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Structures of Linear Multisulphur Systems

V. The Crystal and Molecular Structure of α-[7-(5-*t*-Butyl-1,2--dithiole-3-ylidene)-4,5,6,7-tetrahydro-1,2-benzodithiole-3-ylidene]-acetophenone

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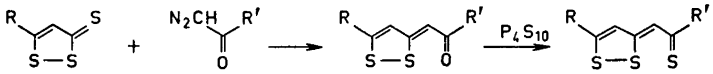
The structure of α-[7-(5-*t*-butyl-1,2-dithiole-3-ylidene)-4,5,6,7-tetrahydro-1,2-benzodithiole-3-ylidene]-acetophenone has been determined by X-ray crystallographic methods. 4759 independent reflections were measured on a diffractometer using the $\theta-2\theta$ scan technique and Mo$K\alpha$ radiation. The structure was solved by direct methods and refined by full-matrix least-squares to $R=0.050$. The four sulphur atoms and the oxygen atom are arranged on an approximately linear row, with interatomic distances: S(1)–S(2) = 2.064(1) Å, S(2)–S(3) = 2.856(1) Å, S(3)–S(4) = 2.110(1) Å, and S(4)–O = 2.327(2) Å. The two short bond lengths are close to a normal S(II)–S(II) single bond of 2.10 Å. The S–O and the long S–S bond are both in the region between single bond and van der Waals distance.

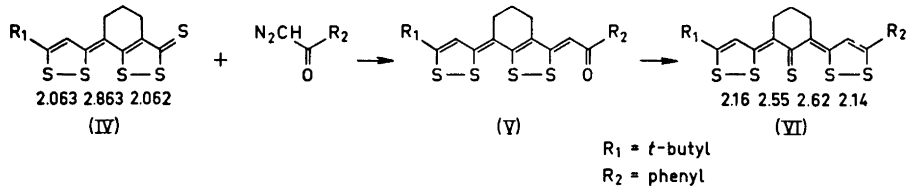
Poirier and Lozac'h have described the synthesis of a dithiafurophthene, II, by the reaction of diazoacetophenone and 1,2-dithiole-3-thione.[1] By the action of P_4S_{10} on II the oxygen is substituted by sulphur, giving a thiathiophthene, III.



(I) (II) (III)

Stavaux and Lozac'h have used an analogous method to synthesize compound V, and the corresponding five-sulphur compound, VI.[2]

A large number of both experimental and theoretical investigations have been undertaken to determine the structures and electronic properties of compounds analogous to I, II, and III. (See references 3–5 for recent review articles.) The structures of compounds IV and VI and five other linear four- and five-sulphur compounds have been determined.[6–12] The sulphur-sulphur



bonding scheme in IV seems to be adequately described in terms of two isolated dithiole systems,[6] while in compound VI delocalized σ-bonding extending across all sulphur atoms apparently exists.[7] A comparison of the UV and visible spectra of II and III shows that the replacement of oxygen by sulphur causes a bathochromic shift in the visible region and introduces a stronger absorption in the UV region.[13] A corresponding change in the spectrum is observed by going from V to VI.[14]

The present structure investigation, of compound V, was undertaken to determine whether this type of molecule may be regarded as an extended thiafurophthene system.

EXPERIMENTAL

The compound was supplied as a micro-crystalline sample. Several attempts to grow crystals suitable for X-ray work failed. Crystallization problems have been encountered with a number of the compounds studied in this series. In the present case the most satisfying results were obtained by evaporation at room temperature from a carbon disulphide solution.

The crystals grew elongated along the a-axis direction. As the crystals fracture easily, a solvent saw [15] was used to cut the crystal chosen for data collection to a suitable length. The dimension of the crystal was 0.76 mm × 0.25 mm × 0.15 mm. Some of the reflections showed splitting, indicating that the crystal was slightly fractured. Determination of space group and preliminary cell dimensions were carried out using Weissenberg and precession photographs. More accurate cell dimensions were determined by measuring setting angles for 16 reflections on an off-line diffractometer using Mo$K\alpha$ radiation. The density were measured by flotation. The crystal data are as follows:

$C_{22}H_{22}OS_4$ $M.W. = 430.67$

Crystal system orthorhombic; space group *Pbca* (No. 61); cell dimensions:

