Crystal structure of tetrapyridinecopper(II) bis(triiodide), $[Cu(C_5H_5N)_4][I_3]_2$

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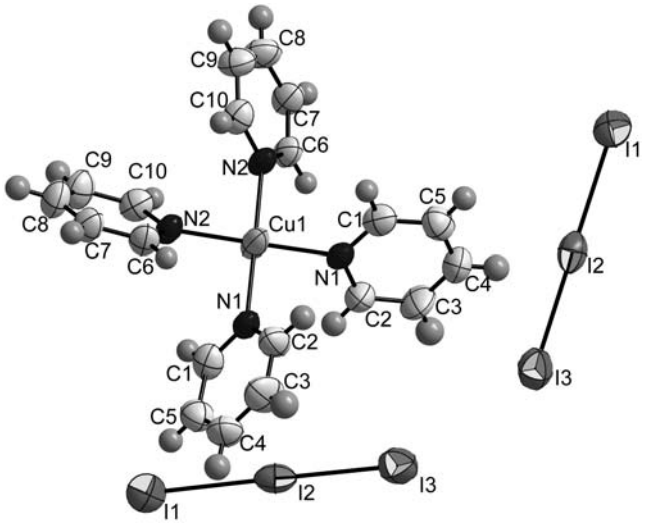

Abstract

$C_{20}H_{20}CuI_6N_4$, monoclinic, *P*2/*n* (no. 13), a = 8.658(1) Å, b = 10.648(2) Å, c = 16.638(2) Å, β = 91.245(2)°, V = 1533.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.037, $wR_{ref}(F^2)$ = 0.094, T = 296 K.

Source of material

A mixture of pyridine-4-boronic acid (61.5 mg, 0.5 mmol), CuI_2 (95.2 mg, 0.5 mmol), HNO_3 (0.5 ml) in water (10 ml) was placed in a Teflon-lined stainless steel Parr bomb. The bomb was heated at 433 K for 4 d and then cooled naturally to room temperature. Suitable single crystal for X-ray analysis was obtained by slow evaporation.

Experimental details

All the hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C—H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C).

Discussion

The previously described structures containing tetrakis-(pyridine)copper cations are the perchlorate [1], hexafluoridophosphate salt [2] and tetrapyridinecopper(II)]-2-naphthalene-1,5-disulfonate [3].

The title crystal structure is composed of discrete $Cu(C_5H_5N)_4$ cation and I^{3-} anions. The Cu(II) atom is coordinated by four nitrogen atoms of four pyridine molecules in square-planar manner differently from the tetrahedral Cu(I) complexes [1,2] and the octahedral Cu(II) complex [3]. The bond lengths d(Cu—N) of 2.012(4) and 2.028(4) Å are in the normal range and similar to those in the literature data [3]. Cu-I interaction with d(Cu—I) = 3.4783(6) Å the coordination of Cu can be described as a distorted octahedron by the Jahn-Teller distortion. The bond angles ∠N–Cu–N are in the range of 89.3° and 90.7°. The pyridine rings are twisted. The d(I—I) of 2.8444 and 3.0032 Å and ∠I–I–I = 178.46(2)° are typical for a triodide anion.

Table 1. Data collection and handling.

Crystal:	green block, size 0.20 × 0.30 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	67.67 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10484, 2983
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2189
$N(param)_{refined}$:	141
Programs:	SHELXS-97, SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*g*	0.7441	0.0343	0.9209	0.071
H(2)	4*g*	0.5411	–0.1067	0.7326	0.085
H(3)	4*g*	0.4020	–0.2407	0.8110	0.110
H(4)	4*g*	0.4408	–0.2399	0.9483	0.093
H(5)	4*g*	0.6219	–0.1040	1.0034	0.079
H(6)	4*g*	0.4434	0.1770	0.8147	0.067
H(7)	4*g*	0.3215	0.3395	0.8777	0.076
H(8)	4*g*	0.4624	0.5111	0.9188	0.085
H(9)	4*g*	0.7282	0.5123	0.9011	0.083
H(10)	4*g*	0.8397	0.3467	0.8362	0.069

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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}
C(1)	4*g*	0.6748(6)	–0.0229(6)	0.8980(4)	0.044(3)	0.059(4)	0.073(5)	–0.004(3)	0.007(3)	–0.009(3)
C(2)	4*g*	0.5553(8)	–0.1035(6)	0.7881(4)	0.103(5)	0.054(4)	0.056(4)	–0.018(4)	0.010(4)	–0.002(3)
C(3)	4*g*	0.473(1)	–0.1857(7)	0.8348(5)	0.122(6)	0.067(5)	0.086(6)	–0.050(5)	0.002(5)	–0.008(4)
C(4)	4*g*	0.4958(9)	–0.1856(6)	0.9158(5)	0.102(5)	0.059(4)	0.071(5)	–0.019(4)	0.018(4)	0.006(3)
C(5)	4*g*	0.6007(7)	–0.1044(6)	0.9484(4)	0.071(4)	0.070(4)	0.056(4)	0.005(3)	0.000(3)	0.005(3)
C(6)	4*g*	0.5011(6)	0.2462(5)	0.8315(4)	0.053(3)	0.048(3)	0.068(4)	–0.012(3)	0.018(3)	–0.004(3)
C(7)	4*g*	0.4273(7)	0.3431(6)	0.8694(4)	0.053(3)	0.056(4)	0.082(5)	0.003(3)	0.020(3)	–0.001(3)
C(8)	4*g*	0.5107(8)	0.4436(6)	0.8943(4)	0.071(4)	0.058(4)	0.084(5)	0.011(3)	0.012(4)	–0.016(3)
C(9)	4*g*	0.6682(8)	0.4452(6)	0.8829(4)	0.068(4)	0.058(4)	0.080(5)	–0.006(3)	0.002(3)	–0.019(3)
C(10)	4*g*	0.7338(6)	0.3456(5)	0.8443(4)	0.044(3)	0.056(4)	0.072(4)	–0.001(3)	0.002(3)	0.004(3)
Cu(1)	2*e*	¾	0.11208(9)	¾	0.0855(8)	0.0368(5)	0.0909(9)	0	0.0494(7)	0
I(1)	4*g*	0.07678(5)	0.11380(5)	–0.12463(3)	0.0622(3)	0.0829(3)	0.0815(4)	–0.0157(2)	–0.0091(2)	–0.0033(2)
I(2)	4*g*	–0.00581(4)	0.26790(4)	0.01938(3)	0.0523(2)	0.0693(3)	0.0783(3)	–0.0093(2)	–0.0065(2)	0.0222(2)
I(3)	4*g*	–0.09185(6)	0.41533(5)	0.15376(3)	0.0771(3)	0.0852(4)	0.0940(4)	–0.0007(2)	0.0178(3)	0.0117(3)
N(1)	4*g*	0.6535(5)	–0.0208(4)	0.8187(3)	0.064(3)	0.034(2)	0.062(3)	–0.005(2)	0.019(2)	–0.004(2)
N(2)	4*g*	0.6524(5)	0.2476(4)	0.8179(3)	0.052(3)	0.034(2)	0.071(3)	–0.003(2)	0.017(2)	–0.003(2)

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