Crystal structure of (2,2′-bipyridine-κ^2N,N')-(*N*,*N*-dimethyldithiocarbamato-κ^2S,S')copper(II) iodide, $CuI(C_{10}H_8N_2)(C_3H_6NS_2)$

Le-Qing Fan* and Ji-Huai Wu

Huaqiao University, Institute of Materials Physical Chemistry, Key Laboratory for Functional Materials of Fujian Higher Education, Quanzhou 362021, Fujian, P. R. China

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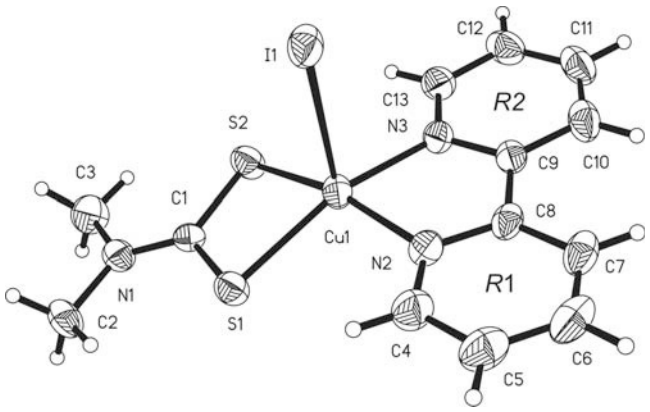

Abstract

$C_{13}H_{14}CuIn_3S_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.8080(1) Å, b = 10.1039(5) Å, c = 11.2436(2) Å, α = 66.70(2)°, β = 76.20(2)°, γ = 66.36(2)°, V = 838.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.108, T = 293 K.

Source of material

A mixture of $Cu(OAc)_2 \cdot H_2O$ (40 mg, 0.2 mmol), $NaS_2CNMe_2 \cdot 2H_2O$ (45 mg, 0.2 mmol), 2,2′-bipyridine (31 mg, 0.2 mmol) and $NaI \cdot 2H_2O$ (37 mg, 0.2 mmol) was stirred in dimethylformamide (20 mL) at room temperature in air for about one day. The vapor of *i*-PrOH was diffused slowly into the resulting solution, and after about one month gray block-shaped crystals (32 mg) were obtained.

Experimental details

The H atoms were positioned geometrically and refined as riding atoms with d(C—H) = 0.93 (aromatic) or 0.96 Å (methyl) and U_{iso}(H) = 1.2 $U_{eq}(C_{aromatic})$ or 1.5 $U_{eq}(C_{methyl})$.

Discussion

Organonitrogen ligands, such as 2,2′-bipyridine (bipy), 1,10-phenanthroline, 4,4′-bipyridine and their derivatives as suitable spacers have been extensively used in the construction of coordination complexes [1-3]. They not only are versatile ligands for coordination bonding but also can form aromatic π-π stacking interactions, C–H···π interactions and hydrogen bonding which are important supramolecular force enhancing the stability of the complexes in both solution and solid states [4,5].

In the title crystal structure, the Cu(II) atom is five-coordinated in a distorted square pyramidal manner by one I atom in the apical position with d(Cu—I) = 2.9485(9) Å, two S atoms from a $S_2CNMe_2^-$ ligand [d(Cu—S1) = 2.296(1) Å and d(Cu—S2) = 2.323(1) Å] and two N atoms from a bipy ligand in the basal plane [d(Cu—N2) = 2.004(4) Å and d(Cu—N3) = 2.015(4) Å]. Two adjacent structural units are linked to a dimer $[CuI(bipy)(S_2CNMe_2)]_2$ by face-to-face π-π interactions between bipy ring *R*1 (N2/C4-C8) to adjacent symmetry-related ring *R*2 (N3/C9-C13) with dihedral angle of 3.68°, centroid-to-centroid distance of 4.470(4) Å, and plane-to-plane distance of 3.372 Å [6]. The dimers are further connected through C2–H2C···π (*R*2) interactions ($\angle$C–H···R = 125.00° and d(C···R) = 3.559(7) Å) and C2–H2B···I hydrogen bonds ($\angle$C–H···I = 164.00° and d(C···I) = 3.963(6) Å) to form 2D structure in the (001) plane [6].

Table 1. Data collection and handling.

