

ANL/MSD/PP-71503

STRUCTURES OF TWO PRECURSORS TO ORGANIC CHARGE-TRANSFER  
SALTS: 1,3-dithiolo[4,5-b][1,4]dithlin-2-thione and 1,3-dithiolo[4,5-b]  
[1,4]dithilin-2-one\*

**DISCLAIMER**

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

M. A. Beno, A. M. Kini, and J. M. Williams  
Argonne National Laboratory  
Materials Science Division  
Argonne, IL 60439

September 1990

The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. W-31-109-ENG-38. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes.

Patent Cleared by Patent Department, ANL	
RELEASE AUTHORIZED BY Technical Publications Services	
Date 10/8/90	DRH
David R. Harris Technical Information Services, ANL	

MASTER

\* Work Supported by the U.S. Department of Energy, BES-Materials Sciences, under Contract W-31-109-ENG-38.

**Structures of Two Precursors to Organic Charge-Transfer Salts:  
1,3-dithiolo[4,5-b][1,4]dithiin-2-thione and 1,3-dithiolo[4,5-b] [1,4]dithiin-  
2-one**

By M. A. Beno\*, A. M. Kini and J. M. Williams

*Materials Science and Chemistry Divisions, Argonne National Laboratory, Argonne,  
Illinois 60439, USA*

The submitted manuscript has been authored  
by a contractor of the U. S. Government  
under contract No. W-31-109-ENG-38.  
Accordingly, the U. S. Government retains a  
nonexclusive, royalty-free license to publish  
or reproduce the published form of this  
contribution, or allow others to do so, for  
U. S. Government purposes.

\*Author to whom correspondence should be addressed.

**Abstract.** C<sub>5</sub>H<sub>4</sub>S<sub>4</sub>O (**1**), M<sub>r</sub> = 208.343, monoclinic, P2<sub>1</sub>/n, *a* = 6.664 (2), *b* = 16.535 (6), *c* = 7.281 (2) Å, β = 97.84 (3), V = 794.8 (5) Å<sup>3</sup>, Z = 4, D<sub>x</sub> = 1.741 Mg m<sup>-3</sup>, λ (Mo Kα) = 0.71073 Å, μ = 1.07 mm<sup>-1</sup>, F(000) = 424, T = 298 K, R(F) = 0.048 for 2338 reflections. C<sub>5</sub>H<sub>4</sub>S<sub>5</sub> (**2**), M<sub>r</sub> = 224.39, monoclinic, P2<sub>1</sub>/n, *a* = 10.765 (2), *b* = 5.879 (2), *c* = 13.479 (2) Å, β = 92.66 (2), V = 852.2 (4) Å<sup>3</sup>, Z = 4, D<sub>x</sub> = 1.749 Mg m<sup>-3</sup>, λ (Mo Kα) = 0.71073 Å, μ = 1.23 mm<sup>-1</sup>, F(000) = 456, T = 298 K, R(F) = 0.059 for 2491 reflections. Structure determinations for the intermediates **1** and **2** were undertaken as part of a qualitative theoretical study of the changes in the geometries of organic donor molecules which occur with charge transfer. Although the geometries of **1** and **2** are nearly identical, the packing of these molecules in the unit cells is quite different. The molecular packing patterns are undoubtedly related to differences in the C-H···chalcogen interactions for the thio and keto substituents.

