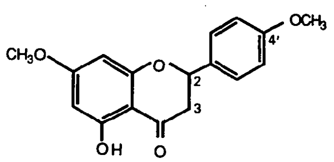
given in Table 1, bond parameters in Table 2 and the structure is illustrated for one enantiomer in Fig. 1.*



Related literature. The structure can be related to those of other flavanone derivatives (Narayanan, Zechmeister, Roehrl & Hoppe, 1971; Mariezcurrena, 1978; Hufford & Lasswell, 1979; Byrne, Cannon, Gawad, Joshi, Skelton, Toia & White, 1982; Cantrell & Hockstein, 1982). The intramolecular hydrogen bonding involving H(5), crystallographically located in the present study for the first time, has little effect on the geometry of the rest of the molecule.

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References

Byrne, L. T., Cannon, J. R., Gawad, D. H., Joshi, B. S., Skelton, B. W., Toia, R. F. & White, A. H. (1982). *Aust. J. Chem.* **35**, 1851–1858.

Cantrell, J. S. & Hockstein, G. S. (1982). *Acta Cryst.* B**38**, 981–983.

Hufford, C. D. & Lasswell, W. L. (1979). *J. Org. Chem.* **44**, 4709–4710.

Mariezcurrena, R. A. (1978). *Acta Cryst.* B**34**, 2322–2324.

Miles, C. O. & Main, L. (1985). *J. Chem. Soc. Perkin Trans.* 2, pp. 1639–1642.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Narayanan, P., Zechmeister, K., Roehrl, M. & Hoppe, W. (1971). *Acta Cryst.* B**27**, 718–725.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters and equations of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51532 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of the Conducting Salt of Pyrazinoethylenedithiotetrathiafulvalene (PEDTTTF): α-(PEDTTTF)$_2$IBr$_2$, at 98 K

By A. Terzis

Institute of Materials Science, 'Demokritos' National Research Center for Physical Sciences, 153 10 Ag. Paraskevi Attikis, Athens, Greece

G. Papavassiliou

National Hellenic Research Foundation, 48 Vassileos Constantinou, Athens, Greece

H. Kobayashi

Department of Chemistry, Toho University, Funabashi, Chiba 274, Japan

and A. Kobayashi

Department of Chemistry, University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113, Japan

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Abstract. Bis{2-(dithiolo[4,5-*b*]dithiin-2-ylidene)dithiolo-[4,5-*b*]pyrazinium} dibromoiodide, $(C_{10}H_6N_2S_6)_2$-IBr_2, $M_r = 979{\cdot}86$, triclinic, $P\bar{1}$, $a = 8{\cdot}850\,(4)$, $b = 5{\cdot}691\,(2)$, $c = 15{\cdot}377\,(7)$ Å, $\alpha = 99{\cdot}04\,(4)$, $\beta = 87{\cdot}23\,(4)$, $\gamma = 108{\cdot}35\,(4)°$, $V = 726{\cdot}0$ Å^3, $Z = 1$, $D_x = 2{\cdot}237$ g cm^{-3}, Mo $K\bar{\alpha}$, $\lambda = 0{\cdot}71069$ Å, $\mu = 48{\cdot}9$ cm^{-1}, $F(000) = 475$, $T = 98$ K. Final $R = 0{\cdot}077$ for 3879 observed reflections. Packing is similar to that of the room-temperature structure. The disorder of the ethylene group observed at room temperature (RT) has disappeared at 98 K. The planarity of the tetrathiafulvalene moiety is better in the low-temperature (LT) structure with the greatest deviation from the mean plane of this moiety being

Table 1. *Positional* ($\times 10^4$) *and equivalent isotropic thermal parameters for the non-*H *atoms with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}(Å^2)
I1	0	0	0	0·9
Br	−2256 (1)	2271 (1)	138·3 (5)	1·3
S1	2370 (2)	2178 (2)	6308 (1)	0·9
S2	4756 (2)	7117 (2)	6325 (1)	0·9
S3	930 (2)	2605 (3)	4505 (1)	0·9
S4	3293 (2)	7665 (2)	4506 (1)	0·9
S5	3298 (2)	1298 (3)	7999 (1)	1·1
S6	6308 (2)	7117 (3)	7974 (1)	1·0
N1	−243 (6)	3121 (10)	2992 (3)	1·1
N2	1986 (6)	7909 (9)	2996 (3)	1·0
C1	3181 (7)	4781 (11)	5780 (4)	1·0
C2	2555 (6)	4989 (10)	5008 (4)	0·8
C3	3670 (6)	3252 (10)	7206 (4)	0·8
C4	4809 (6)	5541 (10)	7205 (4)	0·8
C5	834 (6)	4169 (10)	3622 (4)	0·8
C6	1938 (6)	6546 (10)	3622 (4)	0·8
C7	5097 (7)	2647 (11)	8653 (4)	1·0
C8	5575 (7)	5481 (11)	8896 (4)	1·2
C9	−201 (7)	4465 (12)	2348 (4)	1·2
C10	891 (7)	6843 (12)	2357 (4)	1·2