$a = 10.979(2)$ Å, $b = 17.549(4)$ Å, $c = 21.832(4)$ Å
$V = 4206(2)$ Å^3
$D_m = 1.359$ g cm^{-3}, $D_x = 1.360$ g cm^{-3}, $Z = 8$, $\mu_{MoK\alpha} = 4.52$ cm^{-1}

4759 unique reflections with $2\theta \leqslant 55°$ were measured by the $\theta - 2\theta$ scan technique on an off-line four-circle diffractometer using niobium-filtered Mo$K\alpha$ radiation ($\lambda = 0.71069$ Å). The "five value" measurement procedure was employed, and scan ranges for low and high 2θ sides were calculated according to the tangent relationship given by Alexander and Smith.[16]

The intensities of two reference reflections were recorded for every 50 reflections. These measurements were used to bring the data to a common scale. The uncertainty in scale factors were estimated at 2 %. The standard deviations in intensities were taken as $\sigma_I = [\sigma_c^2 + (0.02\sigma_c^2)^2]^{1/2}$, where σ_c is the error due to counting statistics. σ_F was calculated as $\sigma_I/2(I\ Lp)^{1/2}$. 1264 reflections were measured to be less than $2\sigma_c$. These reflections were given a threshold value of $2\sigma_c$ and included in the refinement only when $|F_{calc}| > |F_{threshold}|$. Data were corrected for Lorentz and polarization effects. An absorption correction according to the method described by Coppens *et al.*[17] was applied.

SOLUTION AND REFINEMENT

The structure was solved by a symbolic addition procedure programmed by Long.[18] Signs for 259 reflections with $|E| \geq 1.70$ were derived by reiterative application of Sayre's equation. An E-map[19] calculated from the most probable set of phases clearly revealed the sulphur atoms, the oxygen and 13 out of the 22 carbon atoms. Smaller peaks were also observed in reasonable positions for the remaining atoms. Carbon atom C(15) was represented by a double peak in the E-map, and was later shown to be disordered. Structure factors were calculated based on four sulphur atoms, one oxygen and thirteen carbon atoms, and in the subsequent Fourier map the remaining carbon atoms were located. The structure was refined by full-matrix least-squares. At an R of 0.17 ($R = \sum||F_o| - |F_c|| / \sum|F_o|$), anisotropic temperature factors were introduced. The thermal parameters of C(15) were abnormally high ($U \approx 0.2$ Å^2), indicating disorder. A difference map confirmed the presence of disorder, and two atoms, C(151) and C(152), each with multiplicity 0.5 and isotropic temperature factor, were introduced in positions as found from the difference map. These positions are in good agreement with those observed in the E-map. The fractional atoms refined satisfactorily. C(16) is also apparently disordered; however, judging from the thermal parameters the separation between the fractional sites are less than for C(15). An attempt to refine two fractional atoms in this case was not successful.

From a difference map all the hydrogen atoms were found, except those on C(14), C(151), C(152), and C(16), which due to the disorder could not be unambigously located. Hydrogen atoms were refined isotropically; however, the *t*-butyl hydrogen atoms refined to unreasonable positions, and were therefore kept fixed at the positions obtained from the difference map and with U values comparable to the carbon atoms to which they are attached. At the end of the refinement a secondary extinction correction was carried out.[20] The extinction coefficient was found to be 0.12×10^{-6}, the maximum correction in F_{obs} being 14 %. The final agreement factor is 0.050. The function minimized in the refinement was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma_F^2$. Scattering factors used were for sulphur, oxygen, and carbon atoms those of Hanson *et al.*[21] and for hydrogen those of Stewart *et al.*[22]

At the end of the refinement a residual difference map was calculated. In the disordered region electron densities up to 0.50 e.Å^{-3} are found.

RESULTS AND DISCUSSION

Atomic coordinates and thermal parameters are listed in Tables 1 and 2. The molecular dimensions are shown in Fig. 1 and in Tables 3 and 4. The four sulphur atoms and the oxygen atom constitute an approximately linear row, the pertinent angles being $\angle S(1)-S(2)-S(3) = 179.57°$, $\angle S(2)-S(3)-S(4) = 175.26°$, and $\angle S(3)-S(4)-O = 174.25°$.

