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Structures of Linear Multisulphur Systems

III. The Crystal and Molecular Structure of 2-(2-*p*-Methoxyphenyl-2-methylthiovinyl)-3,4-trimethylene-5-methylthio-1,6,6a-thiathiophthene

JORUNN SLETTEN

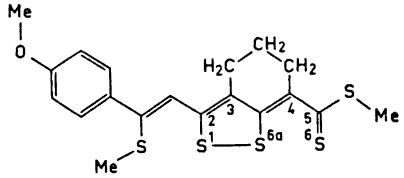
Chemical Institute, Universty of Bergen, N-5000 Bergen, Norway

The structure of $C_{19}H_{20}OS_5$ has been determined by X-ray diffraction methods. The crystals are monoclinic, space group $P2_1/c$, with cell dimensions $a=12.104(7)$ Å, $b=21.717(5)$ Å, $c=7.561(1)$ Å, $\beta=102.23(4)°$. Data were collected on an off-line automatic diffractometer. The structure was solved by the symbolic addition procedure and refined by full-matrix least-squares to an R of 0.029. Four sulphur atoms are arranged approximately in a linear row with interatomic distances S(1)–S(2)=3.0021(8) Å, S(2)–S(3)=2.1563(7) Å, and S(3)–S(4)=2.5510(7) Å. The S(2)–S(3), and S(3)–S(4) distances resemble the sulphur–sulphur distances in some of the unsymmetrically substituted thiathiophthenes.

The present structure determination is part of a program of X-ray crystallographic investigations of linear multisulphur compounds. A special feature of this type of compound is the occurrence of sulphur-sulphur bonds in the region single bond – van der Waals distance. A number of linear four- and five-sulphur compounds have been synthesized by Stavaux and Lozac'h [1] and by Klingsberg.[2] In addition to the present structure, the structures of three other four-sulphur compounds and two five-sulphur compounds have been determined by X-ray crystallographic methods.[3–7]

EXPERIMENTAL

The compound, 2-(2-*p*-methoxyphenyl-2-methylthiovinyl)-3,4-trimethylene-5-methylthio-1,6,6a-thiathiophthene, was supplied by Stavaux and Lozac'h.



(I)

Different procedures for growing crystals were tried. The best results were obtained by slow evaporation from a carbon disulphide solution. Several crystals were tested before a satisfactory single crystal was found. Space group and initial cell dimensions were determined from Weissenberg photographs. Later, final unit cell dimensions were derived from θ, χ, and ϕ measurements of 21 reflections on the diffractometer.

CRYSTAL DATA

$C_{19}H_{20}OS_5$ M.w. = 424.69
Monoclinic, systematic extinctions: $h0l$ when $l = 2n+1$; $0k0$ when $k = 2n+1$

Space group $P2_1/c$
$a = 12.104(7)$ Å, $b = 21.717(5)$ Å, $c = 7.561(1)$ Å, $\beta = 102.23(4)°$
$V = 1942$ Å^3
$D_m = 1.453$ g cm^{-3}, $D_x = 1.465$ g cm^{-3}
$Z = 4$
$\mu_{MoK\alpha} = 5.87$ cm^{-1}

The crystal used for all X-ray measurements had dimensions 0.90 mm × 0.80 mm × 0.17 mm, and was mounted along the a-axis. Intensity data were collected on a Siemens automatic off-line four-circle diffractometer with niobium filtered Mo$K\alpha$ radiation. 3421 unique reflections with $2\theta \leqslant 50°$ were measured using the moving crystal-moving counter technique. A "five value" measurement was employed. By this procedure the background is measured both on high and low 2θ sides and the integrated intensity of the peak is determined twice, once by scanning the two halves of the peak, on low and high 2θ sides, respectively, and once by scanning the total peak. Scan ranges for low and high 2θ sides of the peaks were calculated according to the tangent relationship of Alexander and Smith,[8] $\Delta\theta_1 = A_1 + B_1 \tan\theta$, $\Delta\theta_2 = A_2 + B_2 \tan\theta$. Before the measurements of each reflection the count rate at the top of the peak is measured. Based on the value obtained, the instrument automatically selects scan rate and, if necessary, attenuators so as to obtain similar counting statistics for all reflections, except the very weakest. Due to the use of attenuators the coincidence loss is negligible even for the strongest reflections.