**Introduction.** The charge-transfer salts based on (BEDT-TTF) bis-(ethylenedithio)-tetrathiafulvalene which include metals, semiconductors and superconductors (Williams, Wang, Emge, Geiser, Beno, Leung, Carlson, Thorn, Schultz & Whangbo, 1987), consist of stacks of relatively planar organic donor molecules which are linked by short (less than the van der Waals radii sum, 3.6 Å, Bondi, 1964) inter- and intra-stack S···S interactions. A large number of stacking patterns have been observed and are dependent on the size and symmetry of the counter-anions (Emge, Leung, Beno, Wang, Firestone, Webb, Carlson, Williams, Venturini, Azevedo & Schirber, 1986; Wang, Allen, Schlueter, Hallenbeck, Stupka, Chen, Despotes, Kao, Carlson, Geiser & Williams, 1987) as well as donor···donor and donor···anion hydrogen bonding interactions (Leung, Emge, Beno, Wang, Williams, Petricek & Coppens, 1985; Whangbo, Williams, Schultz, Emge & Beno, 1987; Whangbo, Williams, Schultz & Beno, 1987). In contrast to the BEDT-TTF salts, only one donor packing pattern is observed in the (BEDO-TTF) bis-(ethylenedioxo)tetrathiafulvalene salts (Beno, Wang, Carlson, Kini, Frankenback, Ferraro, Larson, McCabe, Thompson, Purnama, Vashon, Williams, Jung & Whangbo, 1990). The crystal and molecular structures of the intermediates **1** and **2** (see Fig. 1) were determined as part of a qualitative theoretical study of the intermolecular interactions that control donor stacking and are important in determining the molecular geometries of the BEDT-TTF and BEDO-TTF donor molecules (Whangbo, Jung, Ren, Evain, Novoa, Mota, Alvarez, Williams, Beno, Kini, Wang & Ferraro, 1990).

-----

**Figure 1 Here**

-----

**Experimental.** The molecules **1** and **2** were synthesized as reported by Kini (Kini, Beno & Williams, 1987) and recrystallized in THF (tetra-hydrofuran). For **1**, transparent plates,  $0.55 \times 0.33 \times 0.075$  mm. For **2**, transparent needles,  $0.38 \times 0.14 \times 0.07$  mm. Nicolet P2<sub>1</sub> automated diffractometer. Unit cell dimensions from least-squares based on the setting-angles of 25 reflections ( $12^\circ < 2\theta < 20^\circ$ ). Intensity data were collected using the  $\omega$  scan technique with monochromatized Mo K $\alpha$  radiation and variable scan speeds of 2 to  $12^\circ \text{ min}^{-1}$  to  $\sin\theta/\lambda = 0.70$ ,  $0 \leq h \leq 9$ ,  $0 \leq k \leq 23$ ,  $-10 \leq l \leq 10$  for **1** and  $0 \leq h \leq 15$ ,  $0 \leq k \leq 8$ ,  $-18 \leq l \leq 18$  for **2**. Three standard reflections measured every 100 reflections showed  $< 3\%$  intensity variation. Data were corrected for Lorentz and polarization effects, a Gaussian absorption correction (8x8x8) procedure was used and multiply measured reflections (standards and 0kl zone) were averaged. For **1**,  $T_{\min} = 0.71$ ,  $T_{\max} = 0.92$  with  $R_{\text{int}}(F_0) = 0.021$  for 2338 reflections. In the case of **2**,  $T_{\min} = 0.83$ ,  $T_{\max} = 0.93$  with  $R_{\text{int}}(F_0) = 0.027$  for 2491 reflections. In each case the space group was indicated by systematic absences to be P2<sub>1</sub>/n. The structures were solved using MULTAN78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and Fourier methods. Hydrogen atoms, which could be observed in Fourier maps were included at calculated positions with  $B_{\text{iso}} = 5.0 \text{ \AA}^2$ . All non-hydrogen atoms were refined with anisotropic temperature factors. A secondary extinction correction was tested for both structures and found to be significant for **1** with a value of  $0.81(13) \cdot 10^{-5}$ . The function minimized was  $\sum w(|F_0| - |F_c|)^2$  where  $w = 1/\sigma^2(F_0)$  and  $\sigma(F_0) = [\sigma^2(F_0)^2 + (0.02F_0)^2]^{1/2}/2F_0$ , with the value of  $\sigma(F_0)^2$  based on counting statistics.  $\Delta/\sigma \leq 0.05$  in the final least-squares cycle resulted in  $R(F_0) = 0.048$ ,  $wR(F_0) = 0.041$  and  $S = 1.94$  for 92 variable parameters and 2158 data with  $F_0 \geq 0.0$  in the case of **1** and  $R(F_0) = 0.060$ ,  $wR(F_0) = 0.042$  and  $S = 1.77$  for 91 refined parameters and 2302 observed data ( $F_0 > 0.0$ ) in the case of **2**. Atomic scattering factors including anomalous contributions were taken from the *International Tables for X-ray Crystallography* (1974). Calculations were done using a local modification of the *UCLA Crystallographic Package* (Strouse, 1978).

