

Table 7. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>) for (4)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U <sub>eq</sub>
Cu1	0.2306 (2)	1/4	0.1580 (1)	0.025 (4)
S1	0.6009 (3)	1/4	-0.1249 (2)	0.0212 (7)
O1	0.3103 (8)	1/4	-0.0786 (5)	0.026 (2)
O2	0.6394 (6)	0.3139 (2)	-0.2235 (4)	0.033 (2)
O3	0.7840 (8)	1/4	0.0323 (5)	0.027 (2)
N1	0.3341 (7)	0.3527 (2)	0.1965 (4)	0.018 (2)
C3	0.643 (1)	0.4304 (4)	0.3492 (6)	0.033 (2)
N3	0.0071 (9)	0.3998 (2)	0.0034 (5)	0.035 (2)
C2	0.511 (1)	0.4852 (4)	0.2634 (6)	0.037 (2)
C4	0.5497 (9)	0.3644 (4)	0.3116 (6)	0.031 (2)
C1	0.2182 (9)	0.4093 (4)	0.1194 (5)	0.021 (2)
N2	0.2976 (8)	0.4761 (3)	0.1514 (5)	0.031 (2)
O4	0.1031 (9)	1/4	0.3825 (5)	0.032 (2)
O5	0.1168 (6)	0.3520 (2)	0.6096 (3)	0.034 (1)

Table 8. Selected geometric parameters (Å, °) for (4)

Cu1—O4	1.937 (4)	Cu1—N1'	2.045 (5)
Cu1—O1	1.948 (4)	Cu1—N1	2.045 (5)
O4—Cu1—O1	172.7 (2)	O1—Cu1—N1	94.4 (1)
O4—Cu1—N1'	87.6 (1)	N1'—Cu1—N1	147.5 (1)
O4—Cu1—N1	87.6 (1)		

D—H...A	D—H	H...A	D...A	D—H...A
N3—H4...O2 <sup>ii</sup>	0.881 (5)	2.312 (3)	2.946 (5)	135.4 (3)
N3—H4...O3 <sup>ii</sup>	0.881 (5)	2.468 (2)	3.083 (5)	133.5 (3)
N3—H5...N2 <sup>iii</sup>	0.835 (5)	2.193 (5)	3.015 (7)	167.8 (3)
O5—H8...O2 <sup>iv</sup>	0.879 (3)	2.078 (3)	2.887 (5)	152.6 (2)
O5—H9...O2 <sup>v</sup>	1.024 (3)	1.880 (3)	2.889 (4)	167.6 (2)
O4—H6...O5	0.894 (4)	2.166 (4)	2.652 (5)	113.5 (1)

Symmetry codes: (i)  $x, \frac{1}{2} - y, z$ ; (ii)  $x - 1, y, z$ ; (iii)  $-x, 1 - y, -z$ ; (iv)  $x - 1, y, 1 + z$ ; (v)  $x, y, 1 + z$ .

Structure solutions were performed by Patterson and Fourier syntheses using *SHELXS86* (Sheldrick, 1985), refinements by full-matrix least squares using *SHELX76* (Sheldrick, 1976) and *Xtal3.2* (Hall, Flack & Stewart, 1992); anomalous-dispersion was recognized. In compounds (1) and (2), the  $\Delta\rho_{\max} > 1.0 \text{ \AA}^{-3}$  were located close to the metal atoms. All H atoms were located from the difference Fourier maps after the refinements of all heavy atoms. The H-atom parameters were refined with fixed coordinates and displacement parameters ( $U_{\text{iso}} = 0.05 \text{ \AA}^2$ ) because of the untenability of their positions and the lability of the compounds. The high *R* value for compound (2) compared with those of the other structures is due to the instability of the compound, which is also apparent from the high reduction of the used reflections. In the case of compound (3) the refinement of the inverted structure gave *R* = 0.034 and *wR* = 0.037. Figs. 1 to 4 were prepared using *PLATON* (Spek, 1990). Computers used were VAX 8650 and Convex C 3840.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, along with packing diagrams, have been deposited with the IUCr (Reference: HR1034). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1996). **C52**, 56–59

## A Polymeric Cu<sup>I</sup> Complex of Bis(1-pyrazolylmethyl)(2-pyridylmethyl)amine (BPPA), [Cu<sub>2</sub>(μ-BPPA)(μ-I)<sub>2</sub>]<sub>n</sub>

