

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Poly[[μ_2 -1,2-bis(4-pyridyl)ethene- κ^2 N:N']-di- μ_3 -bromido-dicopper(I)]Fwu Ming Shen^a and Shie Fu Lush^{b*}^aDepartment of Biotechnology, Yuanpei University, HsinChu 30015, Taiwan, and^bGeneral Education Center, Yuanpei University, HsinChu 30015, Taiwan

Correspondence e-mail: lush@mail.ypu.edu.tw

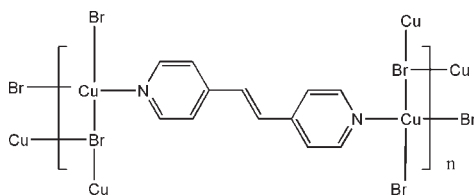
Received 30 July 2010; accepted 2 August 2010

Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.010$ Å; R factor = 0.048; wR factor = 0.096; data-to-parameter ratio = 14.2.

In the title polymeric Cu^{I} compound, $[\text{Cu}_2\text{Br}_2(\text{C}_{12}\text{H}_{10}\text{N}_2)]_n$, the Cu cation is coordinated by an N atom from the 1,2-bis(4-pyridyl)ethene ligand and three Br^- anions in a distorted tetrahedral CuBr_3N coordination geometry. Each Br^- anion bridges three Cu cations related by inversion centers, forming a stair-like polymeric chain along the a axis, and the terminal N atoms of the 1,2-bis(4-pyridyl)ethene ligand, located across an inversion center, coordinate the Cu cations from neighboring chains, forming polymeric sheets.

Related literature

For related structures, see: Yang (2009); Wang (2008); Näther & Greve (2001). For stair-like structures, see: Healy *et al.* (1989); Jasinski *et al.* (1985).



Experimental

Crystal data

 $[\text{Cu}_2\text{Br}_2(\text{C}_{12}\text{H}_{10}\text{N}_2)]_n$ $M_r = 234.56$ Monoclinic, $P2_1/c$ $a = 3.9066$ (3) Å $b = 15.1047$ (13) Å $c = 11.1050$ (9) Å $\beta = 95.149$ (2)° $V = 652.64$ (9) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 9.36$ mm⁻¹ $T = 294$ K $0.40 \times 0.10 \times 0.05$ mm

Data collection

Nonius KappaCCD diffractometer

Absorption correction: multi-scan

(SCALEPACK; Otwinowski &

Minor, 1997)

 $T_{\text{min}} = 0.487$, $T_{\text{max}} = 0.938$

3454 measured reflections

1162 independent reflections

1083 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.037$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.048$ $wR(F^2) = 0.096$ $S = 1.30$

1162 reflections

82 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.61$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.91$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Br—Cu ^I	2.5645 (12)	Br—Cu ^{II}	2.5195 (13)
Br—Cu ^I ⁱ	2.4723 (13)	N1—Cu ^I	2.009 (5)
Br—Cu ^I —N1	105.79 (16)	Br ⁱ —Cu ^I —N1	119.11 (16)
Br—Cu ^I —Br ⁱ	108.79 (4)	Br ⁱⁱ —Cu ^I —N1	109.30 (16)
Br—Cu ^I —Br ⁱⁱ	110.86 (4)	Br ⁱ —Cu ^I —Br ⁱⁱ	102.99 (4)

Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $-x + 1, -y + 1, -z + 1$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

This work was supported financially by Yuanpei University, Taiwan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5009).

References

- Healy, P. C., Kildea, J., Skelton, B. & White, A. (1989). *Aust. J. Chem.* **42**, 79–82.
- Jasinski, J. P., Roth, N. P. & Holt, E. M. (1985). *Inorg. Chim. Acta*, **97**, 91–97.
- Näther, C. & Greve, J. (2001). *Acta Cryst.* **C57**, 377–378.
- Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter & R. M. Sweet, pp. 307–326. London: Academic Press.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Wang, W. (2008). *Acta Cryst.* **E64**, m759.
- Yang, M.-H. (2009). *Acta Cryst.* **C65**, m59–m61.

supplementary materials

Acta Cryst. (2010). E66, m1071 [doi:10.1107/S1600536810030734]

Poly[[μ_2 -1,2-bis(4-pyridyl)ethene- $\kappa^2 N:N'$]-di- μ_3 -bromido-dicopper(I)]

E. M. Shen and S. F. Lush

Comment

In the structural investigations of compounds of Cu^I halide, such as bromide (Yang, 2009; Wang, 2008; Näther & Greve, 2001), has been found. A four coordination polymer, resulted from the hydrothermal treatment of CuBr with 1,2-bis(4-pyridyl)ethene.