**Discussion.** The atomic numbering scheme and conformation of the molecules are shown in Fig. 1. With the exception of the ethylene carbon atoms, the molecules are planar. In the case of **1**, the ethylene carbon atoms are displaced on opposite sides of the molecular plane, C4 on one side by  $-0.22 \text{ \AA}$  and C5, on the opposite side, by  $0.51 \text{ \AA}$ . The same conformation for the five- and six-membered rings is observed in the crystal structure of 5,6-dihydro-5,6-dimethyl-1,3-dithiolo [4,5-b] [1,4]dithiin-2-thione (**3**) (Wallis & Dunitz, 1988), a chiral molecule similar to **2**, but with methyl groups unsymmetrically substituted at carbon atoms C4 and C5. In contrast, both carbon

atoms in **2** are on the same side of the molecular plane, C4 by 0.03 Å and C5 by 0.81 Å. In BEDT-TTF salts both conformations for the six-membered rings are observed (Williams *et al*, 1987). Theoretical calculations show that the different conformations are very close in energy and that the observed conformation is dictated by molecular packing considerations (Jung, Evain, Novoa, Whangbo, Beno, Kini, Schultz, Williams & Nigrey, 1989; Whangbo, Jung, Ren, Evain, Novoa, Mota, Alvarez, Williams, Beno, Kini, Wang & Ferraro, 1990). The final positional and equivalent isotropic thermal parameters are given in Table 1\*. Bond distances and angles appear in Table 2. The bond lengths and angles in **2** are nearly identical to those found in **3** and are similar to those observed in **1** and in the neutral BEDT-TTF molecule (Kobayashi, Kobayashi, Sasaki, Saito & Inokuchi, 1986).

----- **Figure 2 Here** -----

The molecular packing observed in **2**, as depicted in Figure 2, is dominated by the occurrence of centrosymmetrically related pairs of molecules. This packing is similar to that which occurs in the non-centrosymmetric structure **3**. The short intermolecular interactions which occur in both molecules are given in Table 3. As is shown in Figure 3, the keto oxygen of **1** is surrounded by 4 nearest neighbor hydrogen atoms at a distance of less than 2.95 Å (the van der Waals radii sum for H and O atoms, Bondi, 1964). As in the case of **1**, the occurrence of short C-H···O interactions is a controlling factor in the molecular packing observed in neutral BEDO-TTF (Whangbo *et al*, 1990), and is important in BEDO-TTF salts (Beno *et al*, 1990).

----- **Figure 3 Here** -----

Work at Argonne National laboratory is sponsored by the US Department of Energy, Office of Basic Energy Sciences, Division of Materials Sciences, under contract W-31-109-ENG-38.

