Charge Transfer Complexes From 1,2-Dithiolylium Salts, Electrical Conductivity and Structure

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It has been shown that the 4-phenyl-5-thioxo-1,2-dithiol-3-thiolate anion forms charge transfer salts with substituted 1,2-dithiolylium salts. Spectral data are given for a series of complexes. It is observed that the complex derived from the 3,5-diphenyl-1,2-dithiolylium ion has a relatively high d.c. conductivity. The structure of this complex 7 is determined by X-ray diffractometry.

The crystals of compound 7 belong to the orthorhombic space group *Pbca*. Cell dimensions are $a = 22.363(2)$, $b = 11.248(1)$, $c = 17.559(2)$ Å. Refinement by full-matrix least-squares calculations gave an R index of 0.068. Alternate packing of donor and acceptor molecules occurs in the axial directions with the highest packing densities. This explains the low conductivity of 7.

A series of sulfur and selenium containing charge-transfer complexes related to the 1,3-dithiole series has been studied in the attempt to design organic conductors.[1-3]

Only a few charge-transfer complexes in the 1,2-dithiole series have hitherto been reported.[4,5] We have reported that 1,2-dithiolylium ions (1) form salts, which are charge transfer complexes, with 4-phenyl-5-thioxo-1,2-dithiole-3-thiolate anion.[5]
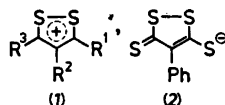
The salts formed from the yellow cations and orange anion are dark-coloured compounds. It is not possible to observe any charge transfer bands in the UV-VIS spectrum of the complexes in the region $250-450$ nm. If present, they are probably obscured by the intense absorption bands, cf. Fig. 1. However, it is evident that charge transfer has taken place as
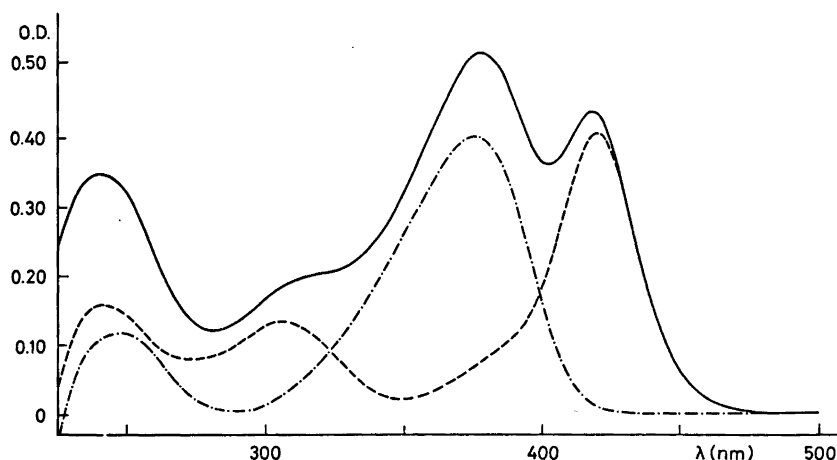

Fig. 1. VIS-spectrum of 7 ———, dimethylammonium 4-phenyl-5-thioxo-1,2-dithiole-3-thiolate $-\,-\,-$ and 3,5-diphenyl-1,2-dithiolylium perchlorate $-\cdot-$ in CH_2Cl_2, $c = ca.$ 10^{-5} mol/l.

Table 1. 1,2-Dithiolylium-4-phenyl-5-thioxo-1,2-dithiole-3-thiolates.

