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Bis(4-methylbenzenecyanamidosulfonato)dipyridinecopper(II)

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Abstract. $[Cu(C_8H_7N_2O_2S)_2(C_5H_5N)_2]$, $M_r = 612.2$, triclinic, $P\bar{1}$, $a = 7.150\,(4)$, $b = 9.314\,(5)$, $c = 10.523\,(7)$ Å, $\alpha = 93.55\,(5)$, $\beta = 97.15\,(5)$, $\gamma = 109.06\,(4)°$, $V = 653.3\,(7)$ Å^3, $Z = 1$, $D_x = 1.556$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0.71069$ Å, $\mu = 1.04$ mm^{-1}, $F(000) = 315$, $T = 293\,(2)$ K, $R = 0.032$ for 2307 observed reflections. The Cu atom lies on a centre of symmetry and is octahedrally coordinated by four N and two O atoms. The Cu—N1, Cu—N2 and Cu—O1 distances are 2.010 (2), 1.936 (2) and 2.616 (4) Å, respectively. Twelve-membered rings are formed by pairs of bidentate cyanamidosulfonato groups which link adjacent Cu atoms through Cu–O–S(O)–N–C–N–Cu bridges into infinite chains running parallel to the *ac* plane.

Experimental. The title compound was prepared from the reaction of an aqueous solution of $Cu(NO_3)_2$ containing pyridine with an aqueous solution of $Na[4\text{-}CH_3C_6H_4SO_2NCN]$ (Köhler & Freude, 1991). Crystals were obtained immediately; m.p. 440 K. Well shaped blue crystal (0.5 × 0.3 × 0.1 mm) was mounted on a Syntex $P2_1$ diffractometer using graphite-monochromated Mo $K\bar{\alpha}$ radiation; θ–2θ-scan technique. Cell parameters by least squares on 15 reflections ($8 \le \theta \le 21°$). Empirical absorption correction based on reflection intensity measurements at different azimuthal angles, transmission range 0.828–1.002. Total 3271 reflections ($1.5 \le \theta \le 27.5°$) measured in the range $0 \le h \le 9$, $-12 \le k \le 11$, $-13 \le l \le 11$. No significant variation in the net intensities of two reference reflections ($2\bar{1}\bar{1}$, $11\bar{3}$) measured every 98 reflections. 3020 unique reflections ($R_{int} = 0.017$ for 336 reflections) and 2307 satisfied $F_o \ge 4\sigma(F)$. Structure was solved by the Patterson method (Sheldrick, 1990), full-matrix least-squares refinement of 227 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and isotropic for H atoms. At convergence $R = 0.0324$, $wR = 0.0366$, $w = 0.32/[\sigma^2(F) + 0.00060F^2]$, $(\Delta/\sigma)_{max} \le 0.033$, $(\Delta\rho)_{max} = 0.29$, $(\Delta\rho)_{min} = -0.25$ e Å^{-3}; isotropic extinction correction $F_c^* = F_c[1 - xF_c^2/\sin(\theta)]$, $x = 2.26\,(1) \times 10^{-6}$. Scattering factors for C, H, N, O and S given in *SHELX*76 (Sheldrick, 1976) and those for neutral Cu corrected for f' and f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). All calculations on an EC-1045 computer system. Atomic parameters are given in Table 1,‡ selected interatomic parameters in Table 2 and

† Deceased.

‡ Lists of structure factors, anisotropic thermal parameters, H-atom parameters, least-squares planes and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54522 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0282]

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Table 1. *Final atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2$) *with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}
Cu	0*	0*	0*	2.6 (1)
N1	−313 (3)	−1686 (2)	1145 (2)	2.6 (5)
N2	−2491 (3)	−1196 (2)	−1080 (2)	3.2 (6)
N3	4289 (3)	−2262 (2)	−2445 (2)	3.2 (6)
S1	2687 (1)	−1406 (1)	−2456 (1)	2.7 (2)
O1	2078 (3)	−1322 (2)	−1220 (2)	3.9 (6)
O2	1170 (3)	−2151 (2)	−3514 (2)	4.1 (5)
C11	25 (4)	−1404 (3)	2419 (2)	3.0 (7)
C12	−346 (4)	−2534 (3)	3219 (3)	3.5 (8)
C13	−1091 (4)	−4016 (3)	2695 (3)	3.9 (10)
C14	−1408 (5)	−4321 (3)	1392 (3)	3.9 (8)
C15	−1013 (4)	−3147 (3)	654 (3)	3.3 (8)
C21	3915 (3)	467 (3)	−2760 (2)	2.4 (6)
C22	4908 (4)	1569 (3)	−1761 (3)	3.1 (7)
C23	5846 (4)	3031 (3)	−2011 (3)	3.7 (8)
C24	5835 (4)	3426 (3)	−3246 (3)	3.4 (8)
C25	4858 (4)	2292 (3)	−4239 (3)	3.5 (9)
C26	3894 (4)	825 (3)	−4010 (2)	3.1 (8)
C27	6828 (7)	5036 (4)	−3491 (5)	5.3 (12)
C3	−4039 (3)	−1657 (2)	−1694 (2)	2.4 (6)

* Parameter fixed.

