Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1024). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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catena-Poly[[(diethylenetriamine-*N*,*N*′,*N*″)-copper(II)]-*μ*-succinato-*O*,*O*′:*O*″]

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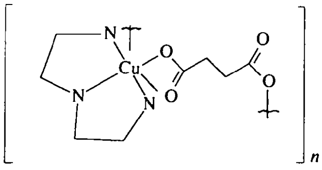
Abstract

The title compound, *catena*-poly[{[*N*-(2-aminoethyl)-1,2-ethanediamine-*N*,*N*′,*N*″]copper(II)}-*μ*-succinato-*O*,*O*′:*O*″], $[Cu(C_4H_4O_4)(C_4H_{13}N_3)]_n$, has a polymeric structure in which the central Cu atom is coordinated by three N atoms from the diethylenetriamine molecule, two O atoms from the succinate ion and one O atom from the succinate ion of the adjacent complex unit. The coordination polyhedron around the Cu atom can be regarded as a severely distorted octahedron.

Comment

Complexes of the tridentate ligand diethylenetriamine (dien) with copper and carboxylic acids exhibit many types of bridging. Most of the complexes are polymeric, such as the formato (Davey & Stephens, 1971), acetato (Towle, Hoffmann, Hatfield, Singh & Chaudhuri, 1988), phthalato (Shakhatreh *et al.*, 1991) and terephthalato (Bakalbassis *et al.*, 1991) complexes. The title compound, (I), which contains a bridging succinate ion, belongs to this family of complexes.



(I)

The structure of the title complex is shown in Fig. 1, which also indicates the atom numbering in the asymmetric unit. The complex possesses a crystallographic mirror plain passing through Cu and N2, and the O and C atoms of the succinate ion. The structure is composed of polymeric chains of complexes running parallel to the *b* axis (Fig. 2). Each unit in the chain is related to the next by a glide operation along the *b* axis. The equatorial plane is composed of the three N atoms of the diethylenetriamine ligand and the O1 atom of the succinate ion. The two apical positions are occupied by the O2 atom of the same succinate ion and the O3^{i} atom of the succinate ion from the next molecule along the chain [symmetry code: (i) $-x, \frac{1}{2}+y, \frac{1}{2}-z$]. Because of the unequal Cu—O2 and Cu—O3^{i} bond lengths [2.663 (2) and 2.393 (2) Å, respectively], and the unequal O1—Cu—O2 and N2—Cu—O2 angles [54.20 (8) and 114.57 (8)°, respectively], the local geometry around the Cu atom is severely distorted. The coordination about the Cu atom can be regarded as distorted octahedral, with a *mer* configuration for the N and O atoms. The largest deviation of atoms N1, N2, N1^{ii} and O1 from their mean basal

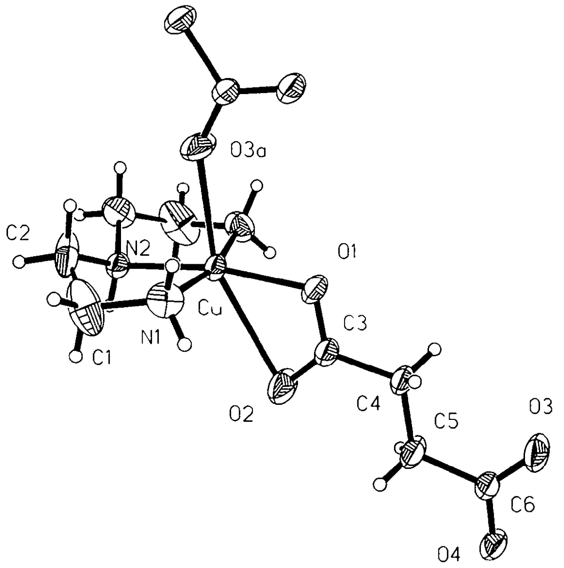


Fig. 1. The structure of compound (I) showing 50% probability displacement ellipsoids.

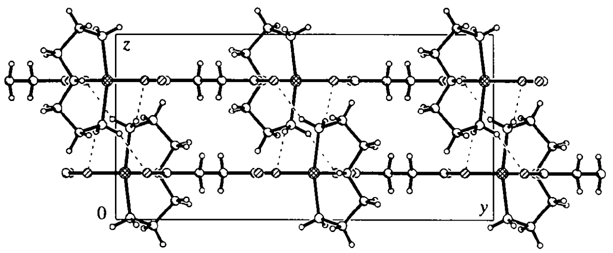


Fig. 2. Packing diagram viewed along the *a* axis.

plane is 0.161 (1) Å, whereas the Cu atom deviates by only 0.048 (1) Å [symmetry code: (ii) $x, y, \frac{1}{2}-z$]. The basal plane is exactly perpendicular to its neighbouring carboxylate group plane (O1—C3—O2).