|	R¹	R²	R³	M.p./°C [a]	λ/nm(ε [b])					
1	4-MeOC₆H₄	H	H	154−155	310	(12 200);	427	(33 200)		
2	H	C₆H₅	H	110−111	325	(12 900);	432	(9800)		
3	C₆H₅	H	H	128−129	330	(13 600);	428	(14 500)		
4	4-BrC₆H₄	H	H	127−128	333	(14 700);	430	(13 300)		
5	4-FC₆H₄	H	H	128−129	333	(14 100);	430	(14 200)		
6	C₆H₅	H	CH₃	106−107	218	(16 000);	323	(12 600);	415	(8400)
7	C₆H₅	H	C₆H₅	214−215	381	(33 300);	420	(25 800)		
8	4-MeC₆H₄	H	C₆H₅	158−159	313	(13 900);	415	(39 800)		
9	4-MeOC₆H₄	H	C₆H₅	164−165	375	(18 900);	426	(47 300)		
10	MeS	H	C₆H₅	71−72	327	(14 700);	380	(17 200);	422	(18 200)
11	4-MeOC₆H₄	H	4-MeOC₆H₄	176−177	307	(13 000);	426	(23 400)		
12	C₆H₅	C₆H₅	H	112−113	303	(14 500);	428	(12 400)		
13	Me	Me	Me	179−180	300	(6800);	380	(7600);	419	(17 500)

[a] All compounds show satisfactory elemental analysis. [b] 10^{-5} M solutions in CH_2Cl_2.

the extinction coefficient of the complex at a given wavelength is higher than the sum of the extinction coefficients of the starting ions at the same wavelength. This is further supported by a distinct shoulder in the absorption spectrum near 60000 nm, where none of the starting salts absorb.

It is found that the extinction coefficients are solvent dependent as they vary in the following order: $\varepsilon_{DMSO} > \varepsilon_{MeCN} > \varepsilon_{CHCl_3} > \varepsilon_{Dioxan}$ which is the same order as is found for the variation of the dielectric constant of the solvents, but no simple correlation between the extinction coefficient and the dielectric constant could be established.

The UV-VIS data of the complexes prepared are given in Table 1.

The charge transfer observed is probably due to the process shown in eqn. (1).

$$(1,2) \rightleftharpoons \quad (1)$$
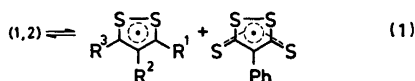
This assumption is supported by the observation that the complex derived from the 3,5-diphenyl-1,2-dithiolylium ion displays a weak ESR signal in the solid phase with an isotropic g value of 2.00. A g value of 2.003 has been observed for the electrochemically generated 3,5-diphenyl-1,2-dithiolyl radical in acetonitrile.[6]

The complex formed from the 3,5-diphenyl-1,2-dithiolylium ion has been found to have a specific d.c. resistance of 6×10^{12} ohm cm measured on a single crystal by the four probe technique along the crystallographic b-axis. The other complexes mentioned in Table 1 have a much higher resistance. This is consistent with the observation that the 3,5-diphenyl-1,2-dithiolylium ion is electrochemically reduced at a higher potential, -0.27 V,[6] than the 3-phenyl-1,2-dithiolylium and the 3,4-diphenyl-1,2-dithiolylium ions which are reduced at -0.45 and -0.46 V, respectively.[7] It has been assumed that electron-transfer calculated from solution redox potentials can be correlated to the conductivity in the solid phase.[8]

A prerequisite for an organic charge-transfer complex of the same type as TTF−TCNQ to be a good conductor is that the crystals are highly ordered, made up of segregated stacks of acceptor molecules and donor molecules, whereas mixing of acceptor and donor molecules in the stacks results in low conductivity.[8]

