Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: AB1161). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(ethanol)-1κO,4κO-hexakis(μ-propionato)-1κO:2$\kappa O'$;2$\kappa^4 O$:3$\kappa^4 O'$;-3κO:4$\kappa O'$-tetrakis(triphenylphosphine)-1$\kappa^2 P$,4$\kappa^2 P$-tetracopper(I,II), $[Cu_4(C_3H_5O_2)_6(C_2H_6O)_2(C_{18}H_{15}P)_4]$

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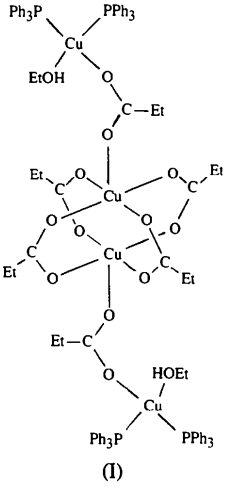
Abstract

The crystal structure of the title compound is formed by centrosymmetric $[Cu_4(prop)_6(Ph_3P)_4(EtOH)_2]$ (prop = $CH_3CH_2CO_2^-$) molecules which contain a dinuclear $Cu_2(\mu\text{-prop})_4$ unit similar to those found in tetrakis(μ-acetato)copper(II) complexes. Both axial positions of this unit are occupied by an O atom of the propionate group of a $Cu(prop)(Ph_3P)_2(EtOH)$ moiety.

Comment

The crystal structure of the title compound, (I), consists of centrosymmetric $[Cu_4(prop)_6(Ph_3P)_4(EtOH)_2]$ molecules (Fig. 1). The two centrosymmetrically related Cu^{II} atoms [Cu(1)] are linked by four bridging propionate groups, this motif being similar to those of copper acetate monohydrate (Meester, Fletcher & Skapski, 1973), $[Cu_4Ac_6(Ph_3P)_4]$ (Ac = acetate) (Koman, Valigura, Ďurčanská & Ondrejovič, 1984) and $[Cu_2Ac_4(Ph_3P)_2]$ (Koman, Valigura & Ondrejovič, 1988). Each Cu^{II} atom forms a further axial coordination bond with the O atom of a propionate group which forms a bridge to the Cu^I atom [Cu(2)]. The Cu^{II} coordination polyhedron is thus a distorted tetragonal pyramid with O atoms at the vertices. The Cu^I coordination polyhedron is a distorted tetrahedron formed by an ethanol O atom, a propionate O atom and two P atoms from the Ph_3P ligands.



(I)

The basal Cu^{II}—O [average 1.971 (9) Å] and apical Cu^{II}—O [2.092 (8) Å] bond lengths are close to those found in $[Cu_4Ac_6(Ph_3P)_4]$. The $Cu^{II}\cdots Cu^{II}$ distance is 2.624 (4) Å. The Cu^I—O(8) bond length of 2.075 (8) Å to the propionate ligand is significantly shorter than the Cu^I—O(9) distance of 2.183 (7) Å to the ethanol group. The remaining distances, including those in the Ph_3P ligand, are not significantly different from values found in $[CuAc(Ph_3P)_2]$ (Drew, Othman, Edwards & Richards, 1975).

The large U_{eq} parameter for C(11) may indicate disorder. The crystals decompose in the X-ray beam at room temperature and lose ethanol at 353 K.

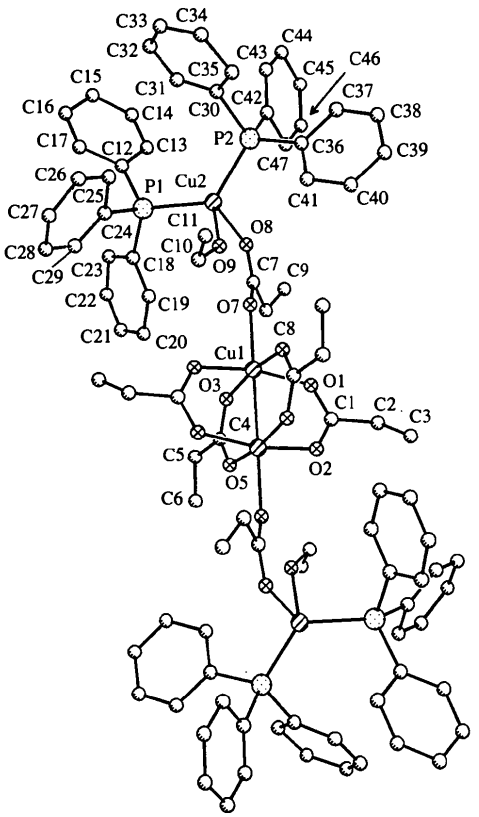


Fig. 1. Structure and atom-numbering scheme of the title molecule.