The net count of each reflection is calculated as $N_{net} = N_{Pk} - N_{Bg}$, where N_{Pk} is the total number of counts in the peak scans, and N_{Bg} is the total background count. The estimated error in net count due to counting statistics is $\sigma_c = (N_{Pk} + N_{Bg})^{\frac{1}{2}}$. Three reference reflections were monitored throughout the data collection, and these measurements were used to scale the data. Also, scale factors according to scan time and attenuators used were applied to bring the data on the same relative scale.

Of the 3421 reflections 376 were less than twice the estimated error in measurement. These reflections were coded unobserved and were given the value $2\sigma_c$. The standard deviations in intensities were calculated as $\sigma_I = k[\sigma_c^2 + (SN_{net})^2]^{\frac{1}{2}}$, where k is the appropriate scale factor and S is an "instability factor" estimated from the variation in intensities of the reference reflections. In this case S was set equal to 0.02. Standard deviations in structure factors were calculated as $\sigma_F = \sigma_I / 2(I\ Lp)^{\frac{1}{2}}$. The data were corrected in

the usual way for Lorentz and polarization effects. Also an absorption correction was applied using the Gaussian integration method described by Busing and Levy.[9]

SOLUTION AND REFINEMENT OF THE STRUCTURE

The structure factors were converted to normalized structure factors using a program written by Shiono.[10] The Wilson method [11] was used to bring the data on absolute scale and to find an overall temperature factor. Three origin determining reflections and three reflections with variable signs were chosen, and a symbolic addition program written by Long [12] was used to calculate the 8 possible sets of signs using Sayre's equation $S(E_h) = S(\sum_{h'} E_{h-h'} E_{h'})$. The 258 reflections with E-values greater than 1.70 were included in this calculation. For each set of signs the consistency index, C, was calculated as

$$C = \frac{\langle |E_h \sum_{h'} E_{h-h'} E_{h'}| \rangle}{\langle |E_h| \sum_{h'} |E_{h-h'}| |E_{h'}| \rangle}.$$

The consistency indexes for the eight sets of signs ranged from 0.44 to 0.97. The set with the highest consistency index was used to calculate an E-map,[13] which clearly revealed peaks corresponding to all non-hydrogen atoms. The successful refinement showed that this was the correct solution.

Structure factor calculation based on atomic positions from the E-map gave an agreement factor of 0.32 ($R = \sum ||F_o| - |F_c|| / \sum |F_o|$). The structure was refined by full-matrix least-squares procedure adjusting scale factor, coordinates and isotropic thermal parameters. At an R of 0.14, anisotropic temperature factors were introduced. All of the 20 hydrogen atoms were located in a three-dimensional difference map and included in the refinement varying positional and isotropic thermal parameters. The refinement converged at an R of 0.029.

The function minimized in the refinement was $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\sigma_F^2$. The scattering factors used were for sulphur those of Dawson,[14] for oxygen and carbon those of Berghuis *et al.*[15] and for hydrogen those of Stewart *et al.*[16]

Table 1a. Final positional parameters with the corresponding standard deviations in parentheses.

	X/a	Y/b	Z/c
S(1)	0.82082(5)	0.10824(3)	0.11360(7)
S(2)	0.65196(4)	0.01317(2)	0.17159(6)
S(3)	0.53130(4)	−0.05403(2)	0.22257(6)
S(4)	0.38468(4)	−0.13069(2)	0.28569(6)
S(5)	0.40285(4)	−0.17868(2)	0.66284(7)
O(1)	1.24864(12)	0.23830(6)	0.68665(18)

Table 1a. Continued.

