Table 2. *Geometric parameters* (Å, °)

S1—C1	1.725 (5)	S8—C6	1.735 (6)
S1—C3	1.717 (5)	S8—C10	1.807 (7)
S2—C1	1.720 (5)	P—F1	1.571 (4)
S2—C4	1.737 (6)	P—F2	1.535 (5)
S3—C2	1.719 (5)	P—F3	1.553 (6)
S3—C5	1.739 (6)	P—F4	1.548 (5)
S4—C2	1.719 (5)	P—F5	1.526 (5)
S4—C6	1.738 (5)	P—F6	1.497 (6)
S5—C3	1.745 (5)	C1—C2	1.381 (8)
S5—C7	1.808 (6)	C3—C4	1.352 (7)
S6—C4	1.737 (5)	C5—C6	1.350 (7)
S6—C8	1.809 (6)	C7—C8	1.501 (7)
S7—C5	1.728 (5)	C9—C10	1.467 (9)
S7—C9	1.812 (8)		
C1—S1—C3	95.8 (2)	S1—C1—S2	114.9 (3)
C1—S2—C4	96.0 (2)	S1—C1—C2	122.9 (4)
C2—S3—C5	96.0 (3)	S2—C1—C2	122.2 (4)
C2—S4—C6	95.7 (3)	S3—C2—S4	115.2 (3)
C3—S5—C7	98.3 (3)	S3—C2—C1	122.7 (4)
C4—S6—C8	103.4 (3)	S4—C2—C1	122.1 (4)
C5—S7—C9	103.9 (3)	S1—C3—S5	116.5 (3)
C6—S8—C10	97.0 (3)	S1—C3—C4	117.6 (4)
F1—P—F2	93.6 (3)	S5—C3—C4	125.8 (4)
F1—P—F3	86.6 (3)	S2—C4—S6	114.7 (3)
F1—P—F4	178.6 (3)	S2—C4—C3	115.8 (4)
F1—P—F5	85.9 (3)	S6—C4—C3	129.4 (4)
F1—P—F6	90.4 (3)	S3—C5—S7	116.5 (3)
F2—P—F3	87.8 (3)	S3—C5—C6	116.2 (4)
F2—P—F4	87.3 (3)	S7—C5—C6	127.2 (5)
F2—P—F5	177.5 (5)	S4—C6—S8	118.0 (3)
F2—P—F6	90.0 (4)	S4—C6—C5	116.8 (4)
F3—P—F4	92.5 (4)	S8—C6—C5	125.1 (4)
F3—P—F5	89.8 (4)	S5—C7—C8	112.6 (4)
F3—P—F6	176.1 (4)	S6—C8—C7	114.9 (4)
F4—P—F5	93.1 (3)	S7—C9—C10	118.0 (5)
F4—P—F6	90.6 (4)	S8—C10—C9	114.7 (5)
F5—P—F6	92.4 (5)		

Structure solved by direct methods and subsequent difference Fourier methods. Anisotropic thermal parameters for all non-H atoms. H atoms were calculated with a C—H distance of 0.95 Å and included in the structure-factor calculation only. The weighting scheme was $w=1/\sigma^2(F)$ where $\sigma(F)=\sigma(F^2)/2F$ and $\sigma(F^2)=[\sigma^2_{\text{counting}}+(0.02|F|^2)^2]^{1/2}$.

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Structure of $(BEDT\text{-}TTF)_2N(CN)_2$

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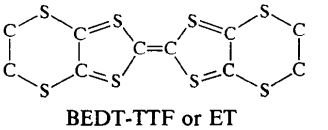
Abstract

The structure consists of layers of partially oxidized BEDT-TTF donor molecules [BEDT-TTF, or ET = 3,4;-3′,4′-bis(ethylenedithio)-2,2′,5,5′-tetrathiafulvalene] separated by dicyanamide anions along the crystallographic *c* axis. Dicyanamide anions are located between BEDT-TTF sheets. The salt showed semiconducting behavior as measured by the four-probe conductivity method.