As Fig. 1, the symmetric unit consists of one copper(I) ion, one bromide ligand and half 1,2-bis(4-pyridyl)ethene ligand, all on general positions. The Cu^I atom is tetrahedral and coordinated by three μ_3 -bridging Br atoms and the each bromide bridges the other two Cu cations, while the N atoms of 1,2-bis(4-pyridyl)ethene ligand coordinate the other Cu cations, forming the three-dimensional polymeric architecture (Fig. 2).

The polymer frameworks has four-membered Cu—Br—Cu—Br units that form the step of a stair (Healy *et al.*, 1989; Jasinski *et al.*, 1985) and 1,2-bis(4-pyridyl)ethene ligand across those stairs, shown as Fig. 2. Cu...Cu distances are between 2.8852 (16)~2.9332 (16) Å and Cu—Br—Cu angles are 71.21 (4)~102.99 (4), respectively.

Experimental

CuBr (0.1097 g, 0.50 mmol) and 1,2-bis(4-pyridyl)ethene (0.0913 g, 0.50 mmol) were mixed in 10 ml deionized water. After being stirred for 30 min, the mixture was placed in a 25 ml Teflon liner reactor and heated at 423 K in an oven for 24 h. The resulting solution was slowly cooled to room temperature. The orange transparent single crystals of the title compound were obtained in 46.45% yield.

Refinement

H atoms were positioned geometrically with C—H = 0.93 Å, and were refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

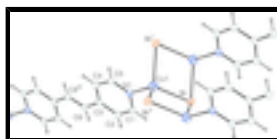


Fig. 1. The coordination environment around the Cu(I) cation with the atom numbering. Displacement ellipsoids are drawn at the 30% probability level.

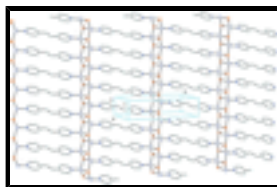


Fig. 2. A diagram of the unit cell packing showing two-dimensional sheet structure.

Poly[[μ_2 -1,2-bis(4-pyridyl)ethene- κ^2 N:N']-di- μ_3 -bromido- dicopper(I)]

Crystal data

[Cu ₂ Br ₂ (C ₁₂ H ₁₀ N ₂)]	$F(000) = 448$
$M_r = 234.56$	$D_x = 2.387 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 2187 reflections
$a = 3.9066 (3) \text{ \AA}$	$\theta = 2.5\text{--}25.0^\circ$
$b = 15.1047 (13) \text{ \AA}$	$\mu = 9.36 \text{ mm}^{-1}$
$c = 11.1050 (9) \text{ \AA}$	$T = 294 \text{ K}$
$\beta = 95.149 (2)^\circ$	Columnar, orange
$V = 652.64 (9) \text{ \AA}^3$	$0.40 \times 0.10 \times 0.05 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	1162 independent reflections
Radiation source: fine-focus sealed tube graphite	1083 reflections with $I > 2\sigma(I)$
Detector resolution: 9 pixels mm^{-1}	$R_{\text{int}} = 0.037$
CCD rotation images, thick slices scans	$\theta_{\text{max}} = 25.1^\circ$, $\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)	$h = -4 \rightarrow 4$
$T_{\text{min}} = 0.487$, $T_{\text{max}} = 0.938$	$k = -17 \rightarrow 17$
3454 measured reflections	$l = -9 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.096$	H-atom parameters constrained
$S = 1.30$	$w = 1/[\sigma^2(F_o^2) + (0.022P)^2 + 3.3443P]$
1162 reflections	where $P = (F_o^2 + 2F_c^2)/3$
82 parameters	$(\Delta/\sigma)_{\text{max}} = 0.010$
0 restraints	$\Delta\rho_{\text{max}} = 0.61 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.90 \text{ e \AA}^{-3}$

