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Comment

In the context of a study on the anticancer properties of several mixed substituted phenanthroline and amino acid complexes, we have determined the structure of the title compound, (I).

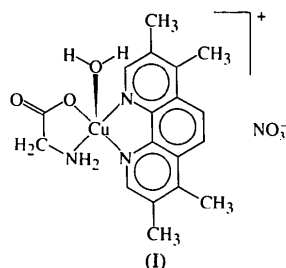


Fig. 1 shows a view of the structure with the atom-numbering scheme. Cu^{II} is surrounded by five ligand atoms in an N₃O₂-type distorted square-pyramidal coordination mode. The water molecule is in the apical position. The N and one of the O atoms from the glycinate moiety, and both phenanthroline N atoms lie in a slightly tetrahedrally distorted plane. The distances to the least-squares plane defined by atoms N1, N2, N3 and O1 are –0.082 (2) for N1, 0.087 (2) for N2, 0.081 (2) for N3 and –0.087 (2) Å for O1. Cu^{II} is 0.160 (2) Å out of this plane. The Cu–N distances are longer than the Cu–O1 distance, as reported for many related complexes (Aoki & Yamasaky, 1980; Antolini, Marcotrigiano, Menabue & Pellacani, 1983; Antolini, Marcotrigiano, Menabue, Pellacani, Saladini & Sola, 1985; Antolini, Battaglia, Corradi, Marcotrigiano, Menabue, Pellacani, Saladini & Sola, 1986; Solans, Ruiz-Ramírez, Gasque & Briansó, 1987; Mendoza-Diaz, Martínez-Aguilera, Pérez-Alonso, Solans & Moreno-Esparza, 1987; Solans, Ruiz-Ramírez, Martínez, Gasque & Briansó, 1988; Solans, Aguiló, Gleizest, Faus, Julve & Verdaguer, 1990; Solans, Ruiz-Ramírez, Martínez, Gasque & Moreno-Esparza, 1992, 1993; Mendoza-Diaz, Martínez-Aguilera, Moreno-Esparza, Pannell & Cervantes Lee, 1993).

The Cu–OW1(aqua) bond is longer than those observed in some related complexes: 2.355 (4) Å for this complex and 2.294 (6) Å for the L-glutamate complex (Antolini, Marcotrigiano, Menabue & Pellacani, 1983) and 2.213 (10) Å for the L-phenylalaninato complex (Solans, Ruiz-Ramírez, Gasque & Briansó, 1987).

The chelate ring formed by atoms N1, C12, C11, N2 and Cu is slightly distorted from planarity, whereas the chelate ring formed by atoms N3, C18, C17, O1 and Cu shows a distorted half-chair conformation, the asymmetry parameter ΔC₂(O1) being 9.3 (4)°, as defined by Duax, Weeks & Roher (1976). The angle between the two chelate-ring mean planes is 9.9 (2)°.

The nitrate ion is not involved in the Cu coordination sphere. The nearest atom, O41(1–x, –y,

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Aqua(glycinato)(3,4,7,8-tetramethyl-1,10-phenanthroline)copper(II) Nitrate

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Abstract

In the title compound, [Cu(C₂H₄NO₂)(C₁₆H₁₆N₂)(H₂O)]NO₃, Cu^{II} displays distorted square-pyramidal coordination where the water molecule is in the apical position and the base is defined by the N and one of the O atoms from the glycinate ligand, and both phenanthroline N atoms. The phenanthroline chelate-ring plane (N1, C12, C11, N2, Cu) is slightly distorted from planarity, whereas the five-membered ring formed by the glycinate ligand (defined by atoms N3, C18, C17, O1 and Cu), presents a distorted half-chair conformation.

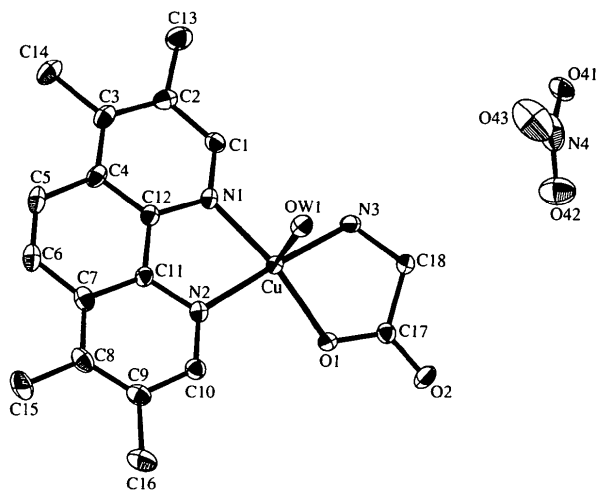


Fig. 1. Molecular structure of the title compound showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

1–z), is at a distance of 3.828 (4) Å. The nitrate is linked to the glycinate moiety by a hydrogen bond: N3—H32 0.88 (5), N3···O42(1–x, –y, 1–z) 2.864 (6), H32···O42 2.01 (4) Å and N3—H32···O42 166 (5)°. Other relevant hydrogen bonds are observed between the glycine carboxylate and the water molecules: OW1—H1W 0.73 (5), OW1···O2(–x, –y, 1–z) 2.788 (5), H1W···O2 2.07 (5) Å and OW1—H1W···O2 165 (6)°; and OW1—H2W 0.75 (5), OW1···O1(x, 1 + y, z) 2.915 (5), H2W···O1 2.17 (5) Å and OW1—H2W···O1 170 (5)°.

