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Tetraethylammonium (2,2-Dicyano-1,1-ethylenedithiolato-*S*,*S*′)bis(triphenylphosphino-*P*)copper(I), $(Et_4N)[Cu(PPh_3)_2\{S_2C_2(CN)_2\}]$

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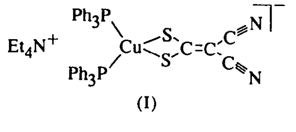
(*Received 28 August 1996; accepted 4 July 1997*)

Abstract

The crystal structure of the copper(I) compound $(C_8H_{20}N)[Cu(C_4N_2S_2)(C_{18}H_{15}P)_2]$ consists of discrete $[Et_4N]^+$ cations and $[Cu(PPh_3)_2\{S_2C_2(CN)_2\}]^-$ anions. The Cu atom in the anion is tetrahedrally coordinated by two phosphine ligands and two S atoms from the 2,2-dicyano-1,1-ethylenedithiolate ligand. The average Cu—P and Cu—S distances are 2.278 (5) and 2.423 (5) Å, respectively.

Comment

In an attempt to prepare a new series of transition metal compounds with phosphine ligands, we isolated a mononuclear copper(I) compound, $(Et_4N)[Cu(PPh_3)_2\{S_2C_2(CN)_2\}]$, (I).



(I)

The crystal structure of (I) consists of discrete $[Et_4N]^+$ cations and $[Cu(PPh_3)_2\{S_2C_2(CN)_2\}]^-$ anions. The univalent Cu atom of the anion is tetrahedrally coordinated by two phosphine ligands and two S atoms from the 2,2-dicyano-1,1-ethylenedithiolate ligand. 2,2-Dicyano-1,1-ethylenedithiolate acts as a chelating ligand; the S—Cu—S angle is 74.5 (5)°. The P—Cu—P angle of 123.74 (6)° is much bigger than the S—Cu—P angles, which range from 107.65 (6) to 118.32 (6)°. The average Cu—P and Cu—S distances are 2.278 (5) and 2.423 (5) Å, respectively; the Cu—S bond lengths are comparable with those found in Cu–$\{S_2C_2(CN)_2\}$ compounds (McCandlish *et al.*, 1968; Zhang & Yu, 1987). Fig. 1 depicts the anion structure.

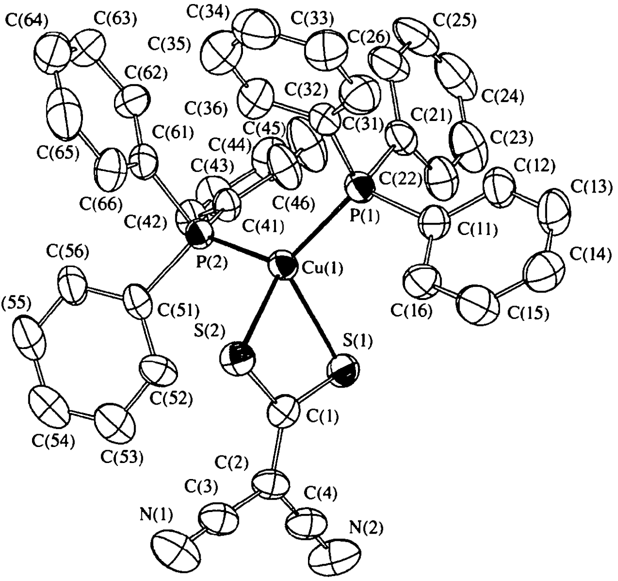


Fig. 1. The structure of $[Cu(PPh_3)_2\{S_2C_2(CN)_2\}]^-$ with displacement ellipsoids plotted at the 50% probability level.

Experimental

The title compound was obtained from the reaction of PPh_3, $K_2S_2C_2(CN)_2$, CuCl and Et_4NCl in CH_3OH, and recrystallized from CH_2Cl_2/CH_3OH.

