Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55145 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1003]

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Structure of $(BEDT\text{-}TTF)_4Hg_2I_6(I_8)$

XIANHUI BU AND PHILIP COPPENS

Department of Chemistry, State University of New York at Buffalo, Buffalo, NY 14214, USA

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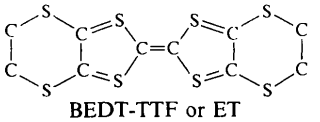
Abstract

The structure consists of two-dimensional sheets containing both BEDT-TTF [BEDT-TTF, or ET = 3,4;3′4′-bis(ethylenedithio)-2,2′,5,5′-tetrathiafulvalene] and centrosymmetric I_8 anions. These mixed (ET)-(I_8) sheets are separated by $Hg_2I_6^{2-}$ anions along the *c* axis. Dimers of ET molecules are tilted towards adjacent dimers, similar to the arrangement in κ-phase ET salts.

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, $(ET)_4Hg_2I_6(I_8)$.



BEDT-TTF or ET

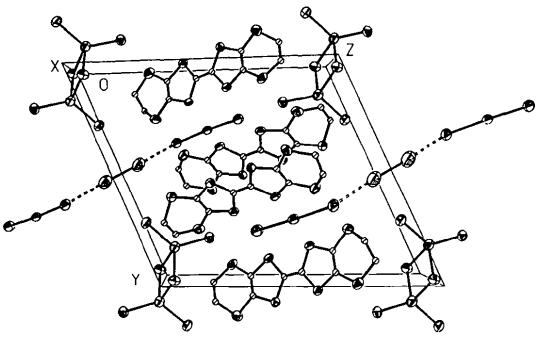


Fig. 1. Packing diagram of the unit cell projected down the *a* axis. Thermal ellipsoids are drawn at the 50% level.

Experimental

Crystal data

$4C_{10}H_8S_8^+.Hg_2I_6^{2-}.I_8^{2-}$	D_x = 2.77 Mg m^{-3}
M_r = 3716.6	Mo $K\alpha$ radiation
Triclinic	λ = 0.71073 Å
$P\bar{1}$	Cell parameters from 25 reflections
a = 8.892 (1) Å	θ = 10-18°
b = 15.627 (2) Å	μ = 8.981 mm^{-1}
c = 17.840 (2) Å	T = 293 K
α = 66.169 (9)°	Needle
β = 79.791 (9)°	0.30 × 0.08 × 0.03 mm
γ = 85.810 (9)°	Black
V = 2231.7 (5) Å^3	
Z = 1	

Data collection

Enraf-Nonius CAD-4 diffractometer	3378 observed reflections [$I > 3\sigma(I)$]
$\theta/2\theta$ scans	R_{int} = 0.034
Absorption correction: by integration from crystal shape	θ_{max} = 23°
T_{min} = 0.47, T_{max} = 0.81	h = 0 → 9
6672 measured reflections	k = −17 → 17
5716 independent reflections	l = −19 → 19
	3 standard reflections
	frequency: 300 min
	intensity variation: −1.4%

Refinement

Refinement on F	$(\Delta/\sigma)_{max}$ = 0.01
Final R = 0.070	$\Delta\rho_{max}$ = 3.6 e Å^{-3}
wR = 0.089	$\Delta\rho_{min}$ = −2.4 e Å^{-3}
S = 3.77	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)
3378 reflections	
297 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*.

