trans pyridines [rings containing N(2) and N(2^{i})] is 90 (3)° and these planes are at 45 (3)° to the M—Cl and M—N directions in the intermediate plane. The *mer* geometry of the indium complex was not predicted by previous vibrational spectroscopic studies (Carty & Tuck, 1975); this has been discussed by us recently (Small & Worrall, 1982).

Bond distances and angles are listed in Tables 3 and 4; the In—N distance, 2·33 (2) Å (av), is almost equal to that observed in the analogous bromide complex, suggesting that the acceptor strengths of the halides are similar with respect to pyridine. The In—Cl distance, 2·474 (4) Å (av), compares with 2·524 (5) Å (av) observed in $InCl_6^{3-}$ (Contreras, Einstein, Gilbert & Tuck, 1977). The Tl—Cl bond lengths, 2·505 (5) Å (av), are similar to those in other six-coordinate Tl compounds [2·48–2·54 Å (Lee, 1971)] as are the Tl—N distances, 2·41 (1) Å, compared with 2·44 (2) Å in $TlBr_3$.2pyridine (Jeffs, Small & Worrall, 1984) and 2·36 (2) Å in $TlCl_3$.(1,10-phenanthroline) (Baxter & Gafner, 1972). The main angular distortions are due to repulsions by Cl(1) which lies *trans* to N(1). The large U values are to be expected for the unbound pyridine.

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Structure of Semiconducting 3,4;3′,4′-Bis(ethylenedithio)-2,2′,5,5′-tetrathiafulvalene–Hexafluoroarsenate (2:1), (BEDT-TTF)$_2$AsF$_6$, ($C_{10}H_8S_8$)$_2$AsF$_6$

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Abstract. $M_r = 958{\cdot}287$, monoclinic, $A2/a$, $a = 14{\cdot}890$ (12), $b = 6{\cdot}666$ (7), $c = 35{\cdot}3789$ (12) Å, $\beta = 111{\cdot}19$ (10)°, $V = 3274$ Å^3, $Z = 4$, $D_x = 1{\cdot}94$ Mg m^{-3}, μ(Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å) $= 2{\cdot}07$ mm^{-1}, $F(000) = 1916$, $T = 298$ K, $R(F) = 0{\cdot}055$ for 1323 observed reflections. The structure consists of two-dimensional sheets of BEDT-TTF ($C_{10}S_8H_8$) molecules separated by AsF_6^- anions. Short S—S contact distances are found normal to the molecular stacks but not along the stacking direction. The material undergoes a low-temperature (a, $2b$, c) phase transition above 125 K.

Introduction. A new family of organic conductors has recently been synthesized, namely, the charge-transfer salts of 3,4;3′,4′-bis(ethylenedithio)-2,2′,5,5′-tetrathiafulvalene (BEDT-TTF): *e.g.* (BEDT-TTF)$_2$ClO$_4$($C_2H_3Cl_3$)$_{0\cdot5}$ (Saito, Enoki, Toriumi & Inokuchi, 1982). These include the first sulfur-based organic superconductor (BEDT-TTF)$_2$ReO$_4$, $T_c = 2$ K at 0·4 G Pa pressure (Parkin, Engler, Schumaker, Lagier, Lee, Scott & Greene, 1983). Crystals of these materials contain segregated stacks of donor and acceptor molecules with significant intermolecular S···S interactions between the organic molecules normal to the stacking direction (Kobayashi, Kato, Mori, Kobayashi, Sasaki, Saito & Inokuchi, 1983; Kobayashi, Mori, Kato, Kobayashi, Sasaki, Saito & Inokuchi, 1983; Parkin, Engler, Schumaker, Lagier, Lee, Scott & Greene, 1983) and as

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a result have been characterized as two-dimensional conductors (Kobayashi, Kato, Mori, Kobayashi, Sasaki, Saito & Inokuchi, 1983). In the conducting salts of tetramethyltetrathiafulvalene, $(TMTTF)_2X$ (Coulon, Delhaes, Flandrois, Lagnier, Bonjour & Fabre, 1982), and tetramethyltetraselenafulvalene, $(TMTSF)_2X$ ($X = PF_4^-$, ClO_4^-, BrO_4^-, BF_4^-, AsF_6^- *etc.*) (Epstein & Conwell, 1982), the electrical conductivities have been related to anion size and symmetry (Williams, Beno, Sullivan, Banovetz, Braam, Blackman, Carlson, Greer & Loesing, 1983). The present study is part of an effort to determine if similar relationships govern the electrical-conductivity behavior of these 2:1 BEDT-TTF congeners.