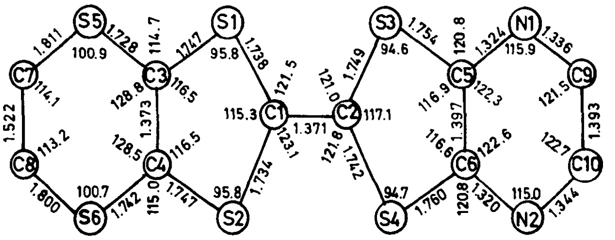

Fig. 1. Atom-numbering scheme of the donor with bond distances (Å) and angles (°). E.s.d.'s are 0·005–0·008 Å for distances and 0·03–0·06° for angles.

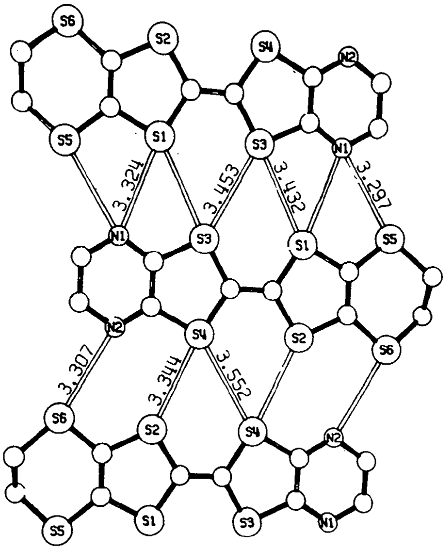

Fig. 2. Intrastack S···S and S···N contacts (Å). E.s.d.'s are 0·003–0·005 Å.

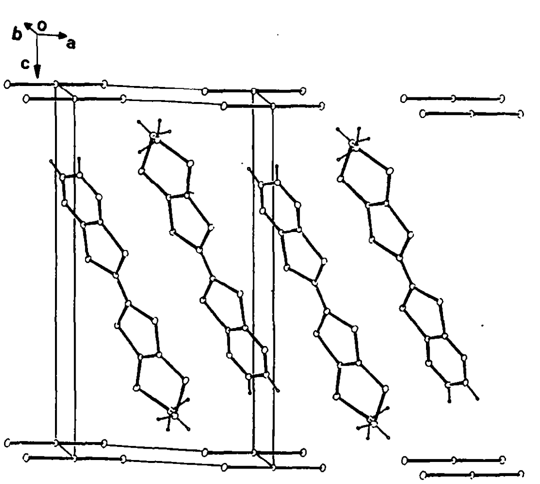

Fig. 3. An *ORTEP* packing diagram (Johnson, 1976).

0·037 (7) Å in the LT and 0·049 (6) Å in the RT structure for C(3) in both cases. There is no dimerization of the donor molecules in the stacking direction since the distances of the mean planes of the tetrathiafulvalene moiety are 3·452 (7) Å within and 3·481 (7) Å between the dimers and the difference of 0·029 (7) Å is not significant. There is no such dimerization in the RT structure either with the corresponding distances being 3·588 (6) and 3·592 (6) Å. There is significant shortening in the LT structure of the S···S and S···N contacts by as much as 0·09 Å.

Experimental. Title compound prepared according to literature (Terzis, Psycharis, Hountas & Papavassiliou, 1988). Crystal was a hexagonal plate 0·3 × 0·3 × 0·1 mm. Intensity data collected on Rigaku automated four-circle diffractometer. Lattice parameters from 20 reflections in the range $20 < 2\theta < 35°$. Data with $2\theta \leq 60°$ (range of $hkl = 0 \rightarrow 12, -7 \rightarrow 7, -21 \rightarrow 21$), $\theta/2\theta$ scan mode, scan speed 8° (2θ) min^{-1}, scan width 1·044° + 0·5° tanθ. Three reflections monitored periodically showed <3% intensity fluctuations. Lorentz and polarization but no absorption correction applied. Data collected/unique/R_{int}/observed $[F_o \geq 3\sigma(F_o)]$, 4526/4395/0·030/3879. Positions for I and Br deduced from Patterson map, remainder from difference map. Full-matrix refinement based on F with *UNICS*III (Sakurai & Kobayashi, 1979) using the weighting scheme $w = (10{\cdot}0 + 0{\cdot}01|F_o|^2)^{-1}$ for $|F_o| < 15{\cdot}0$ and $w = (\sigma^2 + 0{\cdot}01|F_o|^2)^{-1}$ for $|F_o| \geq 15{\cdot}0$, 196 parameters refined including the H positions. Refinement converged to: $R = 0{\cdot}077$, $wR = 0{\cdot}074$, $S = 0{\cdot}6248$, $|\Delta/\sigma|_{max}$