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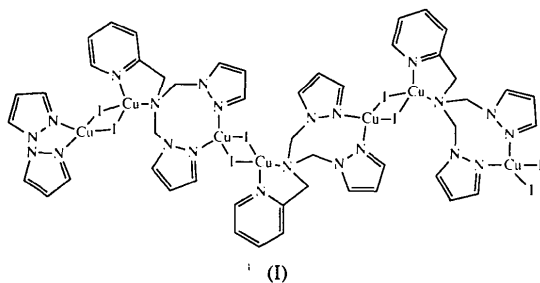
(Received 30 March 1995; accepted 12 July 1995)

### Abstract

The title complex, *catena*-poly[copper-di-μ-iodo-copper-μ-{bis(1-pyrazolylmethyl)(2-pyridylmethyl)amine}-N,N':N<sup>2</sup>,N<sup>2'</sup>], [Cu<sub>2</sub>I<sub>2</sub>(C<sub>14</sub>H<sub>16</sub>N<sub>6</sub>)<sub>n</sub>], is reported. The BPPA ligand, bis(1-pyrazolylmethyl)(2-pyridylmethyl)amine, acts as a bis-bidentate ligand reacting with copper(I) iodide in acetonitrile to form the title complex. In the repeat unit of the title complex, one Cu<sup>I</sup> atom is ligated by two pyrazole groups from one ligand and the other Cu<sup>I</sup> atom is ligated by one pyridine group and an amine from another ligand. The Cu<sup>+</sup> ions are separated by a distance of 2.587 (1) Å.

**Comment**

The ligand bis(1-pyrazolylmethyl)(2-pyridylmethyl)-amine (BPPA) was prepared according to a modification of the literature method (Driessen, 1982) and was designed to act as a tetradentate ligand like tris(3,5-dimethyl-1-pyrazolylmethyl)amine (amtd) (van Driel, Driessen & Reedijk, 1985; Kleywegt *et al.*, 1985) or (1-methyl-2-imidazolyl)bis(2-pyridylmethyl)amine (BPIA) (Oberhausen, O'Brien, Richardson & Buchanan, 1990). The amtd ligand coordinates to divalent transition metal ions through its three pyrazole N atoms and its amine N atom (Driessen, de Graaff, Ochocki & Reedijk, 1988; Spek, Driessen & Wiesmeijer, 1988). Chen, Richardson & Buchanan (1994) investigated copper(II) complexes with imidazolyl-containing ligands. The structure of the [(BPIA)<sub>2</sub>Cu<sub>2</sub>](CF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub> complex, which is formed by sharing two BPIA ligands, is reported by Wei, Murthy, Tyeklár & Karlin (1994). The coordination mode of the BPPA ligand is similar to that of BPIA, where BPPA has a pyridine and two pyrazole substituents. Here, we report the crystal structure of a polymeric copper(I) complex, (I), *catena*-poly[copper-di- $\mu$ -iodo-copper- $\mu$ -{bis(1-pyrazolylmethyl)(2-pyridylmethyl)amine}-*N,N'*:*N*<sup>2</sup>,*N*<sup>2'</sup>] containing a mixed pyrazole/pyridine tripod ligand.



As Fig. 1 shows, (I) crystallizes as a four-coordinate Cu<sup>I</sup> polymer, each Cu atom utilizing part of a BPPA ligand and two I<sup>-</sup> ions. The BPPA ligand acts as a bis-bidentate ligand, reacting with copper(I) iodide to obtain the polymer. In the repeat unit of the title complex, one Cu<sup>I</sup> atom is ligated by two pyrazole groups from one ligand and the other Cu<sup>I</sup> atom is ligated by one pyridine group and an amine from a second ligand. The Cu1—N1 bond length is quite long, indicating that the amine N atom, N1, bonds more weakly to the Cu<sup>I</sup> ion compared with the other three N atoms originating from the pyridyl or pyrazolyl donors. The Cu1—N6 distance is normal and falls within the range of typical Cu—N(py) distances for tetracoordinate Cu<sup>I</sup> complexes (Dyason, Engelhardt, Healy & White, 1984; Healy, Pakawatchai & White, 1983). The Cu—N(pyrazole) (Cu2—N2, Cu2—N4) distances are in the range of those observed for a Cu<sup>I</sup> dimer formed with the *N,N*-bis(pyrazol-1-ylmethyl)benzylamine ligand (Sheu *et al.*, 1995).