Experimental. Crystals of $(BEDT\text{-}TTF)_2AsF_6$ prepared by the electrolytic oxidation of BEDT-TTF in 1,1,2-trichloroethane solution in the presence of $(n\mathrm{Bu}_4N)AsF_6$ at a constant current (0·5 μA) and controlled temperature (296·6 K). Previously, $(BEDT\text{-}TTF)_2PF_6$ was prepared in similar fashion (Kobayashi, Kato *et al.*, 1983; Kobayashi, Mori *et al.*, 1983). D_m not determined. Crystal: thin black plate, 0·08 × 0·22 × 0·48 mm, Picker FACS-I automated four-circle diffractometer (Finger & Hadidiacos, 1982). Unit-cell dimensions from a least-squares refinement of the setting angles of 20 reflections, 2θ range 20–30°. Systematic absences and intensity statistics consistent with space group $A2/a$. The unusual setting was chosen to facilitate direct comparison with the low-temperature (125 K) structure determination for which a phase transition (a, $2b$, c) gives $a = 14{\cdot}706\ (3)$, $b = 13{\cdot}273\ (1)$, $c = 35{\cdot}280\ (2)$ Å, $\beta = 110{\cdot}69°$, $V_c = 6445{\cdot}8$ Å^3 with $Z = 8$. Intensity data collected to $\sin\theta/\lambda = 0{\cdot}48$ Å^{-1} ($h = -14$ to 12, $k = 0$ to -6, $l = -34$ to 34), θ:2θ scans [scan width $= (2{\cdot}0 + \tan\theta)°$], variable scan rates to maintain a constant precision of 0·01 in $I/\sigma(I)$ with max. scan time 2·0 min. Four standard reflections measured at an interval of 90 reflections, only small (2%) random variations. Data corrected for absorption effects using a Gaussian integration procedure; $T_{\min} = 0{\cdot}78$, $T_{\max} = 0{\cdot}92$. 3319 reflections averaged to yield 1709 unique data with $R_{\rm av}(F_o) = 0{\cdot}031$ and $R_{\rm av}(wF_o) = 0{\cdot}020$. Reflections with $F_o > 3\sigma(F_o)$ retained for further calculations. Initial structure solution (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) provided positions of all heavy atoms (As and S). C and F atoms located using Fourier methods. H atoms not located. Atomic and anomalous-scattering factors from *International Tables for X-ray Crystallography* (1974). All computations carried out using a local modification of the *UCLA Crystallographic Program Package* (Strouse, 1978). Anisotropic temperature factors for all non-hydrogen atoms in the final full-matrix least-squares refinement. The anisotropic thermal parameters of C(7)–C(10) were observed to be unusually large in a direction parallel to the molecular plane of the BEDT-TTF molecule (Fig. 1), indicative of disordered ethylene C-atom positions. A refinement of these disordered C atoms led to a very high correlation between the disordered atom pairs (correlation coefficient > ± 0·8). No significant improvement of the agreement factors was observed. Hence, only the ordered model is reported. $\sum w|F_o - F_c|$ minimized, where $w = 1/\sigma(F_o)$ and $\sigma(F_o) = 1/(2F_o)[\sigma_o(F_o^2) + (0{\cdot}02F_o^2)^2]^{1/2}$, with $\sigma(F_o^2)$ based on counting statistics. In final cycle $\Delta/\sigma < 0{\cdot}04$. A final difference Fourier synthesis produced random variations of ± 0·4 e Å^{-3} with the largest peaks 0·60–0·45 e Å^{-3} associated with the disordered ethylene C atoms, C(7)–C(10). No correction for secondary extinction. Final $R(F_o) = 0{\cdot}055$, $wR(F) = 0{\cdot}050$, and $S = 3{\cdot}66$.