Comment

Because of their unusual transport properties, salts of BEDT-TTF have attracted considerable attention (Williams *et al.*, 1987). Of particular interest are the organomineral salts, which show the highest superconducting transition temperatures in the BEDT-TTF family or in other organic salts. We report here on the crystal structure of a newly synthesized salt, $(ET)_2N(CN)_2$.



BEDT-TTF or ET

Crystals were prepared by the electrochemical oxidation of BEDT-TTF in a mixed solvent of 1,1,2-trichloroethane and 10% *v/v* absolute ethanol containing

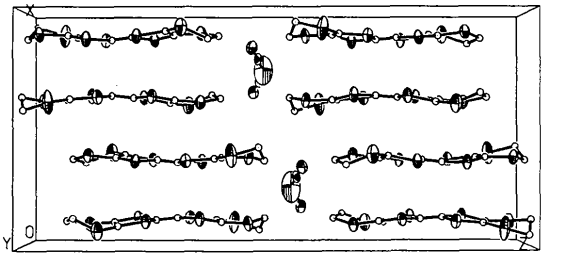


Fig.1. Packing diagram of the unit cell projected along the *b* axis. Thermal ellipsoids are drawn at the 50% level.

1 m*M* ET, 10 m*M* $NaN(CN)_2$, 10 m*M* AgI and 20 m*M* 18-crown-6 with a constant current of 4.0 μA.

As a result of the weak diffracting power of the crystal, only a small fraction of reflections (less than 15%) were observable and thus no anisotropic thermal parameters were used for the C atoms of the ET molecules. The large thermal oscillations or positional disorder indicated by larger-than-normal mean-square displacement parameters of N0 also contribute to the relatively high *R* factors.

Experimental

Crystal data

$2C_{10}H_8S_8^{1/2+}.C_2N_3^-$	Mo $K\alpha$ radiation
M_r = 835.4	λ = 0.71073 Å
Orthorhombic	Cell parameters from 25 reflections
Pcca	θ = 8-16°
a = 14.744 (1) Å	μ = 1.074 mm^{-1}
b = 6.676 (2) Å	T = 293 K
c = 32.151 (3) Å	Needle
V = 3165 (1) $Å^3$	0.31 × 0.075 × 0.03 mm
Z = 4	Black
D_x = 1.75 Mg m^{-3}	

Data collection

Enraf-Nonius CAD-4 diffractometer	θ_{max} = 30°
$\theta/2\theta$ scans	h = 0 → 7
4963 measured reflections	k = 0 → 17
4963 independent reflections	l = 0 → 38
655 observed reflections [$I>3\sigma(I)$]	3 standard reflections frequency: 300 min intensity variation: −0.4%

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.01
Final R = 0.077	$\Delta\rho_{max}$ = 0.59 e $Å^{-3}$
wR = 0.125	$\Delta\rho_{min}$ = −0.55 e $Å^{-3}$
S = 5.05	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)
655 reflections	
136 parameters	
H-atom parameters not refined	