A least-squares plane through the atoms of rings A, B, C, and D (Fig. 1) shows that there is a small but significant deviation from planarity in this part of the molecule. The molecule is slightly twisted at C(4), so as to leave S(2) slightly above and S(3) slightly below the least-squares plane. In addition the

Table 1. Fractional atomic coordinates with the corresponding standard deviations, referring to the last decimal places, listed in parentheses. The standard deviations are derived from the inverse least-squares matrix, except those for H(111) through H(133), which are estimated from the difference Fourier map.

Atom	*X/a*	*Y/b*	*Z/c*
S(1)	0.36393(7)	0.13110(6)	0.41865(5)
S(2)	0.51640(7)	0.09645(5)	0.46647(4)
S(3)	0.72702(6)	0.04740(4)	0.53233(3)
S(4)	0.87622(6)	0.01532(4)	0.58749(3)
O	1.05219(17)	−0.01093(11)	0.64380(9)
C(1)	0.44138(24)	0.19513(15)	0.37182(12)
C(2)	0.56056(25)	0.20036(18)	0.38128(13)
C(3)	0.62155(24)	0.15549(15)	0.42776(12)
C(4)	0.74281(24)	0.15841(15)	0.44162(12)
C(5)	0.80433(23)	0.11425(14)	0.48786(12)
C(6)	0.92669(23)	0.12168(14)	0.50169(12)
C(7)	0.97708(23)	0.07687(15)	0.54986(12)
C(8)	1.09644(25)	0.07882(17)	0.56984(13)
C(9)	1.13102(25)	0.03197(15)	0.61942(12)
C(10)	0.36503(25)	0.23717(18)	0.32371(14)
C(11)	0.44505(36)	0.29401(28)	0.28870(19)
C(12)	0.31060(44)	0.17953(26)	0.27913(19)
C(13)	0.26315(34)	0.28073(23)	0.35547(18)
C(14)	0.82339(29)	0.21363(25)	0.40548(17)
C(151)	0.93275(55)	0.23539(37)	0.43470(29)
C(152)	0.94837(53)	0.19873(35)	0.40322(27)
C(16)	1.00657(27)	0.17673(19)	0.46731(17)
C(17)	1.25662(25)	0.03187(16)	0.64441(12)
C(18)	1.35125(27)	0.07215(19)	0.61802(15)
C(19)	1.46632(30)	0.07157(21)	0.64353(17)
C(20)	1.48836(34)	0.02995(22)	0.69621(17)
C(21)	1.39537(39)	−0.01044(26)	0.72198(18)
C(22)	1.28275(35)	−0.00950(22)	0.69665(17)
H(2)	0.6098(23)	0.2385(16)	0.3633(12)
H(8)	1.1490(22)	0.1138(15)	0.5533(12)
H(111)	0.4670(60)	0.3350(35)	0.3170(30)
H(112)	0.4000(60)	0.3180(35)	0.2670(30)
H(113)	0.5000(60)	0.2600(35)	0.2670(30)
H(121)	0.4000(60)	0.1550(35)	0.2670(30)
H(122)	0.2330(60)	0.1980(35)	0.2500(30)
H(123)	0.2670(60)	0.1290(35)	0.3000(30)
H(131)	0.2190(60)	0.3100(35)	0.3260(30)
H(132)	0.3180(60)	0.3170(35)	0.3850(30)
H(133)	0.2110(60)	0.2360(35)	0.3750(30)
H(18)	1.3328(26)	0.1060(18)	0.5818(14)
H(19)	1.5245(30)	0.1031(20)	0.6238(16)
H(20)	1.5700(32)	0.0284(19)	0.7195(15)
H(21)	1.4077(30)	−0.0405(19)	0.7533(16)
H(22)	1.2273(27)	−0.0310(18)	0.7095(14)