Discussion. Final positional and thermal parameters are given in Table 1.* The calculated interatomic distances and bond angles are summarized in Table 2. The structure is shown in Fig. 1 and the short intermolecular S···S contacts are pictured in Fig. 2. There is one independent BEDT-TTF molecule in the unit cell with the AsF_6^- anion located at an inversion center. The unusually large thermal ellipsoids of the exocyclic C atoms, C(7)–C(10), indicate disordered atomic sites which are common to BEDT-TTF salts (Kobayashi, Kato *et al.*, 1983; Kobayashi, Mori *et al.*, 1983). The bond lengths and angles within the BEDT-TTF molecule (Table 2) are similar to those previously reported. The organic radical-cation molecules form extended sheets parallel to the *bc* plane of the unit cell. Intermolecular S···S contact distances, which are very short compared to the sum of the van der Waals radii (3·6 Å) (side-by-side, in-plane contacts), occur in these sheets (see Fig. 2). However, in contrast to $(TMTTF)_2X$ and $(TMTSF)_2X$ compounds, or $(BEDT\text{-}TTF)_2ClO_4(C_2H_3Cl_3)_{0.5}$, no short contact distances between chalcogenide atoms are observed along the molecular stacking direction (out-of-plane contacts) (Fig. 1). Thus, if S···S contacts are of primary importance in determining the conduction properties, $(BEDT\text{-}TTF)_2AsF_6$ would be expected to be highly one-dimensional. This is indeed the case in β-$(BEDT\text{-}TTF)_2PF_6$ where conductivity measurements have shown that the ratio of the conductivities along the direction of side-by-side contacts to that along the molecular stacking direction is 200:1 (Kobayashi, Kato *et al.*, 1983; Kobayashi, Mori *et al.*, 1983). The structures of $(BEDT\text{-}TTF)_2X$ salts containing octahedral anions, α- and β-PF_6^- (Kobayashi, Kato *et al.*, 1983;

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* Lists of structure factors, anisotropic temperature factors and least-squares-plane details have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39340 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and thermal parameters*

The complete temperature factor is $\exp[-U_{\rm eq}(\sin^2\theta)/(\lambda^2 8\pi^2)]$, where $U_{\rm eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ in units of Å^2.

	x	y	z	$U_{\rm eq}(\times 10^3)$
As	0·0000	0·2500	0·7500	63 (1)
F(1)	0·0383 (5)	0·1165 (11)	0·7939 (2)	87 (3)
F(2)	0·0901 (6)	0·4133 (13)	0·7702 (2)	108 (4)
F(3)	0·0683 (6)	0·1108 (12)	0·7315 (2)	108 (4)
S(1)	1·1427 (2)	1·1097 (4)	1·0568 (1)	48 (1)
S(2)	1·1210 (2)	0·6906 (4)	1·0755 (1)	44 (1)
S(3)	1·1177 (2)	0·9850 (4)	0·9656 (1)	43 (1)
S(4)	1·1144 (2)	0·5627 (4)	0·9866 (1)	44 (1)
S(5)	1·1772 (3)	1·2609 (5)	1·1380 (1)	69 (2)
S(6)	1·1490 (3)	0·7630 (5)	1·1610 (1)	70 (2)
S(7)	1·1071 (2)	0·9120 (4)	0·8823 (1)	49 (1)
S(8)	1·1154 (3)	0·4100 (5)	0·9096 (1)	87 (2)
C(1)	1·1274 (7)	0·8624 (14)	1·0401 (3)	39 (5)
C(2)	1·1213 (7)	0·8116 (14)	1·0023 (3)	35 (4)
C(3)	1·1526 (8)	1·0563 (16)	1·1061 (3)	44 (5)
C(4)	1·1428 (8)	0·8630 (17)	1·1144 (3)	48 (5)
C(5)	1·1150 (7)	0·8106 (15)	0·9287 (3)	34 (4)
C(6)	1·1141 (8)	0·6192 (15)	0·9385 (3)	43 (5)
C(7)	1·1916 (15)	1·1505 (22)	1·1864 (4)	125 (10)
C(8)	1·2057 (15)	0·9585 (20)	1·1946 (4)	136 (11)
C(9)	1·0951 (16)	0·6942 (19)	0·8509 (4)	147 (11)
C(10)	1·0815 (14)	0·5128 (20)	0·8613 (4)	130 (10)

Table 2. *Bond distances* (Å) *and angles* (°)

S(1)–C(1)	1·74 (1)	S(1)–C(3)	1·73 (1)	S(2)–C(1)	1·73 (1)
S(2)–C(4)	1·73 (1)	S(3)–C(2)	1·72 (1)	S(3)–C(5)	1·74 (1)
S(4)–C(2)	1·74 (1)	S(4)–C(6)	1·74 (1)	S(5)–C(3)	1·72 (1)
S(5)–C(7)	1·81 (1)	S(6)–C(4)	1·75 (1)	S(6)–C(8)	1·76 (1)
S(7)–C(5)	1·74 (1)	S(7)–C(9)	1·80 (1)	S(8)–C(6)	1·74 (1)
S(8)–C(10)	1·74 (1)				
C(1)–C(2)	1·35 (1)	C(3)–C(4)	1·34 (1)	C(5)–C(6)	1·32 (1)
C(7)–C(8)	1·31 (2)	C(9)–C(10)	1·30 (2)		
As–F(1)	1·700 (6)	As–F(2)	1·674 (7)	As–F(3)	1·676 (1)