C(1)	0.87627(15)	0.09468(9)	0.34501(23)
C(2)	0.82411(15)	0.05701(8)	0.44309(23)
C(3)	0.72896(14)	0.01550(8)	0.39136(22)
C(4)	0.69422(14)	−0.02378(8)	0.51160(21)
C(5)	0.59626(14)	−0.06113(8)	0.45106(20)
C(6)	0.55343(14)	−0.10102(8)	0.56618(22)
C(7)	0.45372(15)	−0.13428(8)	0.50258(23)
C(8)	0.94458(27)	0.10630(19)	0.01640(38)
C(9)	0.97638(15)	0.13081(8)	0.43375(23)
C(10)	0.98964(17)	0.19263(9)	0.38891(27)
C(11)	1.07964(17)	0.22646(9)	0.47596(26)
C(12)	1.16260(15)	0.20053(8)	0.60961(23)
C(13)	1.33972(21)	0.21292(13)	0.81693(39)
C(14)	1.15270(16)	0.13880(9)	0.65442(25)
C(15)	1.06029(16)	0.10523(9)	0.56697(24)
C(16)	0.75736(17)	−0.02964(9)	0.70643(23)
C(17)	0.73993(16)	−0.09252(10)	0.78391(25)
C(18)	0.61542(16)	−0.10656(10)	0.76071(24)
C(19)	0.29184(21)	−0.22274(12)	0.52655(35)
H(2)	0.8512(15)	0.0598(9)	0.5692(26)
H(81)	0.9939(27)	0.1336(15)	0.0689(43)
H(82)	0.9704(31)	0.0604(19)	0.0395(44)
H(83)	0.9240(26)	0.1311(14)	−0.0907(46)
H(10)	0.9378(19)	0.2110(11)	0.2998(28)
H(11)	1.0865(18)	0.2669(11)	0.4432(27)
H(131)	1.3950(20)	0.2476(11)	0.8613(31)
H(132)	1.3763(26)	0.1825(16)	0.7503(43)
H(133)	1.3155(24)	0.1964(13)	0.9216(40)
H(14)	1.2042(17)	0.1217(9)	0.7398(27)
H(15)	1.0536(15)	0.0617(9)	0.5928(23)
H(161)	0.7301(17)	0.0012(9)	0.7765(28)
H(162)	0.8396(18)	−0.0229(9)	0.7143(24)
H(171)	0.7739(17)	−0.1244(9)	0.7198(26)
H(172)	0.7797(16)	−0.0941(9)	0.9115(26)
H(181)	0.6017(17)	−0.1447(11)	0.8029(28)
H(182)	0.5821(15)	−0.0780(9)	0.8334(24)
H(191)	0.3292(27)	−0.2486(14)	0.4534(42)
H(192)	0.2347(21)	−0.1939(12)	0.4534(33)
H(193)	0.2640(23)	−0.2450(12)	0.6064(34)

Table 1b. Thermal parameters with the corresponding standard deviations in parentheses. The anisotropic thermal parameters are defined by the expression $T_i = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$ and the isotropic parameters by $T_i = \exp[-8\pi^2 U \sin^2\theta/\lambda^2]$. For non-hydrogen atoms the values are multiplied by a factor of 10^4, for hydrogen atoms by 10^3.

	U_{11}	U_{22}	U_{33}	U_{12}	U_{23}	U_{13}
S(1)	498(3)	851(4)	398(3)	−136(3)	210(3)	7(2)
S(2)	489(3)	496(3)	277(2)	−64(2)	59(2)	1(2)
S(3)	464(3)	439(3)	283(2)	−17(2)	11(2)	−22(2)
S(4)	449(3)	496(3)	407(3)	−85(2)	14(2)	−9(2)
S(5)	461(3)	511(3)	462(3)	−65(2)	50(2)	109(2)
O(1)	568(8)	447(8)	588(8)	−118(7)	−13(6)	97(7)
C(1)	396(10)	432(10)	369(10)	60(8)	50(8)	76(8)

Table 1b. Continued.

C(2)	400(9)	378(10)	313(9)	27(8)	6(7)	38(7)
C(3)	384(9)	365(9)	290(9)	63(8)	−1(7)	40(7)
C(4)	353(9)	350(9)	281(9)	51(7)	−9(7)	44(7)
C(5)	366(9)	343(9)	271(8)	66(7)	−19(7)	36(7)
C(6)	361(9)	354(9)	338(9)	38(7)	−8(7)	62(7)
C(7)	382(9)	336(9)	407(10)	41(8)	−9(7)	88(8)
C(8)	783(19)	1114(26)	509(15)	−207(19)	−36(17)	240(13)
C(9)	388(10)	402(10)	359(9)	33(7)	28(8)	107(7)
C(10)	487(11)	437(11)	457(11)	54(9)	98(9)	115(9)
C(11)	560(12)	338(10)	523(11)	11(9)	66(8)	189(9)
C(12)	456(10)	394(10)	413(9)	−32(8)	−28(8)	186(8)
C(13)	517(13)	600(15)	754(17)	−102(12)	−21(13)	15(12)
C(14)	435(10)	412(10)	379(10)	22(8)	29(8)	75(8)
C(15)	458(10)	323(10)	418(10)	4(8)	30(8)	120(8)
C(16)	435(11)	460(11)	297(9)	−32(9)	25(8)	13(8)
C(17)	446(10)	537(12)	309(9)	47(9)	86(8)	1(8)
C(18)	466(11)	475(12)	328(9)	−37(9)	49(9)	61(8)
C(19)	556(13)	517(14)	633(14)	−130(11)	14(12)	145(12)