---

\* Lists of structure factors, anisotropic thermal parameters, and calculated hydrogen atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. xxxx (xx pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Beno, M. A., Wang, H. H., Carlson, D. K., Kini, A. M., Frankenback, G. M., Ferraro, J. R., Larson, N., McCabe, G. D., Thompson, J., Purnama, C., Vashon, M., Williams, J. M., Jung, D. & Whangbo, M.-H. (1990) *Mol. Cryst. Liq. Cryst.* **181**, 145-159.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441-451.
- Emge, T. J., Leung, P. C. W., Beno, M. A., Wang, H. H., Firestone, M. A., Webb, K. S., Carlson, K. D., Williams, J. M., Venturini, E. L., Azevedo, L. J. & Schirber, J. E. (1986). *Mol. Cryst. Liq. Cryst.* **132**, 363-383.
- Kini, A. M., Beno, M. A. & Williams, J. M. (1987). *Chem. Commun.* pp. 335-336.
- Leung, P. C. W., Emge, T. J., Beno, M. A., Wang, H. H., Williams, J. M., Petricek, V. & Coppens, P. (1985). *J. Am. Chem. Soc.* **107**, 6184-6191.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- Jung, D., Evain, M., Novoa, J. J., Whangbo, M.-H., Beno, M. A., Kini, A. M., Schultz, A. J., Williams, J. M. & Nigrey, P. J. (1989). *Inorg. Chem.* **28**, 4516-4522.
- Kobayashi, H., Kobayashi, A., Sasaki, Y., Saito, G. & Inokuchi, H. (1986). *Bull. Chem. Soc. Jpn.* **59**, 301-302.
- Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978), *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Strouse, C. (1978). *UCLA Crystallographic Package*. Univ. of California, Los Angeles.
- Wallis, J. D. & Dunitz, J. D. (1988). *Acta Cryst.* **C44**, 1037-1039.
- Wang, H. H., Allen, T. J., Schlueter, J. A., Hallenbeck, S. L., Stupka, D. L., Chen, M. Y., Despotes, A. M., Kao, H.-C. I., Carlson, K. D., Geiser, U. & Williams, J. M. (1987). Proceedings: The Vth International Conference on the Chemistry of Selenium and Tellurium, August 24-28, 1987, Oak Ridge, Tennessee, to be published in *Phosphorus and Sulfur*.
- Whangbo, M.-H., Jung, D., Ren, J., Evain, M., Novoa, J. J., Mota, F., Alvarez, S., Williams, J. M., Beno, M. A., Kini, A. M., Wang, H. H. & Ferraro, J. R. (1990). in *Proceedings of The First ISSP International Symposium on the Physics and Chemistry of Organic Superconductors*, Berlin, Springer-Verlag, in press.
- Whangbo, M. H., Williams, J. M., Schultz, A. J., Emge, T. J. & Beno, M. A. (1987). *J. Am. Chem. Soc.* **109**, 90-94.
- Whangbo, M. H., Williams, J. M., Schultz, A. J. & Beno, M. A. (1987). *Organic and Inorganic Low-Dimensional Crystalline Materials*, pp. 333-336, New York: Plenum Press.

Williams, J. M., Wang, H. H., Emge, T. J., Geiser, U., Beno, M. A., Leung, P. C. W.,  
Carlson, K. D., Thorn, R. J., Schultz, A. J. & Whangbo, M. H. (1987). *Progress in  
Inorganic Chemistry*, Vol. 35. New York: John Wiley & Sons, pp. 51-218.

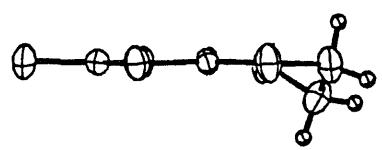
## Figure Captions

Fig. 1. Atom numbering scheme and configuration of **1**, 1,3-dithiolo[4,5-b] [1,4]dithiin-2-one, and **2** 1,3-dithiolo[4,5-b][1,4]dithiin-2-thione. The thermal ellipsoids are drawn at the 50% probability level with hydrogen atoms reduced to  $B_{iso} = 1.0 \text{ \AA}^2$  for clarity.

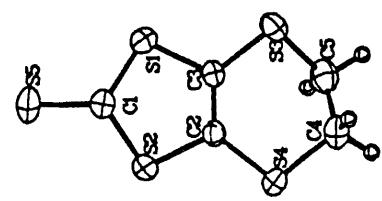
Fig. 2. Stereoscopic views of the molecular packing in **1** (top) and **2** (bottom). The short C-H···O interactions which occur in **1** result in pancake like stacks in which the direction of the molecules alternates along the stacks. In contrast, the absence of these interactions in **2** produces dimerized stacks of molecules, a packing mode similar to the  $\kappa$ -phase BEDT-TTF salts.

Fig. 3. Intermolecular O···H contacts less than 2.95 Å in **1** indicated by the thin lines, serve both to connect molecules in the stack (right hand side of the figure) and to link adjacent stacks (left hand side of the figure).. The thermal ellipsoids are drawn at the 20% probability level.

