° about 90°, are as anticipated except for the angle W1-Cu-W2, 167.9°, which deviates considerably from the expected 180°. This

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(11) C. M. Weeks, A. Cooper, and D. A. Norton, *Acta Crystallogr., Sect. B*, **25**, 443 (1969).

(12) T. Ueki, T. Ashida, Y. Sasada, and M. Kakudo, *Acta Crystallogr., Sect. B*, **24**, 1361 (1968).

is apparently due to the copper being raised toward the fifth ligand. The Cu-Cu distance, 3.33 Å, is long enough to preclude any Cu-Cu bonding.

As previously mentioned, dimerization occurs through the bridging of centrosymmetrically related oxygen atoms of the second hippurate ion. Dimers of copper compounds normally exist as one of two types. In the first type,[13] the two unique Cu-O bond distances are nearly equal, 1.98 and 2.04 Å. This is often the case for four-coordinate square-planar copper complexes. Pentacoordinate [4 + 1] complexes demonstrate the second type of dimerization. Usually the fifth ligand has a bond distance about 0.4 Å longer than the basal plane distances[1] whether or not the pentacoordination was the result of dimerization. In these cases, the two unique distances are about 2.0 and 2.4 Å. In this structure the bridging oxygens have Cu-O distances of 1.93 and 2.37 Å, the latter referring to the oxygen in an apical position. These distances closely parallel those in the pyridine *N*-oxide complex,[6] 1.96 and 2.44 Å. Likewise, the O-Cu-O and the Cu-O-Cu angles between bridging oxygens of 79.0 and 101.0°, respectively, closely parallel those previously reported.

A least-squares fit of the four atoms defining the basal plane (W1, W2, O14, $\overline{\mathrm{O1}}$) indicates that the plane is considerably distorted, esd = ±0.13 Å. A similar distortion, ±0.11 Å, was seen in the isoleucine complex.[11] However, the copper atom is 0.08 Å above this plane in the direction of the fifth ligand as would be anticipated. In previous studies,[11,12] the metal ligands have been found to be 0.13 and 0.17 Å out of the basal plane and yet the resulting metal to ligand distances found are 2.41 and 2.44 Å, respectively. That would imply that the longer the metal to ligand bond, the more removed is the metal from the basal plane.

The two unique hippurate anions have identical bond distances and bond angles to within two esd's. The average of the phenyl C-C bond distances (1.36 ± 0.02 Å) is lower than the accepted literature value[14] (1.396 Å) although our average CCC bond angle (120.3 ± 2.3°) compares more favorably to the accepted value of 120.0°. Each phenyl ring is planar to within 0.01 Å as determined by a best least-squares plane fit. The molecular parameters found in the peptide moieties C4-C8 and C17-C21 parallel those results found in hippuric acid itself.[15] The carbonyl double bond is lengthened from the normal 1.20 Å[16] to 1.24 Å and the C-N single bond is shortened from 1.47[17] to 1.34 Å indicative of delocalization found in amide systems.[18] Likewise, the terminal carboxylic acid parameters closely approximate those found in aliphatic acids.[19] The carboxyl oxygen coordinated to the copper in both cases has C-O bond distances, 1.29 Å, significantly longer than the adjacent C-O bond, 1.22 Å.

Peptides and similar molecules have a sufficient number of C-C linkages so that the molecule may assume many conformations by simply rotating about one or more of these bonds. Although only a limited number of structure determinations have been carried out, a preferred molecular conformation exists, at least in the solid state. Invariably, this results from the hydrogen-bonding pattern and it is usually assumed to hold in solution. The conformation of the two hippurate ions reported herein and the conformation of hippuric acid[15] also suggest that the molecule does have a preferred molecular geometry. The dihedral angles between the phenyl rings (C8-C13 and C21-C26) and the peptide moieties (C4-C8 and C17-C21) are 20 and 10°, respectively, averaging to 15°, and compare well with that found in hippuric acid, 14°. Likewise, the dihedral angles between the carboxylic groups (O1-C4 and O14-C17) and the peptide moieties, 81 and 76°, respectively, averaging to 79°, also agree with the value found in the acid, 83°.

The numerous contact distances less than 3.5 Å are tabulated in Table III. In most cases, the atoms involved suggest hydrogen bonding. This will account for the presence of the two waters of hydration (W3 and W4). There are several intermolecular contact distances between dimer groups (excluding W3 and W4) which are apparently due to the close crystal packing of the dimers. No other intermolecular contacts are sufficiently close to warrant further discussion.[20]

Registry No. Cu[$(C_6H_5)CONHCH_2CO_2]_2 \cdot 4H_2O$, 40215-14-3.

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(13) R. S. Sager, R. J. Williams, and W. H. Watson, *Inorg. Chem.*, 6, 951 (1967).

(14) *Chem. Soc., Spec. Publ.*, No. 11 (1958); Supplement (1964).

(15) H. Ringertz, *Acta Crystallogr., Sect. B*, 27, 285 (1971).

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(20) A listing of observed and calculated structure factors will appear following these pages in the microfilm edition of this volume of the journal. Single copies may be obtained from the Business Operations Office, Books and Journals Division, American Chemical Society, 1155 Sixteenth St., N.W., Washington, D. C. 20036. Remit check or money order for $3.00 for photocopy or $2.00 for microfiche, referring to code number INORG-73-1730.