	U		*U*		*U*
H(2)	46(5)	H(131)	70(7)	H(171)	51(6)
H(81)	102(11)	H(132)	121(12)	H(172)	48(5)
H(82)	133(14)	H(133)	99(10)	H(181)	61(6)
H(83)	108(11)	H(14)	48(5)	H(182)	40(5)
H(10)	64(6)	H(15)	40(5)	H(191)	106(10)
H(11)	59(6)	H(161)	56(6)	H(192)	81(7)
		H(162)	44(5)	H(193)	82(8)

Table 2. Bond lengths with the corresponding standard deviations in parentheses. Bond lengths corrected for rigid body motion are listed in brackets.

Bond	Distance, Å	Bond	Distance, Å
S(1)−S(2)	3.0021(8)	O(1)−C(13)	1.425(3)
S(1)−C(1)	1.761(2)	C(14)−C(15)	1.381(3)[1.386]
S(1)−C(8)	1.803(4)	C(16)−C(17)	1.518(3)
S(2)−S(3)	2.1563(7)[2.1584]	C(17)−C(18)	1.511(3)
S(2)−C(3)	1.725(2)[1.732]	C(2)−H(2)	0.94(2)
S(3)−S(4)	2.5510(7)[2.5534]	C(8)−H(81)	0.87(3)
S(3)−C(5)	1.747(1)[1.754]	C(8)−H(82)	1.05(4)
S(4)−C(7)	1.677(2)[1.684]	C(8)−H(83)	0.96(3)
S(5)−C(7)	1.759(2)	C(10)−H(10)	0.91(2)
S(5)−C(19)	1.788(2)	C(11)−H(11)	0.92(2)
C(1)−C(2)	1.348(3)	C(13)−H(131)	1.02(2)
C(1)−C(9)	1.480(2)	C(13)−H(132)	0.99(4)
C(2)−C(3)	1.450(2)	C(13)−H(133)	0.97(3)
C(3)−C(4)	1.376(2)[1.378]	C(14)−H(14)	0.88(2)
C(4)−C(5)	1.430(2)[1.432]	C(15)−H(15)	0.97(2)
C(4)−C(16)	1.514(2)	C(16)−H(161)	0.96(2)
C(5)−C(6)	1.403(3)[1.405]	C(16)−H(162)	1.00(2)
C(6)−C(7)	1.401(2)[1.404]	C(17)−H(171)	0.98(2)
C(6)−C(18)	1.508(2)	C(17)−H(172)	0.98(2)
C(9)−C(10)	1.402(3)[1.412]	C(18)−H(181)	0.92(2)

Table 2. Continued.

C(9) – C(15)	1.386(2)[1.396]	C(18) – H(182)	0.97(2)
C(10) – C(11)	1.362(3)[1.368]	C(19) – H(191)	0.97(3)
C(11) – C(12)	1.384(2)[1.394]	C(19) – H(192)	1.01(2)
C(12) – O(1)	1.356(2)	C(19) – H(193)	0.89(3)
C(12) – C(14)	1.394(3)[1.404]		

Table 3. Intramolecular bond angles. The corresponding standard deviations are listed in parentheses.