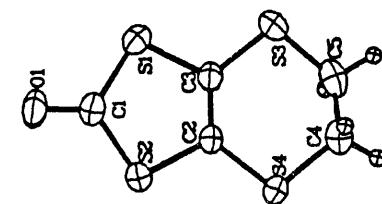
Figure 1



2



1



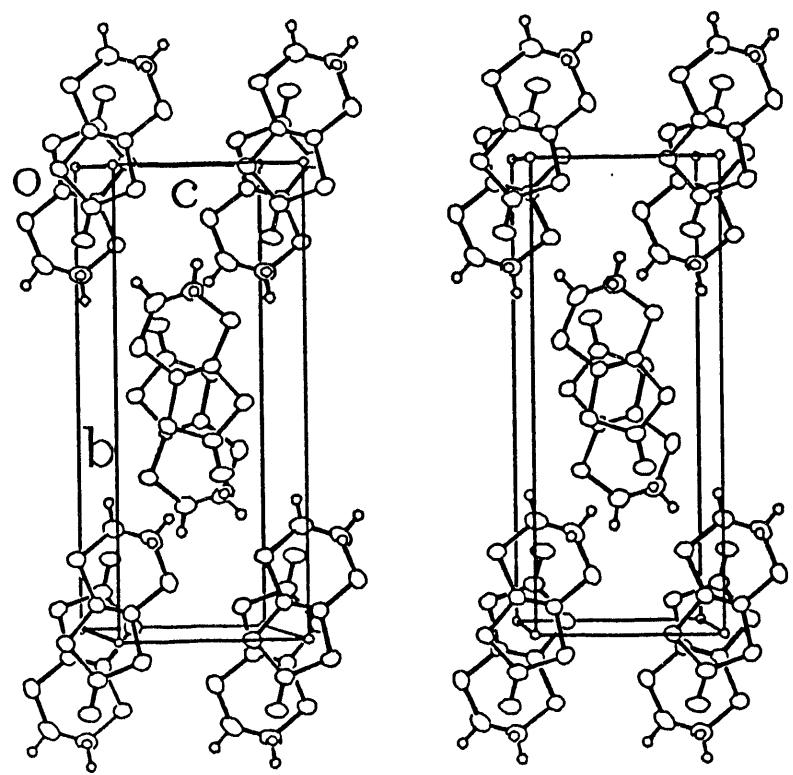
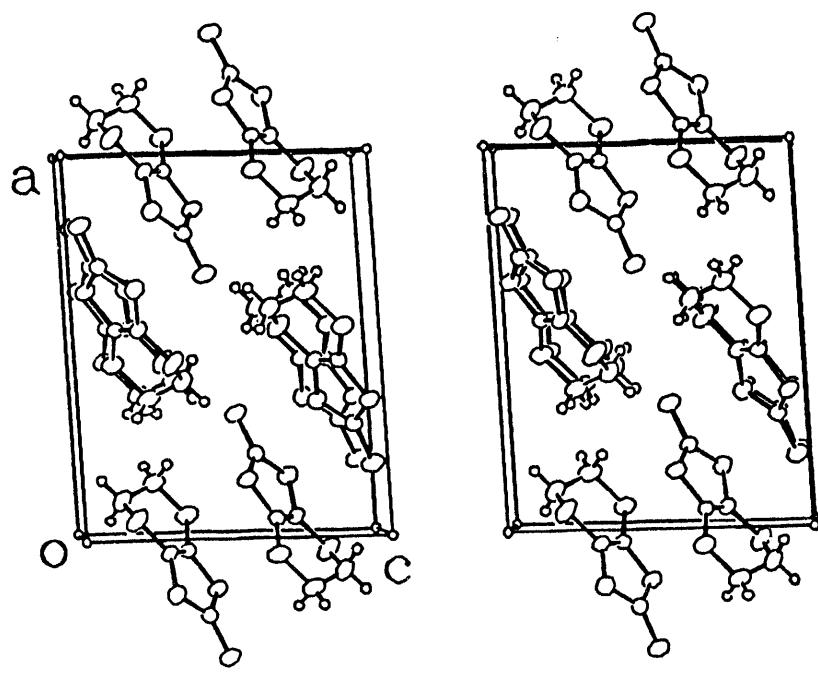