The Cu—N and Cu—O1 bond lengths are comparable to those found in the aforementioned diethylenetriamine complexes. All the C and O atoms of the succinate ion lie on the mirror plane, which means that the ion is planar except for the H atoms. The shortest Cu···Cu distance between the chains is 5.278 (2) Å and the distance between Cu atoms connected to the same succinate ion is 9.008 (2) Å. The chains are joined together by hydrogen bonds between the amine H atoms and the O1 and O4 atoms of the succinate ion (Table 2).

Experimental

Crystals of the title compound were prepared from $CuCO_3$, succinic acid and diethylenetriamine (Pajunen & Näsäkkälä, 1977) and recrystallized from ethanol.

Crystal data

[$Cu(C_4H_4O_4)(C_4H_{13}N_3)$]
$M_r = 282.79$
Orthorhombic
Pbcm
$a = 8.021$ (2) Å
$b = 16.864$ (3) Å
$c = 8.311$ (2) Å
$V = 1124.2$ (4) Å^3
$Z = 4$
$D_x = 1.671$ Mg m^{-3}
$D_m = 1.67$ (2) Mg m^{-3}
D_m measured by flotation in CCl_4/CH_3I solution

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 4.89–10.31°$
$\mu = 1.947$ mm^{-1}
$T = 293$ (2) K
Prismatic
0.30 × 0.25 × 0.15 mm
Blue

Data collection

Enraf–Nonius CAD-4 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
1742 measured reflections
1742 independent reflections
1442 observed reflections [$I > 2\sigma(I)$]

$\theta_{max} = 29.96°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 23$
$l = 0 \rightarrow 11$
2 standard reflections frequency: 60 min intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0279$
$wR(F^2) = 0.0890$
$S = 1.163$
1742 reflections
89 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2 + 1.043P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.454$ e Å^{-3}
$\Delta\rho_{min} = -0.670$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0113 (9)
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 (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	0.19664 (4)	0.52376 (2)	1/4	0.02583 (12)
O1	0.0688 (3)	0.42388 (11)	1/4	0.0380 (5)
O2	0.3256 (3)	0.37823 (14)	1/4	0.0632 (9)
O3	0.0187 (3)	0.12197 (13)	1/4	0.0623 (9)
O4	0.2797 (3)	0.08205 (12)	1/4	0.0430 (6)
N1	0.2026 (2)	0.53743 (12)	0.0085 (2)	0.0390 (4)
N2	0.3662 (3)	0.61155 (13)	1/4	0.0270 (4)
C1	0.3289 (4)	0.5977 (2)	−0.0323 (3)	0.0658 (9)
C2	0.3407 (3)	0.65642 (15)	0.1015 (4)	0.0511 (7)
C3	0.1752 (3)	0.3674 (2)	1/4	0.0318 (6)
C4	0.1021 (3)	0.28413 (15)	1/4	0.0389 (7)
C5	0.2312 (4)	0.2199 (2)	1/4	0.0494 (10)
C6	0.1700 (3)	0.1345 (2)	1/4	0.0296 (5)

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

Cu—O1	1.972 (2)	O4—C6	1.248 (3)
Cu—N2	2.011 (2)	N1—C1	1.475 (3)
Cu—N1	2.021 (2)	N2—C2	1.462 (3)
Cu—O3^i	2.393 (2)	C1—C2	1.492 (4)
Cu—O2	2.663 (3)	C3—C4	1.522 (4)
O1—C3	1.279 (3)	C4—C5	1.499 (4)
O2—C3	1.220 (3)	C5—C6	1.521 (4)
O3—C6	1.232 (3)		
O1—Cu—N2	168.77 (9)	C1—N1—Cu	108.86 (15)
O1—Cu—N1^{ii}	96.30 (5)	C2—N2—C2^{ii}	115.1 (3)
O1—Cu—N1	96.30 (5)	C2—N2—Cu	106.64 (13)
N2—Cu—N1	84.26 (5)	N1—C1—C2	109.2 (2)
N1^{ii}—Cu—N1	166.62 (11)	N2—C2—C1	107.2 (2)
O1—Cu—O3^i	102.45 (9)	O2—C3—O1	123.3 (3)
N2—Cu—O3^i	88.78 (9)	O2—C3—C4	121.2 (3)
N1—Cu—O3^i	86.46 (5)	O1—C3—C4	115.5 (2)
O1—Cu—O2	54.20 (8)	C5—C4—C3	113.6 (2)
N2—Cu—O2	114.57 (8)	C4—C5—C6	117.5 (2)
N1—Cu—O2	95.50 (6)	O3—C6—O4	124.9 (3)
O3^i—Cu—O2	156.65 (8)	O3—C6—C5	118.7 (3)
C3—O1—Cu	106.8 (2)	O4—C6—C5	116.3 (2)
C3—O2—Cu	75.7 (2)		

