Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Cu	0.0243 (1)	0.1307 (1)	0.1693 (1)	0.031 (1)
N(1)	0.0361 (5)	−0.0698 (7)	0.2639 (3)	0.024 (2)
O(2)	0.1434 (5)	0.2435 (6)	0.2710 (3)	0.038 (2)
C(1)	−0.0331 (6)	−0.0641 (9)	0.3222 (4)	0.027 (2)
N(2)	−0.0050 (7)	0.3322 (8)	0.0832 (4)	0.037 (3)
O(1*W*)	0.1697 (5)	0.0325 (8)	0.1189 (4)	0.046 (3)
O(3)	−0.0926 (5)	−0.0155 (7)	0.0826 (3)	0.046 (2)
C(2)	−0.0204 (7)	−0.2024 (10)	0.3851 (5)	0.038 (3)
C(6)	−0.1111 (6)	0.0718 (10)	0.3166 (4)	0.035 (3)
C(5)	−0.1734 (7)	0.0689 (12)	0.3754 (5)	0.046 (3)
C(3)	−0.0900 (8)	−0.1951 (12)	0.4400 (5)	0.049 (4)
C(21)	0.0601 (8)	−0.3565 (12)	0.4005 (5)	0.053 (4)
C(4)	−0.1644 (7)	−0.0621 (13)	0.4357 (5)	0.050 (4)
C(61)	−0.1309 (8)	0.2243 (11)	0.2527 (6)	0.053 (4)
C(8)	0.2051 (6)	0.1342 (12)	0.3293 (4)	0.034 (2)
O(1)	0.2977 (5)	0.1689 (8)	0.3915 (3)	0.054 (2)
O(4)	−0.1225 (5)	−0.2963 (7)	0.0504 (3)	0.049 (2)
C(7)	0.1642 (6)	−0.0540 (9)	0.3209 (4)	0.031 (3)
C(10)	−0.0778 (7)	−0.1765 (11)	0.1040 (5)	0.036 (3)
C(9)	0.0029 (7)	−0.2242 (9)	0.2002 (4)	0.032 (3)

Table 2. *Selected geometric parameters* (Å, °)

Cu—N(1)	2.109 (5)	O(3)—C(10)	1.278 (10)
Cu—O(2)	1.936 (4)	C(2)—C(3)	1.428 (14)
Cu—N(2)	2.000 (6)	C(2)—C(21)	1.509 (12)
Cu—O(1*W*)	2.345 (7)	C(6)—C(5)	1.405 (13)
Cu—O(3)	1.940 (5)	C(6)—C(61)	1.505 (11)
N(1)—C(1)	1.470 (11)	C(5)—C(4)	1.360 (13)
N(1)—C(7)	1.512 (8)	C(3)—C(4)	1.362 (14)
N(1)—C(9)	1.509 (9)	C(8)—O(1)	1.232 (7)
O(2)—C(8)	1.270 (8)	C(8)—C(7)	1.521 (11)
C(1)—C(2)	1.421 (10)	O(4)—C(10)	1.233 (9)
C(1)—C(6)	1.403 (11)	C(10)—C(9)	1.520 (9)
N(1)—Cu—O(2)	84.9 (2)	C(2)—C(1)—C(6)	119.8 (8)
N(1)—Cu—N(2)	172.4 (3)	Cu—O(3)—C(10)	112.2 (4)
O(2)—Cu—N(2)	96.1 (2)	C(1)—C(2)—C(3)	117.1 (7)
N(1)—Cu—O(1*W*)	98.1 (2)	C(1)—C(2)—C(21)	126.5 (8)
O(2)—Cu—O(1*W*)	88.7 (2)	C(3)—C(2)—C(21)	116.4 (7)
N(2)—Cu—O(1*W*)	89.4 (3)	C(1)—C(6)—C(5)	118.6 (7)
N(1)—Cu—O(3)	85.0 (2)	C(1)—C(6)—C(61)	124.1 (8)
O(2)—Cu—O(3)	169.9 (2)	C(5)—C(6)—C(61)	117.3 (8)
N(2)—Cu—O(3)	94.0 (2)	C(6)—C(5)—C(4)	123.2 (8)
O(1*W*)—Cu—O(3)	92.4 (2)	C(2)—C(3)—C(4)	123.2 (8)
Cu—N(1)—C(1)	120.9 (4)	C(5)—C(4)—C(3)	118.0 (9)
Cu—N(1)—C(7)	98.3 (4)	O(2)—C(8)—O(1)	124.5 (8)
C(1)—N(1)—C(7)	110.6 (5)	O(2)—C(8)—C(7)	118.3 (5)
Cu—N(1)—C(9)	100.1 (4)	O(1)—C(8)—C(7)	117.2 (6)
C(1)—N(1)—C(9)	111.1 (6)	N(1)—C(7)—C(8)	111.7 (5)
C(7)—N(1)—C(9)	115.2 (6)	O(3)—C(10)—O(4)	123.9 (6)
Cu—O(2)—C(8)	111.9 (5)	O(3)—C(10)—C(9)	118.3 (6)
N(1)—C(1)—C(2)	118.2 (6)	O(4)—C(10)—C(9)	117.8 (7)
N(1)—C(1)—C(6)	122.0 (6)	N(1)—C(9)—C(10)	113.0 (6)