Two I<sup>-</sup> ions act as bridging ligands to form Cu<sub>2</sub>I<sub>2</sub> rhombohedra. The Cu1—I1—Cu2 bridging angle is 58.85(3)° and the I1—Cu1—I2 angle is 123.74(4)°. The Cu1—I distances are shorter than the Cu2—I distances. The coordination of the copper(I) ion can be described as distorted tetrahedral with angles ranging from 77.92(23) to 123.74(4)° for Cu1 and from 102.06(17) to 120.49(17)° for Cu2. The presence of the five-membered chelate ring and long Cu—N(amine) distance appear to be responsible for the acute N1—Cu1—N6 angle observed for this complex. The angles around the amine N atom, N1, do not diverge much from the ideal tetrahedral angles.

The I1...I2 distance is close to that observed for [Cu(pyridine)<sub>2</sub>]<sub>2</sub> (Dyason *et al.*, 1984). The Cu...Cu

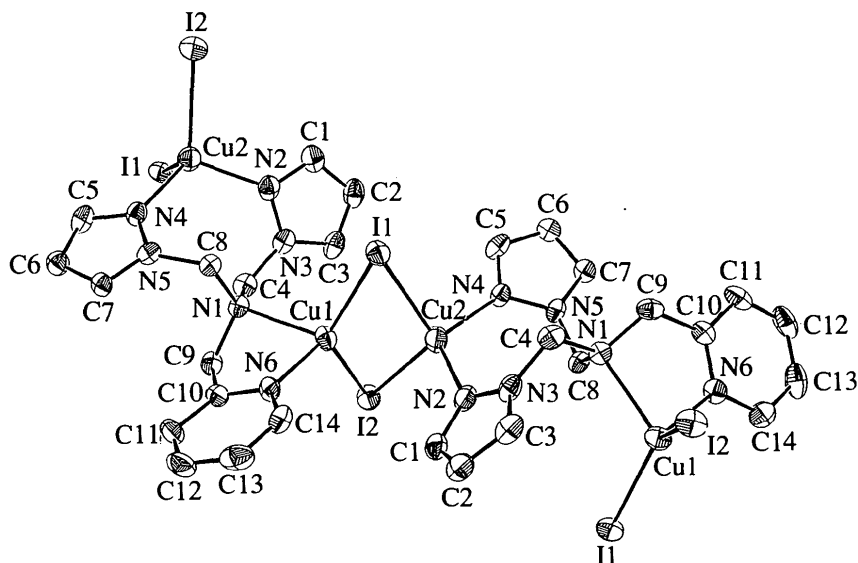


Fig. 1. ORTEP drawing (Johnson, 1965) of the repeated unit of the title compound with 50% probability ellipsoids for non-H atoms.

distance, 2.587 (1) Å, is much shorter than those of [CuI(pyridine)<sub>2</sub>]<sub>2</sub> [2.699 (5) Å; Dyason *et al.*, 1984], [Cu(N,N'-diisopropylethylenediamine)I]<sub>2</sub> [2.733 (2) Å; Haitko, 1984], {CuI[N,N-bis(pyrazol-1-ylmethyl)benzylamine]}<sub>2</sub> [3.028 (3) Å; Sheu *et al.*, 1995] and [CuI(2-methylpyridine)<sub>2</sub>]<sub>2</sub> [3.083 (3) Å; Healy *et al.*, 1983], but is close to the short Cu··Cu distances [2.582 (10) and 2.545 (15) Å] of the [Cu<sub>4</sub>L<sub>4</sub>(quin)<sub>4</sub>] complex, which is considered as having some possible Cu—Cu interaction (Rath, Holt & Tanimura, 1986). This means that some interaction may also occur between Cu1 and Cu2 in the title complex.

## Experimental

The ligand was prepared according to a modification of the literature method (Driessen, 1982). An acetonitrile solution of the ligand (0.268 g, 1 mmol) was mixed with an acetonitrile solution of copper(I) iodide (0.191 g, 1 mmol) and left to stand at room temperature. Colourless crystals of the title complex were obtained; their density  $D_m$  was measured by flotation.

### Crystal data

[Cu <sub>2</sub> I <sub>2</sub> (C <sub>14</sub> H <sub>16</sub> N <sub>6</sub> )]	Mo K $\alpha$ radiation
$M_r = 649.21$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 25 reflections
$P2_1/n$	$\theta = 9.7\text{--}12.4^\circ$
$a = 10.224$ (1) Å	$\mu = 5.447$ mm <sup>-1</sup>
$b = 14.700$ (2) Å	$T = 298$ K
$c = 13.069$ (3) Å	Plate
$\beta = 103.63$ (2) $^\circ$	0.40 × 0.40 × 0.25 mm
$V = 1908.9$ (6) Å <sup>3</sup>	Colourless
$Z = 4$	
$D_x = 2.259$ Mg m <sup>-3</sup>	
$D_m = 2.26$ Mg m <sup>-3</sup>	

