

C(19)—C(20)	1.537 (9)	C(20)—O(7)	1.298 (8)
C(20)—O(8)	1.220 (8)	C(21)—C(22)	1.402 (9)
C(21)—N(5)	1.337 (8)	C(22)—C(23)	1.371 (10)
C(23)—C(24)	1.408 (10)	C(24)—C(25)	1.446 (11)
C(24)—C(31)	1.393 (9)	C(25)—C(26)	1.345 (11)
C(26)—C(27)	1.445 (10)	C(27)—C(28)	1.420 (10)
C(27)—C(32)	1.398 (9)	C(28)—C(29)	1.356 (10)
C(29)—C(30)	1.376 (11)	C(30)—N(6)	1.341 (9)
C(31)—C(32)	1.429 (9)	C(31)—N(5)	1.359 (8)
N(1)—Cu(1)—O(1)	83.6 (2)	N(4)—Cu(2)—O(6)	83.6 (2)
O(1)—Cu(1)—N(2)	93.0 (2)	O(6)—Cu(2)—O(7)	91.7 (2)
O(1)—Cu(1)—N(3)	172.6 (2)	O(6)—Cu(2)—N(5)	91.1 (2)
N(1)—Cu(1)—O(3 ¹)	94.3 (2)	N(4)—Cu(2)—N(6)	101.8 (2)
N(2)—Cu(1)—O(3 ¹)	95.5 (2)	O(7)—Cu(2)—N(6)	98.3 (2)
C(2)—C(1)—O(1)	116.2 (6)	C(18)—C(17)—O(5)	117.6 (6)
O(1)—C(1)—O(2)	124.2 (6)	O(5)—C(17)—O(6)	123.0 (6)
C(1)—C(2)—N(1)	109.8 (5)	C(17)—C(18)—N(4)	107.5 (5)
C(2)—C(3)—C(4)	114.8 (5)	C(18)—C(19)—C(20)	119.7 (5)
C(3)—C(4)—O(4)	118.1 (6)	C(19)—C(20)—O(8)	117.5 (6)
C(6)—C(5)—N(2)	120.5 (7)	C(22)—C(21)—N(5)	121.8 (6)
C(6)—C(7)—C(8)	122.2 (6)	C(22)—C(23)—C(24)	120.6 (6)
C(7)—C(8)—C(15)	115.5 (7)	C(23)—C(24)—C(31)	116.5 (6)
C(8)—C(9)—C(10)	121.6 (7)	C(24)—C(25)—C(26)	121.1 (7)
C(10)—C(11)—C(12)	123.7 (7)	C(26)—C(27)—C(28)	124.6 (7)
C(12)—C(11)—C(16)	117.1 (6)	O(5)—C(17)—C(32)	117.2 (6)
C(12)—C(13)—C(14)	119.6 (7)	C(28)—C(29)—C(30)	120.0 (7)
C(8)—C(15)—C(16)	119.4 (6)	C(24)—C(31)—C(32)	120.4 (6)
C(16)—C(15)—N(2)	117.2 (5)	C(32)—C(31)—N(5)	116.1 (5)
C(11)—C(16)—N(3)	123.0 (6)	C(27)—C(32)—N(6)	122.9 (6)
C(5)—N(2)—C(15)	119.9 (5)	C(21)—N(5)—C(31)	118.7 (5)
N(1)—Cu(1)—N(2)	169.7 (2)	N(4)—Cu(2)—O(7)	85.5 (2)
N(1)—Cu(1)—N(3)	100.0 (2)	N(4)—Cu(2)—N(5)	170.3 (2)
N(2)—Cu(1)—N(3)	82.3 (2)	O(7)—Cu(2)—N(5)	102.8 (2)
O(1)—Cu(1)—O(3 ¹)	91.0 (2)	O(6)—Cu(2)—N(6)	169.0 (2)
N(3)—Cu(1)—O(3 ¹)	95.2 (2)	N(5)—Cu(2)—N(6)	82.2 (2)
C(2)—C(1)—O(2)	119.5 (6)	C(18)—C(17)—O(6)	119.5 (6)
C(1)—C(2)—C(3)	111.6 (5)	C(17)—C(18)—C(19)	111.7 (5)
C(3)—C(2)—N(1)	114.5 (5)	C(19)—C(18)—N(4)	111.0 (5)
C(3)—C(4)—O(3)	117.6 (6)	C(19)—C(20)—O(7)	117.2 (6)
O(3)—C(4)—O(4)	124.3 (6)	O(7)—C(20)—O(8)	125.2 (6)
C(5)—C(6)—C(7)	118.4 (7)	C(21)—C(22)—C(23)	119.0 (6)
C(7)—C(8)—C(9)	125.4 (6)	C(23)—C(24)—C(25)	125.1 (6)
C(9)—C(8)—C(15)	119.1 (6)	C(25)—C(24)—C(31)	118.4 (6)
C(9)—C(10)—C(11)	120.0 (7)	C(25)—C(26)—C(27)	121.4 (7)
C(10)—C(11)—C(16)	119.2 (6)	C(26)—C(27)—C(32)	118.1 (6)
C(11)—C(12)—C(13)	119.6 (7)	C(27)—C(28)—C(29)	119.3 (7)
C(13)—C(14)—N(3)	122.3 (6)	C(29)—C(30)—N(6)	123.3 (6)
C(8)—C(15)—N(2)	123.4 (6)	C(24)—C(31)—N(5)	123.4 (6)
C(11)—C(16)—C(15)	120.5 (6)	C(27)—C(32)—C(31)	120.5 (6)
C(15)—C(16)—N(3)	116.4 (6)	C(31)—C(32)—N(6)	116.6 (6)
C(14)—N(3)—C(16)	118.3 (6)	C(30)—N(6)—C(32)	117.4 (6)

Symmetry code: (i) $-x, y - \frac{1}{2}, 1 - z$.

Non-water H atoms were refined using a riding model and a fixed isotropic U . Water H atoms were found in a ΔF synthesis and refined with constrained distances and isotropic U .