Angle	°	Angle	°
S(2) – S(1) – C(1)	82.40(7)	C(10) – C(11) – C(12)	121.0(2)
S(2) – S(1) – C(8)	133.9(1)	C(10) – C(11) – H(11)	120(1)
C(1) – S(1) – C(8)	103.1(1)	C(12) – C(11) – H(11)	119(1)
S(1) – S(2) – S(3)	178.05(2)	C(11) – C(12) – C(14)	118.8(1)
S(1) – S(2) – C(3)	82.81(6)	C(11) – C(12) – O(1)	116.1(1)
S(3) – S(2) – C(3)	95.50(6)	C(14) – C(12) – O(1)	125.1(1)
S(2) – S(3) – S(4)	178.14(3)	C(12) – O(1) – C(13)	118.3(2)
S(2) – S(3) – C(5)	93.25(6)	O(1) – C(13) – H(131)	108(1)
S(4) – S(3) – C(5)	86.71(6)	O(1) – C(13) – H(132)	106(2)
S(3) – S(4) – C(7)	89.49(7)	O(1) – C(13) – H(133)	113(2)
C(7) – S(5) – C(19)	103.1(1)	H(131) – C(13) – H(132)	109(2)
S(1) – C(1) – C(2)	121.6(1)	H(131) – C(13) – H(133)	107(2)
S(1) – C(1) – C(9)	117.7(1)	H(132) – C(13) – H(133)	115(3)
C(2) – C(1) – C(9)	120.4(1)	C(12) – C(14) – C(15)	119.6(1)
C(1) – C(2) – C(3)	131.8(1)	C(12) – C(14) – H(14)	120(1)
C(1) – C(2) – H(2)	115(1)	C(15) – C(14) – H(14)	120(1)
C(3) – C(2) – H(2)	114(1)	C(9) – C(15) – C(14)	122.1(2)
S(2) – C(3) – C(2)	121.0(1)	C(9) – C(15) – H(15)	117(1)
S(2) – C(3) – C(4)	116.1(1)	C(14) – C(15) – H(15)	121(1)
C(2) – C(3) – C(4)	122.9(1)	C(4) – C(16) – C(17)	111.6(1)
C(3) – C(4) – C(5)	119.3(1)	C(4) – C(16) – H(161)	109(1)
C(3) – C(4) – C(16)	122.2(1)	C(4) – C(16) – H(162)	110(1)
C(5) – C(4) – C(16)	118.5(1)	C(17) – C(16) – H(161)	109(1)
S(3) – C(5) – C(4)	115.8(1)	C(17) – C(16) – H(162)	109(1)
S(3) – C(5) – C(6)	121.3(1)	H(161) – C(16) – H(162)	109(2)
C(4) – C(5) – C(6)	122.8(1)	C(16) – C(17) – C(18)	110.7(2)
C(5) – C(6) – C(7)	120.7(1)	C(16) – C(17) – H(171)	110(1)
C(5) – C(6) – C(18)	118.8(1)	C(16) – C(17) – H(172)	109(1)
C(7) – C(6) – C(18)	120.5(1)	C(18) – C(17) – H(171)	108(1)
S(4) – C(7) – C(6)	121.7(1)	C(18) – C(17) – H(172)	112(1)
S(4) – C(7) – S(5)	121.63(8)	H(171) – C(17) – H(172)	107(2)
S(5) – C(7) – C(6)	116.6(1)	C(6) – C(18) – C(17)	112.0(1)
S(1) – C(8) – H(81)	110(2)	C(6) – C(18) – H(181)	109(1)
S(1) – C(8) – H(82)	102(2)	C(6) – C(18) – H(182)	109(1)
S(1) – C(8) – H(83)	104(2)	C(17) – C(18) – H(181)	113(1)
H(81) – C(8) – H(82)	115(3)	C(17) – C(18) – H(182)	109(1)
H(81) – C(8) – H(83)	92(3)	H(181) – C(18) – H(182)	105(2)
H(82) – C(8) – H(83)	134(3)	S(5) – C(19) – H(191)	105(2)
C(1) – C(9) – C(10)	121.5(1)	S(5) – C(19) – H(192)	109(1)
C(1) – C(9) – C(15)	121.4(1)	S(5) – C(19) – H(193)	104(2)
C(10) – C(9) – C(15)	117.1(1)	H(191) – C(19) – H(192)	113(2)
C(9) – C(10) – C(11)	121.4(2)	H(191) – C(19) – H(193)	111(3)
C(9) – C(10) – H(10)	120(1)	H(192) – C(19) – H(193)	113(2)
C(11) – C(10) – H(10)	118(1)		