C(3)–S(1)–C(1)	95·8 (5)	C(1)–S(2)–C(4)	95·4 (5)
C(2)–S(3)–C(5)	95·9 (5)	C(6)–S(4)–C(2)	94·9 (5)
C(3)–S(5)–C(7)	102·9 (6)	C(4)–S(6)–C(8)	101·7 (6)
C(5)–S(7)–C(9)	103·2 (6)	C(6)–S(8)–C(10)	101·7 (6)
C(2)–C(1)–S(2)	123·4 (8)	C(2)–C(1)–S(1)	121·9 (8)
S(2)–C(1)–S(1)	114·7 (6)	C(1)–C(2)–S(3)	123·4 (8)
C(1)–C(2)–S(4)	122·0 (8)	S(3)–C(2)–S(4)	114·6 (6)
C(4)–C(3)–S(5)	129·1 (8)	C(4)–C(3)–S(1)	116·3 (8)
S(5)–C(3)–S(1)	114·6 (6)	C(3)–C(4)–S(2)	117·8 (8)
C(3)–C(4)–S(6)	126·9 (8)	S(2)–C(4)–S(6)	115·3 (7)
C(6)–C(5)–S(3)	116·5 (8)	C(6)–C(5)–S(7)	128·2 (8)
S(3)–C(5)–S(7)	115·1 (6)	C(5)–C(6)–S(8)	128·1 (8)
C(5)–C(6)–S(4)	117·9 (8)	S(8)–C(6)–S(4)	114·0 (6)
C(8)–C(7)–S(5)	124· (1)	C(7)–C(8)–S(6)	125· (1)
C(10)–C(9)–S(7)	124· (1)	C(9)–C(10)–S(8)	129· (1)
F(2)–As–F(3)	91·2 (4)	F(2)–As–F(3)	88·8 (4)
F(2)–As–F(1)	89·9 (4)	F(2)–As–F(1)	90·1 (4)
F(3)–As–F(1)	91·0 (3)	F(3)–As–F(1)	89·0 (3)

Intermolecular S···S distances

S(2)···S($5^{\rm i}$)	3·528 (4)	S(3)···S($8^{\rm i}$)	3·452 (4)	S(5)···S($6^{\rm i}$)	3·506 (4)
S(7)···S($8^{\rm i}$)	3·447 (4)				

Symmetry code: (i) $x, y+1, z$.

Kobayashi, Mori *et al.*, 1983) and $\mathrm{AsF_6^-}$, possess nearly identical side-by-side interstack contact distances and lack out-of-plane S···S contacts so that similar electrical properties are to be expected. The structures of metallic (BEDT-TTF)$_2X$ salts, $X = \mathrm{ReO_4^-}$ and $\mathrm{ClO_4^-}$, however, contain both (short) side-by-side and out-of-plane S···S contacts. Band-structure calculations on (BEDT-TTF)$_2$ClO$_4$(C$_2$H$_3$Cl$_3$)$_{0.5}$ (Mori, Kobayashi, Sasaki, Kobayashi, Saito & Inokuchi, 1982) have shown that short out-of-plane interactions indicate two-dimensional conduction behavior.

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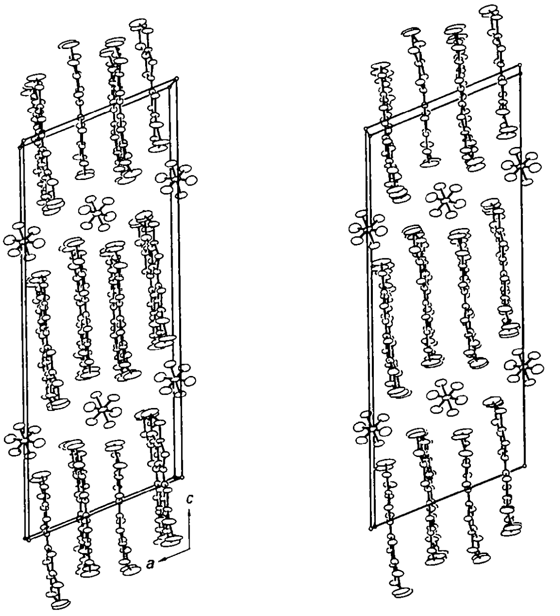

Fig. 1. Stereodrawing of the unit cell viewed down the *b* axis. The thermal ellipsoids are drawn at the 20% probability level.