Experimental

The title compound was crystallized from ethanol. The crystal density D_m was measured by flotation.

Crystal data

$[Cu_4(C_3H_5O_2)_6(C_2H_6O)_2$-$(C_{18}H_{15}P)_4]$	Cu $K\alpha$ radiation
M_r = 1834.0	λ = 1.54178 Å
Triclinic	Cell parameters from 15 reflections
$P\bar{1}$	θ = 5.34–14.55°
a = 13.537 (10) Å	μ = 2.23 mm^{-1}
b = 14.111 (7) Å	T = 188 K
c = 12.060 (10) Å	Prism
α = 99.98 (6)°	0.40 × 0.30 × 0.26 mm
β = 90.24 (7)°	Blue green
γ = 94.02 (6)°	
V = 2263.0 (4) Å^3	
Z = 1	
D_x = 1.35 Mg m^{-3}	
D_m = 1.36 Mg m^{-3}	

Data collection

Syntex $P2_1$ diffractometer	θ_{max} = 50°
θ–2θ scans	$h = -14 \rightarrow 14$
Absorption correction: none	$k = -14 \rightarrow 14$
	$l = 0 \rightarrow 13$
6442 measured reflections	2 standard reflections
6192 independent reflections	monitored every 100 reflections
2530 observed reflections [$I \geq 3\sigma(I)$]	intensity decay: <15%
R_{int} = 0.021	

Refinement

Refinement on F	$w = 13.3124/[\sigma^2(F_o) + 0.000118(F_o)^2]$
R = 0.0697	$(\Delta/\sigma)_{max}$ = 0.465
wR = 0.0734	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
S = 1.58	$\Delta\rho_{min}$ = −0.52 e Å^{-3}
2530 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
497 parameters	
H-atom parameters not refined	

Table 1. *Fractional atomic coordinates and isotropic or 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_{iso}/U_{eq}
Cu(1)	0.0639 (1)	0.9375 (1)	0.0150 (2)	0.043 (1)
Cu(2)	0.2479 (1)	0.7125 (1)	0.2377 (2)	0.042 (1)
P(1)	0.3547 (2)	0.8207 (2)	0.3418 (3)	0.039 (2)
P(2)	0.2186 (3)	0.5583 (2)	0.2525 (3)	0.043 (2)
O(1)	0.0306 (8)	0.8805 (7)	−0.1432 (9)	0.056 (7)
O(2)	−0.0790 (8)	0.9825 (7)	−0.1663 (8)	0.068 (7)
O(3)	0.1657 (6)	1.0247 (6)	−0.0343 (8)	0.050 (6)
O(5)	0.0571 (7)	1.1325 (6)	−0.0615 (9)	0.058 (7)
O(7)	0.1485 (6)	0.8305 (5)	0.0584 (8)	0.048 (6)
O(8)	0.2671 (7)	0.7300 (6)	0.0716 (7)	0.042 (6)
O(9)	0.1097 (6)	0.7843 (5)	0.2547 (8)	0.062 (6)
C(1)	−0.0345 (12)	0.9113 (11)	−0.1979 (13)	0.049 (11)