Data collection: Enraf-Nonius CAD-4 software. Data reduction: Enraf-Nonius (1985) *SDP*. Program(s) used to solve structure: Enraf-Nonius *SDP*. Program(s) used to refine structure: Enraf-Nonius *SDP*. Molecular graphics: Nicolet X-ray products. Software used to prepare material for publication: Enraf-Nonius *SDP*.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* ($Å^2$)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S1	0.3624 (8)	0.161 (1)	−0.1442 (3)	0.049 (3)
S2	0.3711 (8)	−0.260 (2)	−0.1682 (3)	0.047 (3)
S3	0.3683 (8)	0.290 (1)	−0.2412 (3)	0.047 (3)
S4	0.3675 (8)	−0.131 (1)	−0.2653 (3)	0.043 (3)
S5	0.3832 (8)	0.089 (1)	−0.0543 (3)	0.051 (3)
S6	0.394 (1)	−0.412 (2)	−0.0839 (3)	0.092 (5)
S7	0.3997 (9)	0.442 (2)	−0.3259 (3)	0.069 (4)
S8	0.3917 (8)	−0.057 (1)	−0.3554 (3)	0.049 (3)
C1	0.363 (3)	−0.011 (5)	−0.187 (1)	0.04 (1)
C2	0.372 (2)	0.043 (4)	−0.2273 (8)	0.021 (8)
C3	0.382 (2)	−0.011 (6)	−0.105 (1)	0.04 (1)
C4	0.380 (2)	−0.205 (5)	−0.116 (1)	0.04 (1)
C5	0.391 (2)	0.227 (6)	−0.294 (1)	0.04 (1)
C6	0.381 (2)	0.039 (5)	−0.3060 (9)	0.04 (1)
C7	0.370 (3)	−0.129 (5)	−0.024 (1)	0.04 (1)
C8	0.425 (3)	−0.307 (6)	−0.034 (1)	0.06 (1)
C9	0.356 (3)	0.342 (7)	−0.373 (2)	0.10 (2)
C10	0.373 (3)	0.159 (7)	−0.391 (1)	0.08 (2)
C11	0.295 (2)	0.364 (5)	0.038 (1)	0.04 (1)
N1	0.339 (2)	0.237 (6)	0.049 (1)	0.08 (1)
N0	0.250	0.500	0.028 (2)	0.22 (4)

Table 2. *Geometric parameters* (Å, °)

S1—C1	1.80 (3)	S7—C5	1.76 (4)
S1—C3	1.73 (4)	S7—C9	1.78 (5)
S2—C1	1.77 (4)	S8—C6	1.72 (3)
S2—C4	1.72 (3)	S8—C10	1.87 (4)
S3—C2	1.71 (3)	C1—C2	1.34 (4)
S3—C5	1.79 (4)	C3—C4	1.35 (5)
S4—C2	1.69 (3)	C5—C6	1.32 (5)
S4—C6	1.74 (3)	C7—C8	1.47 (5)
S5—C3	1.76 (3)	C9—C10	1.38 (7)
S5—C7	1.76 (3)	C11—N1	1.12 (5)
S6—C4	1.73 (4)	C11—N0	1.17 (4)
S6—C8	1.80 (4)		
C1—S1—C3	98. (2)	S5—C3—C4	128. (3)
C1—S2—C4	99. (2)	S2—C4—S6	115. (2)
C2—S3—C5	91. (2)	S2—C4—C3	117. (3)
C2—S4—C6	95. (1)	S6—C4—C3	127. (3)
C3—S5—C7	102. (2)	S3—C5—S7	112. (2)
C4—S6—C8	104. (2)	S3—C5—C6	118. (3)
C5—S7—C9	99. (2)	S7—C5—C6	128. (3)
C6—S8—C10	106. (2)	S4—C6—S8	118. (2)
S1—C1—S2	109. (2)	S4—C6—C5	114. (2)
S1—C1—C2	124. (3)	S8—C6—C5	127. (3)
S2—C1—C2	125. (3)	S5—C7—C8	119. (3)
S3—C2—S4	118. (2)	S6—C8—C7	112. (3)
S3—C2—C1	121. (2)	S7—C9—C10	129. (4)
S4—C2—C1	120. (2)	S8—C10—C9	117. (3)
S1—C3—S5	115. (2)	N1—C11—N0	178. (4)
S1—C3—C4	116. (3)		

Structure solved by direct methods and subsequent difference Fourier methods. Only unique reflections were measured. No absorption correction applied. Anisotropic thermal parameters were used for S atoms of the ET molecule and three atoms in the $N(CN)_2^-$ group. The weighting scheme was $w=1/\sigma^2(F)$ where $\sigma(F)=\sigma(F^2)/2F$ and $\sigma(F^2)=[\sigma^2_{counting}+(0.02|F|^2)^2]^{1/2}$.

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