Figure 2

Figure 3

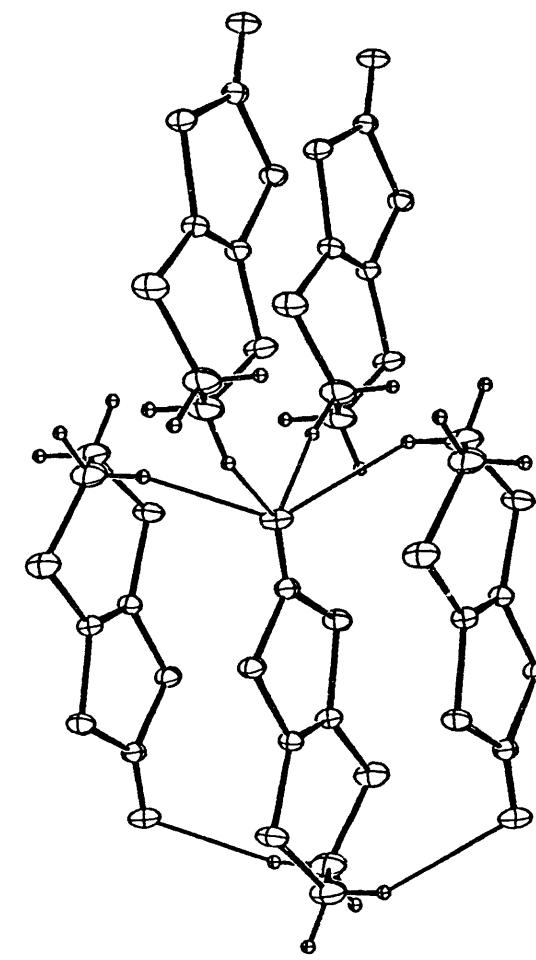


Table 1 *Positional and Equivalent Isotropic Thermal Parameters*

$C_5H_4S_5$				
Atom	x	y	z	$U_{eq} \times 10^4$ a
S1	0.94471(7)	0.18042(12)	0.35378(5)	416(2)
S2	0.79454(7)	-0.22122(12)	0.35127(5)	447(2)
S3	0.86517(8)	0.35619(13)	0.54608(5)	493(2)
S4	0.68131(8)	-0.13391(14)	0.54474(6)	586(3)
S5	0.95871(8)	-0.13776(15)	0.18395(5)	549(3)
C1	0.9025(2)	-0.0651(4)	0.2915(2)	355(7)
C2	0.7853(2)	-0.0493(4)	0.4559(2)	361(8)
C3	0.8553(2)	0.1406(4)	0.4569(2)	318(7)
C4	0.6445(3)	0.1417(6)	0.5934(2)	548(10)
C5	0.7569(3)	0.2648(6)	0.6365(2)	539(10)

$C_5H_4OS_4$				
Atom	x	y	z	$U_{eq} \times 10^4$ a
S1	0.18216(8)	0.05148(3)	0.28596(9)	474(2)
S2	-0.24508(8)	0.00603(3)	0.20600(8)	444(2)
S3	0.33691(8)	-0.11518(4)	0.31790(11)	626(3)
S4	-0.17594(8)	-0.16967(3)	0.22588(10)	537(2)
C1	-0.0701(3)	0.08545(12)	0.2402(3)	417(7)
C2	-0.0696(3)	-0.07267(11)	0.2419(3)	371(6)
C3	0.1257(3)	-0.05184(12)	0.2776(3)	396(6)
C4	0.0395(4)	-0.2319(2)	0.2966(5)	724(11)
C5	0.2292(4)	-0.2085(2)	0.2309(4)	703(11)
O1	-0.1164(3)	0.15637(9)	0.2323(2)	592(6)

a The complete temperature factor is  $\exp(-8\pi^2 U_{eq} \sin^2\theta/\lambda^2)$ , where  $U_{eq} = 1/3 \sum_{ij} U_{ij} a_i^* a_j^* a_i a_j$  in units of  $\text{\AA}^2$ .