Experimental

The title compound was prepared as described previously (Gasque, Ruiz-Ramírez & Moreno-Esparza, 1992). Suitable crystals were grown by slow evaporation from a water solution.

Crystal data

[Cu(C₂H₄NO₂)(C₁₆H₁₆N₂)-
(H₂O)]NO₃

M_r = 453.94

Monoclinic

*P*2₁/*n*

a = 17.842 (3) Å

b = 5.489 (1) Å

c = 20.045 (2) Å

β = 105.18 (1)°

V = 1894.6 (5) Å³

Z = 4

D_x = 1.591 Mg m^{–3}

Data collection

Enraf–Nonius CAD-4
diffractometer

ω/2θ scans

Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 25
reflections

θ = 10.32–14.00°

μ = 1.198 mm^{–1}

T = 293 (2) K

Prism

0.65 × 0.14 × 0.14 mm

Blue

2688 observed reflections

[*I* > 2σ(*I*)]

*R*_{int} = 0.0178

Absorption correction:

ψ scan (North, Philips
& Mathews, 1968)

*T*_{min} = 0.922, *T*_{max} =
0.999

3053 measured reflections

2955 independent reflections

θ_{max} = 24.97°

h = 0 → 21

k = 0 → 6

l = –23 → 22

3 standard reflections

frequency: 60 min

intensity decay: none

Refinement

Refinement on *F*²

R(*F*) = 0.0406

wR(*F*²) = 0.0973

S = 1.253

2955 reflections

280 parameters

Only H-atom *U*'s refined for

H atoms bonded to C

w = 1/[σ²(*F*_o²) + (0.0038*P*)²
+ 3.2038*P*]

where *P* = (*F*_o² + 2*F*_c²)/3

(Δ/σ)_{max} = 0.141

Δρ_{max} = 0.365 e Å^{–3}

Δρ_{min} = –0.280 e Å^{–3}

Atomic scattering factors

from *International Tables*

for *Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Cu	0.02858 (3)	0.18273 (9)	0.33546 (2)	0.02785 (14)
N1	0.0467 (2)	0.4114 (7)	0.2629 (2)	0.0303 (7)
N2	–0.0550 (2)	0.0596 (6)	0.2560 (2)	0.0300 (7)
N3	0.1168 (2)	0.2800 (7)	0.4142 (2)	0.0342 (8)
O1	0.0219 (2)	–0.1010 (5)	0.39220 (14)	0.0354 (7)
O2	0.0695 (2)	–0.2591 (6)	0.4960 (2)	0.0433 (8)
C1	0.1004 (2)	0.5849 (8)	0.2669 (2)	0.0335 (9)
C2	0.1059 (3)	0.7346 (8)	0.2120 (2)	0.0381 (10)
C3	0.0513 (3)	0.7050 (9)	0.1489 (2)	0.0364 (10)
C4	–0.0060 (2)	0.5224 (8)	0.1427 (2)	0.0321 (9)
C5	–0.0653 (3)	0.4692 (10)	0.0802 (2)	0.0435 (11)
C6	–0.1177 (3)	0.2894 (10)	0.0767 (2)	0.0426 (11)
C7	–0.1182 (2)	0.1419 (8)	0.1351 (2)	0.0348 (10)
C8	–0.1719 (2)	–0.0484 (9)	0.1353 (2)	0.0384 (10)
C9	–0.1657 (2)	–0.1777 (9)	0.1960 (2)	0.0373 (10)
C10	–0.1059 (2)	–0.1181 (8)	0.2545 (2)	0.0323 (9)
C11	–0.0609 (2)	0.1886 (7)	0.1967 (2)	0.0271 (8)
C12	–0.0058 (2)	0.3788 (7)	0.2005 (2)	0.0284 (8)
C13	0.1706 (3)	0.9173 (10)	0.2236 (3)	0.0500 (12)
C14	0.0539 (3)	0.8630 (9)	0.0887 (2)	0.0489 (12)
C15	–0.2363 (3)	–0.1013 (11)	0.0718 (3)	0.060 (2)
C16	–0.2198 (3)	–0.3811 (9)	0.2021 (3)	0.0536 (13)
C17	0.0656 (2)	–0.0924 (8)	0.4549 (2)	0.0301 (9)
C18	0.1106 (2)	0.1405 (8)	0.4753 (2)	0.0331 (9)
OW1	–0.0576 (2)	0.4304 (6)	0.3763 (2)	0.0405 (8)
N4	0.7286 (3)	0.0975 (9)	0.5846 (2)	0.0530 (11)
O41	0.7915 (2)	0.2114 (8)	0.5920 (2)	0.0664 (11)
O42	0.7300 (2)	–0.1310 (9)	0.5880 (3)	0.0765 (13)
O43	0.6666 (3)	0.2041 (11)	0.5707 (3)	0.105 (2)