### Data collection

Nonius CAD-4 diffractometer	2104 observed reflections
$\theta/2\theta$ scans	[ $I > 2\sigma(I)$ ]
Absorption correction: $\psi$ scan (North, Phillips & Mathews, 1968)	$\theta_{\max} = 22.5^\circ$
$T_{\min} = 0.745$ , $T_{\max} = 1.00$	$h = -10 \rightarrow 10$
2643 measured reflections	$k = 0 \rightarrow 15$
2482 independent reflections	$l = 0 \rightarrow 14$
	3 standard reflections
	frequency: 60 min
	intensity decay: <4%

### Refinement

Refinement on $F$	$\Delta\rho_{\max} = 0.67$ e Å <sup>-3</sup>
$R = 0.029$	$\Delta\rho_{\min} = -0.57$ e Å <sup>-3</sup>
$wR = 0.026$	Extinction correction: Zachariasen (1967)
$S = 2.89$	Extinction coefficient: $1.28$ (8) × 10 <sup>-4</sup>
2104 reflections	Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
218 parameters	
H-atom parameters not refined	
$w = 1/\sigma^2(F)$	
$(\Delta/\sigma)_{\max} = 0.0014$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$B_{\text{eq}} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	$x$	$y$	$z$	$B_{\text{eq}}$
Cu1	0.10232 (10)	0.55199 (6)	0.24099 (7)	3.89 (5)
Cu2	0.28858 (10)	0.67322 (6)	0.29875 (8)	4.56 (3)
I1	0.26271 (5)	0.56395 (4)	0.12420 (4)	4.03 (3)
I2	0.15593 (6)	0.60958 (4)	0.43588 (4)	4.47 (3)
N1	0.0343 (6)	0.4006 (4)	0.2458 (4)	3.0 (3)
N2	0.1922 (7)	0.7897 (4)	0.2388 (5)	4.4 (4)
N3	0.2366 (7)	0.8499 (4)	0.1763 (5)	4.0 (3)
N4	0.4902 (6)	0.6928 (4)	0.3423 (4)	3.6 (3)
N5	0.5576 (7)	0.7719 (4)	0.3708 (4)	3.4 (3)
N6	-0.0975 (6)	0.5603 (4)	0.1770 (4)	3.5 (3)
C1	0.0616 (9)	0.8081 (6)	0.2245 (7)	4.9 (5)
C2	0.0226 (9)	0.8761 (6)	0.1505 (7)	5.1 (5)
C3	0.1374 (10)	0.9024 (6)	0.1234 (6)	4.5 (5)
C4	0.1246 (9)	0.3483 (5)	0.3312 (6)	4.0 (4)
C5	0.5854 (10)	0.6313 (5)	0.3516 (6)	4.4 (4)
C6	0.7129 (8)	0.6695 (6)	0.3832 (6)	4.0 (4)
C7	0.6911 (8)	0.7588 (5)	0.3950 (5)	3.5 (4)
C8	0.0157 (7)	0.3594 (5)	0.1419 (5)	3.1 (4)
C9	-0.0947 (8)	0.4220 (5)	0.2735 (5)	3.9 (4)
C10	-0.1719 (8)	0.4935 (5)	0.2013 (5)	3.4 (4)
C11	-0.3099 (9)	0.4918 (6)	0.1653 (6)	4.7 (5)
C12	-0.3745 (9)	0.5621 (8)	0.1022 (8)	6.0 (6)
C13	-0.2977 (11)	0.6300 (7)	0.0747 (7)	5.7 (5)
C14	-0.1599 (9)	0.6277 (5)	0.1144 (6)	4.5 (5)

Table 2. Selected geometric parameters (Å, °)

Cu1—Cu2	2.587 (1)	I1—I2	4.509 (1)
Cu1—I1	2.496 (1)	Cu1—I2	2.617 (1)
Cu1—N1	2.337 (6)	Cu1—N6	2.020 (6)
Cu2—I1	2.751 (1)	Cu2—I2	2.659 (1)
Cu2—N2	2.038 (6)	Cu2—N4	2.026 (6)
Cu1—I1—Cu2	58.85 (3)	Cu1—I2—Cu2	58.73 (3)
I1—Cu1—I2	123.74 (4)	I1—Cu1—N1	109.03 (14)
I1—Cu1—N6	119.23 (17)	I2—Cu1—N1	106.11 (13)
I2—Cu1—N6	110.05 (16)	N1—Cu1—N6	77.9 (2)
I1—Cu2—I2	112.88 (4)	I1—Cu2—N2	103.20 (18)
I1—Cu2—N4	102.06 (17)	I2—Cu2—N2	106.1 (2)
I2—Cu2—N4	120.49 (17)	N2—Cu2—N4	110.9 (3)
Cu1—N1—C4	112.1 (4)	Cu1—N1—C8	110.2 (4)
Cu1—N1—C9	95.3 (4)	C4—N1—C8	114.0 (5)
C4—N1—C9	111.0 (5)	C8—N1—C9	112.8 (5)
Cu2—N2—N3	125.5 (5)	Cu2—N2—C1	127.0 (6)
Cu2—N4—N5	128.0 (5)	Cu2—N4—C5	127.7 (5)
Cu1—N6—C10	115.9 (5)	Cu1—N6—C14	125.8 (5)