Table 2. Thermal parameters with the corresponding standard deviations in parentheses. Anisotropic temperature factors are given by:
$T_i = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{23}klb^*c^* + 2U_{13}hla^*c^*)]$,
and isotropic temperature factors by: $T_i = \exp(-8\pi^2 U \sin^2\theta/\lambda^2)$. For non-hydrogen atoms the values are multiplied by 10^4, for hydrogen atoms by 10^3.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
S(1)	375(4)	744(6)	1016(7)	−98(4)	331(5)	−70(5)
S(2)	372(4)	558(5)	841(6)	−59(3)	266(4)	−2(4)
S(3)	373(4)	482(4)	538(4)	−76(3)	124(4)	27(3)
S(4)	416(4)	440(4)	475(4)	−27(3)	98(3)	52(3)
O	509(11)	622(13)	573(13)	−51(10)	115(11)	78(10)
C(1)	461(16)	439(16)	491(16)	−25(13)	−42(13)	−18(14)
C(2)	431(16)	551(18)	481(17)	−88(15)	60(15)	−20(14)
C(3)	423(15)	376(14)	460(16)	−42(12)	−6(12)	49(13)
C(4)	409(14)	440(16)	440(15)	−45(13)	45(13)	12(13)
C(5)	394(14)	379(14)	401(15)	−46(12)	−1(11)	58(11)
C(6)	382(14)	368(14)	402(15)	−30(12)	−2(12)	55(12)
C(7)	397(14)	374(14)	429(15)	−18(12)	−20(12)	81(12)
C(8)	396(15)	447(16)	485(17)	−29(13)	46(14)	42(13)
C(9)	461(16)	434(16)	430(16)	27(13)	−13(13)	78(13)
C(10)	505(17)	597(20)	545(19)	4(15)	−11(15)	−102(15)
C(11)	931(28)	1406(40)	1021(34)	72(29)	713(31)	−152(26)
C(12)	1553(42)	1002(33)	1036(32)	111(32)	−301(26)	−719(32)
C(13)	785(25)	920(28)	962(28)	367(22)	51(24)	−117(23)
C(14)	473(18)	1261(34)	935(28)	−352(21)	641(26)	−127(19)
C(16)	410(16)	479(18)	610(21)	−72(14)	86(17)	12(16)
C(17)	459(15)	429(16)	396(15)	35(13)	−5(13)	25(13)
C(18)	477(18)	592(19)	541(20)	43(15)	88(16)	−15(15)
C(19)	476(19)	724(23)	734(24)	−32(17)	58(20)	−41(18)
C(20)	596(21)	792(25)	708(24)	101(19)	−32(21)	−21(20)
C(21)	827(29)	933(30)	632(24)	42(24)	23(23)	−19(22)
C(22)	609(23)	773(26)	593(22)	−51(20)	220(19)	−30(19)

	U		U		U
C(151)	553(15)	H(113)	120	H(133)	120
C(152)	490(14)	H(121)	120	H(18)	65(10)
H(2)	46(8)	H(122)	120	H(19)	79(12)
H(8)	38(7)	H(123)	120	H(20)	82(11)
H(111)	120	H(131)	120	H(21)	73(11)
H(112)	120	H(132)	120	H(22)	56(11)

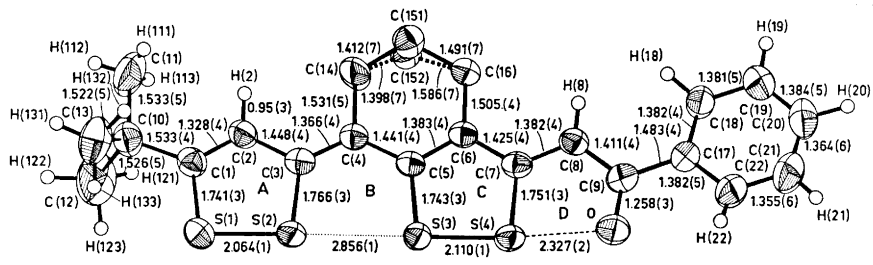


Fig. 1. ORTEP-plot [23] of the molecule showing thermal ellipsoids at the 50 % probability level. Hydrogen atoms are drawn with fixed radius. Bond distances are given with the corresponding standard deviations in parentheses.

Table 3. C–H bond distances in Å units with the corresponding standard deviations in parentheses.

C(2)–H(2)	0.95(3)	C(12)–H(122)	1.11(7)	C(18)–H(18)	1.01(3)
C(8)–H(8)	0.92(3)	C(12)–H(123)	1.11(7)	C(19)–H(19)	0.95(3)
C(11)–H(111)	0.98(7)	C(13)–H(131)	0.96(7)	C(20)–H(20)	1.03(3)
C(11)–H(112)	0.80(7)	C(13)–H(132)	1.08(7)	C(21)–H(21)	0.87(3)
C(11)–H(113)	0.97(7)	C(13)–H(133)	1.07(7)	C(22)–H(22)	0.77(3)
C(12)–H(121)	1.10(7)				

Table 4. Intramolecular angles (in degrees) with the corresponding standard deviations in parentheses.

