Crystal structure of di-μ-iodo-bis{(methylcyanide-*N*)(triphenylselenophosphorane)copper(I)}, $[CuI(Ph_3PSe)(CH_3CN)]_2$

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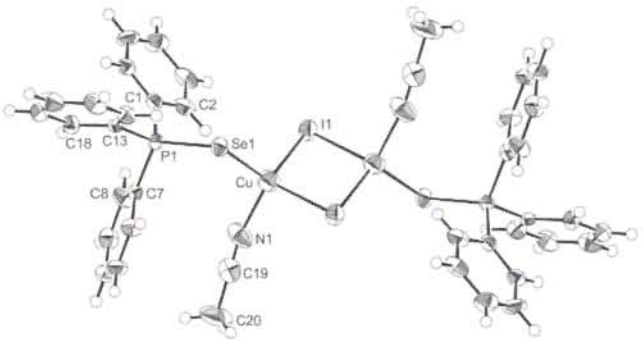
Received March 29, 1999, CCDC-No. 1267/153



Abstract

$C_{20}H_{18}CuINPSe$, monoclinic, $P12_1/n1$ (No. 14), $a = 12.288(5)$ Å, $b = 9.517(8)$ Å, $c = 18.125(7)$ Å, $\beta = 109.26(3)°$, $V = 2001.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.054$, $wR(F) = 0.059$, $T = 293$ K.

Source of material

CuI (0.100 g, 0.5 mmol) was suspended in a dry acetone/acetonitrile mixture (1:1 v/v), to it was added Ph_3PSe (0.180 g, 0.5 mmol) in ethanol and the resulting solution refluxed for 5 h. The clear solution was filtered and allowed to solidify. The creamish-yellow compound was recrystallised from an acetonitrile-ethanol mixture. The yield of colorless crystals was 70 % (mp 422 K – 423 K).

Discussion

The centrosymmetric, dinuclear structure comprises two μ_2-iodo atoms that form almost symmetric Cu–I bonds, d(Cu–I) = 2.646(2) Å and d(Cu–I') = 2.700(2) Å. The remaining donor atoms in the distorted tetrahedral environment of the Cu center are a Se atom, d(Cu–Se) = 2.459(2) Å, and an N, derived from an acetonitrile molecule, d(Cu–N) = 2.02(1) Å. The range of tetrahedral angles is 102.0(4)° to 118.48(7)°, the largest being for I–Cu–Se. Related structures have been reviewed [1].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.08 × 0.13 × 0.19 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	45.37 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC6R, ω/2θ
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5143, 4921
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 2741
$N(param)_{refined}$:	226
Programs:	DIRDIF [2], TEXSAN [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	–0.0015	0.2621	0.1756	0.0466
H(3)	4*e*	0.1670	0.2720	0.2843	0.0487
H(4)	4*e*	0.1532	0.2871	0.4113	0.0549
H(5)	4*e*	–0.0269	0.2882	0.4240	0.0543
H(6)	4*e*	–0.1940	0.2828	0.3168	0.0381
H(8)	4*e*	–0.1003	0.5270	0.1726	0.0486
H(9)	4*e*	–0.1187	0.7370	0.1012	0.0499
H(10)	4*e*	–0.2705	0.7695	–0.0142	0.0554
H(11)	4*e*	–0.4119	0.5940	–0.0578	0.0482
H(12)	4*e*	–0.3907	0.3849	0.0119	0.0436
H(14)	4*e*	–0.4278	0.0799	0.1307	0.0379
H(15)	4*e*	–0.5909	0.0841	0.1656	0.0509
H(16)	4*e*	–0.6298	0.2711	0.2341	0.0556
H(17)	4*e*	–0.4960	0.4606	0.2683	0.0563
H(18)	4*e*	–0.3370	0.4606	0.2278	0.0411
H(20a)	4*e*	–0.0933	0.5632	–0.1327	0.0989
H(20b)	4*e*	–0.2033	0.4777	–0.1760	0.0989
H(20c)	4*e*	–0.2040	0.5825	–0.1106	0.0989