The structure was solved by the heavy-atom method; subsequent Fourier syntheses based on the heavy atom revealed the positions of all non-H atoms. Least-squares refinement including anisotropic displacement parameters for all non-H atoms was performed.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1992). Cell refinement: *CAD-4 Software*. Data reduction: *NRCC-SDP-VAX* (Gabe, Page, White & Lee, 1987). Program(s) used to solve structure: *NRCC-SDP-VAX*. Program(s) used to refine structure: *NRCC-SDP-VAX*. Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *NRCC-SDP-VAX*.

We wish to thank the National Science Council of the Republic of China for support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1186). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1996). **C52**, 59–61

**catena-Poly[dinitratocobalt(II)- $\mu$ -2,5-bis-{2-(2-pyridyl)ethynyl}thiophene- $N:N'$ ], [Co(NO<sub>3</sub>)<sub>2</sub>(C<sub>18</sub>H<sub>10</sub>N<sub>2</sub>S)]<sub>3</sub>**

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(Received 7 February 1995; accepted 26 June 1995)

## Abstract

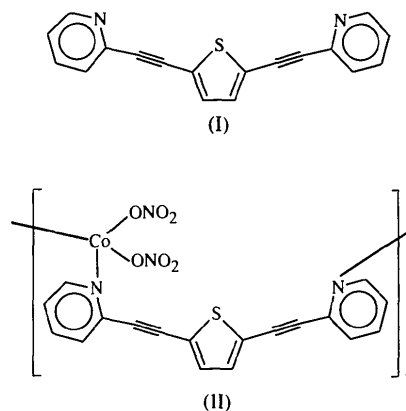
The single-crystal X-ray structure of a one-dimensional chain of nitrate-coordinated Co ions connected by rigid rod-like N donor ligands is described.

## Comment

We have recently begun a program to prepare complexes in which arrays of metal atoms are held together by

rigid electron-rich connecting rods (Neenan, Driessen, & Reedijk, 1995). Materials of this type may find application as the building blocks for conducting and photo-conducting solids, or as molecular wires.

Recently, we prepared the ligand 2,5-bis[2-(2-pyridyl)ethynyl]thiophene, (I), and report here the crystal and molecular structure of the infinite polymer, (II), that the ligand forms with cobalt(II) nitrate (see Figs. 1 and 2).



In the asymmetric unit Co<sub>3</sub>(L)<sub>3</sub>(NO<sub>3</sub>)<sub>6</sub>, each Co ion is coordinated to two nitrate ions and to two pyridine N atoms from two different ligands. The Co ions and the ligands form an infinite one-dimensional chain in the [101] direction.

The coordination geometry of the Co ions can be described as highly distorted octahedral, with both pyridine N atoms at distances between 2.06 and 2.09 Å, with two O atoms, one of each nitrate ion, coordinated at distances between 1.98 and 2.06 Å, and another two O atoms, one of each nitrate ion, completing the octahedral coordination at longer distances between 2.36 and 2.52 Å.

In one of the ligands in the asymmetric unit the donor N atoms point in opposite directions, while in the two other ligands the N atoms point in the same direction. The pyridine–thiophene connection is almost linear, as expected for an *sp*–*sp*-coupled system. The pyridine and thiophene rings are planar.

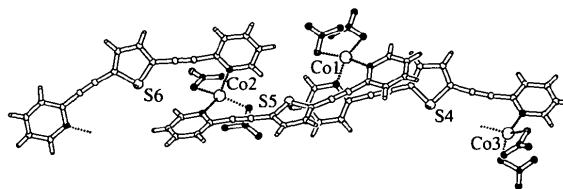


Fig. 1. *PLUTON* (Spek, 1992) projection of the asymmetric unit Co<sub>3</sub>(L)<sub>3</sub>(NO<sub>3</sub>)<sub>6</sub>.