The structure was solved by direct methods and refined by full-matrix least squares. *SHELXTL/PC* (Sheldrick, 1991) was used for all calculations and drawings.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and short intermolecular contacts have been deposited with the IUCr (Reference: HU1069). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Complexation with Ligands Containing a Dipyridylmethane Unit. Structures of a Free Ligand and Two Copper(II) Complexes

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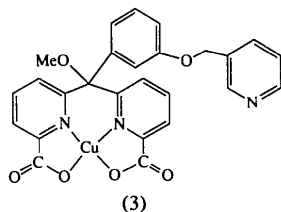
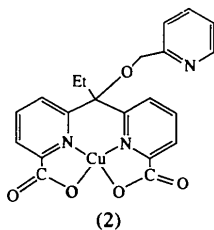
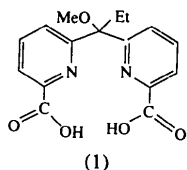
Abstract

The crystal structures of the metal-free ligand 6,6'-(1-methoxypropylidene)bis(2-pyridinecarboxylic acid) monohydrate, $C_{16}H_{16}N_2O_5 \cdot H_2O$ (1), the complex aqua-{6,6'-[1-(2-pyridylmethoxy)propylidene]bis(2-pyridinecarboxylato-*N,O*)}copper(II), $[Cu(C_{21}H_{17}N_3O_5)(H_2O)]$ (2), and the dimeric complex bis[μ -6,6'-{(methoxy)[3-(3-pyridylmethoxy)phenyl]methylene}bis(2-pyridinecarboxylato-*N,N',O,O''*:*N''*)]dicopper(II) monohydrate, $[Cu_2(C_{26}H_{19}N_3O_6)_2] \cdot H_2O$ (3), have been studied by X-ray diffraction. In the crystal of (1) the dipyridylmethane moiety adopts an *anti* conformation, with the two aromatic rings almost perpendicular to each other. This molecular conformation, as well as the crystal packing, are stabilized by hydrogen bonds involving the water molecule. The Cu^{II} ion in (2) has square-pyramidal coordination, with the apex position occupied by the water O atom. The aromatic rings are slightly bent from the plane formed by the two *cis*-positioned N atoms and the two carboxylic O atoms of the ligand molecule. The apical water O atoms link successive molecules into infinite chains *via* hydrogen bonds. The chains, held together by weak electrostatic interactions besides the van der Waals forces, are arranged so as to form a layer structure. (3) also contains a five-coordinate Cu^{II} ion with a square-pyramidal coordination polyhedron. The axial position is occupied by

an N atom, N(22), belonging to the pendant arm of the neighbouring molecule. Thus, (3) forms a dimer although the crystallographic asymmetric unit contains only half the dimeric formula unit. The water O atom, having only a half site occupation, does not coordinate to the Cu ion. In the crystal the dimeric molecules stack, with alternating orientation, along the unique *b* axis.

Comment

The presence of pendant donor groups may extensively alter the properties of metal complexes of macrocyclic ligands, as illustrated by numerous examples (Kaden, 1990; Bernhardt & Lawrance, 1990). In order to study which factors determine the ability of pendant pyridine groups to coordinate apically to a metal atom in a square-planar arrangement formed by a dipyriddyldimethane unit, we have investigated some copper complexes of potentially pentadentate non-cyclic ligands.



We performed X-ray studies on the dipyriddyldimethane-containing compounds (2) and (3) to study the coordination around the Cu^{II} ion and to investigate primarily the effects of the introduction of a pendant arm with an additional donor atom into the structures. Structural work on the metal-free ligand (1) had already become interesting when we were studying related ligands and complexes containing a dipyriddyldimethane unit (Csöreg, Elman, Högberg, Moberg & Nygren, 1988; Moberg, Wärnmark, Csöreg & Ertan, 1991; Csöreg, Ertan, Moberg & Wärnmark, 1991). Here we present the molecular and crystal structures of the ligand (1) and the copper complexes (2) and (3). Crystallographic labelling of the atoms is shown in Fig. 1.

An earlier X-ray study revealed that in the copper(II) complex of ligand (1) [Cu(1)], the bis(2-pyridinecarboxylic acid) moiety is not planar but slightly bent (Csöreg *et al.*, 1988). The dihedral angle (Nardelli, 1983) between the *syn*-positioned aromatic rings is 23.8 (1)°. The present investigation shows

that copper(II) complexes of related ligands, namely (2) and (3), have similar conformations with slightly different inclination angles between the two pyridine rings. The observed dihedral angle between the pyridine rings in (2) is 27.0 (1)°, whereas the corresponding angle in (3) is 20.1 (1)°. Nevertheless, in the absence of the metal ion (Fig. 1*a*), the dipyriddyldimethane moiety adopts an *anti* conformation with the two pyridine rings approximately perpendicular to each other. The observed dihedral angle between the two aromatic rings is 79.9 (1)°. In (1), the carboxyl groups, which are involved in both intra- and intermolecular hydrogen bonds (Table 2), are inclined to the attached ring planes by 3.8 (1) [at C(6)] and 21.4 (1)° [at C(6')]. In (2), the corresponding values are 7.2 (3) and 4.0 (4)° and in (3), 5.0 (2) and 4.9 (3)°. Accordingly, the carboxyl groups in the latter compounds are almost coplanar with the attached pyridine ring planes. In ligand (1), however, the primed carboxylic group significantly deviates from the primed ring plane, in all probability because of the requirements of the hydrogen-bond framework.

In (2) and (3), additional N donor atoms are present. The purpose of the synthesis of these complexes was to design a pendant arm containing a terminal pyridine ring with a structure that would allow the pyridine N atom to coordinate apically to the Cu^{II} ion. The structural investigation showed, however, that in (2) the expected apical coordination was not observed (Fig. 1*b*). Instead of the N atom, the water O atom occupies the unique apical site in a slightly distorted square pyramid. The Cu—N and the Cu—O distances have mean values of 1.964 (1) and 1.942 (2) Å, respectively (Table 4). The Cu^{II} ion in (2) is slightly displaced from the basal plane (−0.218 Å) formed by the four coordinating atoms: two *cis*-positioned N atoms and two carboxylic O atoms, coplanar to within 0.10 (1) Å. The dihedral angles between this basal plane and the pyridine rings are 17.7 (1) (unprimed) and 9.3 (1)° (primed). The dihedral angles between the pyridine ring in the pendant arm and the rings in the dipyriddyldimethane moiety are 30.8 (2)° (with the unprimed ring) and 7.1 (2)° (primed ring). The six-membered chelate ring formed between the two aromatic rings upon metal coordination has a half-boat conformation. The ring-puckering parameters (Cremer & Pople, 1975) are $\Phi = -64.7 (5)^\circ$, $\theta = 97.8 (4)^\circ$ and $Q = 0.442 (4) \text{ \AA}$. The axially situated ethyl group is positioned on the same side of the molecule as the apical water molecule, just as in [Cu(1)] (Csöreg *et al.*, 1988).

