μ-1,2-Ethanediylbis(diphenylphosphine)-$\kappa^2P{:}P'$-bis{acetato[1,2-ethanediylbis(diphenylphosphine)-κ^2P,P']copper(I)} diethyl ether disolvate

Thorsten Morawitz,[a] Hans-Wolfram Lerner,[a] Miriam Bru Roig[b] and Michael Bolte[a]*

[a]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Strasse 11, 60439 Frankfurt/Main, Germany, and [b]Departament de Química Inorgànica i Orgànica, University Jaume I, Campus Riu Sec, Avenida Sos Baynat s/n, 12071 Castellon de la Plana, Spain

Correspondence e-mail:
bolte@chemie.uni-frankfurt.de

Key indicators

Single-crystal X-ray study
$T = 173$ K
Mean σ(C–C) = 0.017 Å
R factor = 0.119
wR factor = 0.291
Data-to-parameter ratio = 15.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[Cu_2(C_2H_3O_2)_2(C_{26}H_{24}P_2)_3]\cdot 2C_4H_{10}O$, crystallizes with one dicopper(I) unit located on a centre of inversion and two diethyl ether molecules in general positions. The Cu atoms exhibit slightly distorted tetrahedral coordination.

Comment

Tris(1-pyrazolyl)borates (1) ('scorpinates') were introduced by Trofimenko (1993) more than 30 years ago and are today well established as ligands in coordination chemistry, but only a limited number of transition metal complexes with the ditopic heteroscorpionate ligand (2) (Bieller *et al.*, 2004) have been structurally characterized. We have become interested in the reaction of ligand (2) with Cu⁺ ions. While Cu^I salts such as $Cu(CH_3CO_2)$ are not soluble in organic solvents, we have prepared the dinuclear complex $[Cu_2(CH_3CO_2)_2(Ph_2PCH_2{-}CH_2PPh_2)_3]$ as a starting material.
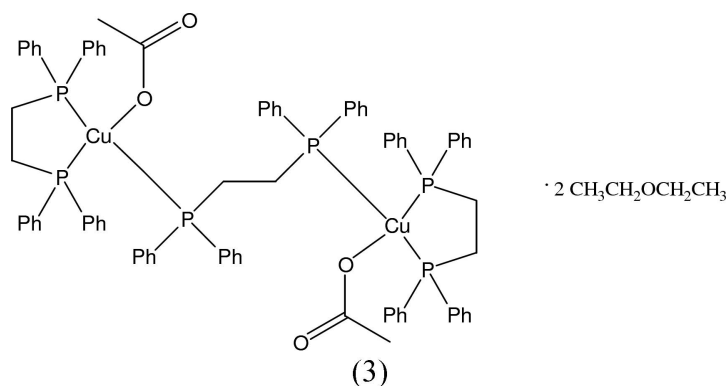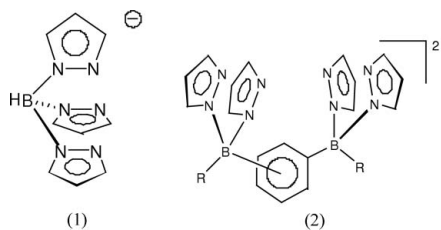
In the structure of the title compound, (3), each Cu atom is coordinated by one O atom of the acetate anion and three P atoms of two 1,2-bis(diphenylphosphino)ethane ligands within a slightly distorted tetrahedron. The asymmetric unit contains one Cu atom, one acetate ligand, one diethyl ether molecule and one and a half crystallographically independent 1,2-bis-(diphenylphosphino)ethane ligands. One of the phosphorus ligands is located in a general position, whereas the other is
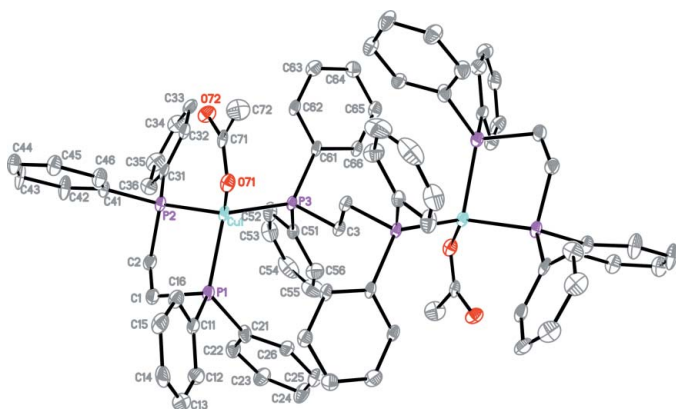
metal-organic papers

Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level and solvent molecules and H atoms have been omitted for clarity. The labelled atoms indicate the asymmetric unit; the complete molecule is generated by a centre of inversion at $(1 - x, 1 - y, 1 - z)$.

located on a centre of inversion. This ligand connects two symmetry-related Cu atoms into the complete $[Cu_2(CH_3CO_2)_2(Ph_2PCH_2CH_2PPh_2)_3]$ unit (Fig. 1). Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.6 plus three updates; *MOGUL* Version 1.0; Allen, 2002). The five-membered ring shows an envelope conformation, with atoms P2, Cu1, P1 and C1 in a plane (r.m.s. deviation = 0.037 Å) and atom C2 deviating by 0.71 (1) Å from this plane. The central methylene bridge adopts an antiperiplanar conformation, but the bridge composed of atoms C1 and C2 is in an anticlinal conformation. Only one of the two acetate O atoms (O71) coordinates to the Cu atom. The other (O72) is found 3.012 (7) Å from Cu1. On the other hand, two weak C—H⋯O contacts are found for O72 (Table 2). The diethyl ether molecules are not coordinated to the Cu atom and are located in holes in the structure.

Experimental

The title compound was obtained from a solution of copper(I) acetate (1 mmol) and 1,2-bis(diphenylphosphino)ethane (1 mmol) in tetrahydrofuran (THF, 10 ml). X-ray quality crystals of the title compound were grown by slow diffusion of diethyl ether into this THF solution at ambient temperature.

Crystal data

$[Cu_2(C_2H_3O_2)_2(C_{26}H_{24}P_2)_3]\cdot 2C_4H_{10}O$
$M_r = 1588.58$
Triclinic, $P\bar{1}$
$a = 12.6520$ (9) Å
$b = 12.8760$ (9) Å
$c = 14.2907$ (11) Å
$\alpha = 85.069$ (6)°
$\beta = 83.287$ (6)°
$\gamma = 61.629$ (5)°
$V = 2033.2$ (3) Å^3

$Z = 1$
$D_x = 1.297$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 42635 reflections
$\theta = 3.6–25.6°$
$\mu = 0.69$ mm^{-1}
$T = 173$ (2) K
Block, colourless
$0.52 \times 0.48 \times 0.44$ mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{min} = 0.714$, $T_{max} = 0.750$
42630 measured reflections

7379 independent reflections
6686 reflections with $I > 2\sigma(I)$
$R_{int} = 0.091$
$\theta_{max} = 25.4°$
$h = -15 \rightarrow 15$
$k = -15 \rightarrow 15$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.119$
$wR(F^2) = 0.291$
$S = 1.13$
7379 reflections
469 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 33.8808P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 3.04$ e Å^{-3}
$\Delta\rho_{min} = -1.48$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O71	2.015 (6)	Cu1—P1	2.343 (2)
Cu1—P2	2.275 (2)	C71—O71	1.233 (10)
Cu1—P3	2.289 (2)	C71—O72	1.281 (10)
O71—Cu1—P2	119.63 (18)	O71—Cu1—P1	110.51 (18)
O71—Cu1—P3	108.90 (18)	P2—Cu1—P1	90.30 (9)
P2—Cu1—P3	116.12 (8)	P3—Cu1—P1	109.61 (8)
P2—Cu1—P1—C1	−4.7 (3)	P1—C1—C2—P2	−55.8 (7)
P1—Cu1—P2—C2	−19.6 (3)	Cu1—P2—C2—C1	46.7 (6)
Cu1—P1—C1—C2	35.3 (6)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C33—H33⋯O72^i	0.95	2.43	3.361 (12)	166
C62—H62⋯O72	0.95	2.47	3.380 (10)	161

Symmetry code: (i) $-x, -y + 1, -z + 1$.

H atoms were refined with fixed individual displacement parameters $[U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5 U_{eq}$(methyl C)] using a riding model, with C—H = 0.95, 0.98 and 0.99 Å for aromatic, methyl and methylene H atoms, respectively. The highest peak in the final difference map is located 0.95 Å from Cu1 and the deepest hole is located 0.77 Å from Cu1.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bieller, S., Zhang, F., Bolte, M., Bats, J. W., Lerner, H.-W. & Wagner, M. (2004). *Organometallics,* **23**, 2107–2113.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus.* Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97.* University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA.* Stoe & Cie, Darmstadt, Germany.
Trofimenko, S. (1993). *Chem. Rev.* **93**, 943–980.