C(2)	−0.0603 (14)	0.8506 (12)	−0.3130 (17)	0.077 (13)
C(3)	−0.1620 (17)	0.8292 (14)	−0.3388 (23)	0.116 (20)
C(4)	0.1417 (10)	1.1026 (10)	−0.0604 (13)	0.049 (10)
C(5)	0.2263 (11)	1.1714 (9)	−0.0845 (13)	0.058 (10)
C(6)	0.2043 (13)	1.2473 (12)	−0.1385 (15)	0.089 (13)
C(7)	0.2268 (11)	0.7848 (9)	0.0246 (13)	0.044 (10)
C(8)	0.2665 (13)	0.8089 (10)	−0.0855 (12)	0.078 (12)
C(9)	0.3267 (12)	0.7403 (8)	−0.1499 (13)	0.078 (11)
C(10)	0.0492 (14)	0.8389 (12)	0.3323 (16)	0.077 (14)
C(11)	−0.0103 (22)	0.797 (2)	0.3894 (20)	0.24 (3)
C(12)	0.4624 (12)	0.7751 (7)	0.3900 (13)	0.051 (10)
C(13)	0.5100 (11)	0.7028 (9)	0.3205 (14)	0.068 (11)
C(14)	0.5930 (12)	0.6657 (9)	0.3567 (14)	0.055 (10)
C(15)	0.6314 (13)	0.6926 (9)	0.4652 (14)	0.053 (4) †
C(16)	0.5855 (12)	0.7662 (9)	0.5354 (13)	0.055 (10)
C(17)	0.5027 (13)	0.8057 (9)	0.5015 (14)	0.045 (10)
C(18)	0.4010 (10)	0.9160 (8)	0.2667 (12)	0.032 (8)
C(19)	0.3347 (11)	0.9592 (9)	0.2117 (13)	0.050 (10)
C(20)	0.3645 (12)	1.0310 (9)	0.1488 (15)	0.067 (11)
C(21)	0.4670 (12)	1.0581 (9)	0.1469 (14)	0.053 (4) †
C(22)	0.5314 (11)	1.0163 (9)	0.1983 (15)	0.076 (11)
C(23)	0.5037 (11)	0.9472 (9)	0.2667 (13)	0.051 (6)
C(24)	0.2997 (9)	0.8873 (9)	0.4695 (11)	0.043 (8)
C(25)	0.2500 (10)	0.8262 (8)	0.5388 (11)	0.042 (9)
C(26)	0.2047 (11)	0.8735 (13)	0.6364 (12)	0.076 (12)
C(27)	0.2037 (12)	0.9717 (12)	0.6593 (12)	0.065 (11)
C(28)	0.2545 (12)	1.0292 (10)	0.5932 (13)	0.062 (4) †
C(29)	0.3016 (10)	0.9830 (9)	0.4960 (12)	0.047 (9)
C(30)	0.2261 (13)	0.5362 (9)	0.3993 (14)	0.054 (12)
C(31)	0.3069 (13)	0.5680 (10)	0.4643 (14)	0.042 (12)
C(32)	0.3098 (16)	0.5584 (10)	0.5756 (14)	0.075 (14)
C(33)	0.2308 (14)	0.5222 (10)	0.6242 (16)	0.063 (4) †
C(34)	0.1488 (14)	0.4855 (11)	0.5650 (16)	0.092 (15)
C(35)	0.1378 (11)	0.4956 (10)	0.4455 (14)	0.061 (12)
C(36)	0.1012 (10)	0.5006 (8)	0.1926 (11)	0.040 (8)
C(37)	0.0863 (11)	0.3995 (8)	0.1613 (16)	0.060 (11)
C(38)	−0.0036 (12)	0.3595 (9)	0.1140 (16)	0.060 (11)
C(39)	−0.0781 (11)	0.4169 (9)	0.0990 (12)	0.049 (3) †