The coordination polyhedron around Cu^{II} in (3) (Fig. 1*c*) is also square-pyramidal, but the axial position is now occupied by a donor N atom which belongs to the pendant arm of the neighbouring molecule related by symmetry ($-x + 1, -y, 1 - z$). Thus, (3) forms a dimer and the crystal

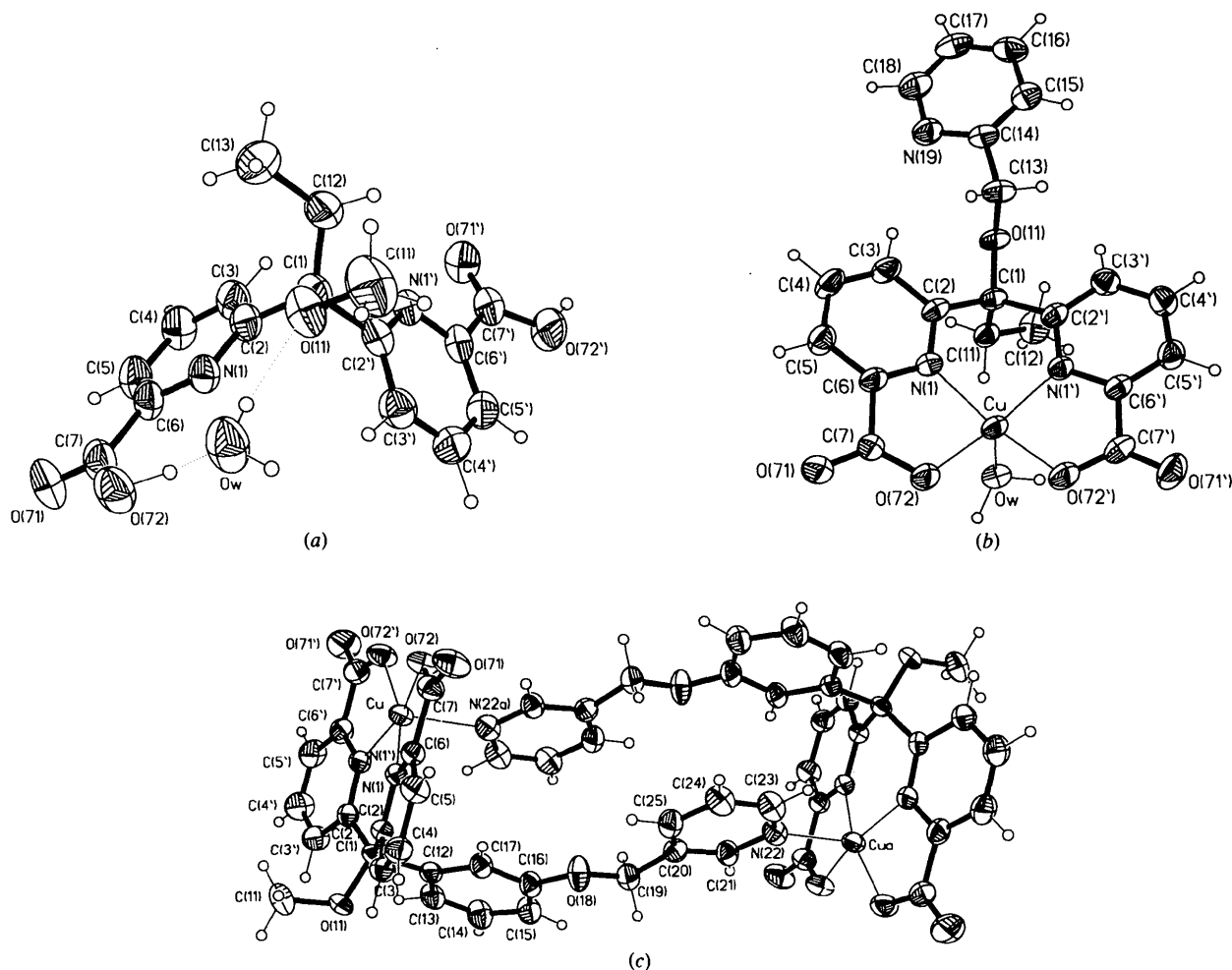


Fig. 1. Perspective views (*SHELXTL/PC*; Sheldrick, 1991) of (a) (1), (b) (2) and (c) (3) [*O*(*W*) atom not included], displaying displacement ellipsoids at the 50% probability level.

water does not coordinate to the metal ion. The Cu^{II} ion in (3) is displaced by $0.285(1) \text{ \AA}$ from the basal plane, which is planar to within $0.03(1) \text{ \AA}$. The dihedral angles between the basal plane and the pyridine rings are $8.6(1)$ (unprimed) and $11.8(1)^\circ$ (primed). The observed dihedral angles between the pyridine ring in the pendant arm and the rings in the dipyridylmethane moiety are $79.4(1)^\circ$ (with the unprimed ring) and $90.2(1)^\circ$ (primed ring). The dihedral angle between the benzene ring and the pyridine ring in the pendant arm is $14.4(1)^\circ$. The distances and angles involving the coordinating atoms around the Cu^{II} ions in (2) and (3) are shown in Tables 4 and 6, respectively. The chelate ring $\text{N}(1)\text{—C}(2)\text{—C}(1)\text{—C}(2')\text{—N}(1')\text{—Cu}$ in (3) has ring-puckering parameters $\Phi = -58.9(5)^\circ$, $\theta = 97.1(5)^\circ$ and $Q = 0.403(3) \text{ \AA}$, indicating a similar half-boat conformation to that observed in (2). The bond lengths and angles are generally in agreement with the expected values.

We have observed that the methoxy C—O bond is approximately coplanar with the nearest C—C and C—N bonds of the pyridine rings in the related dipyridylmethane-containing ligands (Csöreg *et al.*, 1991). This phenomenon has been shown to be due to a stereoelectronic effect (Wärmmark, Moberg, Åkermark & Norrby, 1994). The N—C—C—O and C—C—C—O torsion angles for the present structures are presented in Tables 2, 4 and 6. For comparison, in the $[\text{Cu}(\text{1})]$ complex studied earlier (Csöreg *et al.*, 1988) the mean values of these torsion angles are $\pm 163(1)$ and $\pm 24(1)^\circ$, respectively. The metal coordination leads to a change in the $\text{C}(11)\text{—O}(11)\text{—C}(1)\text{—C}(12)$ torsion angle [involving both C(1) substituents] from $-58(1)^\circ$ in the metal-free ligand (1) to $178(1)^\circ$ in (2) and (3). In $[\text{Cu}(\text{1})]$ the value is $179(1)^\circ$. At the same time, the $\text{C}(12)\text{—C}(1)\text{—C}(2)\text{—N}(1)$ torsion angle changes from $-140(1)$ in (1) to $79(1)$ in (2) and $87(1)^\circ$ in (3), which can be compared with the value of $-82(1)^\circ$ in $[\text{Cu}(\text{1})]$. The sub-

stituent containing the alkoxy O atom seems to prefer the equatorial orientation in the six-membered chelate ring formed upon coordination, unlike the alkyl substituent which seems to prefer the axial position, in accordance to previous findings (Wärmarm *et al.*, 1994). This explains why intramolecular coordination of the pendant-arm N atom to the Cu^{II} ion was not observed, since in this preferred conformation the arm of (2) is too short and that of (3) too long.