RESULTS AND DISCUSSION

Positional and thermal parameters are listed in Tables 1a and 1b. Bond lengths and angles are given in Tables 2 and 3. The standard deviations in bond lengths calculated from the inverse least-squares matrix are as follows: 0.0008 Å in S–S bonds, 0.002 Å in S–C bonds, 0.002–0.003 Å in C–C and C–O bonds, and 0.02–0.04 Å in C–H bonds. A more realistic estimate of the accuracy of the results is probably obtained by multiplying the above calculated standard deviations by a factor of two.[17]

A drawing of the molecule with pertinent bond lengths and angles is shown in Fig. 1. The row of sulphur atoms S(1) to S(4) is found to be approximately

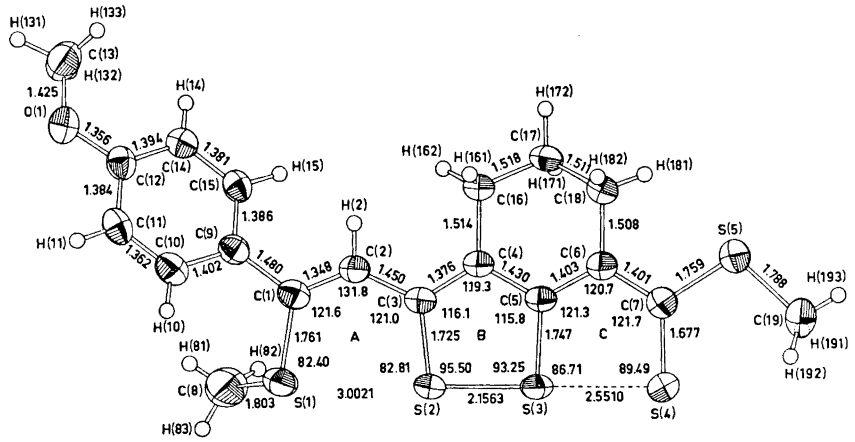


Fig. 1. ORTEP plot [21] of one molecule showing thermal ellipsoids at the 50 % probability level. Hydrogen atoms are plotted with a fixed radius. The most important bond lengths and angles are also shown.

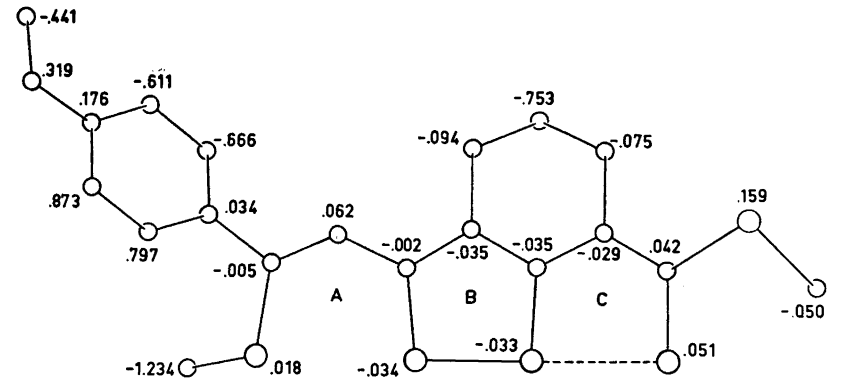
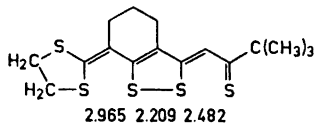


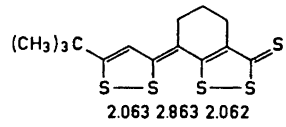
Fig. 2. Atomic deviations from a least-squares plane fitted to the 11 atoms of rings A, B, and C [S(1)–S(4), C(1)–C(7)]. The equation of the plane is $-7.7493\,x+15.9482\,y+2.6913\,z+4.3468=0$. x, y, z are fractional coordinates.

linear. The methyl carbon C(8) deviates from this line, the angle S(2) – S(1) – C(8) being 133.9°. A least-squares plane was fitted to the atoms of rings A, B, and C. This part of the molecule is almost planar with a maximum atomic deviation of 0.06 Å (Fig. 2). C(8) deviates 1.23 Å from this plane. The aromatic ring of the methoxyphenyl group makes an angle of 38° with the ABC plane. The methylthio group S(5)C(19) is slightly twisted out of the plane with a dihedral angle S(4)C(7)S(5)/C(7)S(5)C(19) of 10.5°.

