Crystal structure of di[bis(ethylenedithio)tetrathiafulvalene] dicopper tri-cyanide, $(C_{10}H_8S_8)_2Cu_2(CN)_3$

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Received October 7, 1996, CSD-No. 402662

$C_{23}H_{16}Cu_2N_3S_{16}$, monoclinic, $P2_1/c1$ (No. 14), $a = 16.082(2)$ Å, $b = 8.570(1)$ Å, $c = 13.368(2)$ Å, $\beta = 113.44(1)^\circ$, $V = 1690.4$ Å³, $Z = 2$, $R(F) = 0.051$, $R_w(F^2) = 0.103$.

Table 1. Parameters used for the X-ray data collection

Crystal:	black, plate, size 0.03 x 0.18 x 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	22.72 cm ⁻¹
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	$\theta/2\theta$
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	50°
$N(hkl)_{\text{unique}}$:	2968
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	207
Programs:	SDP, SHELXS-86, SHELXL-93

Source of material: The synthesis procedure for the title compound was described in ref. 1.

The structural description of the title compound has been reported. (see ref. 1). However only the unit cell parameters were provided. The nearly identical crystal structure was also reported by Yamochi (see ref. 2) and Geiser (see ref. 3), the latter being solved from a twinned crystal. Despite the structural similarity, crystals in above three reports showed different electrical conductivity and superconductivity. Thus, the structural information on all three materials are of interest in order to study the subtle differences in their properties. In the structure reported here, one CN^- group is located on the center of inversion and the corresponding site is refined with 50% C and 50% N as shown by the occupancy factors. The C and N sites in the other CN^- group are assigned so that the difference between the refined thermal parameters for the two sites is the minimum.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(7A)	4e	0.0967(6)	0.622(1)	0.4361(8)	0.07(2)
H(7B)	4e	0.1866(6)	0.556(1)	0.5237(8)	0.20(8)
H(8A)	4e	0.1622(6)	0.799(1)	0.556(1)	0.12(4)
H(8B)	4e	0.1596(6)	0.851(1)	0.443(1)	0.3(1)
H(9A)	4e	0.8235(4)	0.6071(9)	0.1844(5)	0.08(3)
H(9B)	4e	0.7567(4)	0.7404(9)	0.1836(5)	0.05(2)
H(10A)	4e	0.8989(4)	0.7549(8)	0.3314(6)	0.07(2)
H(10B)	4e	0.8763(4)	0.5989(8)	0.3761(6)	0.05(2)

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu(1)	4e		-0.00622(6)	0.1465(1)	0.33763(8)	0.0501(5)	0.0338(5)	0.0587(6)	0.0011(4)	0.0280(4)	0.0034(4)
N(11)	4e		0.0147(4)	-0.0257(7)	0.2480(5)	0.039(3)	0.037(3)	0.050(4)	0.004(3)	0.019(3)	0.002(3)
C(11)	4e		-0.0123(4)	0.3532(8)	0.2882(6)	0.035(4)	0.037(4)	0.045(4)	-0.003(3)	0.019(3)	-0.005(4)
C(12)	4e	0.5	-0.0034(4)	0.0367(8)	0.4628(5)	0.044(4)	0.051(4)	0.050(4)	-0.005(3)	0.024(4)	-0.001(3)
N(12)	4e	0.5	-0.0034(4)	0.0367(8)	0.4628(5)	0.044(4)	0.051(4)	0.050(4)	-0.005(3)	0.024(4)	-0.001(3)

Table 3. (Continued)

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
S(1)	4e		0.3684(1)	0.5331(2)	0.3392(1)	0.0291(8)	0.048(1)	0.0360(9)	-0.0088(8)	0.0140(7)	-0.0098(8)
S(2)	4e		0.4615(1)	0.7645(2)	0.5086(1)	0.0297(9)	0.042(1)	0.043(1)	-0.0065(7)	0.0161(7)	-0.0094(8)
S(3)	4e		0.5479(1)	0.5270(2)	0.2776(1)	0.0308(9)	0.043(1)	0.0365(9)	-0.0097(8)	0.0140(7)	-0.0097(8)
S(4)	4e		0.6281(1)	0.7704(2)	0.4415(1)	0.0302(8)	0.041(1)	0.045(1)	-0.0101(7)	0.0194(8)	-0.0156(8)
S(5)	4e		0.1935(1)	0.5564(2)	0.3574(2)	0.0273(9)	0.049(1)	0.061(1)	-0.0063(8)	0.0179(8)	-0.012(1)
S(6)	4e		0.3070(1)	0.8272(2)	0.5656(2)	0.045(1)	0.050(1)	0.072(1)	-0.0072(9)	0.035(1)	-0.022(1)
S(7)	4e		0.7040(1)	0.5047(3)	0.2152(2)	0.063(1)	0.061(1)	0.073(1)	-0.022(1)	0.047(1)	-0.035(1)
S(8)	4e		0.7966(1)	0.8018(2)	0.4093(2)	0.0302(9)	0.054(1)	0.065(1)	-0.0137(8)	0.0248(9)	-0.023(1)
C(1)	4e		0.4644(4)	0.6474(8)	0.4041(5)	0.026(3)	0.037(4)	0.032(3)	-0.004(3)	0.008(3)	-0.005(3)
C(2)	4e		0.5375(4)	0.6450(7)	0.3776(5)	0.028(3)	0.034(3)	0.027(3)	-0.002(3)	0.008(3)	0.001(3)
C(3)	4e		0.3054(4)	0.6201(7)	0.4051(5)	0.021(3)	0.033(4)	0.043(4)	0.000(3)	0.014(3)	0.004(3)
C(4)	4e		0.3485(4)	0.7239(7)	0.4831(5)	0.030(3)	0.031(3)	0.043(4)	-0.003(3)	0.019(3)	0.002(3)
C(5)	4e		0.6558(4)	0.5946(8)	0.2963(5)	0.030(3)	0.037(4)	0.037(4)	-0.002(3)	0.014(3)	-0.003(3)
C(6)	4e		0.6921(4)	0.7072(7)	0.3701(5)	0.028(3)	0.035(4)	0.036(4)	0.000(3)	0.017(3)	-0.003(3)
C(7)	4e		0.1622(6)	0.628(1)	0.4626(8)	0.047(5)	0.123(9)	0.070(6)	-0.032(6)	0.040(5)	-0.037(7)
C(8)	4e		0.1880(6)	0.779(1)	0.503(1)	0.051(5)	0.077(7)	0.14(1)	-0.010(5)	0.060(6)	-0.041(7)
C(9)	4e		0.7875(4)	0.6475(9)	0.2221(5)	0.037(4)	0.053(4)	0.040(4)	-0.005(4)	0.019(3)	-0.003(4)
C(10)	4e		0.8499(4)	0.6928(8)	0.3354(6)	0.033(4)	0.045(4)	0.047(4)	0.000(3)	0.020(3)	0.000(3)

Acknowledgments. Support of this work by the Petroleum Research fund administered by the American Chemical Society (PRF21392-AC6-C) and the National Science Foundation (CHE9021069) is gratefully acknowledged.

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