S(2)–S(1)–C(1)	95.2(1)	C(12)–C(10)–C(13)	109.6(3)
S(1)–S(2)–S(3)	179.57(4)	C(10)–C(11)–H(111)	108(4)
S(1)–S(2)–C(3)	96.6(1)	C(10)–C(11)–H(112)	106(4)
S(3)–S(2)–C(3)	83.6(1)	C(10)–C(11)–H(113)	102(4)
S(2)–S(3)–S(4)	175.26(4)	H(111)–C(11)–H(112)	98(5)
S(2)–S(3)–C(5)	84.9(1)	H(111)–C(11)–H(113)	127(5)
S(4)–S(3)–C(5)	96.9(1)	H(112)–C(11)–H(113)	115(5)
S(3)–S(4)–O	174.25(6)	C(10)–C(12)–H(121)	94(4)
S(3)–S(4)–C(7)	93.4(1)	C(10)–C(12)–H(122)	118(4)
O–S(4)–C(7)	80.9(1)	C(10)–C(12)–H(123)	116(4)
S(4)–O–C(9)	103.8(2)	H(121)–C(12)–H(122)	131(5)
S(1)–C(1)–C(2)	115.8(2)	H(121)–C(12)–H(123)	100(5)
S(1)–C(1)–C(10)	116.5(2)	H(122)–C(12)–H(123)	98(5)
C(2)–C(1)–C(10)	127.8(2)	C(10)–C(13)–H(131)	109(4)
C(1)–C(2)–C(3)	121.8(2)	C(10)–C(13)–H(132)	99(4)
C(1)–C(2)–H(2)	123(2)	C(10)–C(13)–H(133)	102(4)
C(3)–C(2)–H(3)	114(2)	H(131)–C(13)–H(132)	111(5)
S(2)–C(3)–C(2)	110.6(2)	H(131)–C(13)–H(133)	113(5)
S(2)–C(3)–C(4)	123.6(2)	H(132)–C(13)–H(133)	120(5)
C(2)–C(3)–C(4)	125.8(2)	C(4)–C(14)–C(151)	115.5(4)
C(3)–C(4)–C(5)	126.3(2)	C(4)–C(14)–C(152)	117.8(3)
C(3)–C(4)–C(14)	118.2(2)	C(14)–C(151)–C(16)	119.4(4)
C(5)–C(4)–C(14)	115.5(2)	C(14)–C(152)–C(16)	114.2(4)
S(3)–C(5)–C(4)	121.6(2)	C(6)–C(16)–C(151)	111.4(3)
S(3)–C(5)–C(6)	114.5(2)	C(6)–C(16)–C(152)	111.2(3)
C(4)–C(5)–C(6)	123.9(2)	C(9)–C(17)–C(18)	123.0(2)
C(5)–C(6)–C(7)	119.9(2)	C(9)–C(17)–C(22)	119.8(2)
C(5)–C(6)–C(16)	121.2(2)	C(18)–C(17)–C(22)	117.2(3)
C(7)–C(6)–C(16)	119.7(2)	C(17)–C(18)–C(19)	121.1(3)
S(4)–C(7)–C(6)	116.2(2)	C(17)–C(18)–H(18)	118(2)
S(4)–C(7)–C(8)	117.8(2)	C(19)–C(18)–H(18)	120(2)
C(6)–C(7)–C(8)	126.0(2)	C(18)–C(19)–C(20)	119.9(3)
C(7)–C(8)–C(9)	118.9(2)	C(18)–C(19)–H(19)	115(2)
C(7)–C(8)–H(8)	119(2)	C(20)–C(19)–H(19)	125(2)
C(9)–C(8)–H(8)	122(2)	C(19)–C(20)–C(21)	119.1(3)
O–C(9)–C(8)	118.6(2)	C(19)–C(20)–H(20)	125(2)
O–C(9)–C(17)	119.2(2)	C(21)–C(20)–H(20)	116(2)
C(8)–C(9)–C(17)	122.2(2)	C(20)–C(21)–C(22)	120.6(3)
C(1)–C(10)–C(11)	110.2(2)	C(20)–C(21)–H(21)	121(2)
C(1)–C(10)–C(12)	109.4(3)	C(22)–C(21)–H(21)	118(2)
C(1)–C(10)–C(13)	109.4(2)	C(17)–C(22)–C(21)	122.2(3)
C(11)–C(10)–C(12)	109.8(3)	C(17)–C(22)–H(22)	125(2)
C(11)–C(10)–C(13)	108.8(3)	C(21)–C(22)–H(22)	113(2)

molecule is slightly bent around bond S(3)–C(5), the dihedral angle between the planes of A and C+D is 4.4°. A similar and somewhat more pronounced puckering has been observed in molecule IV.[6] The phenyl group is twisted 6.0° relative to the plane of ring D.