The crystal structure of *7* has its highest packing density in the directions of the a and b axes where the average distance between the 1,2-dithiole rings is 5.61 and 5.65 Å, respectively. The packing in these directions consists, however, of stacks with alternating donor and acceptor molecules. The packing in the direction of the c axis consists of isolated stacks of donor and acceptor molecules, but the
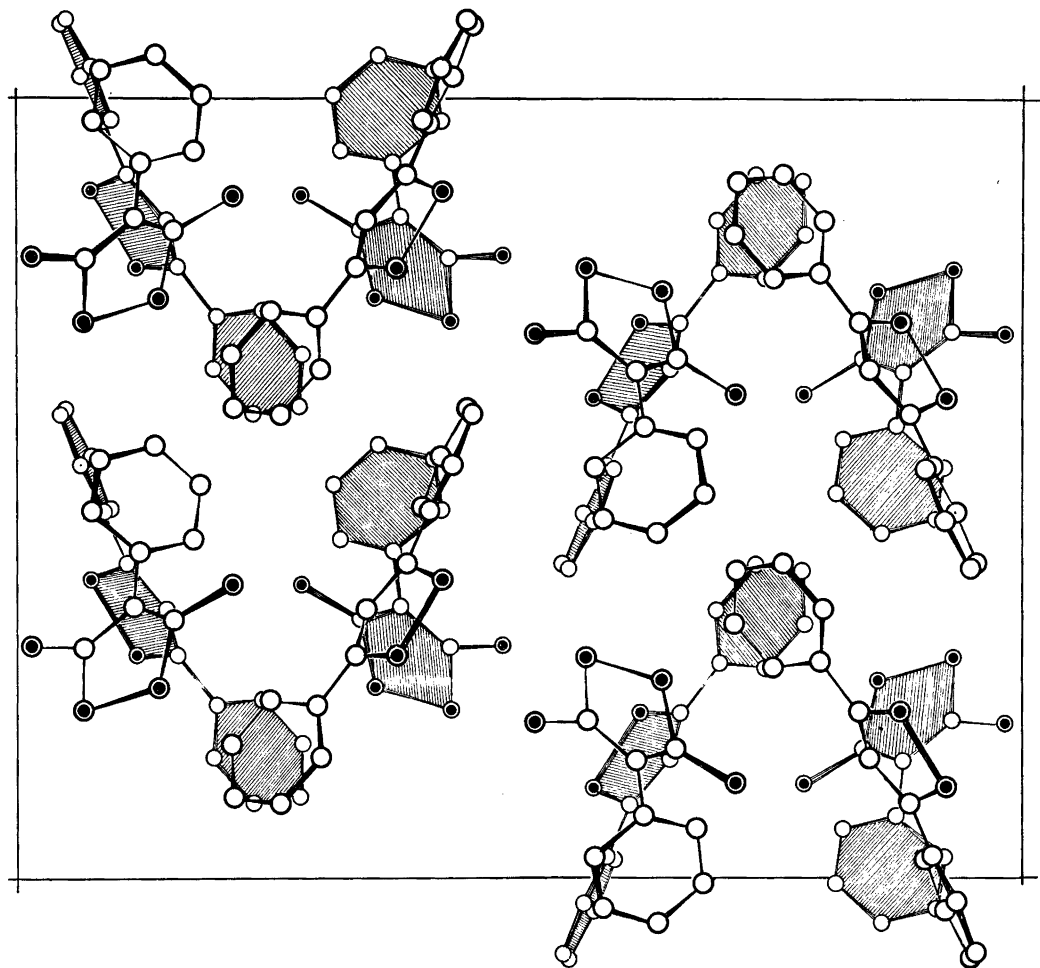
Fig. 2. The crystal structure of 7 as seen along the *b* axis.

distance between the dithiole rings here is 8.78 Å. This can be compared to the stacking distance between TTF molecules in TTF − TCNQ; this distance is 3.47 Å.[9] This explains the low conductivity of this complex.

From model considerations it is obvious that steric hindrance between H(52C) and H(4C) and H(36C) prevents the phenyl groups and the dithiole ring from being coplanar. As can be seen from the angles C(5C) − S(1C) − S(2C) and S(1C) − S(2C) − C(3C) (Table 3) the dithiole nucleus in the acceptor is not symmetrical, this is probably due to crystal effects rotating the phenyl groups out of the dithiole plane to various degrees (A,B = 16.8° and A,C =

24.8°, see Fig. 4) and thus the two phenyl groups do not contribute equally to the resonance of the system.

The structure of simple salts of the 3,5-diphenyl-1,2-dithiolylium ion has not been reported, but the results obtained for this complex are in good agreement with the structure published by Mason *et al.*[10] for bis(3,5-diphenyl-1,2-dithiolylium) tetrachloroferrate(II).