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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}
Hg1	−0.0120 (2)	0.1340 (1)	−0.08060 (9)	0.0626 (5)
I1	0.1324 (3)	0.1725 (2)	−0.2351 (1)	0.0520 (7)
I2	−0.2247 (3)	−0.0145 (2)	−0.0275 (1)	0.0544 (7)
I3	−0.1077 (4)	0.2560 (2)	−0.0117 (1)	0.0761 (9)
I4	−0.0165 (3)	0.2424 (2)	0.5655 (1)	0.0600 (8)
I5	−0.1519 (3)	0.2921 (1)	0.4220 (1)	0.0442 (6)
I6	−0.2959 (3)	0.3506 (2)	0.2701 (1)	0.0596 (8)
I7	−0.0774 (4)	0.4588 (2)	0.0793 (2)	0.095 (1)
S1	0.4724 (9)	0.6862 (5)	0.3823 (5)	0.041 (2)
S2	0.6769 (9)	0.5518 (5)	0.3424 (5)	0.040 (2)
S3	0.5912 (9)	0.6186 (5)	0.5605 (5)	0.039 (2)
S4	0.7932 (9)	0.4836 (5)	0.5173 (4)	0.036 (2)
S5	0.367 (1)	0.7677 (5)	0.2174 (5)	0.051 (3)
S6	0.614 (1)	0.6131 (6)	0.1720 (5)	0.061 (3)
S7	0.701 (1)	0.5810 (6)	0.7174 (5)	0.054 (3)
S8	0.9322 (9)	0.4116 (6)	0.6674 (5)	0.050 (3)
C1	0.608 (3)	0.598 (2)	0.412 (2)	0.030 (7)
C2	0.657 (3)	0.570 (2)	0.487 (2)	0.032 (7)
C3	0.478 (4)	0.685 (2)	0.286 (2)	0.046 (8)
C4	0.581 (4)	0.624 (2)	0.265 (2)	0.049 (9)
C5	0.713 (3)	0.555 (2)	0.632 (2)	0.034 (7)
C6	0.806 (3)	0.491 (2)	0.608 (2)	0.036 (7)
C7	0.467 (6)	0.788 (3)	0.120 (3)	0.11 (2)
C8	0.588 (5)	0.731 (3)	0.100 (3)	0.09 (1)
C9	0.872 (6)	0.517 (4)	0.762 (3)	0.12 (2)
C10	0.911 (6)	0.433 (3)	0.762 (3)	0.11 (2)
S1*B*	0.598 (1)	0.8965 (5)	0.4339 (5)	0.044 (2)
S2*B*	0.6828 (9)	1.0901 (5)	0.3859 (4)	0.041 (2)
S3*B*	0.731 (1)	0.8356 (5)	0.6030 (4)	0.046 (2)
S4*B*	0.8212 (9)	1.0277 (5)	0.5577 (4)	0.040 (2)
S5*B*	0.462 (1)	0.9286 (5)	0.2877 (5)	0.050 (3)
S6*B*	0.582 (1)	1.1569 (6)	0.2225 (5)	0.053 (3)
S7*B*	0.836 (1)	0.7602 (6)	0.7635 (5)	0.067 (3)
S8*B*	0.949 (1)	0.9915 (6)	0.7079 (5)	0.055 (3)
C1*B*	0.674 (3)	0.975 (2)	0.461 (2)	0.032 (7)
C2*B*	0.735 (3)	0.950 (2)	0.532 (2)	0.033 (7)
C3*B*	0.560 (3)	0.975 (2)	0.340 (2)	0.034 (7)
C4*B*	0.598 (3)	1.066 (2)	0.314 (2)	0.029 (7)
C5*B*	0.819 (4)	0.856 (2)	0.675 (2)	0.045 (8)
C6*B*	0.859 (3)	0.947 (2)	0.652 (2)	0.036 (7)
C7*B*	0.516 (5)	1.008 (3)	0.187 (3)	0.09 (1)
C8*B*	0.545 (8)	1.098 (5)	0.160 (4)	0.18 (3)
C9*B*	1.004 (8)	0.807 (4)	0.796 (4)	0.17 (3)
C10*B*	0.970 (4)	0.892 (2)	0.805 (2)	0.07 (1)