Inspection of the non-bonded H-atom distances indicates that there are four possible hydrogen bonds in ligand (1), and the crystal water takes part in three: one to the methoxy O(11), one to the carboxy O(71') and the third from the carboxy O(72) (Table 2). The O···O(W)···O hydrogen bonds form the following angles: O(72)···O(W)···O(11) 110.2(1), O(72)···O(W)···O(71') 129.5(1) and O(11)···O(W)···O(71') 83.9(1)°. Moreover, the hydrate O(W) atom has one more fairly short distance to N(1) [2.985(2) Å], indicating a possible three-centre bond [O(11)···O(W)···N(1)] with a bifurcated H(W2) atom. Inspection of the locality of H(W2) [H(W2)···N(1) 2.38 Å, O(W)—H(W2)···N(1) 126°] and the proximity of N(1) to O(11) [N(1)···O(11) 2.586(3) Å, N(1)···O(W)···O(11) 52.94(5)°] suggest, however, that this latter contact is probably not a hydrogen bond, but a result of the hydrogen-bonding scheme described above. It is worth mentioning that a survey of 1509 N—H···O hydrogen bonds observed in 889 organic crystal structures led Taylor, Kennard & Versichel (1984) to suggest that, in the case of possible three-centre bonds, many of the H···acceptor contacts are due to fortuitous proximity of a proton and an acceptor in the same molecule. They concluded that bonds in which the H···acceptor contact is intramolecular and relatively long should not be described as three-centre bonds (or 'bifurcated').

The fourth hydrogen bond in the crystal of (1) is between the carboxyl groups of different molecules.

The intermolecular hydrogen bonds link the molecules into endless pleated sheets (Fig. 2), perpendicular to the crystallographic *c* direction, and together with the intramolecular hydrogen bonds they also stabilize the molecular conformation. Fig. 2 shows a typical fragment of the hydrogen-bonded layer. It is noteworthy that only identical molecules, belonging to different unit cells, are linked together by hydrogen bonds. Molecules that are related by the crystallographic symmetry operations belong to different sheets, and there are only weaker van der Waals interactions between them.

In the crystal of (2) there are two intermolecular hydrogen bonds (Table 4). The crystal water is involved in both of these bonds: H(W1) is hydrogen bonded to O(71') and H(W2) to O(71). The apically situated water O atom bridges successive oppositely oriented molecules to form infinite chains (Fig. 3). The hydrogen-bonded chains running parallel to the crystallographic *c* direction form a layer structure, which seems to be stabilized by electrostatic interactions (Table 4), besides the ordinary van der Waals forces. The layers are slightly shifted from one to another, so that the pyridyl ring from the pendant arm ($x, -y + \frac{1}{2}, z + \frac{1}{2}$) is placed almost above the Cu^{II} ion. The mean intermolecular distance of the six atoms in the pyridyl ring from the Cu ion is 4.011(1) Å. The shortest intermolecular distance between two Cu^{II} ions is 6.376(1) Å.

The crystal packing in (3) is influenced by dimerization. The dimeric molecules stack, with alternating orientations, along the unique *b* axis (Fig. 4). The intermolecular distance between two Cu^{II} ions in the neighbouring dimers is as short as 4.708(1) Å. The Cu^{II}···Cu^{II} distance within the dimeric 'sandwich'-type

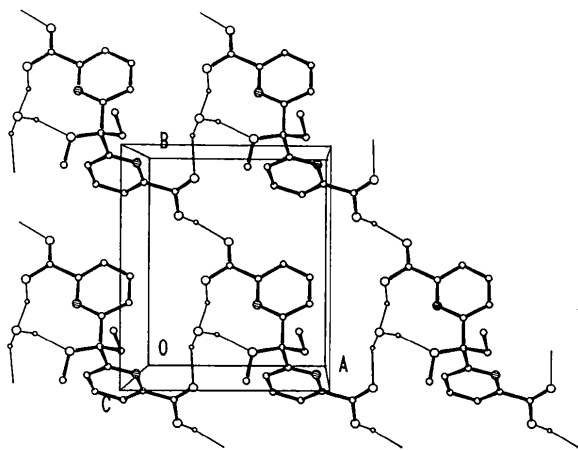


Fig. 2. Packing illustration of (1) showing a typical fragment of the hydrogen-bonded layer (PLUTO; Motherwell & Clegg, 1978).

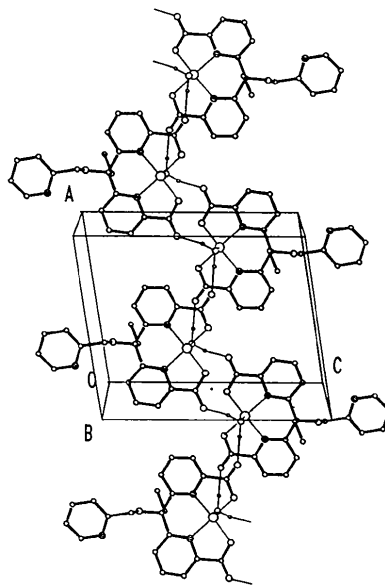


Fig. 3. Packing illustration of (2) showing a typical fragment of the hydrogen-bonded layer (PLUTO; Motherwell & Clegg, 1978).

molecule ($1 - x, -y, 1 - z$) is 11.367 (2) Å. The difference electron density map did not reveal the partially occupied positions of water H atoms. Nevertheless, there are two contacts, O(71)··O(W) [2.88 (1) Å] and O(71)··O(W)($-x, -y, 1 - z$) [3.09 (1) Å], which indicate possible hydrogen bonds involving the crystal water.

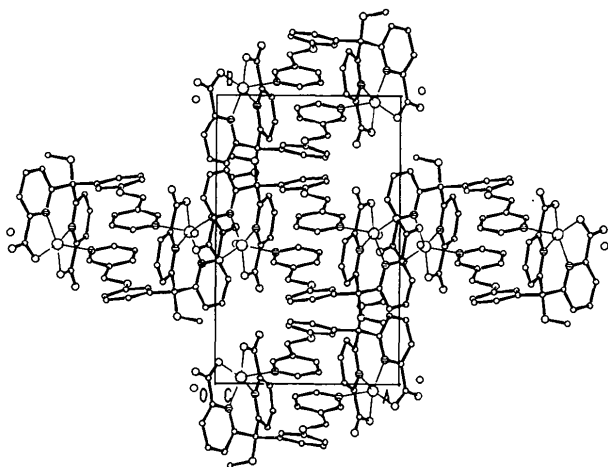


Fig. 4. Packing illustration of the dimeric complex of (3) (PLUTO; Motherwell & Clegg, 1978).

Experimental

Compound (1) was synthesized according to the procedure described by Csöregi *et al.* (1988); (2) and (3) were prepared as described by Adolfsson, Wärnmark & Moberg (1994). Crystals of (1) and (2) were grown as monohydrates from aqueous acetonitrile, while (3) was crystallized from ethanol.