The dithiole part in the donor molecule has experimental, although not crystallographic, mirror symmetry and the phenyl group is rotated 55.93° out of the plane of the dithiole ring. Therefore, there is no mesomeric contribution from this phenyl group.
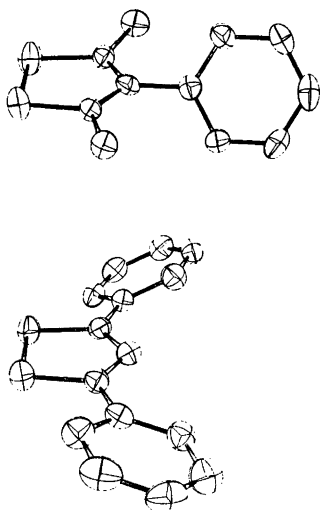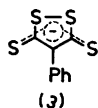
Table 2. Interatomic distances in Å. Estimated standard deviations in parentheses.

Atoms	Distance
S(1A) – S(2A)	2.065(2)
S(2A) – C(3A)	1.738(5)
C(3A) – C(4A)	1.410(7)
C(4A) – C(5A)	1.404(7)
C(5A) – S(1A)	1.735(5)
C(3A) – S(31A)	1.689(5)
C(5A) – S(51A)	1.697(5)
C(4A) – C(41A)	1.482(7)
C(41A) – C(42A)	1.397(7)
C(42A) – C(43A)	1.388(8)
C(43A) – C(44A)	1.385(9)
C(44A) – C(45A)	1.388(9)
C(45A) – C(46A)	1.393(8)
C(46A) – C(41A)	1.394(7)
S(1C) – S(2C)	2.017(2)
S(2C) – C(3C)	1.716(5)
C(3C) – C(4C)	1.374(7)
C(4C) – C(5C)	1.381(7)
C(5C) – S(1C)	1.713(5)
C(3C) – C(31C)	1.473(7)
C(5C) – C(51C)	1.468(7)
C(31C) – C(32C)	1.403(7)
C(32C) – C(33C)	1.374(7)
C(33C) – C(34C)	1.381(9)
C(34C) – C(35C)	1.378(9)
C(35C) – C(36C)	1.388(8)
C(36C) – C(31C)	1.402(7)
C(51C) – C(52C)	1.395(8)
C(52C) – C(53C)	1.386(8)
C(53C) – C(54C)	1.389(9)
C(54C) – C(55C)	1.366(9)
C(55C) – C(56C)	1.377(8)
C(56C) – C(51C)	1.404(7)

Fig. 3. Perspective drawing of the acceptor and donor molecules of *7*. Atoms are represented by ellipsoids enclosing 50 % of the thermal displacements.

Therefore, the correct form of the donor must be formulated as (*3*), and not as (*2*). The symmetrical form is also dominating in the dimethylammonium salt.[11] This is reflected in

the ^{13}C NMR spectrum of the salt which only displays two signals due to the dithiole carbons.

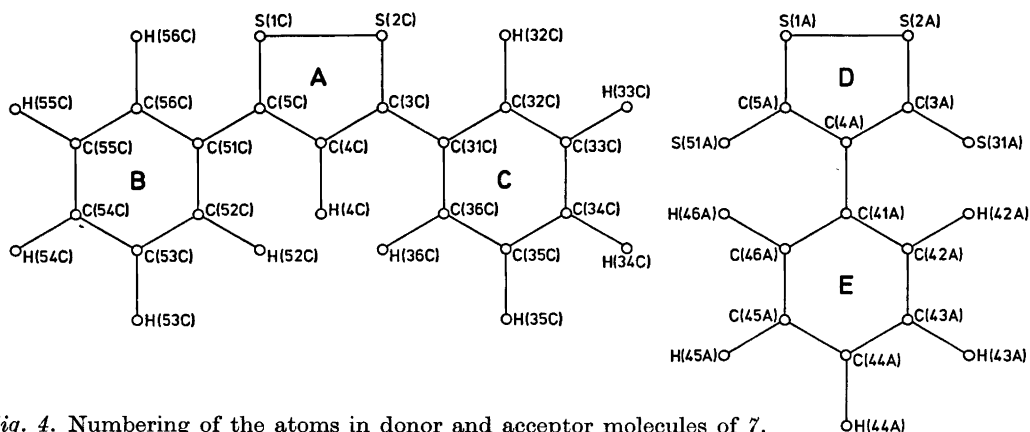The H – C bond distances (Å) range from 0.89(6) to 1.10(7) (mean value 1.01).