Table 2. Selected geometric parameters (Å, °)

Cu—O1	1.950 (3)	C3—C4	1.413 (6)
Cu—N3	1.988 (4)	C3—C14	1.497 (6)
Cu—N2	1.994 (3)	C4—C12	1.401 (6)
Cu—N1	2.012 (3)	C4—C5	1.443 (6)
Cu—OW1	2.354 (4)	C5—C6	1.347 (7)
N1—C1	1.338 (5)	C6—C7	1.425 (6)
N1—C12	1.363 (5)	C7—C11	1.405 (5)
N2—C10	1.328 (5)	C7—C8	1.419 (6)
N2—C11	1.362 (5)	C8—C9	1.388 (6)
N3—C18	1.473 (5)	C8—C15	1.503 (6)
O1—C17	1.295 (5)	C9—C10	1.401 (6)
O2—C17	1.220 (5)	C9—C16	1.503 (6)

C1—C2	1.396 (6)	C11—C12	1.423 (6)
C2—C3	1.390 (6)	C17—C18	1.508 (6)
C2—C13	1.501 (6)		
O1—Cu—N3	84.72 (13)	C12—C4—C3	118.8 (4)
O1—Cu—N2	92.78 (13)	C12—C4—C5	116.5 (4)
N3—Cu—N2	175.22 (15)	C3—C4—C5	124.7 (4)
O1—Cu—N1	165.07 (14)	C6—C5—C4	122.3 (4)
N3—Cu—N1	99.37 (14)	C5—C6—C7	121.8 (4)
N2—Cu—N1	82.05 (14)	C11—C7—C8	117.9 (4)
O1—Cu—OW1	96.51 (12)	C11—C7—C6	117.3 (4)
N3—Cu—OW1	91.28 (15)	C8—C7—C6	124.8 (4)
N2—Cu—OW1	93.05 (13)	C9—C8—C7	118.7 (4)
N1—Cu—OW1	97.74 (13)	C9—C8—C15	120.8 (4)
C1—N1—C12	117.3 (3)	C7—C8—C15	120.5 (4)
C1—N1—Cu	130.4 (3)	C8—C9—C10	118.9 (4)
C12—N1—Cu	112.3 (3)	C8—C9—C16	123.1 (4)
C10—N2—C11	117.8 (3)	C10—C9—C16	118.0 (4)
C10—N2—Cu	129.1 (3)	N2—C10—C9	123.8 (4)
C11—N2—Cu	113.1 (3)	N2—C11—C7	123.0 (4)
C18—N3—Cu	107.7 (3)	N2—C11—C12	116.1 (3)
C17—O1—Cu	115.1 (3)	C7—C11—C12	120.9 (4)
N1—C1—C2	124.6 (4)	N1—C12—C4	122.5 (4)
C3—C2—C1	118.2 (4)	N1—C12—C11	116.5 (4)
C3—C2—C13	123.1 (4)	C4—C12—C11	121.1 (4)
C1—C2—C13	118.7 (4)	O2—C17—O1	122.7 (4)
C2—C3—C4	118.7 (4)	O2—C17—C18	121.4 (4)
C2—C3—C14	120.3 (4)	O1—C17—C18	115.8 (4)
C4—C3—C14	121.0 (4)	N3—C18—C17	111.5 (3)

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software* (Enraf-Nonius, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PC-ORTEP* (Schmid & Brueggemann, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1125). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 854–857

Bis(tetra-*n*-butylammonium) Bis(μ -1,1-dicyanoethene-2,2-diselenolato)- dithallate(I) Acetone Solvate, [(C₄H₉)₄N]₂[Tl₂{Se₂C=C(CN)₂}]₂· 2(CH₃)₂CO

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(Received 9 April 1992; accepted 31 March 1994)

Abstract

In the dimeric anion two Tl atoms are coordinated by Se atoms of two different ligands forming a distorted orthorhombic bipyramid. The Bu₄N⁺ cations form hydrogen bonds with both N atoms but only one of the Se donor atoms of each ligand in the anions: C—H...N and C—H...Se distances are 2.56–2.64 (2) and 2.80–3.19 (1) Å, respectively.

Comment

Thallium shows much greater stability as *M*^I relative to *M*^{III} than the other members of Group III in the Periodic Table. A number of thallium(I) coordination compounds with chalcogene ligands have been prepared (Lee, 1972), but only a few structural data are available. The crystal structures of various thallium(I) dialkyldithiocarbamates have been