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*; *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1039). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diaqua[*N*-(2-ethylphenyl)-*N*-(carboxymethyl)glycinato-*N*,*O*,*O*″]copper(II)

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Abstract

The (4+1) coordination environment around Cu^{II} in the title compound, [Cu($C_{12}H_{13}NO_4$)(H_2O)$_2$] [diaqua-

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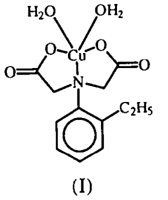
(2-ethylphenyliminodiacetato-*N*,*O*,*O''*)copper(II)], can be described as square pyramidal, distorted towards trigonal bipyramidal. The basal coordination positions are occupied by one N and two O atoms from the doubly deprotonated ligand, and one O atom of a water molecule, the apical position being occupied by the other water molecule.

Comment

The crystal structure of the title compound, (I), consists of discrete neutral [Cu*L*(H_2O)$_2$] molecules, where H_2L is *N*-(2-ethylphenyl)-*N*-(carboxymethyl)glycine. The Cu atom is at the centre of a five-coordinate $CuNO_4$ chromophore and forms four short bonds of *ca* 2.0 Å with one N and two O atoms from the doubly deprotonated ligand L^{2-}, and one O atom of the water molecule, while another water molecule is coordinated in the axial position at a longer distance [2.253 (3) Å]. Bond distances and angles are comparable with those reported for μ-pyrazine-bis[aqua(2,6-xylyliminodiacetato)copper(II)] (Suárez-Varela *et al.*, 1994), [*N*-(*o*-tolyl)iminodiacetato-*N*,*O*,*O*]copper(II) (Chen *et al.*, 1992), diaqua[*N*-(3-methoxyphenyl)iminodiacetato-*N*,*O*,*O*]copper(II) (Liu *et al.*, 1991) and diaqua[(*N*-*p*-methylphenyl)iminodiacetato-*O*,*O*,*N*]copper(II) (Wang, Tian, Miao, Liu & Chen, 1992).



(I)

The angles of the coordination polyhedron are not dissimilar to those of a square-based pyramidal geometry. Distortion can be measured by using the index τ (Addison, Rao, Reedijk, Rijn & Verschoor, 1984), which is unity for perfect trigonal-bipyramidal geometry and zero for square-pyramidal geometry. The calculated value for [Cu*L*(H_2O)$_2$] is 0.203. This result shows that the coordination polyhedron is close to square pyramidal; in this geometry, the basal plane is defined by N(1), O(2), O(6) and O(3), and the apical position is occupied by O(7) from a water molecule, in accordance the relatively large Cu—O(7) distance. Deviations from planarity up to 0.180 (3) Å are observed for the equatorial donor atoms. As usual, the Cu atom is 0.215 (1) Å outside of the basal plane towards the O(7) axial donor.

The tridentate behaviour of the ligand results in the formation of two five-membered rings which have in common the Cu—N(1) bond. The dihedral angle between the mean least-squares planes of these two rings is 17.5 (1)°. The benzene ring is essentially planar, as expected. The dihedral angle between the benzene plane and the mean equatorial plane around the Cu atom is 86.5 (1)°.

Short non-bonding O···O contacts (< 3.0 Å) involving water molecules and O atoms of the carboxylato groups suggest intermolecular hydrogen bonding. Adjacent molecules are linked through O—H···O interactions: O(6)···O(1)(x, $1 + y$, z) 2.649 (4), O(6)···O(4)(x, $\frac{1}{2} - y$, $z - \frac{1}{2}$) 2.676 (4) and O(7)···O(4)($-x$, $y - \frac{1}{2}$, $\frac{1}{2} - z$) 2.765 (4) Å. The structure of [Cu*L*(H_2O)$_2$] is made up of parallel double layers of molecules with phenyl groups pointing outside; the water O atoms form intermolecular bonds with carbonyl O atoms.