Fig. 4. Numbering of the atoms in donor and acceptor molecules of *7*.

EXPERIMENTAL

The compounds in Table 1 were prepared by mixing equimolar acetonitrile solutions of 1,2-dithiolylium perchlorates and dimethyl-ammonium 4-phenyl-5-thioxo-1,2-dithiole-3-thiolate with stirring at room temperature; the precipitates were filtered after 5 min and then washed with acetonitrile and ether. The compounds obtained in this way were normally analytically pure.

The crystals used for the structure determinations were prepared by placing the two starting components in "the legs" of an H-shaped tube. The tube was carefully filled with acetonitrile and left for two weeks. After this time single crystals of the complex could be collected from the horizontal part of the H tube.

The electrical conductivity was found by four probe conductivity measurements using the contracting technique by Coleman.[12]

Accurate unit cell dimensions were determined by a least-squares refinement of data measured from a Guinier powder photograph taken with CuKα_1 radiation ($\lambda = 1.54050$ Å) and calibrated with quartz as an internal standard.

The space group $Pbca$ (No. 61) was unequivocally established from the systematic absences. The three dimensional X-ray data were obtained using a three-circle Enraf-Nonius diffractometer and Zr filtered MoKα radiation. A reflection was designated not observed if $I < 2\sigma(I)$. With this criterion 3880 out of 6316 (independently measured) reflections were regarded as observed. The temperature was $15-20$ °C. A single reflection (14,0,0) was monitored every 10 reflections. Lorentz and polarization corrections were applied, but no extinction or absorption correction was made.

From the needle-shaped crystal a fragment measuring $0.5 \times 0.5 \times 0.75$ mm³ was cut and mounted with the b axis parallel to the ϕ axis of the diffractometer, ω-scan was used. ω-scan range = 1.6°, ω-scan speed = 0.6° min⁻¹. The maximum value of sin θ/λ was 0.703 Å⁻¹.

All calculations were performed on an IBM 370/165 computer using the X-ray system.[13] The structure was solved by direct methods and the structural parameters were refined by full-matrix least squares. An E map computed with 616 phases revealed the 6 sulfur atoms and 12 carbon atoms. An electron density calculation based on the parameters from the identified E map atoms showed all the non-hydrogen atoms.

For the initial cycles of least-squares refinement, the single scale factor, individual isotropic temperature factors, and atomic coordinates for all non-hydrogen atoms were refined.

Position for the hydrogen atoms were calculated. The positional hydrogen parameters were refined in the last refinement cycles. The hydrogen atoms were given the same isotropic

Table 3. Bond angles in degrees with their estimated standard-deviations in parentheses.