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

S1—C1	1.74 (3)	S2*B*—C4*B*	1.77 (3)
S1—C3	1.71 (4)	S3*B*—C2*B*	1.72 (2)
S2—C1	1.67 (3)	S3*B*—C5*B*	1.76 (4)
S2—C4	1.71 (3)	S4*B*—C2*B*	1.73 (3)
S3—C2	1.77 (3)	S4*B*—C6*B*	1.72 (2)
S3—C5	1.75 (3)	S5*B*—C3*B*	1.76 (4)
S4—C2	1.72 (3)	S5*B*—C7*B*	1.73 (4)
S4—C6	1.70 (3)	S6*B*—C4*B*	1.70 (2)
S5—C3	1.77 (3)	S6*B*—C8*B*	1.79 (9)
S5—C7	1.73 (5)	S7*B*—C5*B*	1.71 (3)
S6—C4	1.70 (4)	S7*B*—C9*B*	1.97 (8)
S6—C8	1.80 (4)	S8*B*—C6*B*	1.75 (4)
S7—C5	1.72 (3)	S8*B*—C10*B*	1.83 (3)
S7—C9	1.88 (5)	C1*B*—C2*B*	1.37 (4)
S8—C6	1.76 (3)	C3*B*—C4*B*	1.34 (4)
S8—C10	1.83 (6)	C5*B*—C6*B*	1.35 (4)
C1—C2	1.38 (4)	C7*B*—C8*B*	1.31 (8)
C3—C4	1.39 (5)	C9*B*—C10*B*	1.40 (9)
C5—C6	1.40 (4)	Hg1—I1	2.669 (3)
C7—C8	1.43 (7)	Hg1—I2	2.845 (3)
C9—C10	1.33 (8)	Hg1—I3	2.674 (3)
S1*B*—C1*B*	1.70 (3)	I4—I5	2.821 (3)
S1*B*—C3*B*	1.70 (2)	I5—I6	2.979 (3)
S2*B*—C1*B*	1.75 (2)	I6—I7	3.420 (3)
C1—S1—C3	94. (1)	C2*B*—S4*B*—C6*B*	95. (1)
C1—S2—C4	97. (2)	C3*B*—S5*B*—C7*B*	100. (2)
C2—S3—C5	97. (1)	C4*B*—S6*B*—C8*B*	101. (2)
C2—S4—C6	97. (1)	C5*B*—S7*B*—C9*B*	97. (2)
C3—S5—C7	105. (2)	C6*B*—S8*B*—C10*B*	105. (2)
C4—S6—C8	102. (2)	S1*B*—C1*B*—S2*B*	115. (2)
C5—S7—C9	101. (2)	S1*B*—C1*B*—C2*B*	123. (2)
C6—S8—C10	104. (2)	S2*B*—C1*B*—C2*B*	122. (2)
S1—C1—S2	116. (2)	S3*B*—C2*B*—S4*B*	116. (2)
S1—C1—C2	121. (2)	S3*B*—C2*B*—C1*B*	121. (2)
S2—C1—C2	123. (2)	S4*B*—C2*B*—C1*B*	123. (2)
S3—C2—S4	114. (2)	S1*B*—C3*B*—S5*B*	114. (2)
S3—C2—C1	124. (2)	S1*B*—C3*B*—C4*B*	120. (3)
S4—C2—C1	122. (2)	S5*B*—C3*B*—C4*B*	125. (2)
S1—C3—S5	118. (2)	S2*B*—C4*B*—S6*B*	116. (2)
S1—C3—C4	118. (2)	S2*B*—C4*B*—C3*B*	114. (2)
S5—C3—C4	124. (3)	S6*B*—C4*B*—C3*B*	130. (3)
S2—C4—S6	119. (2)	S3*B*—C5*B*—S7*B*	115. (2)
S2—C4—C3	115. (3)	S3*B*—C5*B*—C6*B*	114. (2)
S6—C4—C3	126. (2)	S7*B*—C5*B*—C6*B*	131. (3)
S3—C5—S7	116. (2)	S4*B*—C6*B*—S8*B*	115. (2)
S3—C5—C6	113. (2)	S4*B*—C6*B*—C5*B*	119. (3)
S7—C5—C6	130. (2)	S8*B*—C6*B*—C5*B*	126. (2)
S4—C6—S8	115. (2)	S5*B*—C7*B*—C8*B*	128. (5)
S4—C6—C5	119. (2)	S6*B*—C8*B*—C7*B*	126. (5)
S8—C6—C5	126. (2)	S7*B*—C9*B*—C10*B*	114. (4)
S5—C7—C8	126. (3)	S8*B*—C10*B*—C9*B*	114. (4)
S6—C8—C7	120. (3)	I1—Hg1—I2	111.5 (1)
S7—C9—C10	122. (5)	I1—Hg1—I3	127.23 (8)
S8—C10—C9	121. (4)	I2—Hg1—I3	111.17 (9)
C1*B*—S1*B*—C3*B*	96. (1)	I4—I5—I6	178.3 (1)
C1*B*—S2*B*—C4*B*	96. (1)	I5—I6—I7	119.3 (1)
C2*B*—S3*B*—C5*B*	96. (1)		

Structure solved by heavy-atom methods and subsequent Fourier methods. Anisotropic thermal parameters for Hg, I and S. No significant improvement in R factors when using anisotropic thermal parameters for C. 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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