Compound (1)

Crystal data

$C_{16}H_{16}N_2O_5 \cdot H_2O$

$M_r = 334.3$

Monoclinic

$P2_1/c$

$a = 8.2341$ (5) Å

$b = 9.7275$ (5) Å

$c = 20.8316$ (12) Å

$\beta = 92.74$ (1)°

$V = 1666.6$ (2) Å³

$Z = 4$

$D_x = 1.3324$ (2) Mg m⁻³

Cu $K\alpha$ radiation

$\lambda = 1.5418$ Å

Cell parameters from 61 reflections

$\theta = 19.8$ – 34.8 °

$\mu = 0.828$ mm⁻¹

$T = 295$ K

Clean faces

$0.32 \times 0.21 \times 0.12$ mm

Colourless

Data collection

Siemens Stoe AED-2

diffractometer

ω - 2θ scans

Absorption correction:

none

3124 measured reflections

2919 independent reflections

1900 observed reflections

$[I > 3\sigma(I)]$

$\theta_{max} = 69.6$ °

$h = 0 \rightarrow 10$

$k = 0 \rightarrow 11$

$l = 0 \rightarrow 25$

4 standard reflections

frequency: 60 min

intensity decay: <5%

Refinement

Refinement on F

$R = 0.043$

$wR = 0.056$

1900 reflections

231 parameters

Only H-atom U 's refined

$w = 1/[\sigma^2(F) + 0.00052F^2]$

$(\Delta/\sigma)_{max} = 0.03$

$\Delta\rho_{max} = 0.06$ e Å⁻³

$\Delta\rho_{min} = -0.10$ e Å⁻³

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (1)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
N(1)	0.6184 (2)	1.3054 (2)	0.1294 (1)	0.041 (1)
C(2)	0.7607 (3)	1.2486 (3)	0.1471 (1)	0.042 (1)
C(3)	0.9082 (3)	1.3119 (3)	0.1354 (1)	0.052 (1)
C(4)	0.9095 (3)	1.4378 (3)	0.1058 (2)	0.058 (1)
C(5)	0.7612 (3)	1.4991 (3)	0.0880 (1)	0.056 (1)
C(6)	0.6215 (3)	1.4285 (2)	0.1008 (1)	0.045 (1)
C(7)	0.4601 (3)	1.4900 (3)	0.0803 (1)	0.052 (1)
O(71)	0.4529 (2)	1.6043 (2)	0.0565 (1)	0.067 (1)
O(72)	0.3284 (2)	1.4188 (2)	0.0894 (1)	0.062 (1)
C(1)	0.7579 (3)	1.1047 (3)	0.1766 (1)	0.046 (1)
O(11)	0.5958 (2)	1.0834 (2)	0.1967 (1)	0.057 (1)
C(11)	0.5652 (5)	0.9528 (4)	0.2265 (2)	0.082 (1)
C(12)	0.8833 (4)	1.0910 (3)	0.2333 (1)	0.060 (1)
C(13)	0.8515 (5)	1.1905 (4)	0.2881 (2)	0.082 (1)
O(W)	0.2934 (1)	1.1788 (1)	0.1473 (1)	0.069 (1)
N(1')	0.9477 (1)	0.9711 (1)	0.1154 (1)	0.042 (1)
C(2')	0.7926 (1)	1.0056 (1)	0.1217 (1)	0.042 (1)
C(3')	0.6678 (1)	0.9604 (1)	0.0795 (1)	0.051 (1)
C(4')	0.7041 (3)	0.8756 (3)	0.0293 (1)	0.053 (1)
C(5')	0.8639 (3)	0.8347 (2)	0.0230 (1)	0.047 (1)
C(6')	0.9808 (3)	0.8851 (2)	0.0670 (1)	0.042 (1)
C(7')	1.1541 (3)	0.8425 (3)	0.0658 (1)	0.047 (1)
O(71')	1.2655 (2)	0.9046 (2)	0.0919 (1)	0.060 (1)
O(72')	1.1709 (2)	0.7263 (2)	0.0341 (1)	0.068 (1)

Table 2. Selected geometric parameters (Å, °) for (1)

C(2)—C(3)	1.394 (3)	C(1)—C(12)	1.537 (4)
C(2)—C(1)	1.530 (4)	C(1)—C(2')	1.534 (3)
C(7)—O(71)	1.219 (3)	C(2')—C(3')	1.391 (2)
C(7)—O(72)	1.308 (3)	C(7')—O(71')	1.206 (3)
C(1)—O(11)	1.433 (3)	C(7')—O(72')	1.320 (3)
C(2)—C(1)—O(11)	106.4 (2)	C(12)—C(1)—C(2')	112.1 (2)
O(11)—C(1)—C(12)	111.4 (2)	N(1)—C(2)—C(1)	117.5 (2)
C(2)—C(1)—C(12)	111.5 (2)	C(1)—C(2')—N(1')	116.4 (1)
C(2)—C(1)—C(2')	105.6 (2)	O(71)—C(7)—O(72)	121.2 (2)
O(11)—C(1)—C(2')	109.6 (2)	O(71')—C(7')—O(72')	124.0 (2)
O(11)—C(1)—C(2')—C(3')	29.6 (2)		
O(11)—C(1)—C(2')—N(1')	-153.2 (2)		
C(3)—C(2)—C(1)—O(11)	166.3 (2)		
N(1)—C(2)—C(1)—O(11)	-18.2 (3)		
N(1)—C(2)—C(1)—C(12)	-139.8 (2)		
C(3)—C(2)—C(1)—C(12)	44.8 (3)		
C(12)—C(1)—C(2')—C(3')	153.8 (2)		
C(12)—C(1)—C(2')—N(1')	-28.9 (2)		
C(11)—O(11)—C(1)—C(12)	-58.1 (3)		
O(11)—C(1)—C(12)—C(13)	-57.4 (3)		

Hydrogen bonds

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O(72)—H(72)··O(W)	1.09	1.62	2.649 (2)	154
O(W)—H(W2)··O(11)	0.88	1.96	2.806 (2)	161
O(W)—H(W1)··O(71')	0.86	2.10	2.910 (2)	155
O(72')—H(72')··O(71 ⁱⁱ)	0.87	1.77	2.629 (3)	169

Symmetry codes: (i) $x - 1, y, z$; (ii) $1 + x, y - 1, z$.

Compound (2)*Crystal data*[Cu(C₂₁H₁₇N₃O₅)(H₂O)] $M_r = 472.9$

Monoclinic

 $P2_1/c$ $a = 11.0497 (6) \text{ \AA}$ $b = 13.1208 (5) \text{ \AA}$ $c = 13.5252 (5) \text{ \AA}$ $\beta = 98.71 (1)^\circ$ $V = 1938.3 (2) \text{ \AA}^3$ $Z = 4$ $D_x = 1.6207 (2) \text{ Mg m}^{-3}$ Cu $K\alpha$ radiation $\lambda = 1.5418 \text{ \AA}$

Cell parameters from 52

reflections

 $\theta = 13.2\text{--}28.7^\circ$ $\mu = 1.836 \text{ mm}^{-1}$ $T = 295 \text{ K}$

Irregular

 $0.59 \times 0.52 \times 0.11 \text{ mm}$

Blue

*Data collection*Siemens Stoe AED-2
diffractometer ω - 2θ scans

Absorption correction:

by integration from crystal
shape $T_{\min} = 0.48$, $T_{\max} = 0.69$

3756 measured reflections

3381 independent reflections

2687 observed reflections

 $[I > 3\sigma(I)]$ $R_{\text{int}} = 0.0274$ $\theta_{\text{max}} = 69.5^\circ$ $h = 0 \rightarrow 13$ $k = 0 \rightarrow 15$ $l = -16 \rightarrow 16$