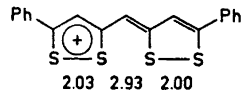
The intramolecular sulphur – sulphur distances in the present compound, I, may be compared with those in the already known linear four-sulphur molecules II, III, and IV.[3–5]



(II)



(III)



(IV)

The bonding scheme of the sulphur atoms in I and II differs markedly from that in III and IV. Molecules I and II both contain a three-sulphur sequence analogous to those found in several isolated thiathiophthenes. The four-sulphur sequences in III and IV may more adequately be described in terms of two separate disulphide systems with terminal S – S bonds of the same lengths as those found in cyclic five-membered disulphides.[18,5] The interaction across the long central S···S distance is apparently too weak to significantly influence the lengths of these bonds. Similarly there is no indication that the S···S interaction across the distances of 3.002 Å and 2.965 Å, in I and II, respectively, perturbs the sulphur – sulphur bonding in the thiathiophthene part of these molecules. Hence, the structural data obtained so far of compounds containing a linear four-sulphur sequence do not give a clear evidence of a delocalized σ-system extending across the four sulphur atoms. In the two known five-sulphur structures,[6,7] on the other hand, the molecular dimensions indicate the presence of an extended thiathiophthene bonding scheme.

The shortest intramolecular H···H and S···H distances are H(2)···H(15), H(2)···H(162) and S(5)···H(181), 2.41 Å, 2.13 Å and 2.53 Å, respectively. The packing of molecules in the unit cell is shown in Fig. 3. Intermolecular sulphur – sulphur close contacts appear between molecules related by a center of symmetry. The distances S(1)···S(4′) and S(2)···S(3′) are 3.515 Å and 3.433 Å, respectively. The unprimed atoms are located in the reference molecule, while primed atoms refer to its inversion at $\frac{1}{2},0,0$. The reference molecule and its inversion at $\frac{1}{2},0,\frac{1}{2}$ overlap considerably with an interplanar spacing of 3.64 Å.

Thermal ellipsoids at the 50 % probability level are illustrated in Fig. 1. The thermal parameters of the atoms were analyzed in terms of rigid body mo-

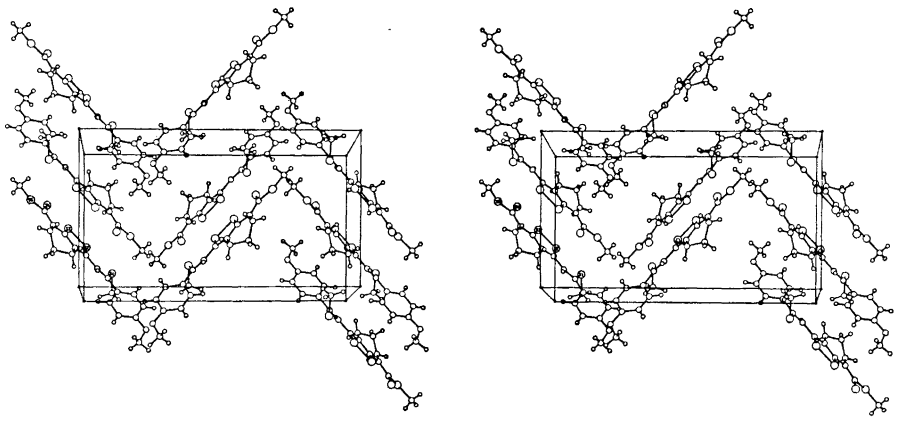


Fig. 3. Stereoview down the c^*-axis. The drawing was done using the ORTEP plotting program.[21]

tion using the method of Schomaker and Trueblood.[19] The results indicate that the whole molecule cannot be regarded as a rigid body. However, the 8 atoms in the thiathiophthene part of the molecule are described reasonably well by the rigid body approximation, the r.m.s. ΔU_{ij} being 0.0012 Å^2. Likewise, the aromatic ring of the methoxyphenyl group may be described as a rigid body. For these atoms the r.m.s. ΔU_{ij} is 0.0015 Å^2. The bond lengths in these parts of the molecule have been corrected for rigid body motion according to Cruickshank's method,[20] and are listed in Table 2 together with the uncorrected bond lengths.

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

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