Tris(1,10-phenanthroline)copper(II) tricyanomethanide

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The crystal structure of $[Cu(C_{12}H_8N_2)_3][C(CN)_3]_2$ is composed of discrete $[Cu(phen)_3]^{2+}$ cations (phen is 1,10-phenanthroline) and $[C(CN)_3]^-$ anions. The Cu^{II} atom is octahedrally coordinated by the three phen ligands. As a consequence of the Jahn–Teller effect, the two axial Cu—N bonds of 2.219 (3) and 2.238 (3) Å are longer than the equatorial Cu—N bonds, which are in *trans* positions, paired in two couples of almost equal distance [2.066 (3)/2.050 (3) and 2.121 (3)/2.121 (3) Å].

Comment

The structure of the five-coordinate Cu^{II} complex $[Cu(L)_2C(CN)_3]C(CN)_3$ ($L = 2,2'$-bipyridine) is known (Potočňák *et al.*, 1997). During an attempt to prepare the analogous complex with $L = 1,10$-phenanthroline (phen), the hexacoordinate Cu^{II} complex $[Cu(phen)_3][C(CN)_3]_2$, the title complex, (I), was isolated. We present here the structure of (I).

(I)
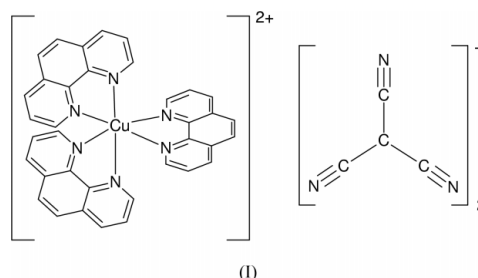
Experimental

Crystals of (I) were prepared by mixing a 0.1 *M* aqueous solution of $Cu(NO_3)_2$ (5 ml) with a 0.1 *M* ethanol solution of phen (10 ml). To the resulting blue solution, a 0.1 *M* aqueous ethanol solution of $KC(CN)_3$ (5 ml) was added (all solutions were warmed before mixing). Light-green dendritic crystals appeared within one week. The crystals were filtered off and dissolved in a warm mixture of ethanol and water (1:1). After one week, light-green prismatic crystals of (I) were filtered off and dried in air.

Crystal data

$[Cu(C_{12}H_8N_2)_3](C_4N_3)_2$	$D_x = 1.391$ Mg m^{-3}
$M_r = 784.29$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 8000
$a = 9.3854$ (12) Å	reflections
$b = 31.179$ (5) Å	$\theta = 1.7$–$26.0°$
$c = 12.7972$ (18) Å	$\mu = 0.63$ mm^{-1}
$\beta = 91.084$ (16)°	$T = 220$ (1) K
$V = 3744.2$ (9) Å^3	Prism, light green
$Z = 4$	$0.30 \times 0.21 \times 0.09$ mm

Data collection

Stoe IPDS diffractometer	6872 independent reflections
φ scans	4834 reflections with $I > 2\sigma(I)$
Absorption correction: numerical	$R_{int} = 0.050$
(*FACE* in *IPDS*; Stoe & Cie, 1999)	$\theta_{max} = 26.0°$
	$h = -11 \rightarrow 10$
$T_{min} = 0.885$, $T_{max} = 0.949$	$k = -38 \rightarrow 38$
19431 measured reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o{}^2) + (0.113P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.044$	$+ 2.769P]$
$wR(F^2) = 0.152$	where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 0.82$	$(\Delta/\sigma)_{max} < 0.001$
6872 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
514 parameters	$\Delta\rho_{min} = -0.62$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cu1—N60	2.050 (3)	C2—C4	1.406 (7)
Cu1—N40	2.066 (3)	C3—N3	1.158 (7)
Cu1—N50	2.121 (3)	C3—C4	1.408 (7)
Cu1—N20	2.121 (3)	C5—N5	1.162 (6)
Cu1—N10	2.219 (3)	C5—C8	1.409 (6)
Cu1—N30	2.238 (3)	C6—N6	1.144 (6)
C1—N1	1.143 (7)	C6—C8	1.421 (6)
C1—C4	1.395 (7)	C7—N7	1.146 (6)
C2—N2	1.173 (6)	C7—C8	1.413 (6)
N60—Cu1—N40	171.67 (11)	N10—Cu1—N30	171.35 (11)
N60—Cu1—N50	80.36 (12)	N1—C1—C4	178.9 (10)
N40—Cu1—N50	95.79 (11)	N2—C2—C4	179.5 (6)
N60—Cu1—N20	94.03 (11)	N3—C3—C4	178.5 (7)
N40—Cu1—N20	90.50 (11)	C1—C4—C3	119.5 (5)
N50—Cu1—N20	172.01 (11)	C1—C4—C2	121.2 (5)
N60—Cu1—N10	93.31 (11)	C3—C4—C2	119.2 (4)
N40—Cu1—N10	94.49 (11)	N5—C5—C8	177.9 (5)
N50—Cu1—N10	96.84 (11)	N6—C6—C8	178.6 (6)
N20—Cu1—N10	77.71 (10)	N7—C7—C8	178.7 (6)
N60—Cu1—N30	94.49 (11)	C7—C8—C5	119.6 (4)
N40—Cu1—N30	77.94 (12)	C7—C8—C6	119.9 (4)
N50—Cu1—N30	88.17 (11)	C5—C8—C6	120.5 (4)
N20—Cu1—N30	97.98 (11)		

The H-atom positions were placed in calculated positions and refined riding on their parent C atoms, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.
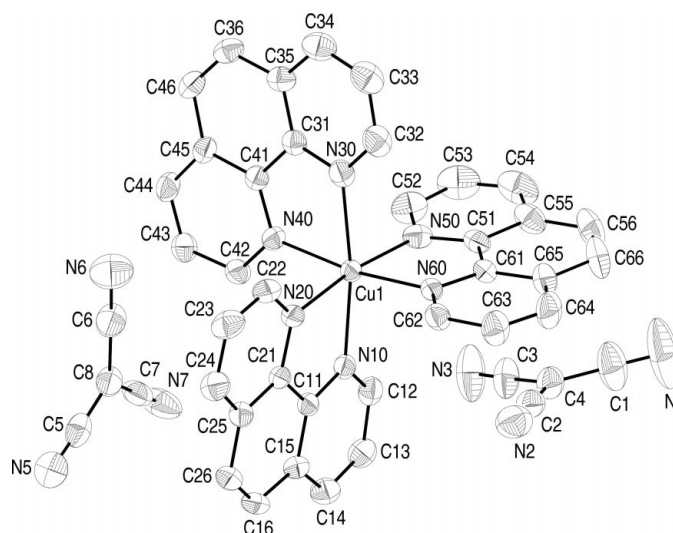

Figure 1
The asymmetric unit of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level. H atoms have ben omitted for clarity.

Data collection: *EXPOSE* in *IPDS* (Stoe & Cie, 1999); cell refinement: *CELL* in *IPDS*; data reduction: *INTEGRATE* in *IPDS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Crystal Impact, 1999); software used to prepare material for publication: *SHELXL*97.

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