5 standard reflections

frequency: 60 min

intensity decay: <2%

*Refinement*Refinement on F $R = 0.048$ $wR = 0.071$

2687 reflections

301 parameters

Only H-atom U 's refined $w = 1/[\sigma^2(F) + 0.00014F^2]$ $(\Delta/\sigma)_{\text{max}} = 0.01$ $\Delta\rho_{\text{max}} = 0.33 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$

Extinction correction: none

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Cu	0.2163 (1)	0.0815 (1)	0.3897 (1)	0.033 (1)
O(W)	0.2112 (3)	-0.0886 (2)	0.4103 (2)	0.042 (1)
N(1)	0.0970 (3)	0.0999 (2)	0.2677 (2)	0.031 (1)
C(2)	0.1153 (4)	0.0891 (3)	0.1714 (3)	0.032 (1)
C(3)	0.0218 (4)	0.1148 (4)	0.0941 (3)	0.040 (1)
C(4)	-0.0870 (4)	0.1536 (4)	0.1164 (3)	0.047 (2)
C(5)	-0.1036 (4)	0.1645 (4)	0.2157 (3)	0.041 (1)
C(6)	-0.0098 (4)	0.1349 (3)	0.2886 (3)	0.034 (1)
C(7)	-0.0191 (4)	0.1383 (4)	0.4000 (3)	0.036 (1)
O(71)	-0.1176 (3)	0.1599 (3)	0.4252 (2)	0.050 (1)
O(72)	0.0789 (3)	0.1151 (3)	0.4577 (2)	0.044 (1)
C(1)	0.2325 (4)	0.0378 (3)	0.1495 (2)	0.031 (1)
C(12)	0.2121 (4)	-0.0784 (3)	0.1593 (3)	0.035 (1)
C(13)	0.3162 (5)	-0.1443 (4)	0.1361 (4)	0.050 (2)
O(11)	0.2448 (3)	0.0529 (2)	0.0470 (2)	0.035 (1)
C(11)	0.2615 (4)	0.1562 (3)	0.0138 (3)	0.041 (1)
C(14)	0.2387 (4)	0.1524 (3)	-0.0988 (3)	0.037 (1)
C(15)	0.3333 (4)	0.1620 (4)	-0.1528 (3)	0.044 (2)
C(16)	0.3093 (5)	0.1541 (4)	-0.2568 (4)	0.052 (2)
C(17)	0.1902 (5)	0.1352 (4)	-0.3004 (3)	0.053 (2)
C(18)	0.1018 (5)	0.1270 (4)	-0.2407 (3)	0.051 (2)
N(19)	0.1222 (4)	0.1350 (3)	-0.1403 (3)	0.044 (1)

N(1')	0.3550 (3)	0.0807 (2)	0.3143 (2)	0.029 (1)
C(2')	0.3537 (4)	0.0662 (3)	0.2156 (3)	0.032 (1)
C(3')	0.4627 (4)	0.0658 (3)	0.1762 (3)	0.040 (1)
C(4')	0.5718 (4)	0.0812 (3)	0.2388 (3)	0.044 (2)
C(5')	0.5732 (4)	0.0970 (3)	0.3400 (3)	0.040 (1)
C(6')	0.4629 (4)	0.0970 (3)	0.3749 (3)	0.032 (1)
C(7')	0.4504 (4)	0.1162 (3)	0.4830 (3)	0.036 (1)
O(71')	0.5417 (3)	0.1372 (3)	0.5437 (2)	0.053 (1)
O(72')	0.3414 (3)	0.1102 (2)	0.5036 (2)	0.042 (1)

Table 4. Selected geometric parameters (\AA , $^\circ$) for (2)

Coordination polyhedron				
Metal-ligand distances				
Cu—N(1)	1.964 (3)	Cu—O(72')	1.944 (3)	
Cu—N(1')	1.965 (3)	Cu—O(W)	2.251 (3)	
Cu—O(72)	1.941 (3)			
Base edges				
N(1)···O(72)	2.615 (4)	N(1)···N(1')	2.836 (4)	
N(1')···O(72')	2.616 (4)	O(72)···O(72')	2.872 (4)	
Face edges				
O(W)···N(1)	3.269 (4)	O(W)···O(72)	3.158 (4)	
O(W)···N(1')	3.126 (4)	O(W)···O(72')	3.148 (4)	
Covalent bonds, angles and torsion angles				
C(2)—C(3)	1.395 (5)	C(1)—C(12)	1.550 (6)	
C(2)—C(1)	1.528 (6)	C(1)—C(2')	1.540 (5)	
C(7)—O(71)	1.222 (5)	C(2')—C(3')	1.388 (6)	
C(7)—O(72)	1.273 (5)	C(7')—O(71')	1.232 (5)	
C(1)—O(11)	1.427 (4)	C(7')—O(72')	1.280 (6)	
O(1W)—Cu—O(72')	97.0 (1)	C(2)—C(1)—O(11)	109.6 (3)	
O(1W)—Cu—O(72)	97.5 (1)	O(11)—C(1)—C(12)	104.8 (3)	
N(1)—Cu—N(1')	92.4 (1)	C(2)—C(1)—C(12)	106.2 (3)	
O(1W)—Cu—N(1)	101.5 (1)	C(2)—C(1)—C(2')	118.0 (3)	
O(1W)—Cu—N(1')	95.5 (1)	O(11)—C(1)—C(2')	109.2 (3)	
O(72)—Cu—N(1')	167.0 (1)	C(12)—C(1)—C(2')	108.3 (3)	
O(72)—Cu—O(72')	95.3 (1)	N(1)—C(2)—C(1)	119.2 (3)	
N(1)—Cu—O(72)	84.1 (1)	C(1)—C(2')—N(1')	119.2 (3)	
N(1')—Cu—O(72')	84.0 (1)	O(71)—C(7)—O(72)	126.6 (4)	
N(1)—Cu—O(72')	161.4 (1)	O(71')—C(7')—O(72')	125.0 (4)	
O(11)—C(1)—C(2')—C(3')		-22.4 (5)		
O(11)—C(1)—C(2')—N(1')		164.7 (3)		
C(3)—C(2)—C(1)—O(11)		18.7 (5)		
N(1)—C(2)—C(1)—O(11)		-168.3 (3)		
N(1)—C(2)—C(1)—C(12)		79.0 (4)		
C(3)—C(2)—C(1)—C(12)		-94.0 (4)		
C(12)—C(1)—C(2')—C(3')		91.2 (4)		
C(12)—C(1)—C(2')—N(1')		-81.8 (4)		
C(11)—O(11)—C(1)—C(12)		178.2 (3)		
O(11)—C(1)—C(12)—C(13)		61.5 (4)		
O(11)—C(11)—C(14)—N(19)		69.3 (5)		
C(1)—O(11)—C(11)—C(14)		-165.6 (3)		
Contact distances				
C(6)···N(19)	3.43 (1)	C(16)···O(72')	3.36 (1)	
C(7)···N(19)	3.44 (1)	C(17)···O(72')	3.36 (1)	
O(71)···C(13)	3.40 (1)	C(5')···O(72')	3.49 (1)	
O(72)···C(17)	3.33 (1)	C(6')···O(71')	3.27 (1)	
C(11)···O(72')	3.20 (1)	C(7')···O(71')	3.35 (1)	
C(15)···N(1')	3.42 (1)			
Hydrogen bonds				
$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
O(W)—H(W1)···O(71')	0.98	1.80	2.781 (4)	179
O(W)—H(W2)···O(71'')	1.01	1.76	2.755 (4)	168
Symmetry codes: (i) $x - 1, y, z$; (ii) $x, \frac{1}{2} - y, \frac{3}{2} + z$.				