Atoms	Angles (°)
C(5A) − S(1A) − S(2A)	95.6(2)
S(1A) − S(2A) − C(3A)	95.5(2)
S(2A) − C(3A) − C(4A)	115.8(3)
S(2A) − C(3A) − S(31A)	114.1(3)
S(31A) − C(3A) − C(4A)	130.0(4)
C(3A) − C(4A) − C(5A)	117.1(4)
C(3A) − C(4A) − C(41A)	121.2(4)
C(41A) − C(4A) − C(5A)	121.6(4)
C(4A) − C(5A) − S(1A)	115.9(4)
C(4A) − C(5A) − S(51A)	130.0(4)
S(51A) − C(5A) − S(1A)	114.1(3)
C(4A) − C(41A) − C(42A)	120.8(4)
C(4A) − C(41A) − C(46A)	120.7(4)
C(42A) − C(41A) − C(46A)	118.5(5)
C(41A) − C(42A) − C(43A)	120.6(5)
C(42A) − C(43A) − C(44A)	120.4(5)
C(43A) − C(44A) − C(45A)	119.8(5)
C(44A) − C(45A) − C(46A)	119.8(5)
C(45A) − C(46A) − C(41A)	121.0(5)
C(5C) − S(1C) − S(2C)	96.2(2)
S(1C) − S(2C) − C(3C)	95.2(2)
S(2C) − C(3C) − C(4C)	115.9(4)
S(2C) − C(3C) − C(31C)	116.5(4)
C(31C) − C(3C) − C(4C)	127.6(4)
C(3C) − C(4C) − C(5C)	117.7(5)
C(4C) − C(5C) − S(1C)	114.9(4)
C(4C) − C(5C) − C(51C)	127.3(4)
C(51C) − C(5C) − S(1C)	117.6(4)
C(3C) − C(31C) − C(32C)	121.5(4)
C(3C) − C(31C) − C(36C)	119.5(5)
C(32C) − C(31C) − C(36C)	118.9(5)
C(31C) − C(32C) − C(33C)	119.8(5)
C(32C) − C(33C) − C(34C)	121.0(5)
C(33C) − C(34C) − C(35C)	119.8(5)
C(34C) − C(35C) − C(36C)	120.3(5)
C(35C) − C(36C) − C(31C)	120.1(5)
C(5C) − C(51C) − C(52C)	120.4(5)
C(5C) − C(51C) − C(56C)	120.0(5)
C(52C) − C(51C) − C(56C)	119.5(5)
C(51C) − C(52C) − C(53C)	119.8(5)
C(52C) − C(53C) − C(54C)	120.3(5)
C(53C) − C(54C) − C(55C)	119.5(6)
C(54C) − C(55C) − C(56C)	121.7(5)
C(55C) − C(56C) − C(51C)	119.1(5)

The H − C − C bond angles (°) range from 111 (4) to 128 (4) (mean value 120).

temperature factors as have the atoms to which they are bonded (initial individual isotropic refinement).

The quantity minimized in the final refinement cycles was $\sum w(|F_o| - |F_c|)^2$, unobserved reflections were not included. The weighting

Table 4. Distances of atoms (in Å) from least-squares planes. The planes are in the form $q_1x + q_2y + q_3z = q_4$. The coordinates x, y and z are expressed parallel to the orthorhombic axial directions a, b and c.[a]

	Plane	q_1		q_2		q_3		q_4	
	A	0.73511		−0.46027		−0.49777		−0.64045	
	B	0.58899		−0.69741		−0.40831		−1.60566	
	C	0.91081		−0.07907		−0.40518		1.30052	
	D	0.58049		−0.63787		−0.50612		1.53814	
	E	0.34186		0.80608		0.48308		6.87636	
S(1C)	.006	C(51C)	.013	C(31C)	.004	S(1A)	.004	C(41A)	−.006
S(2C)	−.009	C(52C)	−.012	C(32C)	−.005	S(2A)	−.002	C(42A)	.000
C(3C)	.010	C(53C)	.002	C(33C)	.006	C(3A)	−.001	C(43A)	.004
C(4C)	−.005	C(54C)	.007	C(34C)	−.006	C(4A)	.005	C(44A)	−.002
C(5C)	−.003	C(55C)	−.006	C(35C)	.005	C(5A)	−.007	C(45A)	−.004
*C(31C)	−.009	C(56C)	−.004	C(36C)	−.004	*S(31A)	.015	C(46A)	.008
*C(51C)	.024	*C(5C)	.077	*C(3C)	−.061	*S(51A)	−.021	*C(4A)	.007
*H(4C)	.11	*H(52C)	−.13	*H(32C)	.18	*C(41A)	.028	*H(42A)	−.01
		*H(53C)	−.03	*H(33C)	.14			*H(43A)	−.11
		*H(54C)	−.06	*H(34C)	−.06			*H(44A)	− 03
		*H(55C)	−.08	*H(35C)	−.17			*H(45A)	.06
		*H(56C)	−.01	*H(36C)	.08			*H(46A)	−.06

[a] Atoms marked with * were not included in the calculations of the least-squares planes.