Table 2. *Interatomic Distances and Angles.*

$C_5H_4S_4O$					
Atoms	Dist. ( $\text{\AA}$ )	Atoms	Dist. ( $\text{\AA}$ )	Atoms	Dist. ( $\text{\AA}$ )
S1-C1	1.760(2)	S2-C1	1.752(2)	S1-C3	1.749(2)
S2-C2	1.745(2)	S3-C3	1.747(2)	S3-C5	1.782(3)
S4-C2	1.751(2)	S4-C4	1.785(3)	C1-O1	1.212(2)
C2-C3	1.337(3)	C4-C5	1.464(4)		
Atoms	Angle ( $^{\circ}$ )	Atoms	Angle ( $^{\circ}$ )	Atoms	Angle ( $^{\circ}$ )
C3-S1-C1	96.30(10)	C2-S2-C1	96.79(10)	C3-S3-C5	100.56(11)
C2-S4-C4	101.95(11)	O1-C1-S2	123.9(2)	O1-C1-S1	123.2(2)
S2-C1-S1	112.82(12)	C3-C2-S2	116.8(2)	C3-C2-S4	128.6(2)
S2-C2-S4	114.60(11)	C2-C3-S3	128.2(2)	C2-C3-S1	117.2(2)
S3-C3-S1	114.53(12)	C5-C4-S4	116.7(2)	C4-C5-S3	115.9(2)

$C_5H_4S_5$					
Atoms	Dist. ( $\text{\AA}$ )	Atoms	Dist. ( $\text{\AA}$ )	Atoms	Dist. ( $\text{\AA}$ )
S1-C1	1.721(3)	S1-C3	1.742(2)	S2-C1	1.712(3)
S2-C2	1.743(3)	S3-C3	1.747(2)	S3-C5	1.807(3)
S4-C2	1.749(2)	S4-C4	1.800(3)	S5-C1	1.653(3)
C2-C3	1.346(3)	C4-C5	1.503(4)		
Atoms	Angle ( $^{\circ}$ )	Atoms	Angle ( $^{\circ}$ )	Atoms	Angle ( $^{\circ}$ )
C1-S1-C3	97.68(12)	C1-S2-C2	97.79(12)	C3-S3-C5	103.19(13)
C2-S4-C4	98.76(13)	S5-C1-S2	123.9(2)	S5-C1-S1	123.1(2)
S2-C1-S1	112.94(14)	C3-C2-S2	115.8(2)	C3-C2-S4	127.2(2)
S2-C2-S4	116.84(15)	C2-C3-S1	115.7(2)	C2-C3-S3	128.6(2)
S1-C3-S3	115.68(14)	C5-C4-S4	112.8(2)	C4-C5-S3	114.5(2)

**Table 3. Intermolecular Contact Distances.****C<sub>5</sub>H<sub>4</sub>S<sub>4</sub>O**

Atom1	Atom2	Dist (Å)	Symmetry Operation (Atom2)
S3	S4	3.5205(13)	x+1, y, z
H4A	O1	2.773(2)	-x-1/2, y-1/2, -z+1/2
H4B	O1	2.706(2)	-x, -y, -z+1
H5A	O1	2.431(2)	-x+1/2, y-1/2, -z+1/2
H5B	O1	2.489(2)	-x, -y, -z

**C<sub>5</sub>H<sub>4</sub>S<sub>5</sub>**

Atom1	Atom2	Dist (Å)	Symmetry Operation (Atom2)
S3	S5	3.3882(12)	x-1/2, -y-1/2, z+1/2
S3	S4	3.5916(12)	x, y-1, z
H4A	S5	3.190(2)	x-1/2, -y-1/2, z+1/2
H4B	S5	3.134(2)	-x+1.5, y+1/2, -z+1/2
H5B	S5	3.053(2)	-x+2, -y, -z+1

Supplementary information for:

**Structures of Two Precursors to Organic Charge-Transfer Salts:  
1,3-dithiolo[4,5-b][1,4]dithiin-2-thione and 1,3-dithiolo[4,5-b] [1,4]dithiin-  
2-one**

By M. A. Beno\*, A. M. Kini\* and J. M. Williams\*

*Chemistry and Materials Science Divisions, Argonne National Laboratory, Argonne,  
Illinois 60439, USA*

Table 4. Anisotropic thermal parameters.