C(40)	−0.0646 (10)	0.5141 (9)	0.1296 (14)	0.051 (10)
C(41)	0.0272 (11)	0.5573 (8)	0.1806 (14)	0.050 (10)
C(42)	0.3094 (10)	0.4782 (9)	0.1809 (12)	0.051 (9)
C(43)	0.3484 (11)	0.4064 (10)	0.2200 (13)	0.062 (10)
C(44)	0.4144 (12)	0.3436 (9)	0.1582 (15)	0.073 (12)
C(45)	0.4335 (13)	0.3601 (10)	0.0456 (13)	0.062 (4)†
C(46)	0.3970 (11)	0.4310 (9)	0.0063 (13)	0.063 (10)
C(47)	0.3317 (11)	0.4960 (9)	0.0679 (12)	0.057 (10)

† Refined isotropically.

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

Cu(1)···Cu(1^{i})	2.624 (4)	O(5)—C(4)	1.250 (17)
Cu(1)—O(1)	1.975 (11)	C(4)—C(5)	1.511 (16)
Cu(1)—O(2^{i})	1.975 (8)	C(5)—C(6)	1.39 (2)
Cu(1)—O(3)	1.947 (7)	O(7)—C(7)	1.308 (14)
Cu(1)—O(5^{i})	1.987 (10)	O(8)—C(7)	1.195 (17)
Cu(1)—O(7)	2.092 (8)	C(7)—C(8)	1.517 (16)
Cu(2)—O(8)	2.075 (8)	C(8)—C(9)	1.436 (16)
Cu(2)—O(9)	2.183 (7)	O(9)—C(10)	1.406 (16)
Cu(2)—P(1)	2.238 (4)	C(10)—C(11)	1.25 (3)
Cu(2)—P(2)	2.222 (3)	P(1)—C(12)	1.769 (17)
O(1)—C(1)	1.244 (18)	P(1)—C(18)	1.825 (10)
O(2)—C(1)	1.214 (16)	P(1)—C(24)	1.849 (10)
C(1)—C(2)	1.52 (3)	P(2)—C(30)	1.854 (17)
C(2)—C(3)	1.41 (3)	P(2)—C(36)	1.821 (14)
O(3)—C(4)	1.258 (14)	P(2)—C(42)	1.844 (11)
O(1)—Cu(1)—O(2^{i})	167.2 (5)	O(9)—Cu(2)—P(1)	102.6 (3)
O(1)—Cu(1)—O(3)	90.4 (4)	O(9)—Cu(2)—P(2)	109.7 (2)
O(1)—Cu(1)—O(5^{i})	88.9 (4)	P(1)—Cu(2)—P(2)	126.8 (2)
O(2^{i})—Cu(1)—O(5^{i})	91.0 (4)	Cu(2)—P(1)—C(12)	116.1 (4)
O(2^{i})—Cu(1)—O(3)	87.4 (4)	Cu(2)—P(1)—C(18)	112.6 (5)
O(3)—Cu(1)—O(5^{i})	169.2 (4)	Cu(2)—P(1)—C(24)	113.7 (4)
O(1)—Cu(1)—O(7)	99.8 (4)	Cu(2)—P(2)—C(30)	113.5 (5)
O(2^{i})—Cu(1)—O(7)	93.0 (4)	Cu(2)—P(2)—C(36)	115.8 (4)
O(3)—Cu(1)—O(7)	101.5 (3)	Cu(2)—P(2)—C(42)	114.1 (3)
O(5^{i})—Cu(1)—O(7)	89.3 (4)	C(12)—P(1)—C(24)	105.2 (6)
O(1)—C(1)—O(2)	125.8 (17)	C(12)—P(1)—C(18)	104.7 (6)
O(3)—C(4)—O(5)	127.8 (10)	C(18)—P(1)—C(24)	103.5 (5)
O(7)—C(7)—O(8)	126.7 (11)	C(30)—P(2)—C(36)	107.1 (7)
O(8)—Cu(2)—O(9)	93.9 (3)	C(30)—P(2)—C(42)	102.3 (6)
O(8)—Cu(2)—P(1)	106.6 (3)	C(36)—P(2)—C(42)	102.6 (6)
O(8)—Cu(2)—P(2)	111.9 (2)		

Symmetry code: (i) $-x, 2-y, -z$.

The crystal specimen was of poor quality and no correction was made for crystal decomposition. Cu-atom positions were obtained from a Patterson function and other non-H-atom positions were obtained from Fourier syntheses. H atoms were placed in calculated positions. Calculations were performed using *SHELX*76 (Sheldrick, 1976).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: MU1081). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Dichlorobis(1-ethyltetrazole-N^4)-copper(II)

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Abstract

In the title compound, $[CuCl_2(C_3H_6N_4)_2]$, the tetrazole ligand is coordinated terminally by the N4 ring atom. The coordination polyhedron of the Cu atom takes the form of an elongated square bipyramid with $\bar{1}$ site symmetry. The equatorial positions are occupied by two Cl atoms [Cu—Cl 2.290 (1) Å] and two ethyltetrazole ligands [Cu—N 1.990 (4) Å]. In addition, there are two Cl atoms in axial positions [Cu—Cl 2.993 (1) Å]. Hence, the Cl atoms play the role of non-symmetrical bridges and connect the molecules into infinite layers located in the *yz* plane.

Comment

The tetrazoles (Tz) are an interesting class of ligands which have various coordination abilities because of the presence of four N atoms. There are only a few reports of substituted tetrazoles being unable to coordinate to metal atoms (Baenziger & Schultz, 1971; van den Meuvel, Franke, Verschoor & Zuur, 1983; Aliev, Goncharov, Grachev & Roschupkin, 1990; Aliev, Goncharov, Grachev, Kurmaz & Roschupkin, 1991). It can be concluded that these ligands have a tendency towards terminal monodentate coordination, whereas their nearest analogues, the 1,2,4-triazoles, typically form bridges between two metal atoms (Bietmeijer, Van Albada, de Graaff, Haasnoot & Reedijk, 1985; Sinditskii *et al.*, 1987; Vreugdenhil, Haasnoot & Reedijk, 1990; Grap, Kuz'mina, Porai-Koshits, Kurbakova & Efimenko, 1993). Previously, we found that in *trans*-$[Cu(1\text{-PhTz})_2(NO_3)_2(H_2O)_2]$, where 1-PhTz is 1-phenyltetrazole (Lavrenova, Virovets, Podberezskaya & Bikzhanova, 1994), the Cu atom has two terminal N^4-coordinated 1-PhTz ligands and two water molecules in equatorial positions, and two NO_3^- ligands in axial positions. We thought it of interest to investigate the structure of a complex of copper(II) chloride with another 1-