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br	0.19561 (17)	0.49650 (5)	0.33505 (6)	0.0347 (2)
Cu1	0.2671 (3)	0.56165 (6)	0.54908 (9)	0.0473 (3)
N1	0.3103 (14)	0.6931 (3)	0.5275 (5)	0.0343 (17)
C1	0.1767 (19)	0.7337 (4)	0.4255 (6)	0.039 (2)
C2	0.2046 (19)	0.8230 (5)	0.4055 (7)	0.041 (2)
C3	0.3818 (18)	0.8766 (4)	0.4912 (6)	0.034 (2)
C4	0.520 (2)	0.8355 (5)	0.5972 (6)	0.040 (2)
C5	0.4799 (18)	0.7450 (4)	0.6095 (6)	0.037 (2)
C6	0.4128 (19)	0.9711 (4)	0.4647 (7)	0.036 (2)
H1	0.05970	0.69940	0.36560	0.0470*
H2	0.10420	0.84770	0.33410	0.0490*
H4	0.63640	0.86840	0.65860	0.0480*
H5	0.57850	0.71860	0.67990	0.0440*
H6	0.30120	0.99200	0.39280	0.0440*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0345 (4)	0.0376 (4)	0.0312 (4)	−0.0036 (3)	−0.0006 (3)	0.0001 (3)
Cu1	0.0589 (6)	0.0302 (5)	0.0520 (6)	−0.0054 (4)	0.0000 (4)	0.0062 (4)
N1	0.041 (3)	0.023 (3)	0.039 (3)	−0.001 (2)	0.005 (3)	0.000 (2)
C1	0.048 (4)	0.029 (4)	0.038 (4)	0.002 (3)	−0.006 (3)	−0.002 (3)
C2	0.049 (4)	0.036 (4)	0.035 (4)	0.002 (3)	−0.006 (3)	0.007 (3)
C3	0.038 (4)	0.023 (3)	0.041 (4)	0.003 (3)	0.011 (3)	0.006 (3)
C4	0.050 (4)	0.035 (4)	0.035 (4)	−0.006 (3)	−0.001 (3)	−0.006 (3)
C5	0.043 (4)	0.029 (3)	0.037 (4)	−0.004 (3)	−0.003 (3)	0.001 (3)
C6	0.043 (4)	0.029 (4)	0.036 (4)	0.002 (3)	−0.001 (3)	0.000 (3)

Geometric parameters (\AA , $^\circ$)

Br—Cu1	2.5645 (12)	C3—C6	1.465 (9)
Br—Cu1 ⁱ	2.4723 (13)	C4—C5	1.384 (10)

supplementary materials

Br—Cu1 ⁱⁱ	2.5195 (13)	C6—C6 ⁱⁱⁱ	1.321 (10)
N1—Cu1	2.009 (5)	C1—H1	0.9300
N1—C1	1.351 (8)	C2—H2	0.9300
N1—C5	1.332 (8)	C4—H4	0.9300
C1—C2	1.373 (10)	C5—H5	0.9300
C2—C3	1.386 (10)	C6—H6	0.9300
C3—C4	1.396 (10)		
Br…C1	3.724 (6)	C4…H6 ⁱⁱⁱ	2.7000
Br…H1	3.1300	C6…H4 ⁱⁱⁱ	2.7800
Br…H2 ^{iv}	3.0900	H1…Br	3.1300
Br…H6 ^{iv}	3.0500	H2…H6	2.3800
C1…C5 ^v	3.551 (10)	H2…Br ^{ix}	3.0900
C2…C3 ^v	3.527 (10)	H4…C6 ⁱⁱⁱ	2.7800
C2…C4 ^v	3.570 (11)	H4…H6 ⁱⁱⁱ	2.2000
C3…C2 ^{vi}	3.527 (10)	H5…C2 ^x	3.0800
C4…C2 ^{vi}	3.570 (11)	H6…H2	2.3800
C5…C1 ^{vi}	3.551 (10)	H6…Br ^{ix}	3.0500
C6…C6 ^{vii}	3.498 (10)	H6…C4 ⁱⁱⁱ	2.7000
C2…H5 ^{viii}	3.0800	H6…H4 ⁱⁱⁱ	2.2000
Cu1—Br—Cu1 ⁱ	71.21 (4)	C2—C3—C6	118.5 (6)
Cu1—Br—Cu1 ⁱⁱ	69.15 (4)	C4—C3—C6	124.8 (6)
Cu1 ⁱ —Br—Cu1 ⁱⁱ	102.99 (4)	C3—C4—C5	118.9 (6)
Br—Cu1—N1	105.79 (16)	N1—C5—C4	124.6 (6)
Br—Cu1—Br ⁱ	108.79 (4)	C3—C6—C6 ⁱⁱⁱ	124.9 (7)
Br—Cu1—Br ⁱⁱ	110.86 (4)	N1—C1—H1	118.00
Br ⁱ —Cu1—N1	119.11 (16)	C2—C1—H1	118.00
Br ⁱⁱ —Cu1—N1	109.30 (16)	C1—C2—H2	120.00
Br ⁱ —Cu1—Br ⁱⁱ	102.99 (4)	C3—C2—H2	120.00
Cu1—N1—C1	121.2 (4)	C3—C4—H4	121.00
Cu1—N1—C5	122.8 (4)	C5—C4—H4	121.00
C1—N1—C5	115.9 (5)	N1—C5—H5	118.00
N1—C1—C2	123.5 (6)	C4—C5—H5	118.00
C1—C2—C3	120.3 (7)	C3—C6—H6	118.00
C2—C3—C4	116.8 (6)	C6 ⁱⁱⁱ —C6—H6	118.00
Cu1 ⁱ —Br—Cu1—N1	-129.06 (17)	Br ⁱ —Cu1—N1—C1	-97.7 (5)
Cu1 ⁱⁱ —Br—Cu1—N1	118.37 (17)	Cu1—N1—C1—C2	-178.8 (6)
Cu1 ⁱ —Br—Cu1—Br ⁱ	0.00 (4)	C5—N1—C1—C2	-1.0 (10)
Cu1 ⁱⁱ —Br—Cu1—Br ⁱ	-112.57 (5)	Cu1—N1—C5—C4	179.0 (6)
Cu1 ⁱ —Br—Cu1—Br ⁱⁱ	112.57 (5)	C1—N1—C5—C4	1.2 (10)
Cu1 ⁱⁱ —Br—Cu1—Br ⁱⁱ	0.00 (4)	N1—C1—C2—C3	1.1 (11)
Cu1 ⁱ —Br ⁱ —Cu1—N1	121.22 (19)	C1—C2—C3—C4	-1.3 (11)
Cu1 ⁱⁱ —Br ⁱⁱ —Cu1—Br	0.00 (4)	C1—C2—C3—C6	178.4 (7)