C<sub>5</sub>H<sub>4</sub>S<sub>4</sub>O

Atom	U <sub>11</sub> ×10 <sup>4</sup>	U <sub>22</sub> ×10 <sup>4</sup>	U <sub>33</sub> ×10 <sup>4</sup>	U <sub>12</sub> ×10 <sup>4</sup>	U <sub>13</sub> ×10 <sup>4</sup>	U <sub>23</sub> ×10 <sup>4</sup>
S1	433(3)	340(3)	634(4)	-69(2)	97(3)	-1(3)
S2	385(3)	319(3)	611(4)	45(2)	19(2)	3(3)
S3	337(3)	466(4)	1056(6)	65(2)	78(3)	39(4)
S4	413(3)	287(3)	890(5)	-21(2)	40(3)	-58(3)
C1	509(12)	305(10)	427(12)	9(9)	102(10)	12(9)
C2	371(10)	270(9)	463(12)	23(8)	64(9)	-24(9)
C3	369(10)	305(10)	503(12)	15(8)	99(9)	19(9)
C4	571(16)	367(13)	1200(26)	48(11)	-31(16)	67(15)
C5	586(15)	449(14)	1053(23)	192(12)	131(15)	-17(15)
O1	691(11)	270(8)	799(12)	43(7)	157(9)	28(8)

C<sub>5</sub>H<sub>4</sub>S<sub>5</sub>

Atom	U <sub>11</sub> ×10 <sup>4</sup>	U <sub>22</sub> ×10 <sup>4</sup>	U <sub>33</sub> ×10 <sup>4</sup>	U <sub>12</sub> ×10 <sup>4</sup>	U <sub>13</sub> ×10 <sup>4</sup>	U <sub>23</sub> ×10 <sup>4</sup>
S1	494(4)	402(4)	351(3)	-104(3)	108(3)	-28(3)
S2	464(4)	419(4)	458(4)	-113(3)	88(3)	-136(3)
S3	645(5)	434(4)	401(4)	-142(4)	119(3)	-125(3)
S4	615(5)	485(4)	663(5)	-151(4)	349(4)	-95(4)
S5	609(5)	652(5)	386(4)	65(4)	141(3)	-117(4)
C1	361(13)	383(13)	319(12)	49(11)	9(10)	-19(11)
C2	354(13)	389(14)	341(12)	-10(11)	89(10)	-34(11)
C3	331(12)	351(12)	271(11)	-4(10)	22(9)	-9(10)
C4	471(17)	610(19)	565(18)	68(15)	181(14)	-92(16)
C5	659(20)	581(19)	377(14)	-13(17)	130(14)	-68(14)

The complete temperature factor is

$$\exp[-2\pi^2(a^*2U_{11}h^2 + b^*2U_{22}k^2 + c^*2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl)].$$

Table 5. *Calculated hydrogen atom positional parameters.* $C_5H_4S_4O$ 

Atom	x	y	z
H4A	0.0058	-0.2880	0.2506
H4B	0.0637	-0.2320	0.4350
H5A	0.3306	-0.2522	0.2674
H5B	0.2029	-0.2045	0.0928

 $C_5H_4S_5$ 

Atom	x	y	z
H4A	0.5794	0.1241	0.6466
H4B	0.6041	0.2402	0.5391
H5A	0.7313	0.3963	0.6778
H5B	0.8025	0.1512	0.6848

100-  
75-  
50-

4/8/96

FILED  
SEARCHED  
INDEXED  
SERIALIZED  
FILED  
APR 14 1989  
FBI - LOS ANGELES