$= 0{\cdot}18$, $\Delta\rho_{\max}/\Delta\rho_{\min} = 0{\cdot}6/-0{\cdot}6$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,* bond lengths and angles in Fig. 1, the intrastack S···S and S···N contacts in Fig. 2, and the packing arrangement in Fig. 3.

Related literature. The room-temperature structure is reported in Terzis *et al.* (1988).

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* Lists of observed and calculated structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51508 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

SAKURAI, T. & KOBAYASHI, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–74.

TERZIS, A., PSYCHARIS, V., HOUNTAS, A. & PAPAVASSILIOU, G. (1988). *Acta Cryst.* C**44**, 128–132.

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Structure of a Sulfonylated Sulfamate: Cyclohexyl *N*-Benzyl-*N*-(*p*-bromophenylsulfonyl)sulfamate

BY TIM HIGGINS

Department of Physical Sciences, Regional Technical College, Galway, Ireland

AND PADRAIG O. BURKE, WILLIAM J. SPILLANE, PATRICK D. CUNNINGHAM AND PATRICK MCARDLE

Chemistry Department, University College, Galway, Ireland

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Abstract. $C_{19}H_{22}BrNO_5S_2$, $M_r = 488{\cdot}4$, triclinic, space group $P\bar{1}$, $a = 9{\cdot}184\,(3)$, $b = 10{\cdot}049\,(3)$, $c = 12{\cdot}3825\,(4)$ Å, $\alpha = 90{\cdot}90\,(2)$, $\beta = 91{\cdot}58\,(2)$, $\gamma = 115{\cdot}9\,(3)$°, $V = 1027{\cdot}12$ Å^3, $Z = 2$, $D_x = 1{\cdot}579$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $F(000) = 488{\cdot}0$, $\mu(\text{Mo }K\alpha) = 21{\cdot}52$ cm^{-1}, $T = 291\,(1)$ K, $R = 0{\cdot}053$ for 1378 independent reflections with $I_o > 3\sigma(I)$. The most notable feature of the structure is the essentially trigonal planar geometry about the nitrogen, the bond-angle sum being 359·4°. The deviation of nitrogen out of the S_1—S_2—C_1 plane is 0·072 (3) Å. The four S—O bonds: S_1—O_1 [1·434 (6) Å], S_2—O_3 [1·407 (7) Å], S_1—O_2 [1·418 (6) Å] and S_2—O_4 [1·410 (6) Å] are somewhat shorter than similar S—O bonds in related N—S(VI) structures. The two N—S bonds: S_1—N_1 [1·662 (7) Å] and S_2—N_1 [1·672 (7) Å] are also shorter than typical N—S bonds.

Experimental. The title compound was prepared as previously reported (Spillane & Burke, 1986) and crystals were obtained from a saturated *n*-hexane–ethyl acetate (3:1) solution at 273 K. Colorless data crystal 0·30 × 0·40 × 0·50 mm mounted on glass fiber. Intensities were measured with a Hilger & Watts Y290 diffractometer, lattice parameters determined from least-squares data for 12 independent reflections with $2\theta > 20°$. Scan range $2 < \theta < 24°$, standard reflection variation over data collection time was 2%. Data collected in $\theta/2\theta$ mode and $(\sin\theta)/\lambda = 0{\cdot}57$ Å^{-1}, $-10 < h < 10$, $-10 < k < 10$, $0 < l < 13$. 1774 total reflections, 1378 independent reflections with $I > 3\sigma(I)$.

Bromine and sulfur atoms were located by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Structure refinement, using full-matrix least-squares techniques, was carried out using *SHELX*76 (Sheldrick, 1976). Data were corrected for Lorentz and polarization effects but not for absorption. Hydrogen atoms were included in calculated positions. The bromine and sulfur atoms were refined anisotropically. The thermal parameters were terms U_{ij} of $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$. The atomic scattering factors for non-hydrogen and hydrogen atoms, and the anomalous-dispersion correction factors for non-hydrogen atoms

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