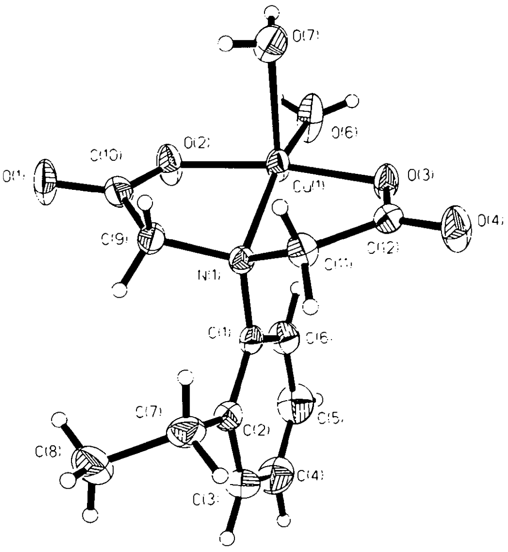


Fig. 1. *ORTEP* (Johnson, 1965) drawing of (I). Ellipsoids are drawn at the 50% probability level.

Experimental

N-(2-Ethylphenyl)-*N*-(carboxymethyl)glycine was synthesized by a modification of the method of Skubin, Kutepov & Valgin (1970), using a concentrated solution of Cu(NO_3)$_2$.3H_2O for precipitation of [Cu(*L*)(H_2O)$_2$]. 0.3g (0.87 mmol) of this complex was added to 50 ml of ethanol and heated until boiling. The resulting solution was filtered and allowed to stand at room temperature for 6 days, whereupon well developed blue crystals of [Cu(*L*)(H_2O)$_2$] were obtained. The crystal density D_m was measured by flotation.

Crystal data

[Cu($C_{12}H_{13}NO_4$)(H_2O)$_2$]
M_r = 334.8
Monoclinic
$P2_1/c$
a = 12.430 (3) Å
b = 7.601 (2) Å
c = 15.219 (5) Å
β = 108.10 (4)°

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 12.5–17.5°
μ = 1.623 mm^{-1}
T = 294 K
Prism

$V = 1366.8\,(7)$ Å^3
$Z = 4$
$D_x = 1.627$ Mg m^{-3}
$D_m = 1.639$ Mg m^{-3}

$0.24 \times 0.21 \times 0.18$ mm
Blue

Data collection

Stoe Siemens AED-2 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.689$, $T_{\max} = 0.747$
6210 measured reflections
2161 independent reflections

2033 observed reflections [$F > 4\sigma(F)$]
$R_{\text{int}} = 0.0144$
$\theta_{\max} = 32.5°$
$h = -14 \rightarrow 13$
$k = -8 \rightarrow 8$
$l = -12 \rightarrow 17$
3 standard reflections
frequency: 150 min
intensity decay: none

Refinement

Refinement on F
$R = 0.0315$
$wR = 0.0385$
$S = 1.22$
2033 reflections
210 parameters
Only H-atom U's refined
Unit weights applied
$(\Delta/\sigma)_{\max} = 0.119$

$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.35$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 55, 99 & 149)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Cu(1)	0.1442 (1)	0.1451 (1)	0.1617 (1)	0.025 (1)
O(3)	0.1392 (2)	0.2967 (3)	0.2616 (1)	0.029 (1)
O(6)	0.1477 (3)	0.3295 (3)	0.0740 (2)	0.044 (1)
O(4)	0.1201 (2)	0.2797 (3)	0.4012 (2)	0.038 (1)
O(2)	0.1614 (2)	−0.0358 (3)	0.0792 (2)	0.037 (1)
O(7)	−0.0458 (2)	0.1247 (3)	0.1200 (2)	0.034 (1)
O(1)	0.1479 (3)	−0.3229 (3)	0.0623 (2)	0.043 (1)
N(1)	0.2073 (2)	−0.0411 (3)	0.2633 (2)	0.021 (1)
C(12)	0.1368 (3)	0.2117 (5)	0.3327 (2)	0.025 (1)
C(2)	0.3916 (3)	−0.1489 (5)	0.3725 (2)	0.028 (1)
C(1)	0.3313 (3)	−0.0313 (4)	0.3027 (2)	0.023 (1)
C(9)	0.1682 (3)	−0.2088 (4)	0.2126 (2)	0.026 (1)
C(10)	0.1595 (3)	−0.1898 (5)	0.1102 (2)	0.028 (1)
C(11)	0.1490 (3)	0.0122 (4)	0.3319 (2)	0.025 (1)
C(5)	0.5035 (3)	0.1199 (5)	0.3074 (3)	0.042 (2)
C(3)	0.5091 (3)	−0.1242 (5)	0.4064 (3)	0.038 (1)
C(6)	0.3875 (3)	0.0997 (5)	0.2713 (2)	0.031 (1)
C(4)	0.5643 (3)	0.0073 (6)	0.3755 (3)	0.044 (2)
C(7)	0.3410 (3)	−0.2979 (5)	0.4128 (2)	0.035 (1)
C(8)	0.3786 (4)	−0.4789 (5)	0.3887 (3)	0.051 (2)