The bond C(10)–C(11) in the *t*-butyl group and bond C(2)–H(2) are eclipsed. The fractional atoms C(151) and C(152) are situated 0.4 and −0.5 Å, respectively, out of the plane through C(4), C(5), C(6), C(14), C(16). Since the disorder of atom C(16) could not be resolved, the bond distances between C(16) and the adjacent atoms are not realistic. In a case like this, the standard deviations obtained from the inverse least-squares matrix are underestimated.

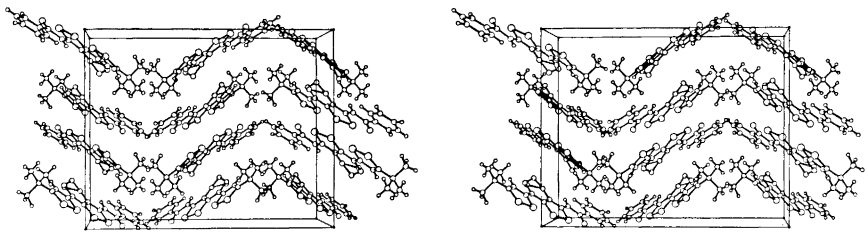


Fig. 2. Stereoscopic drawing showing the molecular packing as viewed down the *a*-axis.

The packing of molecules in the crystal is shown in a stereoplot (Fig. 2). Centrosymmetrically related molecules, *e.g.* the reference molecule and its inversion through 1,0,½, partially overlap, the distance between the least-squares planes being 3.4 Å. The shortest intermolecular sulphur-sulphur distances occur between the reference molecule and its inversion through ½,0,½, the short contacts being: S(1)–S(3)′ = 3.458 Å, S(1)–S(4)′ = 3.684 Å, S(2)–S(3)′ = 3.676 Å, and S(2)–S(2)′ = 3.706 Å.

In compound IV,[6] which is closely related to the present structure, the two terminal S–S bonds are of the same lengths as those found in isolated 1,2-dithiolium rings,[3] while the central S···S distance is slightly shorter than Huggins' constant energy distance of 2.92 Å.[24] When considering the possibility of partial bonding between atoms held in a covalent framework, it is probably more relevant to compare an experimentally determined distance with Huggins' constant energy distance than with the usually quoted sum of van der Waals radii. The effect of extending the linear row by introducing an oxygen atom is clearly demonstrated in the present structure. There is a pronounced lengthening of the bond S(3)–S(4) adjacent to the oxygen atom from 2.062 Å to 2.110 Å, the latter being significantly longer than the single bond distance of 2.10 Å.[25] The S(4)···O distance of 2.327 Å is appreciably shorter than the sum of the constant energy radii of 2.58 Å,[24] indicating a significant interaction between S(4) and O. Rings C+D constitute a system analogous to the dithiafurophthenes, II. For these compounds S–S and S–O distances in the regions 2.101–2.137 Å and 2.184–2.443 Å, respectively, have been found.[26–30] The S(3)–S(4) and S(4)···O distances observed in the

present compound fit nicely into this pattern. The influence of the oxygen atom on the bonding between the sulphur atoms can also be detected in the slight change in the S(2)···S(3) distance. However, the effect is, as might be expected, minor and within the experimental error. The terminal S(1)–S(2) bonds in the two compounds are identical. This indicates that a delocalized σ-system in the present compound does not include all the atoms in the linear row.

In compound VI, on the other hand, where the oxygen has been replaced by a sulphur atom the extention of the chain profoundly affects the bonding between all the atoms in the row. Thus compound VI, in contrast to the present compound, V, possesses an extended delocalized σ-system including all five atoms in the linear row.

Lists of observed and calculated structure factors may be obtained from the authors.

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