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu	4*e*	–0.0788(1)	0.1232(2)	0.02169(9)	0.051(1)	0.043(1)	0.046(1)	0.0006(8)	0.0136(8)	0.0015(8)
I(1)	4*e*	0.13153(7)	0.04272(9)	0.10614(5)	0.0343(4)	0.0442(5)	0.0373(4)	0.0032(4)	0.0105(3)	–0.0058(4)
Se(1)	4*e*	–0.2358(1)	0.0905(1)	0.07617(7)	0.0395(7)	0.0264(6)	0.0321(6)	–0.0006(5)	0.0101(5)	–0.0075(5)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P(1)	4*e*	–0.2383(2)	0.2695(3)	0.1474(2)	0.026(1)	0.021(2)	0.022(1)	–0.002(1)	0.007(1)	–0.004(1)
N(1)	4*e*	–0.0930(9)	0.317(1)	–0.0257(7)	0.045(7)	0.042(8)	0.09(1)	0.004(6)	0.028(7)	0.020(7)
C(1)	4*e*	–0.1153(9)	0.275(1)	0.2360(6)	0.030(6)	0.022(6)	0.032(6)	0.003(5)	0.012(5)	0.003(5)
C(2)	4*e*	–0.007(1)	0.269(1)	0.2266(6)	0.051(8)	0.022(7)	0.030(7)	–0.004(6)	–0.006(6)	–0.002(5)
C(3)	4*e*	0.0937(9)	0.274(1)	0.2912(7)	0.018(6)	0.039(7)	0.052(8)	0.003(5)	–0.004(6)	0.008(6)
C(4)	4*e*	0.086(1)	0.283(1)	0.3664(7)	0.054(8)	0.042(8)	0.029(7)	–0.007(7)	–0.003(6)	–0.002(6)
C(5)	4*e*	–0.021(1)	0.284(1)	0.3732(6)	0.060(9)	0.050(9)	0.020(6)	0.000(7)	0.006(6)	0.006(6)
C(6)	4*e*	–0.1211(9)	0.281(1)	0.3092(6)	0.028(6)	0.033(7)	0.030(6)	0.005(5)	0.004(5)	0.004(5)
C(7)	4*e*	–0.2439(8)	0.436(1)	0.0983(6)	0.015(5)	0.024(6)	0.029(6)	–0.002(4)	0.011(4)	0.000(5)
C(8)	4*e*	–0.163(1)	0.540(1)	0.1255(7)	0.039(7)	0.030(7)	0.047(7)	–0.002(6)	0.005(6)	0.001(6)
C(9)	4*e*	–0.174(1)	0.665(1)	0.0827(7)	0.042(7)	0.036(8)	0.047(8)	–0.005(6)	0.016(6)	–0.005(6)
C(10)	4*e*	–0.265(1)	0.685(1)	0.0147(7)	0.071(9)	0.029(7)	0.043(8)	0.005(7)	0.025(7)	0.010(6)
C(11)	4*e*	–0.348(1)	0.581(1)	–0.0117(6)	0.043(7)	0.046(8)	0.029(6)	0.007(6)	0.007(6)	0.012(6)
C(12)	4*e*	–0.3350(9)	0.457(1)	0.0303(6)	0.035(6)	0.036(7)	0.036(7)	–0.007(6)	0.007(5)	0.003(6)
C(13)	4*e*	–0.3667(9)	0.268(1)	0.1758(6)	0.031(6)	0.023(6)	0.018(5)	0.003(5)	0.007(5)	0.003(5)
C(14)	4*e*	–0.4425(9)	0.159(1)	0.1582(6)	0.029(6)	0.027(7)	0.038(7)	–0.005(5)	0.011(5)	0.002(5)
C(15)	4*e*	–0.539(1)	0.161(1)	0.1793(7)	0.044(8)	0.027(7)	0.054(8)	–0.009(6)	0.013(6)	0.003(6)
C(16)	4*e*	–0.562(1)	0.270(1)	0.2195(8)	0.034(7)	0.051(9)	0.056(9)	0.004(7)	0.017(6)	0.020(7)
C(17)	4*e*	–0.483(1)	0.384(1)	0.2390(7)	0.052(8)	0.053(9)	0.038(7)	0.025(7)	0.018(6)	0.010(7)
C(18)	4*e*	–0.3883(9)	0.383(1)	0.2162(6)	0.034(6)	0.033(7)	0.039(7)	0.006(5)	0.016(6)	0.002(6)
C(19)	4*e*	–0.121(1)	0.405(1)	–0.0692(8)	0.033(7)	0.045(9)	0.064(9)	–0.009(7)	0.019(7)	–0.001(8)
C(20)	4*e*	–0.159(1)	0.517(2)	–0.1272(9)	0.11(1)	0.05(1)	0.08(1)	0.00(1)	0.02(1)	0.030(9)

Acknowledgment. The Australian Research Council is thanked for support of the crystallographic facility.

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