Table 2. *Selected geometric parameters* (Å, °)

Cu(1)—O(3)	1.924 (2)	N(1)—C(11)	1.500 (5)
Cu(1)—O(6)	1.946 (3)	C(12)—C(11)	1.524 (5)
Cu(1)—O(2)	1.919 (3)	C(2)—C(1)	1.412 (4)
Cu(1)—O(7)	2.253 (3)	C(2)—C(3)	1.403 (5)
Cu(1)—N(1)	2.062 (3)	C(2)—C(7)	1.515 (5)
O(3)—C(12)	1.269 (4)	C(1)—C(6)	1.384 (5)
O(4)—C(12)	1.238 (4)	C(9)—C(10)	1.535 (5)
O(2)—C(10)	1.265 (4)	C(5)—C(6)	1.383 (5)
O(1)—C(10)	1.229 (4)	C(5)—C(4)	1.375 (5)
N(1)—C(1)	1.472 (4)	C(3)—C(4)	1.376 (6)
N(1)—C(9)	1.491 (4)	C(7)—C(8)	1.534 (6)
O(3)—Cu(1)—O(6)	97.1 (1)	O(3)—C(12)—C(11)	118.2 (3)
O(3)—Cu(1)—O(2)	169.8 (1)	O(4)—C(12)—C(11)	117.7 (3)
O(6)—Cu(1)—O(2)	92.1 (1)	C(1)—C(2)—C(3)	116.2 (3)
O(3)—Cu(1)—O(7)	88.6 (1)	C(1)—C(2)—C(7)	126.0 (3)
O(6)—Cu(1)—O(7)	95.8 (1)	C(3)—C(2)—C(7)	117.8 (3)
O(2)—Cu(1)—O(7)	95.0 (1)	N(1)—C(1)—C(2)	121.2 (3)
O(3)—Cu(1)—N(1)	85.3 (1)	N(1)—C(1)—C(6)	118.2 (3)
O(6)—Cu(1)—N(1)	157.6 (1)	C(2)—C(1)—C(6)	120.5 (3)
O(2)—Cu(1)—N(1)	84.6 (1)	N(1)—C(9)—C(10)	110.7 (3)
O(7)—Cu(1)—N(1)	106.5 (1)	O(2)—C(10)—O(1)	123.6 (3)
Cu(1)—O(3)—C(12)	112.5 (2)	O(2)—C(10)—C(9)	117.5 (3)
Cu(1)—O(2)—C(10)	113.6 (2)	O(1)—C(10)—C(9)	118.9 (3)
Cu(1)—N(1)—C(1)	112.1 (2)	N(1)—C(11)—C(12)	110.2 (3)
Cu(1)—N(1)—C(9)	102.3 (2)	C(6)—C(5)—C(4)	119.4 (4)
C(1)—N(1)—C(9)	112.7 (3)	C(2)—C(3)—C(4)	123.0 (3)
Cu(1)—N(1)—C(11)	100.6 (2)	C(1)—C(6)—C(5)	121.3 (3)
C(1)—N(1)—C(11)	111.9 (2)	C(5)—C(4)—C(3)	119.6 (3)
C(9)—N(1)—C(11)	116.2 (3)	C(2)—C(7)—C(8)	112.2 (4)
O(3)—C(12)—O(4)	124.0 (3)		

Data collection: *DIF*4 (Stoe & Cie, 1991*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1991*b*). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1988). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*; *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1241). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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