Table 2. *Bond lengths* (Å) *and valence angles* (°) *with e.s.d.'s in parentheses*

Cu—N1	2.010 (2)	C12—C13	1.359 (4)
Cu—N2	1.936 (2)	C13—C14	1.358 (5)
Cu—O1	2.616 (4)	C14—C15	1.355 (4)
N1—C11	1.326 (3)	C21—C22	1.367 (4)
N1—C15	1.332 (3)	C21—C26	1.376 (3)
N2—C3	1.142 (3)	C22—C23	1.366 (4)
N3—S1	1.594 (3)	C23—C24	1.372 (5)
S1—O1	1.428 (3)	C24—C25	1.377 (4)
S1—O2	1.418 (2)	C24—C27	1.489 (5)
S1—C21	1.748 (3)	C25—C26	1.366 (4)
C11—C12	1.367 (4)		

N1—Cu—N2	90.2 (2)	C12—C13—C14	118.6 (4)
Cu—N1—C15	121.1 (2)	C13—C14—C15	119.4 (3)
Cu—N1—C11	121.8 (3)	N1—C15—C14	123.1 (3)
C11—N1—C15	117.0 (3)	S1—C21—C26	119.8 (3)
Cu—N2—C3	167.3 (3)	S1—C21—C22	120.1 (3)
N3—S1—C21	106.4 (2)	C22—C21—C26	120.1 (3)
N3—S1—O2	105.9 (2)	C21—C22—C23	119.6 (3)
N3—S1—O1	110.8 (2)	C22—C23—C24	121.7 (3)
O2—S1—C21	109.0 (2)	C23—C24—C27	120.6 (4)
O1—S1—C21	107.1 (2)	C23—C24—C25	117.8 (3)
O1—S1—O2	117.2 (3)	C25—C24—C27	121.6 (4)
N1—C1—C12	123.0 (3)	C24—C25—C26	121.5 (3)
C11—C12—C13	119.0 (3)	C21—C26—C25	119.4 (3)

hydrogen-bond contacts are in Table 3. The numbering scheme used is shown in Fig. 1 which was drawn with *ORTEP*II (Johnson, 1971) with 50% probability ellipsoids.

Table 3. *Hydrogen bonds and short interionic contacts* (Å, °)

X—H···Y	X—H	X···Y	H···Y	X—H···Y
C(15)—H(15)···N(2)	0.92 (3)	2.989 (4)	2.60 (3)	106 (2)
C(15)—H(15)···O(1)	1.08 (3)	3.272 (4)	2.82 (3)	112 (2)
C(22)—H(22)···O(1)	0.91 (3)	2.930 (4)	2.64 (2)	100 (2)
C(26)—H(26)···O(2)	0.99 (3)	2.942 (3)	2.54 (3)	104 (2)
C(15)—H(15)···N(2^{i})	0.92 (3)	3.035 (4)	2.61 (3)	109 (2)
C(12)—H(12)···O(2^{ii})	0.90 (3)	3.434 (4)	2.57 (3)	160 (2)
C(27)—H(273)···O(2^{iii})	1.08 (3)	3.349 (5)	2.63 (4)	125 (3)
C(27)—H(273)···O(2^{iii})	1.06 (3)	3.349 (5)	2.63 (4)	125 (3)

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

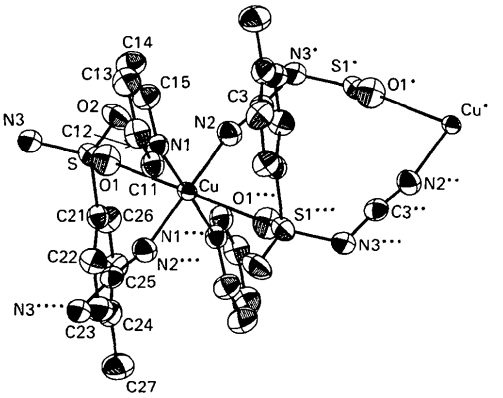

Fig. 1. 50% probability *ORTEP*II plot of bis(4-methylbenzenecyanamidosulfonato)dipyridinecopper(II) and atom numbering. [Symmetry code used: (*) $-1+x, y, z$; (**) $-1-x, -y, -z$; (***) $-x, -y, -z$; (****) $1-x, -y, -z$.]

Related literature. The 4-methylbenzenecyanamidosulfonate anion belongs to the family of acylcyanamide ligands which are generally regarded as pseudohalide in character (Golub, Köhler & Skopenko, 1986). Thus, the anion forms a colourless silver salt which is insoluble in water; it also forms complexes of the type $M(4\text{-}CH_3C_6H_4SO_2NCN)_2(C_5H_5N)_n$ ($n = 2,4$; M = Mn, Fe, Co, Cu, Zn) with 3*d* metal ions in the presence of pyridine (Köhler & Freude, 1991).

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