Table 5. Final atomic coordinates with estimated standard deviations in parentheses.

Atom	x	y	z	Atom	x	y	z
S(1A)	.43463(7)	.22339(13)	.28092(8)	C(31C)	.0897(2)	.3628(4)	.0281(3)
S(2A)	.36007(7)	.12440(12)	.25269(9)	C(32C)	.0649(2)	.3019(5)	.9659(3)
S(31A)	.28476(6)	.14906(12)	.12080(8)	C(33C)	.0462(2)	.3641(6)	.9029(3)
S(51A)	.48599(7)	.42078(12)	.19942(8)	C(34C)	.0500(3)	.4866(6)	.9003(3)
C(3A)	.3449(2)	.2006(4)	.1688(3)	C(35C)	.0743(3)	.5479(5)	.9608(4)
C(4A)	.3845(2)	.2939(4)	.1507(3)	C(36C)	.0937(3)	.4872(5)	.0251(3)
C(5A)	.4312(2)	.3164(4)	.2022(3)	C(51C)	.1996(2)	.2846(5)	.2774(3)
C(41A)	.3772(2)	.3648(4)	.0802(3)	C(52C)	.2449(3)	.3694(5)	.2714(3)
C(42A)	.3237(2)	.4245(5)	.0652(3)	C(53C)	.2858(3)	.3828(6)	.3300(4)
C(43A)	.3173(3)	.4919(5)	.9995(3)	C(54C)	.2811(3)	.3142(6)	.3956(3)
C(44A)	.3638(3)	.5005(5)	.9476(3)	C(55C)	.2356(3)	.2337(6)	.4019(3)
C(45A)	.4173(3)	.4419(5)	.9618(3)	C(56C)	.1945(3)	.2171(6)	.3444(3)
C(46A)	.4239(2)	.3755(5)	.0282(3)	H(4C)	.176(3)	.417(6)	.144(4)
H(42A)	.288(3)	.417(6)	.105(4)	H(32C)	.069(3)	.214(6)	.964(4)
H(43A)	.281(3)	.526(6)	.982(4)	H(33C)	.037(3)	.316(6)	.864(4)
H(44A)	.355(3)	.555(7)	.901(4)	H(34C)	.032(3)	.526(6)	.850(4)
H(45A)	.455(3)	.454(6)	.922(4)	H(35C)	.073(3)	.631(6)	.971(4)
H(46A)	.458(3)	.331(6)	.036(4)	H(36C)	.114(3)	.531(6)	.066(4)
S(1C)	.11771(7)	.13506(12)	.21392(8)	H(52C)	.250(3)	.429(6)	.232(4)
S(2C)	.07408(7)	.16596(12)	.11530(8)	H(53C)	.320(3)	.449(6)	.324(4)
C(3C)	.1082(2)	.3000(4)	.0978(3)	H(54C)	.310(3)	.328(7)	.443(4)
C(4C)	.1480(2)	.3379(5)	.1522(3)	H(55C)	.227(3)	.186(6)	.450(4)
C(5C)	.1580(2)	.2646(4)	.2140(3)	H(56C)	.160(3)	.157(6)	.347(4)

scheme was $w = 1.0/1 + (F_o - B)/A)^2$ with $A = 80$ and $B = 40$. The resulting R index $(R = \sum ||F_o| - |F_c||/\sum|F_o|)$ was 6.8 %.

Atomic scattering factors were taken from *International Tables*.[14]

Crystal data. 3,5-diphenyl-1,2-dithiolylium-4-phenyl-5-thioxo-1,2-dithiole-3-thiolate $(C_{24}H_{16}S_6)$ has a formula weight of 496.78. The crystals form black needles.