Compound (3)*Crystal data*[Cu₂(C₂₆H₁₉N₃O₆)₂].H₂O $M_r = 1084.0$ Cu $K\alpha$ radiation $\lambda = 1.5418 \text{ \AA}$

Monoclinic
 $P2_1/c$
 $a = 10.2034 (5) \text{ \AA}$
 $b = 15.4648 (13) \text{ \AA}$
 $c = 14.7073 (9) \text{ \AA}$
 $\beta = 102.40 (1)^\circ$
 $V = 2266.3 (2) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.5883 (1) \text{ Mg m}^{-3}$

Cell parameters from 38 reflections
 $\theta = 18.4\text{--}29.5^\circ$
 $\mu = 1.688 \text{ mm}^{-1}$
 $T = 295 \text{ K}$
 Irregular
 $0.46 \times 0.24 \times 0.19 \text{ mm}$
 Blue

C(3')	0.2862 (5)	-0.1515 (3)	-0.0336 (3)	0.045 (2)
C(4')	0.3161 (5)	-0.0928 (3)	-0.0968 (3)	0.053 (2)
C(5')	0.2964 (5)	-0.0053 (3)	-0.0860 (3)	0.049 (2)
C(6')	0.2450 (4)	0.0212 (2)	-0.0112 (2)	0.037 (1)
C(7')	0.2131 (4)	0.1145 (3)	0.0063 (3)	0.042 (1)
O(71')	0.2393 (4)	0.1701 (2)	-0.0462 (2)	0.062 (1)
O(72')	0.1575 (3)	0.1264 (2)	0.0756 (2)	0.046 (1)
O(W)	-0.1140 (9)	-0.0176 (6)	0.5023 (6)	0.088 (2)

Table 6. Selected geometric parameters (\AA , $^\circ$) for (3)

Data collection

Siemens Stoe AED-2 diffractometer
 ω - 2θ scans
 Absorption correction: by integration from crystal shape
 $T_{\min} = 0.79$, $T_{\max} = 0.93$
 4385 measured reflections
 3967 independent reflections
 2674 observed reflections
 $[I > 3\sigma(I)]$

$R_{\text{int}} = 0.0308$
 $\theta_{\text{max}} = 69.6^\circ$
 $h = 0 \rightarrow 12$
 $k = 0 \rightarrow 18$
 $l = -17 \rightarrow 17$
 5 standard reflections
 frequency: 60 min
 intensity decay: $<3\%$

Coordination polyhedron

Metal-ligand distances

Cu—N(1)	1.971 (3)	Cu—O(72')	1.945 (3)
Cu—N(1')	1.960 (3)	Cu—N(22)	2.237 (3)
Cu—O(72)	1.943 (3)		

Base edges

N(1)···O(72)	2.618 (4)	N(1)···N(1')	2.824 (5)
N(1')···O(72')	2.630 (4)	O(72)···O(72')	2.858 (5)

Face edges

N(22)···N(1)	3.257 (4)	N(22)···O(72)	3.086 (4)
N(22)···N(1')	3.246 (4)	N(22)···O(72')	3.125 (4)

Covalent bonds, angles and torsion angles

C(2)—C(3)	1.394 (5)	C(1)—C(2')	1.546 (5)
C(2)—C(1)	1.546 (6)	C(2')—C(3')	1.404 (6)
C(7)—O(71)	1.222 (6)	C(7')—O(71')	1.224 (6)
C(7)—O(72)	1.279 (5)	C(7')—O(72')	1.281 (6)
C(1)—O(11)	1.416 (4)	N(1)—C(2)	1.337 (4)
C(1)—C(12)	1.534 (5)	N(1)—C(6)	1.354 (5)
N(1)—Cu—N(1')	91.8 (1)	C(2)—C(1)—O(11)	109.4 (3)
O(72)—Cu—N(1')	164.0 (1)	O(11)—C(1)—C(12)	104.6 (3)
O(72)—Cu—O(72')	94.6 (1)	C(2)—C(1)—C(12)	108.1 (3)
N(1)—Cu—O(72)	84.0 (1)	C(2)—C(1)—C(2')	117.3 (3)
N(1')—Cu—O(72')	84.7 (1)	O(11)—C(1)—C(2')	108.8 (3)
N(1)—Cu—O(72')	162.3 (1)	C(12)—C(1)—C(2')	107.6 (3)
N(22)—Cu—O(72')	96.5 (1)	N(1)—C(2)—C(1)	120.0 (3)
N(22)—Cu—O(72)	94.9 (1)	C(1)—C(2')—N(1')	120.8 (3)
N(22)—Cu—N(1)	101.2 (1)	O(71)—C(7)—O(72)	125.7 (4)
N(22)—Cu—N(1')	101.1 (1)	O(71')—C(7')—O(72')	126.4 (4)

O(11)—C(1)—C(2')—C(3')	-22.9 (5)
O(11)—C(1)—C(2')—N(1')	161.4 (3)
C(3)—C(2)—C(1)—O(11)	29.1 (4)
N(1)—C(2)—C(1)—O(11)	-159.9 (3)
N(1)—C(2)—C(1)—C(12)	86.6 (4)
C(3)—C(2)—C(1)—C(12)	-84.4 (4)
C(12)—C(1)—C(2')—C(3')	89.9 (4)
C(12)—C(1)—C(2')—N(1')	-85.8 (4)
C(11)—O(11)—C(1)—C(12)	177.7 (3)
O(11)—C(1)—C(12)—C(13)	59.8 (4)
O(16)—O(18)—C(19)—C(20)	166.3 (3)
O(18)—C(19)—C(20)—C(21)	175.5 (3)
C(19)—C(20)—C(21)—N(22)	-177.8 (4)

Contact distances

C(3)···O(72)	3.22 (1)	C(19)···O(71')	3.35 (1)
C(4)···O(72)	3.13 (1)	N(22)···C(17)	3.40 (1)
O(71)···C(24)	3.37 (1)	C(23)···N(1')	3.45 (1)
O(72)···C(21)	3.16 (1)	C(24)···O(71)	3.37 (1)
O(11)···C(25)	3.37 (1)	C(7)···O(W)	3.37 (1)
C(17)···N(22)	3.40 (1)	C(19)···O(W)	3.41 (1)