Crystal data

$(C_8H_{20}N)[Cu(C_4N_2S_2)$-
$(C_{18}H_{15}P)_2]$
$M_r = 858.6$
Triclinic
$P\overline{1}$
$a = 10.532 (2)$ Å
$b = 12.384 (2)$ Å
$c = 18.095 (5)$ Å
$\alpha = 88.71 (2)°$
$\beta = 99.55 (2)°$
$\gamma = 99.99 (2)°$
$V = 2293.4 (13)$ Å^3
$Z = 2$
$D_x = 1.244$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.0$–$12.5°$
$\mu = 0.667$ mm^{-1}
$T = 296$ K
Prism
$0.35 \times 0.20 \times 0.20$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical ψ scan (North *et al.*, 1968)
$T_{\min} = 0.803$, $T_{\max} = 0.875$
8062 measured reflections
8050 independent reflections
6163 reflections with $I > 3\sigma(I)$
$R_{\rm int} = 0.043$
$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -21 \rightarrow 21$
3 standard reflections every 250 reflections intensity decay: none

Refinement

Refinement on F
$R = 0.052$
$wR = 0.059$
$S = 1.44$
6163 reflections
485 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o) + 0.01(F_o^2) + 1.0]$
$(\Delta/\sigma)_{\max} = 0.04$
$\Delta\rho_{\max} = 1.12$ e Å^{-3}
$\Delta\rho_{\min} = -0.88$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Cu(1)—S(1)	2.4188 (17)	P(1)—C(31)	1.825 (6)
Cu(1)—S(2)	2.4265 (15)	P(2)—C(41)	1.835 (6)
Cu(1)—P(1)	2.2672 (17)	P(2)—C(51)	1.845 (6)
Cu(1)—P(2)	2.289 (2)	P(2)—C(61)	1.827 (6)
S(1)—C(1)	1.719 (5)	N(1)—C(3)	1.171 (8)
S(2)—C(1)	1.713 (5)	N(2)—C(4)	1.158 (8)
P(1)—C(11)	1.826 (6)	C(1)—C(2)	1.405 (8)
P(1)—C(21)	1.829 (6)	C(2)—C(4)	1.408 (9)
S(1)—Cu(1)—S(2)	74.5 (5)	C(11)—P(1)—C(31)	101.1 (3)
S(1)—Cu(1)—P(1)	108.61 (6)	C(21)—P(1)—C(31)	107.5 (3)
S(1)—Cu(1)—P(2)	113.60 (6)	Cu(1)—P(2)—C(51)	115.2 (2)
S(2)—Cu(1)—P(1)	118.32 (6)	Cu(1)—P(2)—C(61)	115.1 (2)
S(2)—Cu(1)—P(2)	107.65 (6)	C(51)—P(2)—C(61)	100.8 (2)
P(1)—Cu(1)—P(2)	123.74 (6)	S(1)—C(1)—S(2)	117.5 (3)
Cu(1)—S(1)—C(1)	82.6 (2)	S(1)—C(1)—C(2)	120.0 (4)
Cu(1)—S(2)—C(1)	82.5 (2)	S(2)—C(1)—C(2)	122.3 (4)
C(11)—P(1)—C(21)	102.0 (3)		

The structure was solved by direct methods. All non-H atoms were refined with anisotropic displacement parameters. The positions of all H atoms were generated geometrically (C—H = 0.96 Å) and assigned isotropic displacement parameters; they were not refined but included in R-value calculations. All calculations were performed on an HP586/75 computer.

Data collection: *CONTROL* (Molecular Structure Corporation, 1988). Cell refinement: *MolEN* (Fair, 1990) and *CAD*-4 *SDP/VAX* (Enraf-Nonius, 1989). Data reduction: *CAD*-4 *SDP/VAX*. Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *LSFM* (B. A. Frenz & Associates Inc., 1985) in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *GCIF* (local program).

This work was supported by the National Natural Scientific Foundation of China and Natural Scientific Foundation of Fujian Province.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BS1018). Services for accessing these data are described at the back of the journal.

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Chloro(N,N',N''-trimethyl-1,5,9-triazacyclododecane-κ^3N)zinc(II) Hexafluorophosphate

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(*Received 1 October 1997; accepted 3 November 1997*)

Abstract

The preparation and crystal structure of $[ZnCl(C_{12}H_{27}N_3)]PF_6$ are described. The Zn atom has a tetrahedral environment, coordinated to three N atoms of the triaza macrocyclic ligand and to one Cl^- ion. The Zn—N distances are in the range 2.037 (1)–2.048 (1) Å, with Zn—Cl 2.2010 (4) Å.

Comment

Macrocyclic triamine complexes of zinc(II) have been studied as model structures of the zinc-containing active

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