Crystal:	gray block, size 0.20 × 0.20 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	33.87 cm^{-1}
Diffractometer, scan mode:	Rigaku Mercury CCD, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6208, 3630
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2629
$N(param)_{refined}$:	181
Programs:	PLATON [6], SHELXS-97, SHELXL-97, SHELXTL [7]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	2*i*	0.0220	0.0026	0.3699	0.098
H(2B)	2*i*	0.0638	–0.1127	0.2961	0.098
H(2C)	2*i*	–0.0750	0.0490	0.2513	0.098
H(3A)	2*i*	0.2669	0.0973	0.0173	0.117
H(3B)	2*i*	0.0815	0.1104	0.0254	0.117
H(3C)	2*i*	0.2202	–0.0515	0.0696	0.117
H(4A)	2*i*	0.2266	0.2015	0.5966	0.086
H(5A)	2*i*	0.2799	0.2047	0.7853	0.095
H(6A)	2*i*	0.5078	0.2650	0.7841	0.094
H(7A)	2*i*	0.6743	0.3250	0.5909	0.079
H(10A)	2*i*	0.8203	0.3801	0.3994	0.084
H(11A)	2*i*	0.9495	0.4448	0.1900	0.089
H(12A)	2*i*	0.8502	0.4302	0.0251	0.088
H(13A)	2*i*	0.6290	0.3466	0.0720	0.074

* Correspondence author (e-mail: lqfan@hqu.edu.cn)

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(1)	2*i*	0.38048(7)	0.26380(7)	0.31194(6)	0.0478(3)	0.0563(4)	0.0495(3)	–0.0235(3)	–0.0069(2)	–0.0162(3)
I(1)	2*i*	0.16459(5)	0.58903(4)	0.24460(4)	0.0689(3)	0.0518(2)	0.1009(3)	–0.0102(2)	–0.0336(2)	–0.0332(2)
S(1)	2*i*	0.2063(2)	0.1229(2)	0.3901(1)	0.0604(7)	0.0587(7)	0.0491(7)	–0.0298(6)	0.0005(6)	–0.0180(6)
S(2)	2*i*	0.3735(2)	0.2040(2)	0.1344(1)	0.0700(8)	0.0665(8)	0.0504(7)	–0.0383(7)	0.0039(6)	–0.0193(6)
N(1)	2*i*	0.1553(5)	0.0571(5)	0.1985(4)	0.062(2)	0.047(2)	0.058(3)	–0.023(2)	–0.008(2)	–0.018(2)
N(2)	2*i*	0.4165(5)	0.2561(5)	0.4842(4)	0.055(2)	0.055(2)	0.052(2)	–0.017(2)	–0.012(2)	–0.015(2)
N(3)	2*i*	0.5888(5)	0.3186(4)	0.2586(4)	0.041(2)	0.050(2)	0.059(3)	–0.015(2)	–0.011(2)	–0.014(2)
C(1)	2*i*	0.2352(6)	0.1179(5)	0.2357(4)	0.049(2)	0.036(2)	0.049(3)	–0.013(2)	–0.009(2)	–0.009(2)
C(2)	2*i*	0.0308(7)	–0.0065(6)	0.2865(6)	0.066(3)	0.055(3)	0.088(4)	–0.032(3)	–0.002(3)	–0.029(3)
C(3)	2*i*	0.1834(9)	0.0529(8)	0.0664(6)	0.107(5)	0.086(4)	0.062(4)	–0.046(4)	–0.015(3)	–0.029(3)
C(4)	2*i*	0.3185(8)	0.2252(7)	0.5960(5)	0.076(4)	0.087(4)	0.051(3)	–0.033(3)	–0.006(3)	–0.018(3)
C(5)	2*i*	0.3493(9)	0.2274(8)	0.7092(6)	0.100(5)	0.079(4)	0.054(3)	–0.022(4)	–0.012(3)	–0.023(3)
C(6)	2*i*	0.4840(9)	0.2636(7)	0.7084(6)	0.094(5)	0.068(4)	0.068(4)	0.000(3)	–0.036(4)	–0.029(3)
C(7)	2*i*	0.5839(7)	0.2980(6)	0.5937(6)	0.066(3)	0.053(3)	0.081(4)	0.000(3)	–0.035(3)	–0.031(3)
C(8)	2*i*	0.5485(6)	0.2918(5)	0.4835(5)	0.051(3)	0.040(2)	0.066(3)	–0.002(2)	–0.026(2)	–0.019(2)
C(9)	2*i*	0.6447(6)	0.3293(5)	0.3552(5)	0.041(2)	0.035(2)	0.071(3)	–0.005(2)	–0.018(2)	–0.017(2)
C(10)	2*i*	0.7814(7)	0.3751(7)	0.3319(7)	0.049(3)	0.061(3)	0.107(5)	–0.014(3)	–0.023(3)	–0.032(3)
C(11)	2*i*	0.8585(7)	0.4131(7)	0.2074(7)	0.052(3)	0.061(3)	0.114(5)	–0.026(3)	–0.009(3)	–0.028(3)
C(12)	2*i*	0.8003(7)	0.4038(7)	0.1098(7)	0.055(3)	0.065(4)	0.088(4)	–0.028(3)	0.002(3)	–0.013(3)
C(13)	2*i*	0.6667(6)	0.3549(6)	0.1384(5)	0.052(3)	0.064(3)	0.062(3)	–0.024(3)	–0.004(2)	–0.012(3)

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