Data reduction included corrections for background, Lorentz, polarization and absorption effects, although the rather low absorption effects for (1) were ignored. Absorption corrections for (2) and (3) were carried out by numerical integration, using the program *STOEABS* (Norrestam, 1989). The structure of the metal-free ligand (1) was solved by direct methods (Sheldrick, 1990), whereas the Cu complexes were solved by a combination of heavy-atom and direct methods, using the programs *SHELXS90* (Sheldrick, 1990) for (2) and *DIRDIF* (Beurskens *et al.*, 1992) for (3). The structural models were refined by full-matrix least-squares treatment using the *SHELXL76* (Sheldrick, 1976) program system. All H atoms,

Refinement

Refinement on F^2
 $R = 0.042$
 $wR = 0.059$
 2674 reflections
 350 parameters
 Only H-atom U 's refined
 $w = 1/[\sigma^2(F) + 0.00107F^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.11$

$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
 Extinction correction: none
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 5. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2) for (3)

U_{iso} for O(W); $U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$ for other atoms.

	x	y	z	$U_{\text{iso}}/U_{\text{eq}}$
Cu	0.1411 (1)	0.0228 (1)	0.1471 (1)	0.032 (1)
N(1)	0.0752 (3)	-0.0869 (2)	0.1888 (2)	0.028 (1)
C(2)	0.1074 (4)	-0.1682 (2)	0.1716 (2)	0.028 (1)
C(3)	0.0529 (4)	-0.2382 (2)	0.2104 (3)	0.034 (1)
C(4)	-0.0362 (4)	-0.2234 (2)	0.2666 (3)	0.037 (1)
C(5)	-0.0678 (4)	-0.1390 (2)	0.2855 (3)	0.038 (1)
C(6)	-0.0112 (4)	-0.0728 (2)	0.2454 (3)	0.031 (1)
C(7)	-0.0366 (4)	0.0215 (2)	0.2625 (3)	0.035 (1)
O(71)	-0.1053 (3)	0.0408 (2)	0.3181 (2)	0.052 (1)
O(72)	0.0192 (3)	0.0749 (2)	0.2163 (2)	0.041 (1)
C(1)	0.2220 (4)	-0.1857 (2)	0.1202 (2)	0.030 (1)
O(11)	0.2079 (3)	-0.2701 (2)	0.0819 (2)	0.036 (1)
C(11)	0.0852 (5)	-0.2860 (3)	0.0130 (3)	0.054 (2)
C(12)	0.3550 (4)	-0.1881 (2)	0.1926 (2)	0.030 (1)
C(13)	0.4731 (4)	-0.2080 (3)	0.1630 (3)	0.039 (1)
C(14)	0.5923 (4)	-0.2153 (3)	0.2265 (3)	0.046 (1)
C(15)	0.6009 (4)	-0.2023 (3)	0.3211 (3)	0.042 (1)
C(16)	0.4855 (4)	-0.1780 (2)	0.3497 (2)	0.036 (1)
C(17)	0.3632 (4)	-0.1723 (2)	0.2862 (2)	0.033 (1)
O(18)	0.4803 (3)	-0.1579 (2)	0.4399 (2)	0.047 (1)
C(19)	0.6060 (4)	-0.1385 (3)	0.5010 (3)	0.040 (1)
C(20)	0.5778 (4)	-0.0977 (2)	0.5873 (3)	0.036 (1)
C(21)	0.6863 (4)	-0.0790 (2)	0.6597 (2)	0.035 (1)
N(22)	0.6746 (3)	-0.0450 (2)	0.7417 (2)	0.37 (1)
C(23)	0.5507 (4)	-0.0286 (3)	0.7531 (3)	0.043 (1)
C(24)	0.4371 (4)	-0.0441 (3)	0.6855 (3)	0.045 (1)
C(25)	0.4504 (4)	-0.0792 (3)	0.6005 (3)	0.040 (1)
N(1')	0.2172 (3)	-0.0364 (2)	0.0524 (2)	0.033 (1)
C(2')	0.2388 (4)	-0.1210 (2)	0.0434 (2)	0.032 (1)

except those on the methyl groups in (1), were located from difference electron density (Δ) maps. In (2), ten H atoms, including the water H atoms, were found from the Δ map. The remaining H atoms, and all H atoms in (3), were given geometrically assumed positions (C—H = 1.00 Å) which were recalculated after each refinement cycle. Refinement of the water O atom in (3) with full site occupancy gave rise to unacceptably high displacement parameters ($U_{iso} = 0.205 \text{ \AA}^2$) for that atom. Moreover, there is an unusually short O(W)··O(W) contact distance of 2.40 (1) Å, indicating possible disorder for the water O atom. Subsequent refinement of the site occupation factor and the displacement parameter of O(W) in consecutive cycles yielded an approximately 50% occupancy for each O(W) position in the unit cell and a decrease of the vibrational parameter to $U_{iso} = 0.088 \text{ \AA}^2$. The partially occupied water H-atom positions, however, could not be located. In the last stage of the refinement the positions of the non-H atoms were refined together with their anisotropic displacement parameters, and isotropic vibrational parameters were refined for the O(W) position. The methyl groups were treated as rigid with free rotation around the O—C_{methyl} bond (Sheldrick, 1976). In the final calculation the mean shift/e.s.d. of the three rotation parameters of the methyl C(11) group of complex (3) was 0.51 (3). Six and 15 low θ reflections with F_o systematically lower than F_c , indicating extinction, were excluded from the last refinements of (1) and (3), respectively. The geometric features were calculated with the program PARST (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry, as well as stereo drawings of the structures, have been deposited with the IUCr (Reference: AB1160). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(benzoato-O)bis(thiourea-S)zinc(II)

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Abstract

The title complex crystallizes from an aqueous solution containing sodium benzoate, zinc sulfate and thiourea in the ratio 2:1:2. The structure is molecular and contains two crystallographically different [Zn(C₆H₅COO)₂{CS(NH₂)₂}₂] molecules. The coordination geometry of each Zn atom, by one O atom from each benzoate anion and by one S atom from each thiourea ligand, is that of a deformed tetrahedron [Zn—O 1.964 (2) and 1.963 (2) Å; Zn—S 2.368 (1) and 2.366 (1) Å; angles 103.25 (9)–126.6 (2)°]. The Zn··O distances to the non-coordinated O atoms are long: 2.957 (3) (×2) and 2.955 (3) Å (×2). Most of the H atoms of the amidic groups are involved in hydrogen bonds of the N—H··O type.

Comment

As a part of our study of zinc carboxylates with or without additional ligands (Potočňák, Dunaj-Jurčo & Černák, 1993; Potočňák, Dunaj-Jurčo, Petříček & Černák, 1994), we prepared the title complex, (I). The