D—H···A	H···A	D···A	D—H···A
N1—H1···O4^{iii}	2.18	3.009 (2)	152
N1—H2···O1^{iv}	2.34	3.127 (2)	147
N2—H3···O4^{v}	2.02	2.883 (3)	161

Symmetry codes: (i) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $x, y, \frac{1}{2}-z$; (iii) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (iv) $-x, 1-y, -z$; (v) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$.

H atoms were included in calculated positions with fixed displacement parameters (1.2 times the displacement parameter of the host atom).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXTL/PC*

(Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1387). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[1,3-Bis(diphenylphosphino)propane-*P*,*P*′](1,3-propanedithiolato-*S*,*S*′)-palladium(II) Acetonitrile Solvate, $[Pd\{Ph_2P(CH_2)_3PPh_2\}(SC_3H_6S)].CH_3CN$

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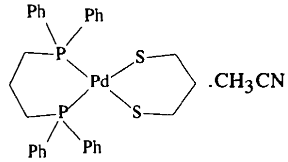
Abstract

The structure of the title compound, $[Pd(C_3H_6S_2)(C_{27}H_{26}P_2)].CH_3CN$, consists of discrete mononuclear palladium(II) complex and acetonitrile molecules. The Pd atom is fourfold coordinated by two P atoms from the phosphine ligand and two S atoms from the $C_3H_6S_2^{2-}$ ligand in a distorted square-planar geometry. The average Pd—S and Pd—P distances are 2.326 (8) and 2.299 (7) Å, respectively.

Comment

Transition metal compounds with mixed sulfur and phosphine ligands have attracted much attention due to their importance in a wide range of chemical and industrial systems. In the nickel-group metals, many nickel compounds with such mixed ligands have been reported. Surprisingly few palladium compounds, such as $[Pd_2(SC_6F_5)_2(PPh_3)_2]$ (Fenn & Segrott, 1972), have been structurally characterized. We reported recently the palladium compounds $[Pd(SCH_2CH_2SCH_2CH_2S)(PPh_3)_2]$ and $[Pd_2(PPh_3)_2(HOC_6H_4S)_2Cl_2]$ (Cao, Hong, Jiang, Xie & Liu, 1996), and $[Pd_2(PPh_3)_2(SC_2H_4S)_2]$ (Cao, Hong, Jiang & Liu, 1995). We report here the crystal structure of a mononuclear palladium complex, namely, $[Pd\{Ph_2P(CH_2)_3PPh_2\}(SC_3H_6S)].CH_3CN$, (I).



(I)

The title compound, (I), consists of a discrete mononuclear palladium(II) complex and an acetonitrile molecule (Fig. 1). The Pd atom is fourfold coordinated by two P atoms from the phosphine ligand and two S atoms from the $C_3H_6S_2^{2-}$ ligand in a distorted square-planar geometry. The displacements from the least-squares plane formed by the atoms Pd(1), P(1), P(2), S(1) and S(2) are 0.007, −0.067, 0.064, −0.65 and

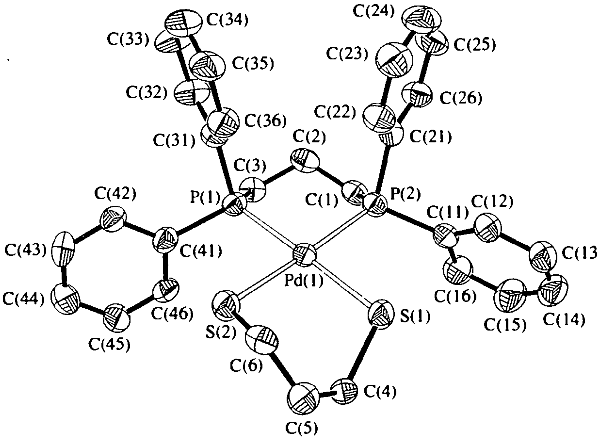


Fig. 1. The crystal structure of the title complex with ellipsoids drawn at the 30% probability level.