Cu1 ⁱⁱ —Br ⁱⁱ —Cu1—N1	-116.22 (17)	C2—C3—C4—C5	1.4 (10)
Cu1 ⁱ —Br ⁱ —Cu1—Br	0.00 (5)	C6—C3—C4—C5	-178.3 (7)
Br ⁱⁱ —Cu1—N1—C1	144.4 (5)	C2—C3—C6—C6 ⁱⁱⁱ	-176.7 (7)
Br ⁱⁱ —Cu1—N1—C5	-33.2 (6)	C4—C3—C6—C6 ⁱⁱⁱ	3.0 (12)
Br ⁱ —Cu1—N1—C5	84.7 (5)	C3—C4—C5—N1	-1.5 (11)
Br—Cu1—N1—C1	25.0 (5)	C3—C6—C6 ⁱⁱⁱ —C3 ⁱⁱⁱ	-180.0 (7)
Br—Cu1—N1—C5	-152.6 (5)		

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y+2, -z+1$; (iv) $-x, y-1/2, -z+1/2$; (v) $x-1, y, z$; (vi) $x+1, y, z$; (vii) $-x, -y+2, -z+1$; (viii) $x, -y+3/2, z-1/2$; (ix) $-x, y+1/2, -z+1/2$; (x) $x, -y+3/2, z+1/2$.

Fig. 1

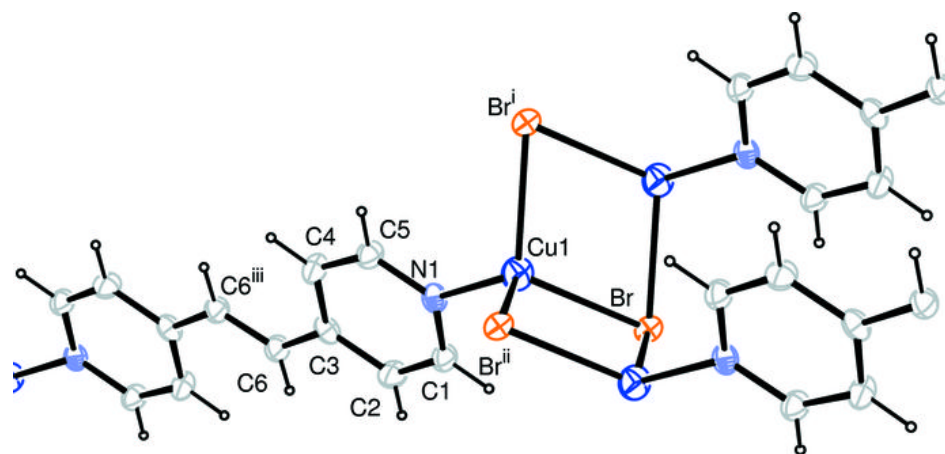


Fig. 2