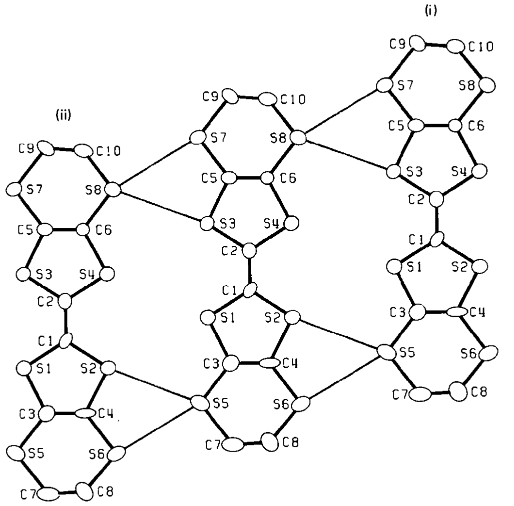

Fig. 2. *ORTEP* drawing (Johnson, 1965) of the molecular packing in the *bc* plane (50% probability level). Intermolecular S···S contacts less than the sum of the van der Waals radii (3·60 Å) are shown. Symmetry code: none x, y, z; (i) $x, y+1, z$; (ii) $x, y-1, z$.

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Structure of 3,4;3′,4′-Bis(ethylenedithio)-2,2′,5,5′-tetrathiafulvalene–Tetrabromoindate (2:1), $(BEDT\text{-}TTF)_2InBr_4$, $(C_{10}H_8S_8)_2InBr_4$

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Abstract. $M_r = 1203{\cdot}83$, triclinic, $P\bar{1}$, $a = 6{\cdot}618\ (1)$, $b = 16{\cdot}040\ (3)$, $c = 17{\cdot}470\ (3)$ Å, $\alpha = 95{\cdot}29\ (2)^\circ$, $\beta = 92{\cdot}43\ (1)$, $\gamma = 99{\cdot}12\ (1)^\circ$, $V = 1819{\cdot}9$ Å^3, $Z = 2$, $D_x = 2{\cdot}20$ Mg m^{-3}, μ(Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å) $= 5{\cdot}89$ mm^{-1}, $F(000) = 1162$, $T = 298$ K, $R(F) = 0{\cdot}095$ for 2965 observed reflections ($F_o > 0{\cdot}0$). The structure consists of dimerized stacks of BEDT-TTF ($C_{10}S_8H_8$) molecules separated by sheets of tetrahedral $InBr_4^-$ anions. Different, and short ($d \leq 3{\cdot}6$ Å) S—S contacts occur normal to the molecular-stacking direction for the two independent BEDT-TTF molecules. Short anion–sulfur non-bonding contacts, and dimerization of the BEDT-TTF molecules within the stacks, may be responsible for this behavior. An activated conductivity ($E_a = 0{\cdot}15$ e V) with a room-temperature value of 0·05–0·5 Ω^{-1} cm^{-1} is observed along the needle (crystal a^*) axis.

Introduction. The family of organic conductors based on BEDT-TTF includes $(BEDT\text{-}TTF)_2X$ charge-transfer salts where X is a monovalent anion with tetrahedral or octahedral geometry. Crystals of these materials derived from tetrahedral anions include the first sulfur-based organic superconductor $(BEDT\text{-}TTF)_2ReO_4$ [$T_c \simeq 2$ K at 0·4 GPa pressure (Parkin, Engler, Schumaker, Lagier, Lee, Scott & Greene, 1983)] and $(BEDT\text{-}TTF)_2ClO_4\,(TCE)_{0{\cdot}5}$ (TCE = 1,1,2-trichloroethane) which is metallic to 1·4 K (Kobayashi, Kobayashi, Sasaki, Saito, Enoki & Inokuchi, 1983). Both of these derivatives contain segregated stacks of donor and acceptor molecules having significant intermolecular S—S interactions between the organic molecules both along and normal to the stacking direction (Kobayashi, Kato, Mori, Kobayashi, Sasaki, Saito & Inokuchi, 1983; Parkin *et al.*, 1983) and as a result have been characterized as two-dimensional electrical conductors (Kobayashi, Kato *et al.*, 1983). In contrast, salts containing octahedral anions, *e.g.* α- and β-$(BEDT\text{-}TTF)_2PF_6$ (Kobayashi, Kato *et al.*, 1983;

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