Space group *Pbca* (No. 61), $a = 22.363(2)$ Å, $b = 11.248(1)$ Å, $c = 17.559(2)$ Å, $Z = 8$, $D_m = 1.50$ g cm^{-3}, $D_c = 1.49$ g cm^{-3}. $\mu(MoK\alpha) = 6.65$ cm^{-1}.

A list of observed and calculated structure factors is available on request from Ole Simonsen, Kemisk Institut, Odense Universitet, Niels Bohrs Alle 75, DK-5000 Odense.

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Table 6. Thermal parameters μ_{ij} (Å)^2 with estimated standard deviations in parentheses. The expression used is: exp $[-2\pi^2(h^2a^{*2}U_{11} + \cdots + 2hka^*b^*U_{12} + \cdots)]$.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1A)	.0549(9)	.0412(8)	.0402(7)	− .0024(6)	− .140(6)	.0082(6)
S(2A)	.0587(9)	.0351(7)	.0416(7)	− .0070(6)	− .0065(7)	.0089(6)
S(31A)	.376(7)	.0394(7)	.0445(8)	− .0029(6)	− .0029(6)	− .0053(6)
S(51A)	.0424(8)	.0429(8)	.0420(7)	− .0049(6)	− .0017(6)	− .0015(6)
C(3A)	.037(3)	.027(2)	.027(2)	.004(2)	.002(2)	.001(2)
C(4A)	.029(2)	.028(2)	.038(2)	.008(2)	.001(2)	.000(2)
C(5A)	.033(2)	.030(2)	.032(2)	.002(2)	.003(2)	.001(2)
C(41A)	.037(2)	.030(2)	.030(2)	.001(2)	− .002(2)	− .003(2)
C(42A)	.041(3)	.039(3)	.042(3)	.010(2)	− .003(2)	.002(2)
C(43A)	.049(3)	.047(3)	.048(3)	.008(3)	− .011(3)	.010(3)
C(44A)	.076(4)	.037(3)	.032(3)	.001(3)	− .012(3)	.003(2)
C(45A)	.058(4)	.051(3)	.029(2)	− .002(3)	.010(2)	.000(2)
C(46A)	.042(3)	.043(3)	.035(3)	.011(2)	.007(2)	.000(2)
S(1C)	.0503(8)	.0364(7)	.0416(8)	− .0069(6)	− .0041(6)	.0033(6)
S(2C)	.0445(8)	.0354(7)	.0426(7)	− .0099(6)	− .0045(6)	− .0008(6)
C(3C)	.028(2)	.034(2)	.036(2)	.000(2)	.000(2)	− .003(2)
C(4C)	.033(2)	.037(3)	.040(3)	.000(2)	− .001(2)	− .002(2)
C(5C)	.035(2)	.031(2)	.038(3)	.000(2)	.004(2)	− .005(2)
C(31C)	.032(3)	.032(2)	.040(3)	− .002(2)	.002(2)	.000(2)
C(32C)	.034(3)	.041(3)	.040(3)	− .006(2)	.001(2)	− .003(2)
C(33C)	.040(3)	.056(4)	.036(3)	− .007(3)	− .001(2)	− .002(3)
C(34C)	.041(3)	.056(3)	.039(3)	− .006(3)	− .001(2)	.010(3)
C(35C)	.045(3)	.035(3)	.063(4)	− .003(3)	− .008(3)	.007(3)
C(36C)	.046(3)	.041(3)	.043(3)	− .001(2)	− .008(2)	− .003(2)
C(51C)	.040(3)	.033(3)	.038(3)	.006(2)	.001(2)	− .002(2)
C(52C)	.046(3)	.038(3)	.040(3)	.002(2)	− .003(2)	− .006(2)
C(53C)	.058(4)	.052(4)	.046(3)	− .003(3)	− .004(3)	− .014(3)
C(54C)	.047(3)	.070(4)	.042(3)	.008(3)	− .016(3)	− .017(3)
C(55C)	.062(4)	.068(4)	.037(3)	.015(3)	− .005(3)	.003(3)
C(56C)	.045(3)	.053(3)	.041(3)	− .001(3)	− .002(2)	.001(3)

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